Case Studies of Faulty Laboratory Results Due to a Lack of a Strong Quality Management System

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Who is TNI?

- A 501(c)3 non-profit organization.
- A voluntary consensus standards development organization accredited by the American National Standards Institute (ANSI).
- An organization that administers the National Environmental Laboratory Accreditation Program (NELAP) which accredits over 1400 laboratories.
- Focus is on **reliable** data (*i.e., data of known and documented quality generated according to accepted professional practices of the industry)*.

New Strategic Initiative

- Develop a long-range plan for promoting the use of the TNI accreditation program to data users.
  - Show the value/benefits.
  - Demonstrate the improvement in performance and data quality.
- Phase One: White Paper, *Laboratory Accreditation Makes a Difference*, completed in 2020.
  - https://nelac-institute.org/docs/comm/advocacy/White%20Papers/WP-Value_101420.pdf
- Phase Two: Case Studies of Faulty Laboratory Data

Many Decisions Are Based on Having Reliable Data

- Demonstrate compliance to a regulated limit.
- Continue or cease remediation.
- Assess risk to human health or environment.
- Health surveillance.
- Water and wastewater engineering and technology implementation.

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What Is "Reliable" Data?

- What characterizes reliable data?
- How do we know that it is reliable?
- We only have an estimate of the true concentration.
- Quality Control results can be misleading for a variety of factors.

Laboratory Data Quality

- Laboratories say they generate
  - High quality data,
  - Definitive data,
  - Data of known and documented quality,
  - Legally defensible data, or
  - Valid data.
- What do any of these terms mean? How do laboratories ensure and document reliability?
- Are there any documents that can help ensure reliable data?

TNI's Quality Management System - Module 2 of the Laboratory Standard

- Developed over a 25-year period by a consensus body, the TNI Quality Management Systems committee.
- Committee has a balanced representation from all affected stakeholders: Accreditation Bodies, laboratories, data users, and other interests.
- Based on ISO/IEC 17025 (2005) with specificity added for environmental testing.
- Significant revisions in development, including update to 17025 (2017).
- Technical Modules 3-7 provide additional detail for specific types of testing.

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Guiding Principles

- **Flexible**: Allow laboratories freedom to use their experience and expertise in performing their work and allow for new and novel approaches. Specify the *What* and avoid where possible the *How To*.
- **Auditable:** Sufficient detail included so that the assessors can evaluate laboratories consistently.
- **Practical and Essential:** Necessary policies and procedures that should not place an unreasonable burden upon laboratories.
- **Widely Applicable:** Applicable to laboratories regardless of size and complexity.
- **Appropriate:** Ensure that data is of known quality and that the quality is adequate for the intended use.

Module 2 - Organization

- Introductory Material
- Management Requirements (Section 4)
- Technical Requirements (Section 5)

Module 2 - Introductory Material

- Introduction, scope, references, etc.
- A few key points:
  - Mandated test methods
  - EH&S not included

4.0 Management Requirements

- Organization
- Quality System
- Document Control
- Review of Work
- Subcontracting
- Purchasing
- Complaints
- Control of Nonconforming Work
- Corrective Action
- Preventive Action
- Records Control
- Internal Audits
- Management Review

Comparable to ISO 9000, these requirements are good management practices to ensure analyses are performed in an orderly and structured way.

5.0 Technical Requirements

- General
- Personnel
- Facilities
- Test Methods and Method Validation
- Equipment
- Traceability
- Sampling
- Handling of Samples
- Assuring the Quality of Results
- Reporting the Results

These requirements focus on technical issues in generating measurements.

Comparison to ISO 17025

- ISO 17025
  - Management Requirements
  - Technical Requirements
  - 35 pages

- TNI
  - Management Requirements
  - Technical Requirements
  - 150 pages
  - Includes **ALL** language from 17025

PLUS

  - Specific Requirements for Environmental Laboratories

AND

  - Data Integrity

Modules 3-7 Key Elements

- Method Selection
- Method Validation
- Demonstration of Capability
- Instrument Calibration
- Quality Control
- Data Acceptance/Rejection
- Sample Handling

Example: Instrument Calibration

ISO 17025

- Before being placed into service, equipment shall be calibrated to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications. It shall be checked and/or calibrated before use.

