A Small Laboratory's Experience

Becoming NELAP Accredited

Mary Johnson, RRWRD

Rock River Water Reclamation District

NELAP

- National Environmental Laboratory Accreditation Program
- NELAP is a voluntary program.
- IEPA is an approved accrediting authority
- NELAP requires Quality Management Systems in a lab

Why should a small, municipal lab apply for NELAP Accreditation?

Illinois does not require wastewater laboratories to be accredited.

Even our IEPA auditor asked why we were applying for accreditation.

NELAP Accreditation Purpose*

"To foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community."

Quote is from TNI (The NELAC Institute) Website.

Promoted Benefits

- Smoother, more efficient laboratory operations
- Improved public trust
- Eliminates need for multiple certifications
- More business from outside companies

Why RRWRD applied for Accreditation

District Management Directive

What is a small laboratory?

- Number of analytes
- Number of analyses
- Number of employees
- Types of analyses/equipment

RRWRD Laboratory Workload

- 50 Analytes
  - 19 wet lab
  - 24 metals
  - 7 anions
- 80,000 analyses per year
  - 30,000 metals analyses
  - 30,000 quality control
- 4.5 FTEs

RRWRD Laboratory Equipment (as of 2009)

- Perkin Elmer GFAA
- Varian ICP
- Perkin Elmer GCMS
- Varian micro-GC
- Thermo UV/VIS

One hot block digestion system

Three specific ion meters

Three autotitrators

Two dishwashers

Another suggestion for defining a small laboratory:

A laboratory in which one person serves as the laboratory manager, the quality assurance officer, and often times as the analyst.

Laboratory in the 80’s

- SOPs were written documentation of oral procedures
- QC was infrequent (QC solutions made in house)
- No computers to track data or qc
- Safety rules were looser

Laboratory in the 90’s

- SOPs traceable to 40 CFR 136 methods
- Variety of QC measures
- Computer tracking of sample data and quality control
- Stricter safety measures

Laboratory in new Millennium

- SOPs for routine procedures other than analytical methods
- Method detection limits
- Detailed tracking of sample and qc data
- Expanded safety program including regular training and audits

Laboratory Accreditation Preparation

- Wrote QAP based on IWEA Model QAP
- Two Performance Testing (PT) samples annually
- Made sure Method Detection Limits (MDL) were up-to-date
- Documented Technician Method Performance (IDMP)

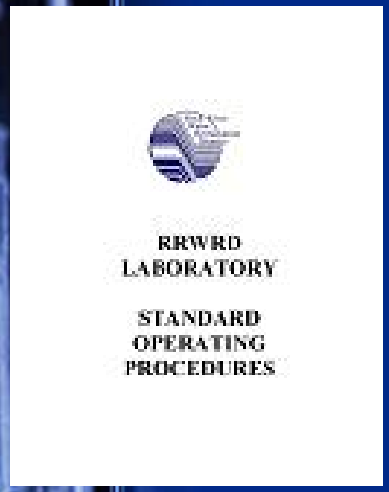
Laboratory Accreditation Preparation continued

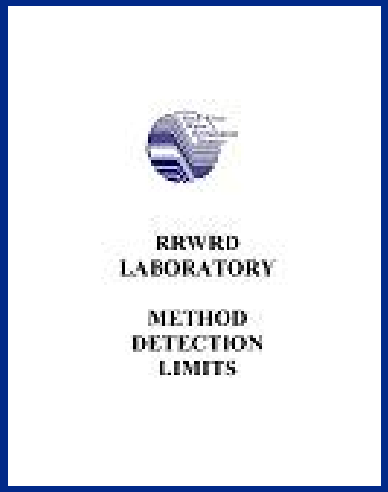
- Annual internal audit
- Corrective actions
- Complaint forms
- Consistent, appropriate formats for benchsheets and qc spreadsheets

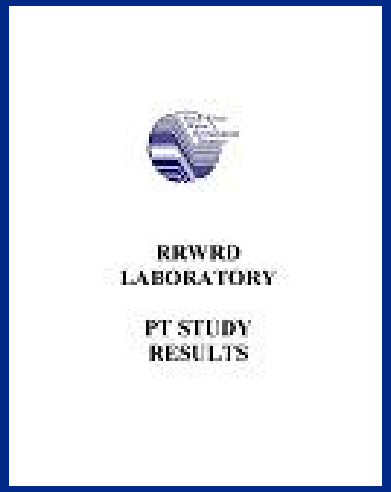
Laboratory Accreditation Preparation continued

