



# **Monitoring Data Quality in Laboratory Deliverables**

in the

## **Contract Laboratory Program**

### **Data of Known and Documented Quality**

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Analytical Services Branch



# Agenda

- CLP and the QA Tool Box
- Performance Monitoring
- Evaluating Performance
- Poor, Improper, or Inappropriate
- Investigating Poor Performance
- Case Study



# Superfund Contract Laboratory Program

- National network of environmental testing laboratories
- Full spectrum of client-driven services:
  - Analytical Services:
    - Routine Organic and Inorganic (CASC)
    - Ultra Trace Organic (HRSM)
    - Modified Analyses: Modifications of Statements of Work
  - Centralized sample scheduling, tracking, and invoicing (SMO)
  - Electronic data delivery, evaluation, review, and reporting
  - Comprehensive Quality Assurance Services (QATS)



# CLP's QA Toolbox

- Electronic Sample Management Tools
  - Scribe
- Electronic Data Management Tools
  - EXES
  - LIMS
  - Scribe
  - Data Processing Tools





# CLP's QA Toolbox

- Statement of Work
- Performance Testing and Lab Reference Samples
- On-site Audits
- Staged Electronic Data Deliverable (SEDD)
  - Contract Compliance Screening (CCS)
  - Electronic Data Exchange and Evaluation System (EXES)
- Data Package and Electronic Media Audits
- National Functional Guidelines for Data Review (NFG)
- Data Validation



# Statement of Work

## Pre-defines:

- Analytical Methods
- Calibration
- Method Quality Control
- Performance Criteria
- Quantitation Limits
- Data and Documentation Management
- Data Reporting Elements



# Performance Testing Samples

Designed, developed, tested, and manufactured for CLP by EPA QATS Program

## Type:

- Pre and Post Contract Award
- Single blind or double blind
- Concentrated or full volume

## Purpose:

- Test laboratory's capability
- Provide a metric for data validation process
- Monitor and document performance





# On-site Audits

- Pre- and Post- Contract Award
- ISO9001 Lead Auditors and Certified Quality Auditors
- Identify contractual technical and reporting deficiencies
- Evaluate laboratory facilities, equipment, instrumentation, operations, and personnel
- Assess laboratory's continued capability
- Performed regularly





# Staged Electronic Data Deliverable

## CCS and EXES

- Completeness and compliance with technical, reporting, and administrative contract requirements
- Laboratory Self-Assessment
- Data Qualified by EXES
- Electronic Data Mining Tools