TNI Standard

- 7 pages of specific details related to initial calibration and calibration verification, including:
  - Removal of Calibration Standards – Low/High
  - Removal of Calibration Standards – Interior
  - Linear range
  - Minimum number of standards
  - Replacement of Calibration Standards
  - Measure of Relative Error

But We Know We Generate Good Data

- "We follow the method and do the QC."
- "Why must we do all this 'management' stuff that does not relate to quality?"

Quality System Vulnerabilities

- Expired standards
- Sample temperature
- Equipment not matched to sample
- No trip blanks for volatiles
- Internal audits do not cover all aspects of testing
- Interference check sample not analyzed
- SOP does not reflect actual practice
- DI water bottle not labeled
- Corrections not dated or initialed

These types of Vulnerabilities indicate a problem with the quality system which may or may not affect the quality of the data.

Examples of Faulty Data

- Inaccurate or incorrect result
- Insufficient documentation
- Non-conformance to mandated method
- Diminished confidence in result
- Not meeting customer requirements

Does not include Inappropriate Practices that may or may not have a direct impact on data quality, e.g.:

- Inappropriate manual integrations,
- Selective removal of calibration points,
- Spiking LCS/Surrogates into extract, not sample, or
- Adjusting time clocks.

However, these all relate to not having a robust data integrity system.

The PT Sample – Part 1

- Engineering firm asked lab to analyze sample for 8 specific volatile organics using the low-level option of SW-846 Method 8260 (25mL purge).
- The engineering firm sent a double-blind performance evaluation sample to the lab.
- The laboratory analyzed the sample using the normal method option for all volatile organics in the method (5 mL purge).
- The laboratory reported everything not detected. (This was the correct result under that option.)

The PT Sample – Part 2

- The engineering firm called the lab and said it was a PE sample. Could they look harder?
- The laboratory supervisor went into the computer system and was able to find 4 compounds below their normal reporting limit.
- The engineering firm called back and told the lab which 8 compounds were actually present.
- The laboratory supervisor “found” the other 4 compounds.

The PT Sample - Outcome

- Who committed fraud?
  - The engineering firm?
  - The sample log-in person?
  - The supervisor?
- Who was charged with fraud?
  - The analyst

QMS Failures

4.2.8 – Data Integrity

4.4 – Review of Requests, Tenders, and Contracts

4.13.2 – Technical Records

Changed QC Limits

- Laboratory copied method verbatim into internal SOP but changed QC limit from 80-120 to 60 – 140.
- Analyst performed improper manual integration to change a 69 to a 70.
- Why? Passed SOP limits before and after; failed Method limits before and after.
- Likely reason; 70-130 for a particular customer not

QMS Failures

4.2.8 – Data Integrity

4.2.8.5 – SOPs

4.3.2 – Document Control

Newborn Screening for Propionic Acidemia

- State health lab obtained result of 19.99830.
- Results greater than 20 indicate abnormal results and medical attention required.
- Results were reported as **Normal**, so no action taken.
- Mel, now 10, has severe brain damage.

QMS Failures

5.4.6 – Uncertainty

5.10.3 – Test Reports

Brain Eating Amoeba
Naegleria fowleri

- 2 deaths in St. John's Parish due to lack of chlorine in the distribution system attributed to lung exposure to amoeba (neti pot)
- Water utility decided to collect samples at the far ends of the system and check for residual chlorine.
- Two utility workers indicted for failing to test the water supply and then lying about it (after).
- Branch did not stop at 30 of the 48 water inspections he claimed to have done and Roussel did not stop for three of the six inspections.

QMS Failures

4.2.8 – Data Integrity

5.7.3 – Sample Recording

Coliform Outbreak in Walkerton, Canada

- Seven dead, 2,300 ill
- PUC manager Stan Koebel did not report lab results and did not inform public that well had been operating without a chlorinator
  - Did not want to interfere with Victoria Day
  - Did not think coliform was that bad
- Koebel sentenced to one year in jail
- $5 million in legal fees
- $1 billion class action lawsuit
- Ontario minister blamed for not regulating water quality

QMS Failures

4.2.1 – Management

5.10.1 – Reporting Results

High Coliform Results

- A large municipality had a MAJOR leak in a raw wastewater pipe under a river that resulted in fish kills across state lines.
- The laboratory was not prepared for handling samples that had high results outside of their normal range.
- An investigation revealed that the results had not been calculated correctly based on dilution factors.