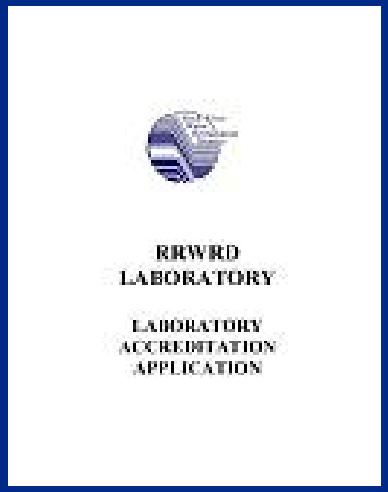
- Completed application
- 54 page audit checklist
  - 5 check lists
  - 707 items to reference to QAP
- Sent check ($4900)
  - $1500 initial application assessment
  - $2400 administrative assessment
  - $1000 for each field of testing

DOCUMENTATION









Drinking Water vs. Wastewater

- Originally planned to seek both drinking water and wastewater certification for inorganics
- Downgraded to wastewater only on recommendation of IEPA

IEPA Audit February 2009

- Auditor – Scott Siders
- Relaxed and friendly
- Reviewed general lab quality rather than being picky about every point
- Two critical findings; 34 other findings

Audit Critical Findings

- No data integrity training
- PT samples run multiple times and treated as “special”

Other Audit Findings

- Easy to fix findings
  - Add lab director’s phone number to QAP title page
  - Appoint deputy technical director and quality assurance officer
  - Add instrument serial numbers to logbooks
  - Calibrate dispensers quarterly rather than annually
  - Need access log for archived records

Other Audit Findings

- More involved audit findings
  - IDMP signing statements
  - SOP read receipts
  - Standard preparation documentation
  - Improve internal audit
  - Annual management review
  - Preventive actions

Other Audit Findings

- More involved audit findings
  - Annual demonstrations of proficiency
  - Calculation documentation
  - Complaint documentation
  - Need to expand on several QAP sections
  - Improved analyst training files

Other Audit Findings

- Information Services Issues
  - Unique sample log-ins
  - Data qualifiers
  - Analyst initials

Forms, Forms, Forms

ROCK RIVER WATER RECLAMATION DISTRICT LABORATORY
Method Detection Limit

Analyte: ICP Cadmium
Spike Conc: 0.005 mg/L
Method: EPA 200.7
Analyst: Sue Heinke

Replicate	Prep Date	Test Date	Analysis Result	units	Percent Recovery
1	27-Aug-09	1-Sep-09	0.0053	mg/L	106.0
2	31-Aug-09	1-Sep-09	0.0052	mg/L	104.0
3	2-Sep-09	3-Sep-09	0.0050	mg/L	100.0
4	4-Sep-09	8-Sep-09	0.0054	mg/L	108.0
5	11-Sep-09	14-Sep-09	0.0050	mg/L	100.0
6	17-Sep-09	21-Sep-09	0.0053	mg/L	106.0
7	22-Sep-09	23-Sep-09	0.0051	mg/L	102.0
Average			0.005186		103.7
Std Dev			0.000157		3.1
% RSD					3.0

calculated MDL	0.0005	
MDL upper confidence limit	0.0011	
MDL lower confidence limit	0.0003	
suggested reporting limit*	0.005	
LOQ, limit of quantitation	0.002	
Calculated MDL > 0	yes	
Calculated MDL > 0.1 * spike conc	yes	
Spike conc > calculated MDL	yes	
Spike conc between 1 and 10 times the MDL	yes	(0.0005 - 0.005)
acc replicate percent recovery, upper limit**		109.8
acc replicate percent recovery, lower limit**		97.6
all spikes within replicate percent recovery limits		yes
SDWA Dection Limit	na	
meets SDWA required detection limit	na	

* suggested reporting limit is MDL *3 or 0.005, which ever is greater

** acceptable replicate percent recovery is range of percent mean recovery ± 2 times the percent relative standard deviation

