



Internal and Electronic Audits

Satisfying Regulatory Requirements

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October 28, 2009



This Training

- Presents techniques for auditing
 - Planning, Implementation and Performing
- Review of things to audit
 - Audit trail
 - Spreadsheets
 - Data changes to electronic records
- Applicable to drinking water and wastewater regulatory data reporting



OFFICE OF INSPECTOR GENERAL

Catalyst for Improving the Environment

Evaluation Report

Promising Techniques Identified to Improve Drinking Water Laboratory Integrity and Reduce Public Health Risks

Report No. 2006-P-00036

September 21, 2006



EPA IG Report

EPA IG Report Expert Panel - Appendix E

[www.epa.gov/oig/reports/
2006/20060921-2006-
P-00036.pdf](http://www.epa.gov/oig/reports/2006/20060921-2006-P-00036.pdf)



Vulnerabilities

- Date entry errors
- Vague requirements for LIMS security and entry
- Adherence to SOP (Laboratory)
- SOP has ambiguities
- Lack of meaningful ethics training and testing
- Backdating
- Indiscriminate use of software to correct problems

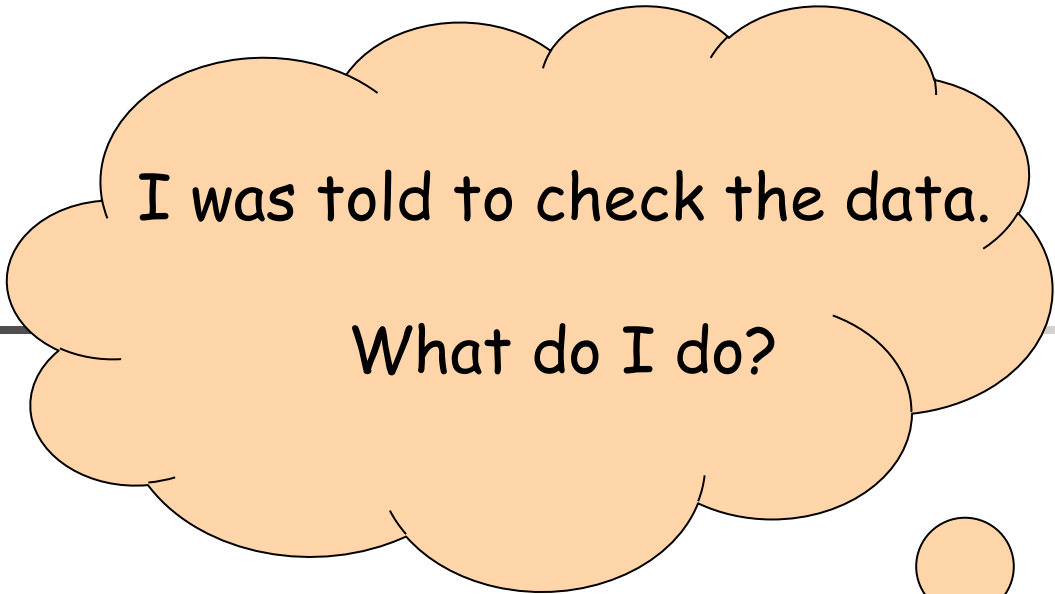
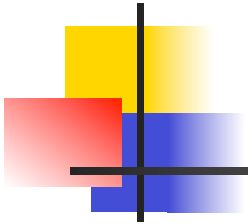
EPA IG Report Expert Panel - Appendix E - Partial



Vulnerabilities

- Improper calibration
- Recordkeeping errors
 - Missing calibration or standards logs
- Inadequate training (sensitivity loss)
- Rerunning QC without rerunning samples
- Failure to review data, records
- Backups not timely or secured

EPA IG Report Expert Panel - Appendix E - Partial



What do you do?





