

Wastewater Laboratory Certification

Overview



What is laboratory certification?

“Laboratory certification is a process that provides formal recognition to the managerial and technical competence of a laboratory performing specific analyses defined by a certifying body (or environmental program)”

Wastewater Regulation Status

- SB120 → HB385
- Statute 224.10-670 – signed on 6/8/2011
- Regulation – promulgate 9/5/2013
- Wastewater Laboratory Manual – promulgate 9/5/2013
- Effective dates
 - Effective date of January 1, 2014 for general labs
 - Compliance date of January 1, 2015
 - Effective date of January 1, 2015 for field only labs
 - Compliance date of January 1, 2016
- First year – interim certification
- Second through fifth year – on-site audits performed

How laboratories become certified

- Submit application to the Division of Water
 - Every other year (even years for general labs, odd years for field only labs)
- Provide list of requested method- analyte pairings for certification
 - Minimum Reporting Limit (MRL) – must meet specific program Required Reporting Limit (RRL)
 - Must use an approved method.
- Submit required documentation:
 - Quality Assurance Plan (QAP)
 - Must address the required QA/QC
 - Organizational chart
 - List of instrumentation and other resources
 - IDC / MDL Studies
 - Performed annually
 - Proficiency test results for all analytes and methods
 - Performed annually
- On-site evaluation (audit)
 - Audits performed a least once every five years

Reference Methods

- 40 CFR 136.3 – list of EPA approved methods
- 40 CFR 136.4 & 136.5 – ATP Procedures
- 40 CFR 136.6 – Method Modification
- EPA Method Modification Rule (5/18/2012)
- SM Reference Nomenclature 4500-H⁺ B-2000
- Use QC from SM 20th, 21st or 22nd Ed. only
- SM 22nd Ed. has useful QC (e.g. 2020B)

Tiers of certification

- Interim Certification
- Full Certification
- Provisional Certification
- Not Certified/Revocation of Certification

Sections of the Manual

- General
- Chemistry
- Microbiology
- Whole Effluent Toxicity (WET)
- Contact Information
- Appendices
 - Glossary & Acronyms
 - Definitions & Laboratory Terms
 - ASTM Type I, II, III, & IV Water Specifications

Quality Assurance

An integrated system of management activities involving planning, implementation, documentation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client (intended use).

Quality Control

The overall system of technical activities that measure the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality.

Quality Assurance Plan

QAP

- Documents the planning, implementation, and assessment procedures for a particular project, as well as any specific quality assurance and quality control activities. It integrates all the technical and quality aspects of the project in order to provide a "blueprint" for obtaining the type and quality of environmental data and information needed for a specific decision or use.

Quality Assurance Plan

- Quality Assurance Plan
- A laboratory analyzing wastewater compliance samples shall adhere to quality control procedures established in the analytical methods in 40 C.F.R. 136. Each laboratory shall prepare, implement, and maintain a written Quality Assurance Plan (QAP). The QAP shall be kept current by conducting an annual review and making necessary revisions. Laboratory personnel shall be familiar with the contents of the QAP. If the annual review results in substantive updates or revisions, the amended QAP shall be submitted to the cabinet with the application for renewal of certification.
- The laboratory QAP shall be a stand-alone document. However, it may reference other documents such as Standard Operating Procedures (SOP), published methods, or other published literature.

Quality Assurance Plan

- The following items shall be addressed in the laboratory QAP:
 - Laboratory organization and responsibility
 - Process used to identify clients' data quality objectives (DQO).
 - SOPs with dates of last revision
 - Field sampling procedures
 - Laboratory sample receipt and handling procedures
 - Instrument calibration procedures (may reference an SOP)
 - Analytical procedures (may reference an SOP)
 - Data reduction, validation, reporting and verification (may reference an SOP)
 - Type of quality control checks and the frequency of their use (may reference an SOP)
 - List schedules of internal and external system and data quality audits and inter-laboratory comparisons (may reference an SOP).
 - Preventive maintenance procedures and schedules
 - Corrective action contingencies
 - Record keeping procedures