ROCK RIVER WATER RECLAMATION DISTRICT LABORATORY
Initial Demonstration of Method Performance

Analyte: ICP Cadmium
Method: SOP 127, Metals by ICP (EPA 200.7)
Analyst: Connie Potter

Replicate	QC Source	QC Lot No.	Certified Value	units	Analyst Result	Percent Recovery	Prep Date
1	SPEX LFS-1	8-180VY	0.100	mg/L	0.112	112	8/21/2007
2	SPEX-LFS-1	8-180VY	0.100	mg/L	0.116	116	8/27/2007
3	SPEX-LFS-1	8-180VY	0.100	mg/L	0.112	112	8/29/2007
4	SPEX-LFS-1	8-180VY	0.100	mg/L	0.114	114	9/4/2007
Average Recovery						113.5	
Standard Deviation						1.9	

Lab Generated Method Acceptance Limits*: 96 - 127 %
Lab Generated Standard Deviation Limit*: 5.2 %

* lab generated limits are based on analyses performed Jan. 1, 2006 - July 31, 2007.

SPEX solution is diluted 50 mL to 500 mL in-house.

ROCK RIVER WATER RECLAMATION DISTRICT
Demonstration of Continued Proficiency

Analyst: Connie Potter
Year 2009

Note: Continued Proficiency Demonstration can be either acceptable performance on PT sample or four consecutive laboratory control samples with acceptable results

Parameter	ERA PT Performance		Laboratory Control Samples			
	Study	Date	LCS ID	test dates		
Ammonia, ISE			era wp 172	08/24/09	08/31/09	09/08/09	09/11/09
Ammonia, titration							
BOD	WP 172	05/14/09					
BOD, carbanaceous	WP 172	05/14/09					
Chromium, hexavalent			era p161-984a	08/10/09	08/17/09	08/24/09	08/31/09
COD	WP 172	05/15/09					
Cyanide			era p165-502	08/04/09	08/10/09	08/17/09	08/24/09
Fecal Coliform	WP 167	12/29/08					
Fluoride			era p166-506	08/05/09	08/14/09	09/08/09	
Hardness			era p167-507	07/07/09	07/08/09	07/14/09	07/15/09
HEM	WP 167	12/29/08					
HEM, non-polar							
Nitrate			era wp 172	08/24/09	08/31/09	09/08/09	09/14/09
pH	WP 172	05/14/09					
Phosphorus	WP 172	05/18/09					
TKN			nist 2781	08/17/09	09/14/09	09/15/09	10/02/09
TDS	WP 172	05/14/09					
TS	WP 172	05/21/09					
TSS	WP 172	05/14/09					
TVS	WP 172	05/15/09					
GFAA Cadmium	WP 167	01/06/09					
GFAA Copper	WP 167	01/06/09					
GFAA Lead			NIST 1640	12/05/08	12/08/08	01/05/09	03/20/09
GFAA Nickel	WP 167	01/06/09					
GFAA Selenium	WP 167	01/07/09					
GFAA Silver	WP 167	01/07/09					
ICP Antimony	WP 167	01/07/09					
ICP Arsenic	WP 167	01/07/09					
ICP Barium	WP 167	01/07/09					
ICP Beryllium	WP 167	01/07/09					
ICP Cadmium	WP 167	01/07/09					
ICP Calcium	WP 167	01/26/09					
ICP Chromium	WP 167	01/07/09					
ICP Cobalt	WP 167	01/07/09					
ICP Copper	WP 167	01/07/09					
ICP Iron	WP 167	01/07/09					
ICP Lead	WP 167	01/07/09					
ICP Magnesium	WP 167	01/26/09					
ICP Manganese	WP 167	01/07/09					
ICP Molybdenum	WP 167	01/07/09					
ICP Nickel	WP 167	01/07/09					
ICP Potassium			SPEX LPC-1	10/19/09	10/23/09	10/28/09	11/11/09
ICP Selenium	WP 167	01/07/09					
ICP Silver	WP 167	01/07/09					
ICP Sodium			SPEX LPC-1	10/19/09	10/23/09	10/28/09	10/30/09
ICP Tin	WP 167	01/07/09					
ICP Thallium	WP 167	01/07/09					
ICP Titanium	WP 167	01/07/09					
ICP Vanadium	WP 167	01/07/09					
ICP Zinc	WP 167	01/07/09					

* Actual results can be seen on PT Study Reports or appropriate quality control spreadsheets.