QMS Failures
4.4.1 – Adequate Resources

Another Coliform Example

- A total coliform result was obtained by the laboratory. Instead of following state protocol to report the positive result, the laboratory vacated the result as "laboratory error" and informed the client to submit another sample.

QMS Failure
5.10.13 – Reporting Results

Train Car Derailment

- A train carrying many cars filled with lime spilled and lime spread over the ground.
- EPA Region 9 analyzed samples and found the pH to be 12.5 and thus the spill was classified as hazardous waste.
- Lime is calcium hydroxide and is used to make pH 12 buffer and at 25 C has a pH of 12.454, or less than 12.5
- EPA laboratory did not correct for temperature or do an expanded readout as required by the technique.

QMS Failure

5.4.1 – Method Deviation

Pesticide Remediation

- A major remediation project at a pesticide manufacturing facility generated hundreds of test results for organophosphate pesticides.
- During a pre-trial deposition, a review of the thousands of pages of raw data, the records to link the initial instrument calibration to the continuing calibrations could not be found.
- All of the data were ruled inadmissible by the court.

QMS Failure
4.13.3 – Historical Reconstruction

Pesticide Misidentification

- Analyst incorrectly identified dieldrin in soil samples because the analyst did not know how to establish retention time windows correctly.
- Engineering firm performed unnecessary remediation.

QMS Failures

4.1.5 – Management

4.2.8.4 – Experienced personnel

5.2.1 – Management of personnel

1.6 (Module 4) – Demonstration of Capability

Incorrect Spreadsheet

- Unprotected cell got changed resulting in dry weight correction to be off by a factor of 2.
- 18 months of incorrect data reported which affected decisions made by a large federal entity.

QMS Failure
4.3.3 – Document Control

Data Review

- Verbal results reported no volatile organics detected in several train cars of waste.
- Waste was then discarded in a municipal landfill not licensed for hazardous wastes.
- One week later, final report showed volatile organics exceeded action level.
- Verbal results were associated with different samples.

QMS Failure
5.10.2 – Test Reports

Mixed Waste

- Salesperson assumed “mixed waste” to be a mixture of organic and inorganic substances and RFP did not have a technical review by laboratory staff.
- Mixed waste actually refers to a mixture of radioactive and non-radioactive materials.
- Luckily, an assessor checked out the laboratory before samples were shipped and discovered the laboratory did not have the capability to handle radioactive samples.

QMS Failures

4.1.5 – Technical Management

4.4 – Review of Requests

Incorrect Reagent

- Some methods require use of reagents of specified purity (e.g., EPA 1664 requires 85% purity for hexane).
- Laboratory violated requirement in 40 CFR 136 to follow the method exactly as written.
- Result was likely accurate, but not acceptable.

QMS Failure
5.9.3– Mandated Methods

Benzidine? Really?

- Laboratory reported benzidine (4,4′-diaminobiphenyl) in 100's of samples from petroleum contaminated sites.
- Identification based on retention time and mass spectrum of benzidine standard purchased from a vendor.
- Upon investigation, standard was actually dibenzothiophene, a compound with the same melting point.

QMS Failures

5.6.3.2 – Reference Materials

1.7.1.1 (Module 4) – Second Source Verification

The Sludge Pond Sample

- Sent in for CLP soils analysis.
- Sample had 2 % solids.
  - Representative 30 g sample?
- GPC correction factor not applied – 2X multiplier.
- Results corrected to dry weight – 50X multiplier.
- MS performed on another unrelated sample in the batch.
- Result passed data validation but made no logical sense.

QMS Failures

4.4.1 – Review of Requests, Tenders, and Contracts

5.4 – Methods and Method Validation

5.4.7 – Control of Data

6 and 7-Day BOD

- Analyst did not want to come in on weekends and take readings for samples set up on Tuesday and Wednesday.
- Oxygen levels measured on Monday resulting in 6 or 7-Day BOD.