Standard Operating Procedures

- SOPs with dates of last revision
 - The laboratory shall maintain SOPs that **accurately reflect all phases of the current laboratory activities.**
 - Keep a list of SOPs and their **effective dates.**
 - Ensure that **current copies** of SOPs are in the laboratory and in the QA manager's files.
 - Ensure that SOPs are **reviewed annually** and revised as changes to the procedure are made.
 - Ensure that SOPs have signature pages and revision dates.
 - Ensure that SOPs **are read, understood, and used** by applicable laboratory personnel.

Standard Operating Procedures Continued

Table of Contents

1. Title Page
2. Table of Contents
3. Procedures
 - a. Scope and Applicability
 - b. Summary of Method
 - c. Definitions
 - d. Health & Safety Warning
 - e. Cautions
 - f. Interferences
 - g. Personnel Qualifications / Responsibilities
 - h. Equipment and Supplies
 - i. Step by Step Procedure
 - Instrument or Method Calibration and Standardization
 - Sample Collection
 - Sample Handling and Preservation
 - Sample Preparation and Analysis
 - Troubleshooting
 - Data Acquisition, Calculations & Data Reduction Requirements
 - Computer Hardware & Software
 - j. Data and Records Management
4. Quality Control and Quality Assurance Section
5. Reference Section

Chain of Custody

- A COC is an accurate written legal record to track the possession, handling, and location of samples and data from collection **through** reporting
- A proper COC will answer the following 6 questions about the “life” of a sample.
 1. **what** (the type of sample i.e. drinking water, soil, wastewater)
 2. **where** (the location)
 3. **when** (date and time)
 4. **how** (grab/composite & deviations)
 5. **why** (what analysis)
 6. **who** (signatures)

COC

Internal COC

Sample ID	Analyst	Out		Analysis	Location	In	
		Date	Time			Date	Time
AT110001- AT110020	FCH	01/01/11	10:00	TSS	WET CHEM BENCH	01/01/11	11:00
AT1100040- AT110060	PJG	01/01/11	14:00	VOC	GC/MS ROOM	01/02/11	08:00
AT110001- AT110020	FCH	01/02/11	08:00	ALK	REF # 1	01/04/11	16:00

SAMPLE RECEIVING

- Check actual samples against COC & sign receiving samples
 - Check temperature at receipt
 - Containers
 - Preservation
 - Hold times
 - ID
 - Location
 - Date/Time
 - Collector
- Rejection of sample criteria

TEMPERATURE LOGS

- Refrigerator/Oven #
- Date
- Time
- Corrected temperature
- Analyst
- Corrective actions

TEMPERATURE LOGS

REF # 1

Date	Time	Temp (°C)	Analyst	Corrective Action
01/01/11	08:00	4.1	FCH	N/A
01/02/11	10:05	4.6	FCH	Adj. temp down
01/03/11	11:00	3.8	FCH	N/A
01/04/11	8:20	4.9	PJG	N/A
01/05/11	13:10	5.1	PJG	N/A
01/06/11	16:30	6.1	FCH	Adj. temp down & notified QA manager
01/06/11	16:45	5.8	FCH	N/A
01/06/11	17:00	5.8	FCH	Adj. temp down
01/07/11	08:00	4.5	PJG	Adj. temp down
01/08/11	09:05	4.0	PJG	N/A

BALANCE LOGS

- Balance #
- Date
- Time
- Actual Weight
- Balance Reading
- Analyst
- Corrective actions

BALANCE LOGS

BAL # 1					
Date	Time	Analyst	Weight (g)	Reading (g)	Corrective Action
01/01/11	08:00	FCH	50.0	50.0001	N/A
01/01/11	08:02	FCH	1.0	0.9999	N/A
01/01/11	08:04	FCH	0.1	0.1001	N/A
01/04/11	8:20	PJG	1.1	1.1000	N/A
01/05/11	8:22	PJG	51.1	50.1001	N/A
01/06/11	16:30	FCH	50.0	50.0100	Cleaned balance pan, adjusted level, zeroed
01/06/11	16:45	FCH	50.0	50.0002	N/A
01/06/11	16:47	FCH	1.0	1.0001	N/A
01/07/11	16:50	FCH	0.1	0.9999	N/A