And More Forms

ROCK RIVER WATER RECLAMATION DISTRICT

Standard Operating Procedure Signing Statement

Test Method: Total Kjeldahl Nitrogen

Reference: *Standard Methods, 20th Edition*, 4500-N_{org}

SOP Number: 306

I, the undersigned, have read, understand, and agree to perform the above referenced method as it is written in the SOP.

_____ _____

Signature Date

ROCK RIVER WATER RECLAMATION DISTRICT

Investigation and Corrective Actions Report for Proficiency Testing (PT) Studies

Laboratory:

Study Number:

Lab ID Number:

Analyte: Method:

Reported Value: True Value:

Acc. Limits:

Analyst: Date:

Laboratory Manager _____ Initials _____

Analyst _____ Initials _____

Analyst _____ Initials _____

Instructions

Upon receipt of an unacceptable PT study evaluation report, the Laboratory Supervisor conducts an investigation. The Supervisor will follow this procedure to investigate the cause of the unacceptable result. He/She will proceed through the checklist and initial or check each step to indicate completion, or write "NA" if the step does not apply. The investigation should be terminated if the causative error is found.

Corrective actions must follow all investigations. When an error is discovered the corrective actions should be appropriate. If no causative error is found then corrective actions should focus on measures to improve quality control such as analyst training, new standards or reagents, and instrument maintenance.

Include in the Investigation and Corrective Action Report all information deemed essential for the report. If the PT sample is reanalyzed (Step 11) include the result and a determination of acceptable accuracy. When an independent standard is analyzed (Step 12) include the results and a determination of acceptable accuracy. Additional pages and attachments should identify the study, analyte, and laboratory.

The Laboratory Supervisor shall complete all Investigation and Corrective Reports within 30 days of receipt of the PT study results. After review, the Laboratory Supervisor will send copies of completed reports for the WP studies the Illinois EPA Environmental Laboratory Accreditation Program Manager. Copies of completed reports for the DMR-QA studies will be sent to the IEPA DMR-QA Program Coordinator.

ROCK RIVER WATER RECLAMATION DISTRICT

Laboratory Preventive Action Plan

Laboratory:

Analyte: Method:

Analyst: Start Date:

Laboratory Manager _____ Date _____

Analyst _____ Date _____

Instructions

Upon notification of a potential source of non-conformance, the Laboratory Supervisor and/or Analyst will conduct an investigation.

If the source of non-conformance is identified, the laboratory will take steps to prevent the problem in the future. If no source of non-conformance is identified, laboratory will focus on measures to improve quality control such as analyst training, new standards or reagents, and instrument maintenance.

The Laboratory Supervisor shall complete all investigation and the Preventive Action Report within 30 days of notification of potential source of non-conformance. Report will be kept on file at the District laboratory and will be available for review by internal and external auditors.

Potential Source of non-conformance

Investigation of potential source of non-conformance

NELAP ACCREDITED LABORATORY

TNI

January 5, 2010

2010 Notable Improvements

- We better aligned some of our procedures with EPA methods
- Improved documentation
- Finally got some of our Information Services issues addressed

What's Happened Since 2010?

- NELAC program changes
- Added drinking water accreditation for anions and metals
- New equipment & methods at RRWRD lab
- RRWRD personnel changes
- Three more audits

NELAP Standard

Labs are now evaluated against the 2009 TNI Standard rather than the 2003 NELAC standard.

To address this change, RRWRD used a TNI developed template to completely update/rewrite our Quality Assurance Plan.