Data Surveillance

- In-depth Data Review
- Training of Assessors Underway
 - Improved data review skills
- Laboratory must adopt better data review as part of internal audit
 - Laboratory must have program on reporting data review weaknesses and vulnerabilities



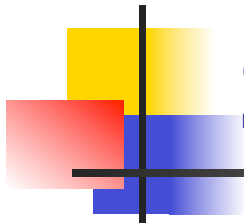
Internal Audits

- Qualified Assessor
- Schedule and Plan each Audit
- Timeframe to notify staff
- Audit to:
 - Lab's system
 - NELAC standard
 - Reference methods
 - Client requirements (permit)



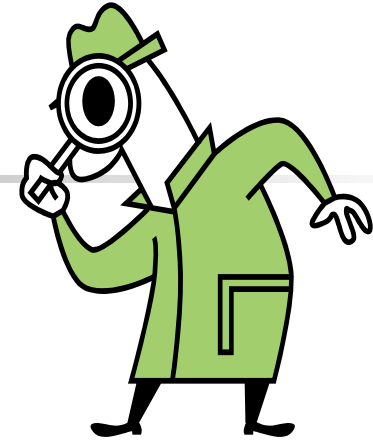
Technical Review

- Read SOP
- Read regulation or mandated method
- Read checklist
- Make list of questions
- Interview personnel
- Review data
- Identify findings or conformance
- Record information



SOP

- Clear Instructions
- Details Activity
- Specific to Function
- Handling Routine Problems
- Assessing Data Integrity



If not ... report observation so
system improves



Audit Elements

- Review Test Methods for Audit
- Review Questions for SOP
- Review Calibration Data
- Review Batch Test Data
- Review DOC Data
- Review MDL Data, If Applicable
- Review PT Data
- Review Related Test Data Records
 - E.g.; support equipment, reagent/standards logs



Audit Elements

- Track sample from receipt to report
- Track data from report to raw data records
- Interview personnel
- Develop listing of findings (deficiencies)
 - Opportunities for Improvements



Audit Procedure

- Opening meeting (Define scope of audit)
 - NELAC specific requirements
 - State (permit) specific requirements
 - Procedures included in audit
- Keep records of data/information reviewed
 - State (permit) specific requirements
- Closing meeting
 - NELAC specific requirements
 - State (permit) specific requirements



Review Records

- Define personnel responsible
- Review the records, data, etc.,
 - Factual
 - Complete
 - In compliance with SOPs, SAP, or QAPP
- Identify errors or omissions



Control of Data

- Data Transfers (NELAC 5.5.4.7.1)
 - Manual and Electronic
 - Quality Control Reviewed and Evaluated

What to look for....



Data Set

- Proficiency Testing Data
- Detection/Quantitation Limits
- Demonstration of Capability (DOC)
- Calibration
- Preparation
 - Sample
 - Standards, QC materials, reagents
- Sample Batch

HOW MUCH?



NELAC Definition

- Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.
 - A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours.
 - An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.



Computers (NELAC 5.5.4.7.2)

- Software Development & Validation
 - Laboratory
- Procedures for Data Protection
 - Integrity
 - Confidentiality
- Maintain Equipment
 - Proper functioning
- Procedures for
 - Security
 - Access
 - Changes to Test Records

Does the lab retain
original electronic
data?



Electronic Data Review



- Do not be afraid...
 - Look at what is on screen as you would if the data were on paper
 - Ask lab personnel to show you how the data is processed
 - Listen for places in the process where data may be changed without documentation
 - Do not forget spreadsheets!
 - Documented procedures must be available
 - NELAC requires documented procedures for data handling