QMS Failure

5.4.1 – Deviation of Test Methods

Another BOD Example

- A laboratory analyzes three blanks when running samples for BOD. The laboratory reports the results, without qualifying, as long as one blank passes (<0.20 mg/L).

QMS Failure
1.7.3.1 (Module 4) – Negative Control

Passing PT Result, Really?

- Laboratory purchased QC check sample from PT vendor.
- Laboratory "corrected" result based on measured value in QC check and did not use calibration curve.

QMS Failures

4.2.2 (Module 1) – Analyze PTs like regular samples

1.7 (Module 4) – Calibration

Arsenic at Elementary School

- Laboratory reported high levels of arsenic in soil at elementary school.
- Laboratory had modified method without validating or receiving authorizations.
- School was shut down.
- Another laboratory analyzed samples and showed well below action levels.
- The first laboratory had not applied required Zeeman background correction due to high aluminum in soil.

QMS Failures

5.4.4 – Method Validation

1.5.1 (Module 4) – Method Validation

Mercury in Tuna

- In the 1990's FDA issued an advisory suggesting pregnant or breast-feeding women should avoid eating tuna due to high levels of mercury.
- The mercury was coming from the can due to the solder.
- Tuna does contain mercury, but not at the levels reported.
- Pregnant and breast-feeding women now should moderate their intake of king mackerel, swordfish, …
- Albacore and yellow fin tuna are now considered "good" choices and canned light tuna is now a "best" choice.

QMS Failures

5.9.3 – Negative Controls

1.5.2 (Module 4) – Limit of Detection

USEPA Region 5
Central Regional Laboratory

- Data were provided to the regional program offices for decision making and enforcement actions that were of "unknown quality and indefensible."
  - Lack of an approved Quality Management Plan
  - Little or no oversight of day-to-day operations
  - Low priority to QC and customer needs in favor of analyzing samples
  - SOPs out of date or non-existent
  - Staff not evaluating the quality of data
  - Plus 18 more areas of concern

QMS Failures
4.0 Management
5.0 Technical

US Geological Survey
Energy Geochemistry Laboratory

- QC procedures inadequate to detect quality issues.
- Analysts had violated method required activities without detection.
- "Chronic pattern of mis-conduct."
- Impacted 24 research projects with $108 million of funding, including:
  - trace metals analysis of water in the greater Everglades ecosystem;
  - assessment of uranium in the environment in and around Grand Canyon National Park for possible groundwater restoration; and
  - analysis of metals released into waters associated with natural gas production activities in Alaska.

QMS Failures

4.2.8.1 – Data Integrity Monitoring

4.14 – Internal Audits

FBI Forensic Laboratory

- 2600 convictions, including 45 on death row, in the 1980's and 1990's.
- Flawed results on hair analysis.
- FBI examiners *"exceeded the limits of science"* when linking hair to crime-scene evidence.
- The FBI knew as early as 1970 that these methods were not appropriate.