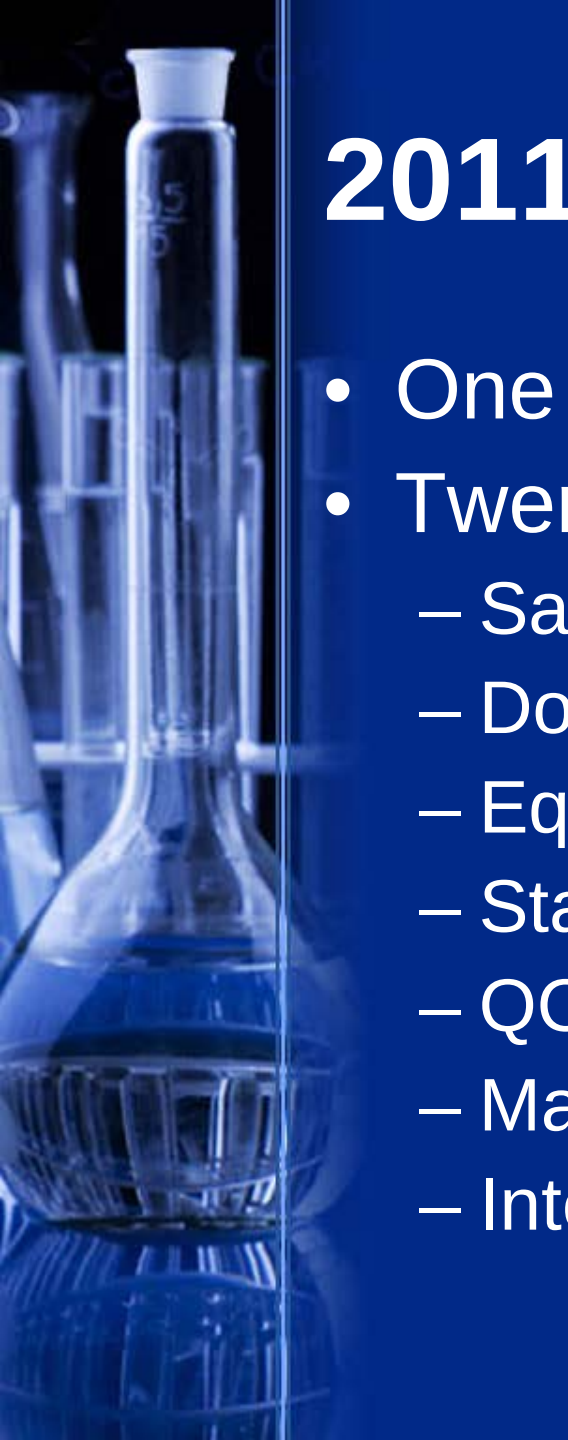
New Equipment

- Metrohm Ion Chromatograph
  - EPA 300.0
- Thermo ICP-MS
  - EPA 200.8
- Multiple small equipment replacements
  - DO meter
  - Hotblock for cyanide distillation
  - Support equipment including balance, incubators, ovens, digestion units

Personnel Changes

Promoted senior technician to a “Quality Assurance Analyst.” This position shares Quality Manager responsibilities with Laboratory Supervisor, specifically:

- Reviewing analysis data on a daily basis
- Monitoring corrective actions
- Performing internal audits
- Ensuring management system is implemented and followed.



2011 Audit

- One Critical Finding: HEM analysis
- Twenty Other Findings
  - Sample collection and check-in issues
  - Documentation of sample spot checks
  - Equipment checks
  - Standard preparation documentation
  - QC acceptance criteria not in SOPs
  - Maintenance logs for small equipment
  - Internal audits need more depth