REAGENT/STANDARD LOGS

- Reagent/Standard number
- Reagent/Standard name
- Reagent/Standard Concentration
- Received date (purchased reagents/standards)
- Reagent/Standard number of each component
- Reagent/Standard name of each component
- Exact weights/volumes of each component
- Certificate of Analysis on file
- Analyst
- Date
- Time
- Expiration date
- Disposal date

REAGENT/STANDARD LOGS

Reagent/Standard Log

Tracking #	Rgt/std Name	Rgt/Std Conc.	Rec Date	Component Name	Comp Tracking #	Weight/ Volume Used	Final Vol	Analyst	Date	Time	C of A On file	Exp Date	Disposal Date
RGT1101001	Barbituric acid	n/a	01/05/11	n/a	n/a	n/a	n/a	FCH	01/05/11	14:00	Y	01/05/16	
RGT1101002	Pyridine	n/a	01/05/11	n/a	n/a	n/a	n/a	FCH	01/05/11	14:00	Y	01/05/12	
RGT1101003	HCl	Conc.	01/05/11	n/a	n/a	n/a	n/a	FCH	01/05/11	14:00	Y	01/05/12	
RGT1101004	Chloramine T	n/a	01/05/11	n/a	n/a	n/a	n/a	FCH	01/05/11	14:00	Y	01/05/16	
RGT1101005	NaOH	Conc	01/05/11	n/a	n/a	n/a	n/a	FCH	01/05/11	14:00	Y	01/05/16	
STD1101001	CN Stock std	1000ppm	01/05/11	n/a	n/a	n/a	n/a	FCH	01/05/11	14:00	Y	04/11	03/15/11
RGT1101006	Barbituric acid/pyridine	n/a	n/a	Barbituric acid	RGT1101001	15.0001g	250 mL	FCH	01/06/11	08:05	n/a	06/06/11	
				Pyridine	RGT1101002	75 mL							
				HCl	RGT1101003	15 mL							
RGT1101007	Chloramine T sol	n/a	n/a	Chloramine T	RGT1101004	1.0010g	100 mL	FCH	01/06/11	08:15	n/a	01/13/11	01/11/11
RGT1101008	NaOH solu	0.025 N	n/a	NaOH	RGT1101005	1.6001g	1L	FCH	01/06/11	08:25	n/a	07/06/11	01/07/11
STD1101002	CN working std	100 ppm	n/a	CN Stock std	STD1101001	10.0 mL	100 mL	FCH	01/06/11	08:35	08:35	01/07/11	01/07/11
STD1101003	0.01 CN STD	0.01 ppm	n/a	CN Stock std	STD1101002	0.01 mL	100 mL	FCH	01/06/11	08:40	08:35	01/07/11	01/07/11
STD1101004	0.25 CN STD	0.25 ppm	n/a	CN Stock std	STD1101002	0.25 mL	100 mL	FCH	01/06/11	08:45	08:35	01/07/11	01/07/11
STD1101005	0.5 CN STD	0.5 ppm	n/a	CN Stock std	STD1101002	0.5 mL	100 mL	FCH	01/06/11	08:50	08:35	01/07/11	01/07/11
STD1101006	0.75 CN STD	0.75 ppm	n/a	CN Stock std	STD1101002	0.75 mL	100 mL	FCH	01/06/11	08:55	08:35	01/07/11	01/07/11
STD1101007	1.0 CN STD	1.0 ppm	n/a	CN Stock std	STD1101002	1.0 mL	100 mL	FCH	01/06/11	09:00	08:35	01/07/11	01/07/11