QMS Failure
5.4.2 – Selection of Methods

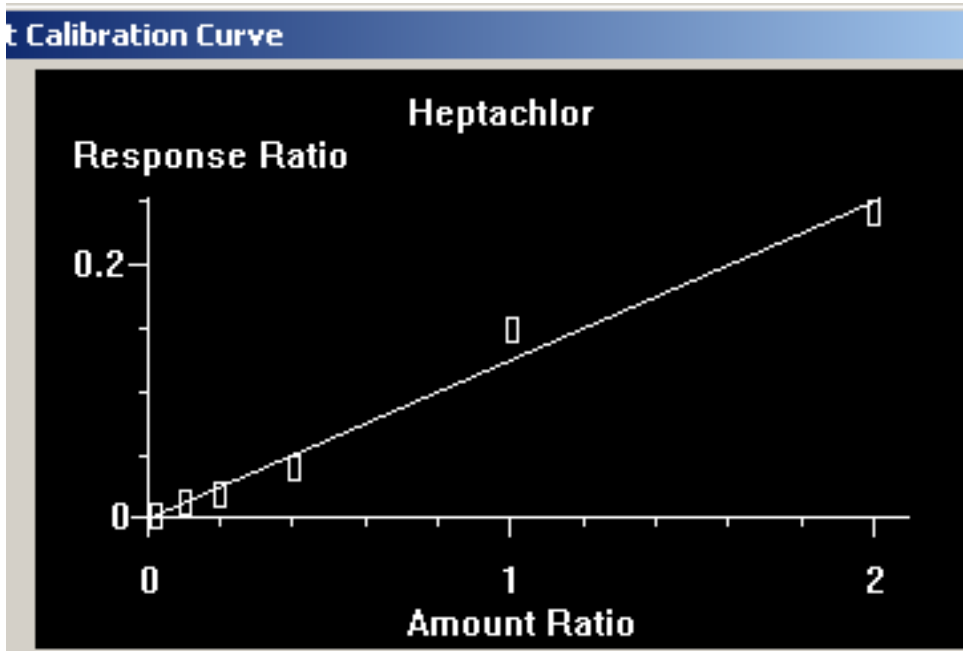
Aleutian Islands Project

- Phase 1 investigation into possible contamination from WW2.
- Because of holding times, decision made to extract samples in start-up lab in Anchorage and then ship extracts to continental US lab.
- All QC checks (LCS, MS, Surrogates) were 5-10% recovery (data of known and documented quality!)

QMS Failure
1.6 (Module 4) – Demonstration of Capability

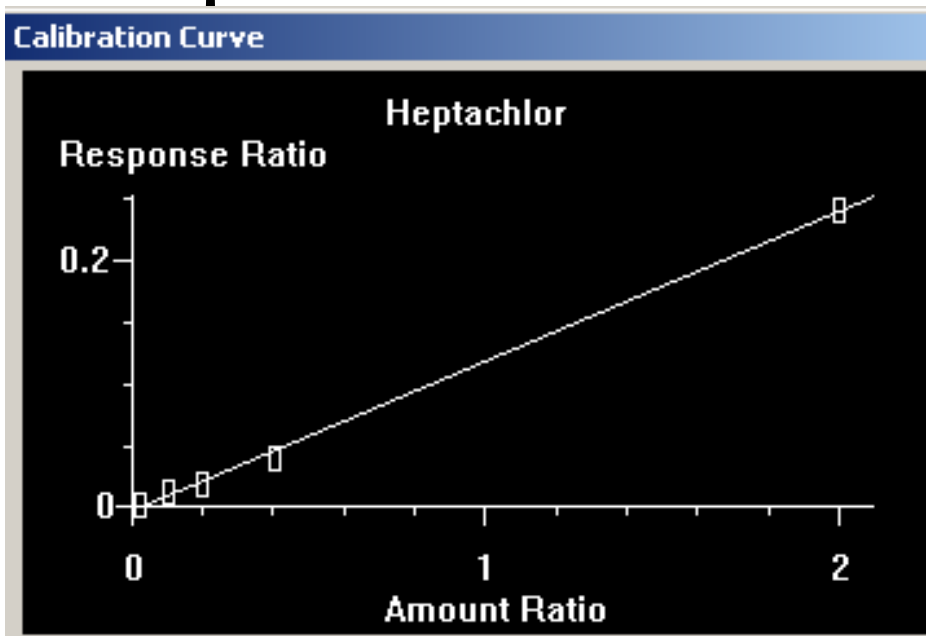
Removal of Interior Level to Pass Calibration Criteria