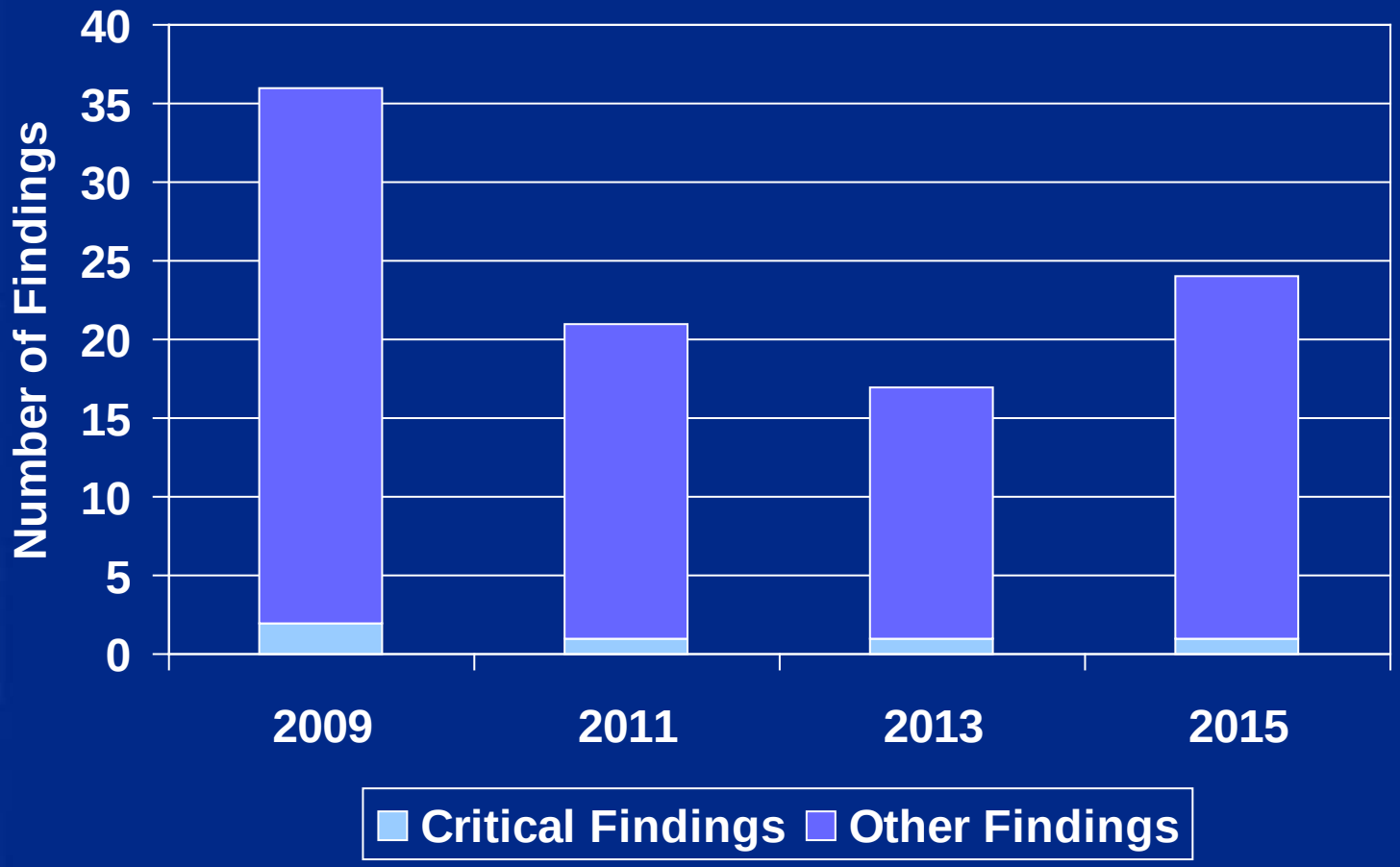
2013 Audit

- 1 Critical Finding
  - Electronic validation on data (Information Services Department)
- 16 Other Findings
  - Minor documentation issues such as recording temp checks and SOPs with handwritten notes
  - Issues with agreement between some SOPs and their reference methods
  - Laboratory Supervisor annual demonstration of competency

2015 Audit

- 1 Critical Finding
  - chlorine residual procedure (Operations Department)
- 23 Other Findings
  - All accredited methods not audited annually
  - Need to audit quality system as well as methods
  - All SOPs not reviewed biennially
  - Use if two point calibration curves
  - Analysis report format
  - Issues related to qualifying data
  - Support equipment temperatures out of range
  - Minor issues with some SOPs
  - Customer feedback

IEPA Audit History



Are NELAP requirements value added?

…. Maybe not all

- Tracking analyst ongoing competency
  - (We do QC everyday, isn't that enough?)
- Customer feedback requirements
  - (We're an in-house lab.)
- Two PT samples annually.
  - (We do QC everyday and PT samples are different matrix from routine samples.)
- Calculation documentation
  - (You can see formulas in spreadsheet.)
- Annual internal audits on every accredited method.
  - (Would like this reduced to every technique.)
- Required documentation is structured to meet NELAP requirements rather than Laboratory needs.