BENCH SHEETS

- Must be able to use the bench sheet to reproduce the analysis results.
 - Analyst
 - Analytical method used
 - Results of sample & QC analysis
 - All data to reproduce calculations must be present
 - Calibration & standards information

BENCH SHEETS

Alkalinity
SM 2320B

Analyst	FCH	QCS	STD1012050	100 mg/L	
Date	01/06/11	LCS/MS	STD1012050	1000 mg/L	Spiked 5.0 mL
Time	14:00	H2SO4	RGT1006550	0.1 N	

Sample ID	mL sample	mL titrant	Calc. value	RPD	% REC
BLK	100	0.1	5		
QCS	50	1.0	100		100
LCS	50	0.9	90		90
1101000550	50	2.0	200		
1101000550DUP	50	2.1	210	4.88%	
1101000551	50	3.0	300		
1101000551MS	50	3.9	390		90
1101000552	50	2.1	210		
1101000553	50	2.2	220		
1101000554	50	5.0	500		
1101000555	50	3.0	300		
1101000556	50	0.8	80		
1101000557	50	1.1	110		
1101000558	50	1.5	150		
1101000559	50	1.0	100		
BLK	100	0.1	5		
QCS	50	1.0	100		100

$$\text{Alkalinity, mg CaCO}_3 / L = \frac{A \times N \times 50000}{\text{mL sample}}$$

where:

A = mL standard acid used N = normality of standard acid



MAINTENANCE LOGS

- Types of maintenance
 - Preventive maintenance
 - Trouble shooting
 - Service Contract
- What information is needed
 - Instrument
 - Analyst
 - Date
 - Time
 - Problem
 - Maintenance performed

MAINTENANCE LOGS

Spec 20 – Instrument ID # 00025

Analyst	Date	Time	Problem	Maintenance
FCH	01/05/2011	14:00	No signal	Replaced lamp
PJG	01/20/2011	08:15	High blanks	Replaced sample cells
FCH	02/10/2011	09:25	Annual check	Labtronics
FCH	02/15/2011	10:35	Broke sample cell in instrument	Cleaned sample cell compartment
FCH	2/28/2011	11:05	No signal	Replaced lamp



DOCUMENTATION & DATA RETENTION

- Report Requirements and Record Keeping
 - The following are required for a certified wastewater laboratory analyzing wastewater compliance sample results:
- **Legal Defensibility:** Compliance monitoring data shall be made legally defensible by keeping thorough and accurate records. The QAP or SOPs shall describe the policies and procedures used by the facility for record integrity, retention, and storage. Chain of custody procedures shall be utilized.
- **Maintenance of Records:** A certified laboratory shall maintain records for **five (5) years or until the next on-site audit, whichever is longer**. All references in this manual to a five (5) year records retention period shall include “or until the next on-site audit, whichever is longer.” A change in ownership, mergers, or closure of laboratory does not eliminate this requirement. Records include all raw data, calculations, and quality control data. These data files may be hard copy, microfiche, or electronic. If the laboratory changes its computer hardware or software, it shall make provisions for transferring old data to the new system so that it remains retrievable.
- **Sample & Analytical records:** Data shall be recorded in indelible ink with any changes lined through once, such that the original entry is visible. Changes shall be initialed and dated. Data may also be kept electronically.

DOCUMENTATION & DATA RETENTION

Continued

- **Reconstruction of Data:** Adequate information shall be available to allow the auditor to reconstruct the final results for compliance and PT samples, including:
- **Computer Programs:** Computer programs shall be verified initially and be available for inspection. Access to computer programs and electronic data shall be limited to authorized personnel.
- **Sub-contracting to another Kentucky wastewater certified laboratory:** Any DOW- certified wastewater laboratory may sub-contract wastewater compliance samples to another DOW- certified wastewater laboratory for analysis. The initial (primary) laboratory is responsible for ensuring that any wastewater compliance sample analysis is sub-contracted to a laboratory that is certified by DOW for the specific method-analyte pairing that has been requested. If the primary lab reports data from the sub-contract laboratory they must clearly identify that data on the report.