With 1.0 level standard



R^2 =0.983

Drop 1.0 level standard



R^2 = 0.998

QMS Failure
1.7.1.1 (Module 4) – Initial Calibration

Selective Instrument Calibration

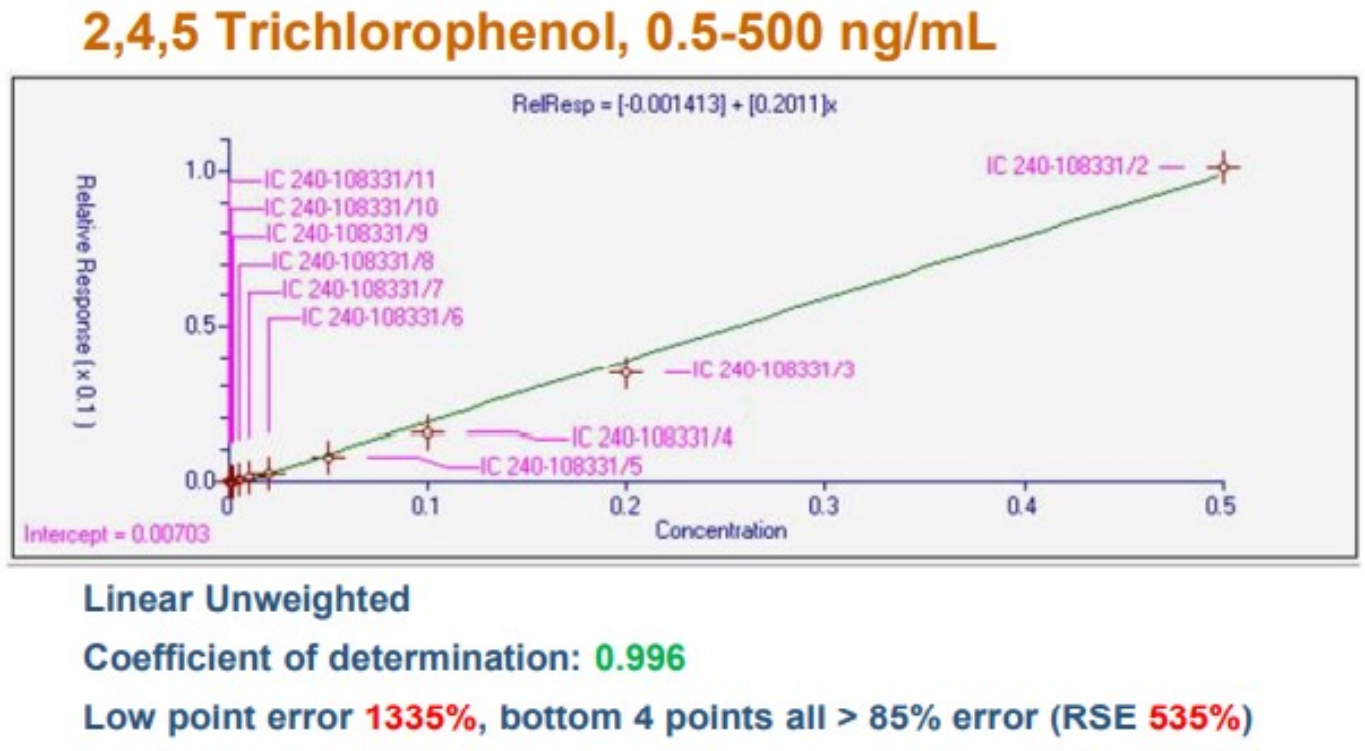
```
*  CALIBRATION FILES
   1   =CAL1.D  2   =CAL2.D  3   =CAL3.D  4   =CAL4.D  5   =CAL5.D  6   =CAL6.D

*        COMPOUND              1      2      3      4      5      6    *  AVG       %RSD
   ---------------------------------------------------------------------------------

   1) I  p-Terphenyl-d14          ----------------ISTD--------------------

   2)    Acenapthene-d10          ----------------ISTD--------------------
   3)    Hexachlorocycl...  0.331         0.267  0.248  0.242  0.226  0.263    15.56
   4)    Propachlor         0.623  0.559  0.510  0.462  0.454  0.528  0.523    12.12
   5)    Hexachlorobenzene  0.510  0.493  0.501  0.507  0.494  0.546  0.509     3.87

   6) I  Chrysene-d10             ----------------ISTD--------------------
   7)    Simazine           0.285  0.277         0.140  0.153  0.113  0.194    42.01
   8)    Atrazine           0.418  0.446  0.299  0.292  0.359  0.282  0.349    20.01
   9)    Pentachlorophenol  0.202  0.164  0.078  0.040  0.037  0.028  0.092    80.85
  10)    Lindane            0.250  0.272  0.208  0.211  0.292  0.256  0.248    13.51
  11)    Metribuzin         0.261  0.278  0.176  0.128  0.141  0.101  0.181    40.35
  12)    Alachlor           0.209         0.177  0.172  0.209  0.166  0.187    11.18
  13)    Heptachlor         0.120         0.097  0.097  0.127  0.107  0.110    12.31
  14)    Metalochlor        0.618  0.680  0.504  0.468  0.549  0.489  0.552    14.97
  15)    Aldrin             0.122  0.146  0.119  0.125  0.171  0.142  0.137    14.45
  16)    Heptachlor Epo...  0.087         0.084  0.089  0.116  0.114  0.098    15.76
  17)    Butachlor          0.273  0.286  0.207  0.190  0.200  0.161  0.219    22.48
  18)    Nonachlor          0.140         0.128  0.136  0.180  0.153  0.148    13.87
  19)    4,4-DDE            0.234  0.257  0.221  0.222  0.285  0.315  0.256    14.84
  20)    Dieldrin           0.140  0.150  0.143  0.148  0.190  0.202  0.162    16.49
  21)    Endrin             0.042         0.034  0.032  0.039  0.037  0.037    10.53
```