LAB	METHOD	QC_TYPE	TYPE	ANALYTE_NAME	Result	LIMIT_LOW	LIMIT_HIGH
Lab1	Semivolatiles	Method_Blank	Surrogate	4-Chloroaniline-d4	1	1	145
Lab1	Trace Volatiles	Method_Blank	Surrogate	1,1-Dichloroethene-d2	60	60	125
Lab1	Trace Volatiles	Method_Blank	Surrogate	1,1-Dichloroethene-d2	60	60	125
Lab1	Trace Volatiles	Method_Blank	Surrogate	1,1-Dichloroethene-d2	62	60	125
Lab2	Pesticides	CCV	Surrogate	Decachlorobiphenyl	24.4	-25	25
Lab2	Pesticides	CCV	Target	beta-BHC	24.8	-25	25
Lab2	Pesticides	CCV	Target	beta-BHC	24.8	-25	25
Lab2	Pesticides	CCV	Target	beta-BHC	24.8	-25	25
Lab2	Pesticides	CCV	Target	beta-BHC	23.5	-25	25
Lab2	Pesticides	CCV	Surrogate	Tetrachloro-m-xylene	18.9	-20	20
Lab2	Pesticides	CCV	Target	Endrin ketone	-19.9	-20	20
Lab2	Pesticides	CCV	Target	Endrin ketone	-19	-20	20
Lab2	Pesticides	CCV	Target	4,4'-DDE	18.7	-20	20
Lab2	Pesticides	CCV	Surrogate	Tetrachloro-m-xylene	18.9	-20	20
Lab2	Pesticides	CCV	Target	4,4'-DDE	18.7	-20	20
Lab2	Pesticides	CCV	Target	Methoxychlor	19.7	-20	20
Lab2	Pesticides	CCV	Surrogate	Tetrachloro-m-xylene	18.9	-20	20
Lab2	Pesticides	CCV	Target	4,4'-DDE	18.7	-20	20
Lab2	Pesticides	CCV	Surrogate	Tetrachloro-m-xylene	18.9	-20	20
Lab2	Pesticides	CCV	Target	4,4'-DDE	18.7	-20	20
Lab2	Pesticides	CCV	Target	4,4'-DDT	19.2	-20	20
Lab2	Pesticides	CCV	Target	Methoxychlor	19.8	-20	20
Lab2	Pesticides	CCV	Target	4,4'-DDT	18.7	-20	20
Lab2	Pesticides	CCV	Surrogate	Tetrachloro-m-xylene	18.3	-20	20
Lab2	Pesticides	CCV	Target	4,4'-DDT	-24.9	-20	20
Lab2	Pesticides	CCV	Target	alpha-BHC	19.5	-20	20
Lab2	Pesticides	CCV	Target	gamma-BHC (Lindane)	19.4	-20	20
Lab2	Pesticides	CCV	Target	alpha-BHC	19.6	-20	20
Lab2	Semivolatiles	CCV	Target	Caprolactam	-28.6	-30	30
Lab2	Semivolatiles	CCV	Target	Caprolactam	-28.6	-30	30
Lab3	Aroclors	CCV	Target	AR1016-1	13.7	-15	15
Lab3	Pesticides	CCV	Target	Endrin	-24.2	-25	25
Lab3	Pesticides	CCV	Target	Endrin	-24.2	-25	25
Lab3	Semivolatiles	Method_Blank	Surrogate	4,6-Dinitro-2-methylphenol-d2	11	10	130
Lab3	Semivola by SIM	CCV	Target	Benzo(g,h,i)perylene	-48.2	-50	50
Lab3	Semivola by SIM	CCV	Target	Benzo(g,h,i)perylene	-48.2	-50	50
Lab3	Semivola by SIM	CCV	Target	Benzo(g,h,i)perylene	-48.2	-50	50
Lab3	Trace Volatiles	CCV	Surrogate	1,2-Dichlorobenzene-d4	-19.7	-20	20
Lab3	Trace Volatiles	CCV	Target	Trichlorofluoromethane	-29	-30	30
Lab3	Trace Volatiles	Method_Blank	Surrogate	1,1-Dichloroethene-d2	60	60	125
Lab3	Volatiles	CCV	Target	1,1,2-Trichloroethane	-19.7	-20	20
Lab3	Volatiles	CCV	Target	Bromochloromethane	-23.4	-25	25
Lab4	Aroclors	CCV	Target	Aroclor-1260	23.6	-25	25
Lab5	Aroclors	CCV	Target	Aroclor-1248	23.5	-25	25
Lab5	Aroclors	CCV	Target	Aroclor-1248	23.6	-25	25
Lab5	Aroclors	CCV	Target	Aroclor-1248	24.6	-25	25



# Data Package & Electronic Media Audits

## Frequency:

- Data Package/Electronic Media Audits: Approx. 2 per year / lab / method

## Purpose:

- Identify contractual technical and reporting deficiencies
- Monitor data quality and integrity

## Process:

- Reprocessing of electronic media files
- Review of SEDD deliverables
- In-depth review of hardcopy data package





# National Functional Guidelines

## Purpose:

- Define data elements necessary for assessing data quality and usability
- Logical and thorough approach to data validation
- Consistency in data quality decision making
- Consistency in documenting data quality