12 Mandatory QC Elements

1. Demonstration of Capability (DOC);
2. Method Detection Limit (MDL);
3. Laboratory reagent blank (LRB), also referred to as method blank (MB);
4. Laboratory fortified blank (LFB), also referred to as a spiked blank, or laboratory control sample (LCS);
5. Matrix spike (MS) and matrix spike duplicate (MSD), or laboratory fortified matrix (LFM) and LFM duplicate, may be used for suspected matrix interference problems to assess precision;
6. Internal standards (for GC/MS analyses), surrogate standards (for organic analysis) or tracers (for radiochemistry);
7. Calibration (initial and continuing), also referred to as initial calibration verification (ICV) and continuing calibration verification (CCV);
8. Control charts (or other trend analyses of quality control results);
9. Corrective action (root cause analysis);
10. QC acceptance criteria;
11. Definitions of preparation and analytical batches that may drive QC frequencies; and
12. Minimum frequency for conducting all QC elements.

These twelve quality control elements shall be clearly addressed in the written SOP or QAP for each analytical method, where applicable.

10 Mandatory QC Elements Micro*

1. Demonstration of Capability (DOC), initial and on-going
2. Method blanks and sterility checks
3. QC samples and laboratory fortified blanks
4. Matrix spike and matrix spike duplicates
5. Calibration
6. Control charts – trend analysis
7. Corrective actions
8. QC acceptance criteria
9. Definition of a batch or test run
10. Minimum frequency of QC checks

* M. Hunt, “What are the Critical Elements in the Microbiology Laboratory?”, *National Environmental Monitoring Conference*, San Antonio, TX, August 2013.

Demonstration of Capability

- Allows laboratories to demonstrate that an EPA approved method is “sensitive enough” to meet the required reporting limits specified in a discharge permit
- Laboratories must submit DOC prior to analyzing compliance samples
 - Method Detection Limit (MDL) Study
 - 40 CFR Part 136 Appendix B
 - Initial Demonstration of Capability (IDC)
 - Reporting Limit Standard (RLS) at the required reporting limit specified in the permit

Method Detection Limit Study

- Must analyze seven (7) replicates over 2 to 3 non-consecutive days
- Determine concentration of replicates (2-5 x est MDL)
- Calculate MDL using student t-test equation
- Verify that calc MDL is within 10 times est MDL
- $MDL < MRL \leq$ Permit Required Reporting Limit (RRL)
- Must be performed annually per method
- **Note:** if laboratory is not performing MDL per instrument they must successfully demonstrate that the MDL is achievable by all instruments.

Initial Demonstration of Capability

- Four (4) mid-range replicates – LCS / QCS
- Analyze over one to four days
- Calculate mean concentration
- Each replicate must be $\pm 20\%$ of the mean concentration
- The percent relative standard deviation (%RSD) must be $\leq 15\%$
- **Note:** Must be performed initially by each analyst
- **Note:** Then ODC (ongoing demonstration of capability) annually by the primary analyst & 1/5 years by all back up analysts