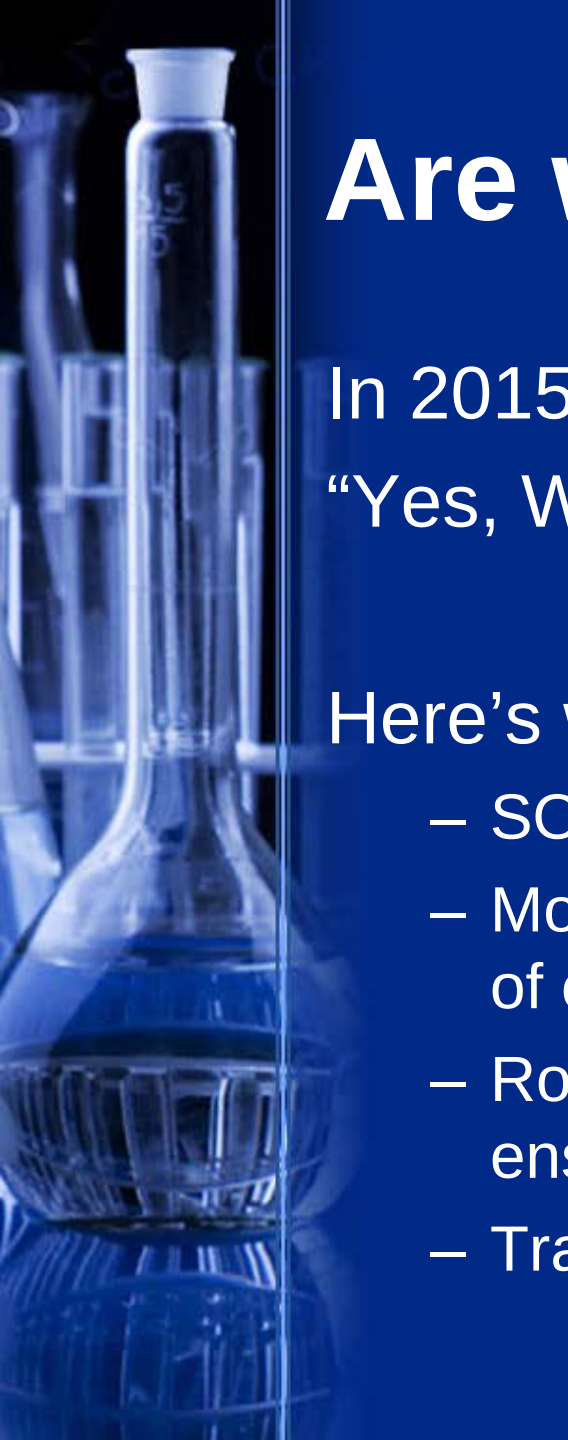
Did RRWRD Lab Achieve Promoted Benefits?

- Are laboratory operations smoother and more efficient?
- Have we improved public trust?
- Have we eliminated need for multiple certifications?
- Do we have more business from outside companies?

Are We a Better Lab?

- Are our analyses “better?”
  - More reliable?
  - More accurate?
  - More precise?
- In 2010, my answer was

  “No, but we are surely better documented.”



Are we a better lab?

In 2015, my answer to this question is:

“Yes, We are a better lab.”

Here’s why

- SOPs are aligned with methods.
- More documentation helps us identify sources of error associated with analyses
- Routine audits of SOPs and procedures ensure continuous quality improvement.
- Training is easier.

Additional Benefits

- Reduced “questioning” of District data by regulated industries.
- Increased revenue stream.
  - There is no contract lab in easy driving distance of Rockford. We do analyses for other municipalities and some local industries.

Benefits come with Challenges

- Paperwork
- Increase in time per analysis
- Dollar Costs
  - Annual fee
  - Proficiency Tests
  - Quality Control Solutions
  - Staff
    - Promoted one technician to a Quality Assurance Analyst
    - Hired an additional intern

Achieving NELAC Accreditation is only possible because of RRWRD's great Laboratory Staff.

References

- The NELAC Institute
  - www.nelac-institute.org
- Illinois Environmental Laboratory Accreditation Program
  - www.epa.state.il.us/labs
- Rock River Water Reclamation District
  - mjohnson@rrwrd.dst.il.us