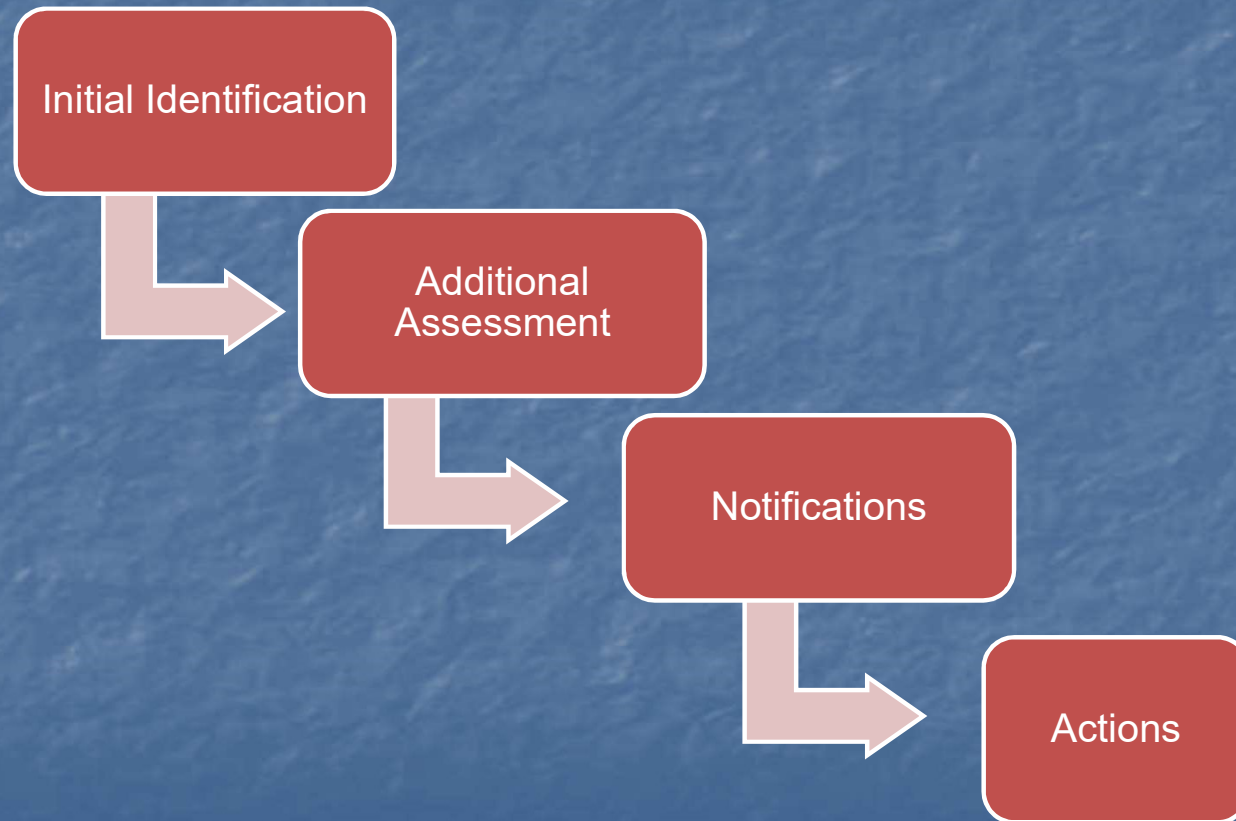


# Data Verification and Validation

- Focused on Regional data users' needs
- Utilizes National Functional Guidelines and project DQOs
- Enhanced by information from EXES
- Final tool in the CLP toolbox to obtain data that are
  - Complete and compliant
  - Consistent, accurate and precise
  - Representative
  - Usable



# Performance Monitoring Process





# Definitions

## ■ Poor Performance

- Decrease in, or not meeting, acceptable performance

## ■ Improper Practice

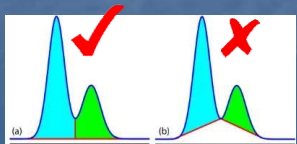
- An unauthorized deviation from acceptable procedures or practices; non-conformance with a specifications written in the CLP SOWs or contract

## ■ Inappropriate Practice

- A technically unjustified omission, manipulation, or alteration of data that bypasses the required QC parameters, making the results appear acceptable.

### Performance Categories

- ☐ Superior
- ☐ Good
- ☐ Acceptable
- ☒ Marginal



Peak Integration



# What is Laboratory Fraud?

- Laboratory fraud is defined as the deliberate falsification of analytical and quality assurance results, where failed method and contractual requirements are made to appear acceptable during reporting.
  - Intentional misrepresentation of lab data to hide known or potential problems
  - Making data look better than they really are