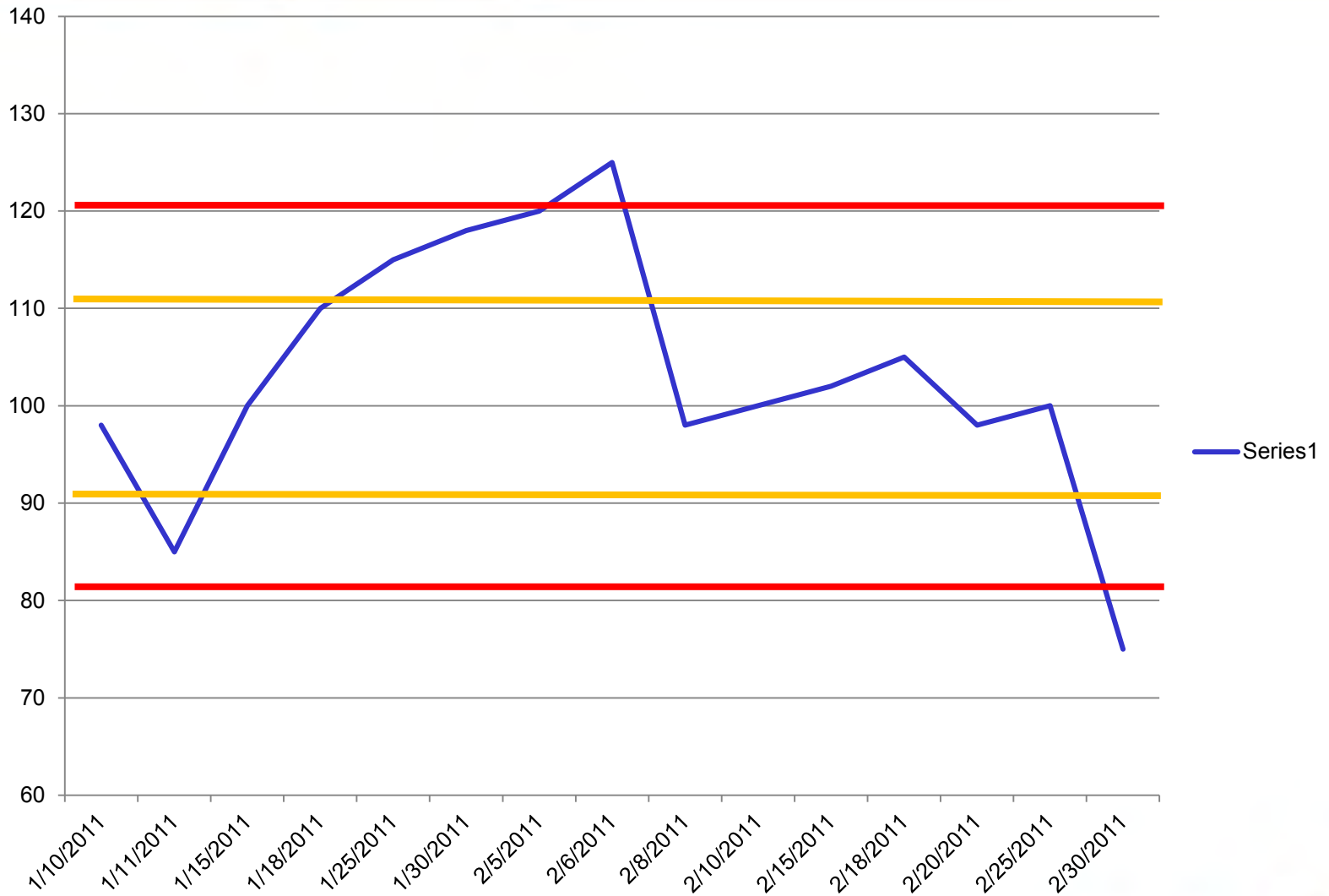
Reporting Limit Standard

- Initial calibration (low standard) or verification must be at or below the permit required reporting limit
- Standard must be part of the calibration – can not be disregarded for failure to meet calibration criteria
- Calculate reporting limit standard as a sample – must be within 70 to 130% of expected value
- RLS must be analyzed/calculated with each calibration and at least quarterly
- Results must be available for DOW review during an on-site audit or by request

CONTROL CHARTS

- Include the following information:
 - Method
 - QC being charted
 - Upper control limit
 - Upper warning limit
 - Lower control limit
 - Lower warning limit

CONTROL CHARTS



Proficiency Test Study (PT)

- Annual submission of a Proficiency Test Study to DOW
- Laboratory must submit at least one ‘acceptable’ result for each and every analyte-method for which they are certified (or requested certification for)
- DMR-QA Program is an acceptable PT
- If laboratory receives an ‘unacceptable’ result for an analyte-method for which they are certified – they must perform additional studies until ‘acceptable’ result.
- Repeated failures may result in change in certification status