GALP

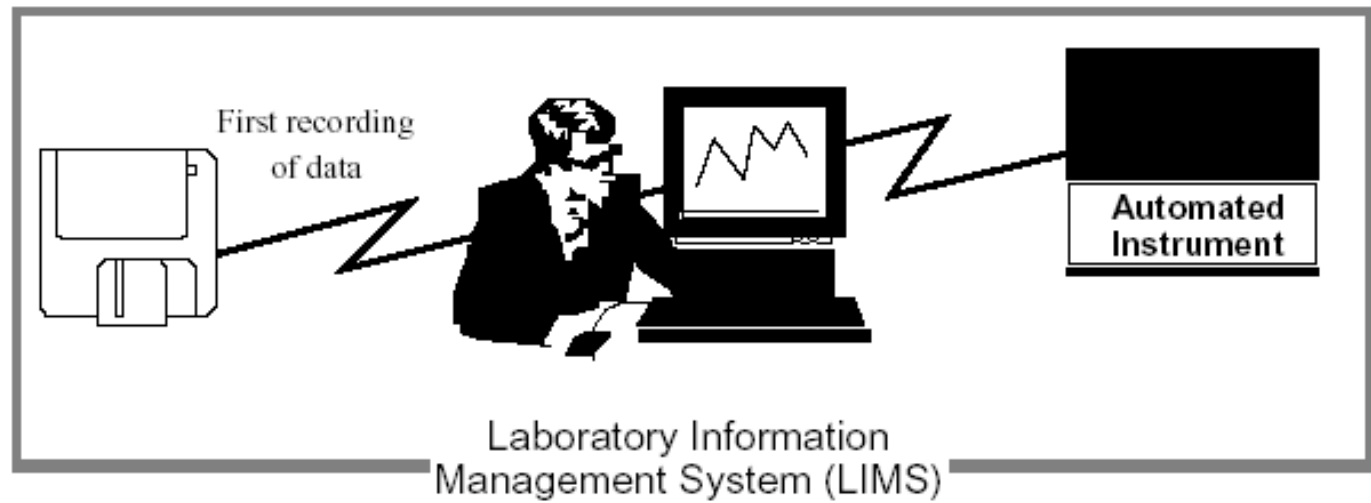


Figure 1.3. Automated Laboratory Systems Subject to the GALPs

Not GALP

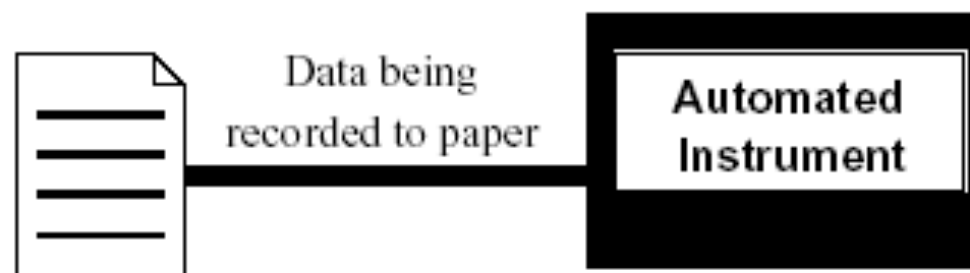


Figure 1.2. Automated Laboratory Systems NOT Subject to the GALPs

- Often overlooked ---
 - Required to ensure data integrity



Computer QA Elements

- Control of electronic documents
- Record keeping
- Software validation
- Maintenance of equipment
- Procedures for protecting data
 - Confidentiality, integrity
- Procedures for data security
 - Access control and authorization for changes



Six Principles

- Integrity of data
- Appropriate formulas and algorithms
- Track entry, modification and recording
- Change control – software and operations
- Documented procedures
- Minimize and manage LIMS failure



Sample Handling

- Inorganic Records
 - Preservation
 - Distillation, pH adjustment
 - Timing
 - Digestion Records
 - # samples per hour
 - Reagents, Standards
- Electronic Records Maintained
 - As applicable



Sample Handling

- Organic Records
 - Preservation
 - Extraction, pH adjustment, Clean-up
 - # samples per hour
 - Timing, Temperature
 - Equipment Records
 - Tune, purge rates, injection amounts
 - Reagents, Standards
- Electronic Records Maintained
 - As applicable



All Data

- Method Blank
- Control Sample (LCS, LFB, SB)
- Duplicate
- Matrix Spike
- Control Limits
 - How set?
 - What do the limits tell you?

Look at data



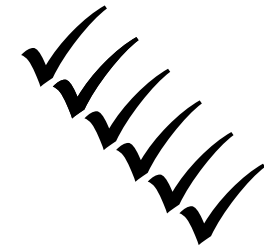
Quality Control

- Check calculations
 - R, RPD, RSD, SD, Many Others
 - Check spreadsheets
 - Check macros
 - Check values
 - Control limits
 - Statistical parameters, as used by lab
- HOW?



Control of Data

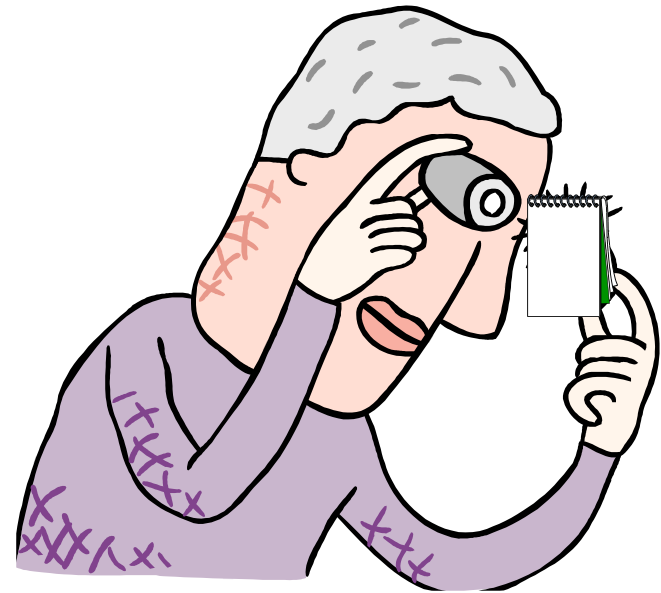
- Appropriate Checks (5.5.4.7.1)
 - Calculations
 - Data transfers
 - Transcription errors
 - QC measures
 - Manual integrations