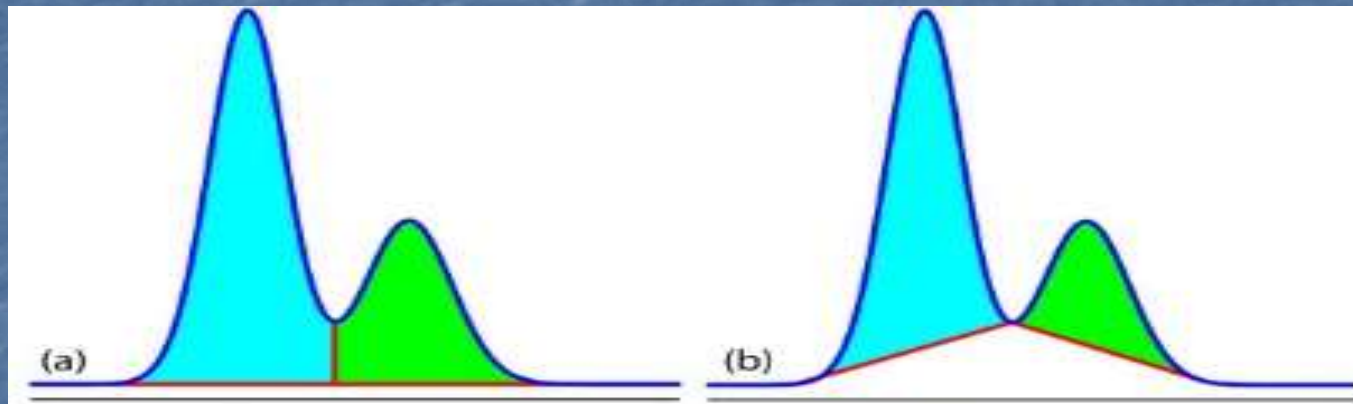


# Types of Laboratory Fraud

- Procedural Deceptions:
  - Not following critical steps of methodology
  - Short-cutting sample prep, calibration, analysis
- Measurement Deceptions:
  - Directly altering results
  - Time and date, conditions of experiment



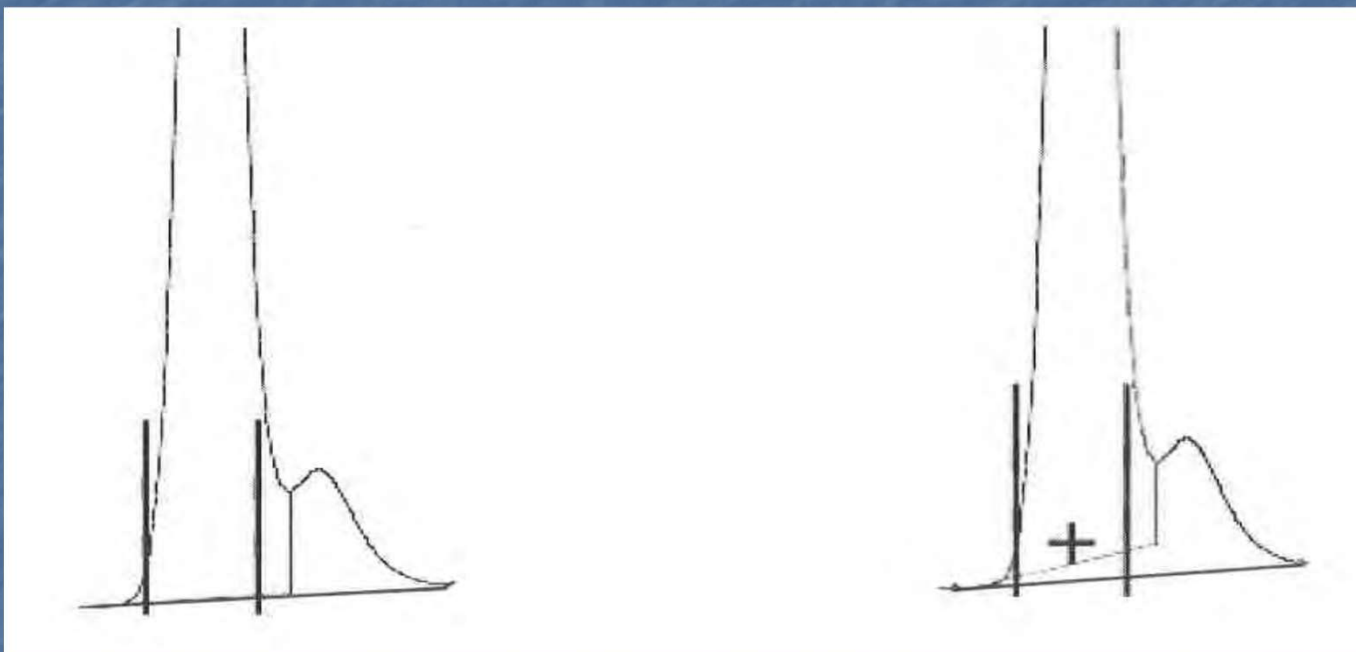
# What is Manual Integration?