Field Parameters

- Field parameters must be performed by a certified lab
- Field parameters are as follow:
 - pH
 - Conductivity
 - Temperature
 - Dissolved oxygen
 - Chlorine
 - Turbidity

Requirements for Field Analysis

- Applies to: Certified operators, laboratory field personnel, environmental consultants, etc
- If a facility performs only field analysis for their own facility, then only an initial Laboratory Certification audit will be performed. From that point forward only the normal field inspections, if deficiencies are found the field may then request a certification audit.
- Documentation of standard operating procedures, calibration procedures, quality assurance plans, and sampler training; and
- PT results for pH, residual chlorine, conductivity, and turbidity.

QC Requirements for Field Analysis

- The following quality control measures apply to all laboratories performing residual chlorine, pH, conductivity and turbidity analyses in the field:
 - Calibration of instruments shall be performed daily, using a primary standard.
 - A calibration verification shall be performed daily using a second source. This may be accomplished using a secondary standard.
 - A method blank shall be analyzed daily prior to analysis.
 - A quality control sample (QCS) shall be analyzed at least quarterly.

QC Requirements for Field Analysis

- The following quality control measures apply to all laboratories performing dissolved oxygen, residual chlorine, pH, temperature, conductivity, flow, and turbidity analyses in the field:
 - Duplicate analysis of samples shall be performed once per batch of twenty samples, at a minimum. Sample batches may extend beyond one day.

On-site evaluation (audit)

- Laboratories will be audited at least once every five years
- Laboratories will be audited for technique on analytical methods, documentation, & quality control performance
- DOW will use audit techniques similar to drinking water program
- DOW utilizes method checklists to ensure that all laboratories are held to the same standard of quality

Fees

Wastewater Laboratory Certification Fee	
Category	Annual Fee
Application Fee	\$1000
Inorganic/General Chemistry	\$500
Inorganic/Metals	\$500
Organic Chemistry/Volatiles	\$500
Organic Chemistry/Semi-Volatiles	\$500
Organic Chemistry/Pesticides, Herbicides, PCB	\$500
Organic Chemistry/Dioxins	\$750
Microbiology	\$500
Whole Effluent Toxicity	\$1000
Field Analysis Only	\$250

Fees

- Fees shall be received by November 15 each calendar year (excluding initial application).
- A fee received after November 15 and on or before December 15 shall incur a surcharge of 15% (excluding initial application).
- Laboratory certification shall be revoked if fees are not received by December 15 (excluding initial application).
- To reinstate a laboratory certification that was revoked shall pay a surcharge of 25% of the certification fee.
- A laboratory shall pay a \$500 fee if an additional audit is necessary.
- An out of state laboratory, that does not qualify for equivalency certification must bear the reasonable cost of an audit.

Discounts

- In state DW Labs = 20 % of total fee
- Equivalency = 20% of total fee
- Only Field = No Application fee
- Only Field by the permitted facility = No fee
- **Note:** Discounts are not cumulative, only the greatest discount applies.

Discounts

A laboratory operated by a municipality that only provides service for its own facility shall receive the following reduction to the administrative application fee, based on the maximum permitted million gallons per day (MGD) flow value:

- \leq to 0.10 MGD shall receive a 100% discount;
- \leq to 0.50 MGD but greater than 0.10 MGD shall receive a 75% discount;
- \leq to 1.0 MGD but greater than 0.5 MGD shall receive a 50% discount;
- \leq to 2.0 MGD but greater than 1.0 MGD shall receive a 25% discount; and
- $>$ than 2.0 MGD shall receive a 10% discount.

Wastewater Website

<http://water.ky.gov>

Laboratory Certification (under programs)

- Application
- Updates section
- Demonstration of capability procedure
- MDL / IDC Worksheet
- Calibration curve worksheet

Coming soon

- QAP Template
- SOP Template
- Method-Analyte list
- Control charting worksheet

Questions?? Comments??

DOWLabCertification@ky.gov

or call

Frank Hall

(502)564-3410 extension 4959

