

Quality Assurance Oversight Team – Laboratory Assessment Findings (FY12 and FY13)

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Who are/is the QAOT

- *Quality Assurance Oversight Team: Multi-agency group (USACE, SFWMD, USGS, USFWS, USEPA, FDEP) concerned with the quality and consistency of data that CERP produces.**
- *CERP: Comprehensive Everglades Restoration Plan.*

*: QAOT Fact Sheet (October 2013)



What is CERP?

- *Provides a framework and guide to restore, protect and preserve the water resources of Central and southern Florida, including the Everglades.*
- *Goal is to capture fresh water that now flows unused to the ocean and the gulf and redirect it to areas that need it most.*

Website: Evergladesplan.org



Purpose of Lab Assessments

To assess, at the bench level, the proficiency that the laboratory has to perform chemical analysis and to ensure that the analytical chemistry laboratories meet the Quality Assurance/Quality Control requirements defined in the USEPA methods that are specified in CERP project documents.



Desired Outcome

Enhances assurance that laboratories are producing defensible data for CERP samples by following the analytical methods requested for the project.



Program Structure

- *Desk – Documentation Review. Standard Operating Procedure vs. Method.*
- *On-Site Assessment – In-depth Discussion with Analysts. Implementation at the bench level.*
- *Performance Evaluation Samples – Provided by the QAOT at no cost to lab.*



Findings Categories

- *Observation: Information or clarification purposes.*
- *Recommendation: Represent deviations that if corrected, may facilitate an improvement in data.*
- *Deficiency: Deviations that must be corrected to assure compliance with a cited requirement.*



Desk: Requested Information

- *Quality Manual (QM)*
- *Standard Operating Procedures (SOPs)*
- *Method Detection Limits (MDLs)*
- *Control Charts*
- *NELAC PT Results/Audits w/Corr. Action*
- *Data Package (Instrument Raw Data)*
- *Example Customer Report*



OnSite: Key Focus Areas

- *Discuss Desk Assessment Report with QAO and Supervisors.*
- *Walk-through of sample receipt area.*
- ***Implementation presented at the bench by the individuals that normally perform both sample preparation and sample analysis.***
- *Check consistency with lab's SOPs and referenced methods.*



Assessment Summary

- *Seven Laboratories in FY12 + FY13.*

(WET, Radiochemistry, Microbiological)

- *Four environmental chemistry labs -*

Test Methods/Frequency:

Chromatography – 6

Metals/Mercury – 6

Wet Chemistry – 15

Total Organic Carbon – 3

Other - 5



Common Critical Findings

Calibration:

- *Initial Calibration Curves (ICAL) not being evaluated properly, (Avg. vs. Linear vs. Quadratic).*
- *Low standard not being quantitated against the new ICAL. Example (pesticides): DDT spike at 2 ug/L calculated to 7 ug/L. Result: high bias at low levels detected. Potential false positives.*
- *Continuing Calibration Verification (CCVs) failing, but analysis continues; results reported.*
-- Potential false negatives.



Common Critical Findings

Method Detection Limit (MDL) studies: Not being verified w/ low level spike (MDL-V). Some results extremely high (400%). Some compounds not detected. Corrective action not taken.

Standard Operating Procedures/Quality Manuals (Documentation): Combination of methods (must be very specific with subtleties).



Common Critical Findings

- *QC not analyzed at correct frequency, or data qualified. Labs have difficult time keeping requirements separate for same technologies, (SW846 vs. DW).*
 - *Blanks: Samples analyzed/reported even if blank(s) are contaminated.*
 - *Matrix Spikes, duplicates not analyzed at correct frequency.*



• ***Pesticides: Endrin/DDT Breakdown.***



Common Critical Findings

- *Control charts: Limits not reviewed; appear to be generated for the assessment, (LCL 0% - calculation error). Multiple units for same test. 30 data points – all 100% R (rounding before graphing/calculations).*
- *Method requires standards and/or samples to be analyzed twice; lab only analyzing once.*



Specific Findings

- *Method 8081B (pesticides): DDT deleted, not reported for a soil sample.*
 - *Column 1: 0.022 $\mu\text{g}/\text{mL}$ = 7.3 ug/kg*
 - *Column 2: 0.024 $\mu\text{g}/\text{mL}$ = 8.0 ug/kg*
(note: RL = 3.3 ug/kg)
- *Lab deleted with no explanation. Second level review did not catch this deletion.*
- *Note: Three samples from same site. DDT was reported in the other two samples.*



Specific Findings

- *Metals-200.7: LCS interpretation for high recoveries. Lab interpreting NELAC wrong.*
- *Metals and inorganic anions: Blanks - If contamination >MDL, analysis can continue per SOP. Methods say to stop analysis, determine source of contamination, and reanalyze.*
- *200.7: Many metals results in samples <0 mg/L due to blank used for zeroing instrument artificially high in beginning.*
- *200.7: MDL determined off different wavelength than wavelength for reported results.*



Specific Findings

- *1311: TCLP Rotation & Temperature of tumbling not documented.*
- *1311: MB not filtered; MS not tumbled/analyzed. Combine waters & TCLP into one batch.*
- *1631E: Lab reporting two sig figures; method states three. On MB Report, MDL>LRL.*
- *1631E: Analyst unsure of instrument software/blank subtraction; QA officer intervened.*
- *3510C: Only two aliquots of methylene chloride for extraction; method states three.*



Specific Findings

- *Alkalinity: “..air space above solution at a minimum.” Lab changed process.*
- *Ortho-Phos/TP: r^2 for ICAL 1.000000 for twelve graphs in a row. Software graphing incorrectly, but results are accurate (verified on-site). QAO signed off graphs.*
- *Sulfide: MDL determined without dilution taken into account; true MDL 2X higher.*



Specific Findings

- *Conductivity: LCS – SOP text: 95-105%; SOP Table: 98-102%; QM Table 90-110%. Discrepancy.*
- *TDS: Not drying to constant weight in dessicator. Balance tolerance limits not posted.*
- *Chlorophyll (HPLC): Use of Magnesium carbonate. Not in method.*



Specific Findings

- *Alkalinity: Number of significant figures recorded & used in calculation.*

DET U.FP{1} VOL..... 5.562622045628877

DET U.TITER.....0.02132882399439139

units = mL.

- *Calibrations: Point-to-point for sulfate, chloride (method 300). Use of internal standards for ICP AES (SM 3120B).*



Summary

- *Desk: Ensures SOPs adhere to method requirements, and quality systems are in place.*
- *Onsite: Verifies laboratory practices follow SOPs/methods, and personnel have knowledge & skills to follow procedures.*
- *PE samples: Acceptable results equals added confidence with laboratory performance.*



Conclusion

Desk review of laboratory documentation and onsite review of laboratory operations at the bench level, along with evaluation of Performance Evaluation samples, provides an excellent opportunity to judge laboratory performance, and helps to support the defensibility of data generated for CERP samples by focusing on compliance with method requirements.



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Questions?