Peak Integration



# Example of Improper Manual Integration



Delta BHC  
12.45 min 24.641 ng/ml  
response = 45837737  
%D = 23.2

Delta BHC  
12.45 min 23.915 ng/ml m  
response = 44486890  
%D = 19.6%



# Improper Manual Integration

Modified : Tue Feb 12 14:35:46 2013

Event : Manual Integration

Message : Changed peak amount for delta-BHC #2 from 24.6412ng/mL to 24.4597ng/mL

QuantFile: PESTC0046955.RES

Severity : 1

23.290 22.390

1

Modified : Tue Feb 12 14:35:51 2013

Event : Manual Integration

Message : Changed peak amount for delta-BHC #2 from 24.4597ng/mL to 24.275ng/mL

QuantFile: PESTC0046955.RES

Severity : 1

21.490

2

Modified : Tue Feb 12 14:35:55 2013

Event : Manual Integration

Message : Changed peak amount for delta-BHC #2 from 24.275ng/mL to 24.0887ng/mL

QuantFile: PESTC0046955.RES

Severity : 1

20.490

3

Modified : Tue Feb 12 14:35:58 2013

Event : Manual Integration

Message : Changed peak amount for delta-BHC #2 from 24.0887ng/mL to 23.915ng/mL

QuantFile: PESTC0046955.RES

Severity : 1

19.690

4





# Example of Time Travel

## G003493 epatemp Quantitation Report

(QT Reviewed)

Data File : \\Inst\GCMS\G-5973.net\DATA\G003493.d  
Acq On : 19 Apr 2013 14:34  
Sample : 3D18004-CCV3  
Misc : SSTD0208P  
DataAcq Meth:CLPACQ.M

Vial: 22  
Operator: RS  
Inst : G-5973  
Multiplr: 1.00

Quant Time: Apr 19 13:58:21 2013  
Quant Results File: SOMG003493.RES  
Integration File: RTEINT.P  
Quant Method : \\Inst\gcms\G-5973.net\METHOD\SOMG003493.M  
Quant Title : CLP SOM1.2 BNA Calibration  
QLast Update : Fri Apr 19 14:29:19 2013  
Response via : Initial Calibration



# Example of Time Travel

## G003493 epatemp Quantitation Report

(QT Reviewed)

Data File : \\Inst\GCMS\G-5973.net\DATA\G003493.d  
Acq On : 19 Apr 2013 14:34  
Sample : 3D18004-CCV3  
Misc : SSTD0208P  
DataAcq Meth:CLPACQ.M

Vial: 22  
Operator: RS  
Inst : G-5973  
Multiplr: 1.00

Quant Time: Apr 19 13:58:21 2013  
Quant Results File: SOMG003493.RES  
Integration File: RTEINT.P  
Quant Method : \\Inst\gcms\G-5973.net\METHOD\SOMG003493.M  
Quant Title : CLP SOM1.2 BNA Calibration  
QLast Update : Fri Apr 19 14:29:19 2013  
Response via : Initial Calibration



# Improper Laboratory Practices Examples

```
                                C010212 epatemp
                                Quantitation Report      (QT Reviewed)

Data File : \\Inst\qcms\C-5973.net\DATA\C010212.d
Acq On    : 2 Jun 2013 16:03
Sample    : 3F04004-CAL5
Misc      : VSTD10057 5GM SOIL
DataAcq Meth:VOAC.M

                                Vial: 8
                                Operator: SP
                                Inst : C-5973
                                Multiplr: 1.00

Quant Time: Jun 06 17:21:25 2013
Quant Results File: SOSC010207.RES
Integration File: Rteint.p
Quant Method : \\Inst\qcms\C-5973.net\METHOD\SOSC010207.M
Quant Title  : CLP SOM1.2-VOA-SOIL- 5GM Heated Purge
QLast Update : Thu Jun 06 17:21:16 2013
Response via : Initial Calibration
```

```
                                C010212 audit
\\Inst\qcms\C-5973.net\DATA\C010212.d\audit.txt
Created Fri May 31 16:09:20 2013
```

```
Modified : Fri May 31 16:09:20 2013
Event    : Quantitation
Message  : Calculation using initial calibration
QuantFile: SOSC010207.RES
Severity : 0
```

```
Modified : Fri May 31 16:09:23 2013
```





# Case Study: Organic Laboratory

## ■ Observations:

- Large number of manual integrations performed in QC samples.
- Manually integrated results just within technical acceptance limits.
- Inappropriate manual integrations: peak shaving and enhancement.
- Reprocessing of raw data produced acceptable integrations that did not meet technical acceptance criteria.
- Electronic audit trail files showed multiple integrations of many analytes.