QMS Failure

1.7.1.1 (Module 4) – Initial Calibration

Use of R^2 Without Checking Error

- 0.5 ng/mL true value measured as 7.2 ng/mL



QMS Failure
1.7.1.1 (Module 4) – Measure of Relative Error

Wrong Method

- Client asked laboratory to test for PBDEs, but did not specify the specific analytes, the method, or any data quality objectives.
- Laboratory used internally developed method which did not meet client's needs.
  - Wrong analytes,
  - LOQ too high, and
  - Bias too high.

QMS Failure

5.4.4 – Non-Standard Methods

Reasons for Data Quality Problems

- Causes
  - Inadequate training
  - Inadequate management
  - Insufficient resources
  - Many, many more
- **Root Cause**
  - Lack of a Strong Quality Management System

Summary

- The QMS requirements in the TNI standard have a direct impact on both data quality and laboratory performance.
- Failures to correctly implement a robust QMS can result in loss of accreditation, decreased revenue, reanalysis, or data rejection.
- Failures can result in unnecessary remediation, illegal disposal, or other bad decisions based on faulty data.

Reliable Data

- Implementing a QMS provides confidence in the data
  - The reported result is good estimate of the true concentration.
  - The reported result is of known and documented quality.
  - The laboratory complied with mandated method requirements.
  - The laboratory implemented a strong quality management system to ensure confidence in the result.
  - The laboratory met customer requirements.
- Implementing a QMS improves laboratory performance
  - Better trained analysts
  - Better systems

Reliable Data

- Result can be reconstructed.
  - Sufficient documentation for sample, calibration, QC results, and SOP in use to fully reconstruct the processes leading to the result.
- Traceable.
  - Reference materials, reference standards, and reagents are all traceable.
- Competent analysts.
  - Training records, PT results, DOC results all demonstrate competency of analyst.
- Sample handled correctly.
  - Ability to trace sample from receipt to reported result.
- Quality control results document data quality.
- Reliable and transparent data through known laboratory activities.

Reliable Data

- Meets Daubert standards for data admissibility (e.g., "legal defensibility"):
  - technique has been tested,
  - there is a known rate of error, and
  - there are professional standards controlling the technique's operation.
- Reported correctly.
  - Met requirements relating to quantitation limits and data flagging.

TNI White Paper

- **Laboratory Accreditation Makes a Difference**
- Accreditation is not just about a quantitative improvement in data quality and a Quality Management System that is committed to the maintenance of quality. Rather these system aid in generating reliable data that can be used for making high confidence decisions.

https://nelac-institute.org/docs/comm/advocacy/White%20Papers/WP-Value_101420.pdf

New White Paper in Development: **Ways That a Strong Quality Management System Prevents Faulty Laboratory Results**

Recommendation

- TNI believes **ALL** environmental laboratories in the US should be accredited to the TNI standard.
- Texas - 151 Accredited Laboratories, including:
  - All commercial Laboratories,
  - All drinking water laboratories,
  - 68 municipalities and river authorities, and
  - A few laboratories from regulated industry and research groups.
- What about all the others? How many are there?
- How much faulty data is generated each year in Texas?
- Lack of a strong QMS can affect frequently analyzed parameters like BOD and coliform.

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THANK YOU!

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