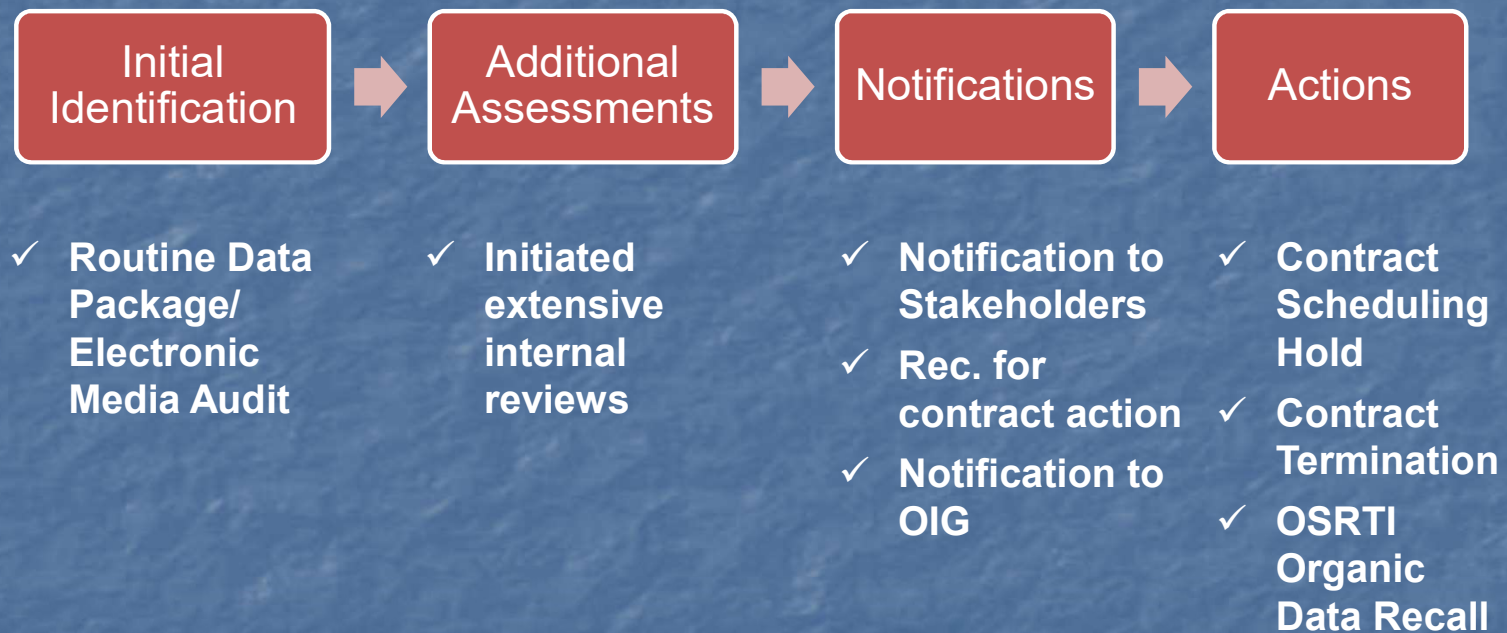
# Case Study: Organic Laboratory

## ■ Impact:

- Severe impact on many site decisions, past and future.
- Cost of rejected data, re-sampling, and associated costs in the millions of dollars.
- Investigation of laboratory on-going.
- CLP Program taking steps to prevent a re-occurrence.



# Investigation Steps





# The Path Forward

- **Growing the Tool Box**
  - Enhanced Contract Language
  - More PT Samples
  - New Electronic Tools
    - Electronic Data Mining Tools
    - Automated Data Review Tools
  - More Data Review Training
  - Better, more consistent communication
    - Initiation of the “Water Cooler Conversation”







# Questions?

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- Shari Myer, Analytical Services Lead Chemist, ASB: [myer.shari@epa.gov](mailto:myer.shari@epa.gov)