Data Reduction

- Procedures
- Calculations
- Computers
 - Audit Trails
 - On/off
 - Talk with vendor
 - Method Files





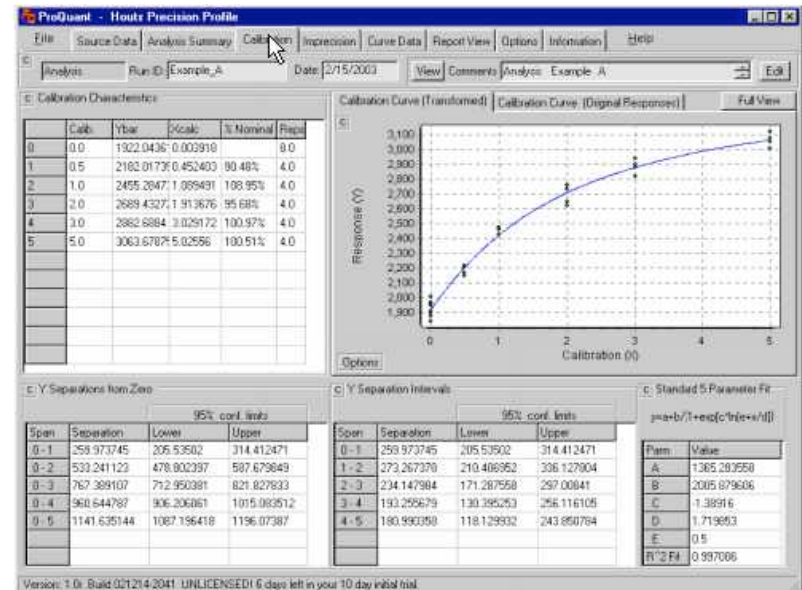
Improper Practice

- A scientifically unsound or technically unjustified omission, manipulation, or alteration of procedures or data that bypasses the required QC parameters, making the results appear acceptable
- Any alteration of data such that the data are unauthentic or untrue representations of the experiment or test performed



Calibration

- Instrument (5.5.5.2.2)
 - Initial
 - Criteria
 - Frequency
 - Standards





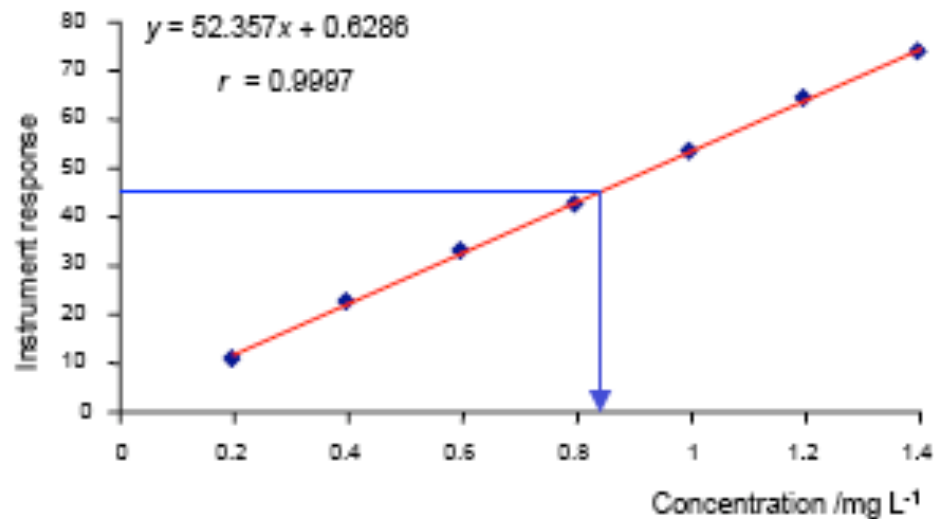
Calibration Records

- Complete
 - Person(s), Date
 - Method, Equipment
 - Calculations
- Continuing
 - Acceptance
 - Qualifiers



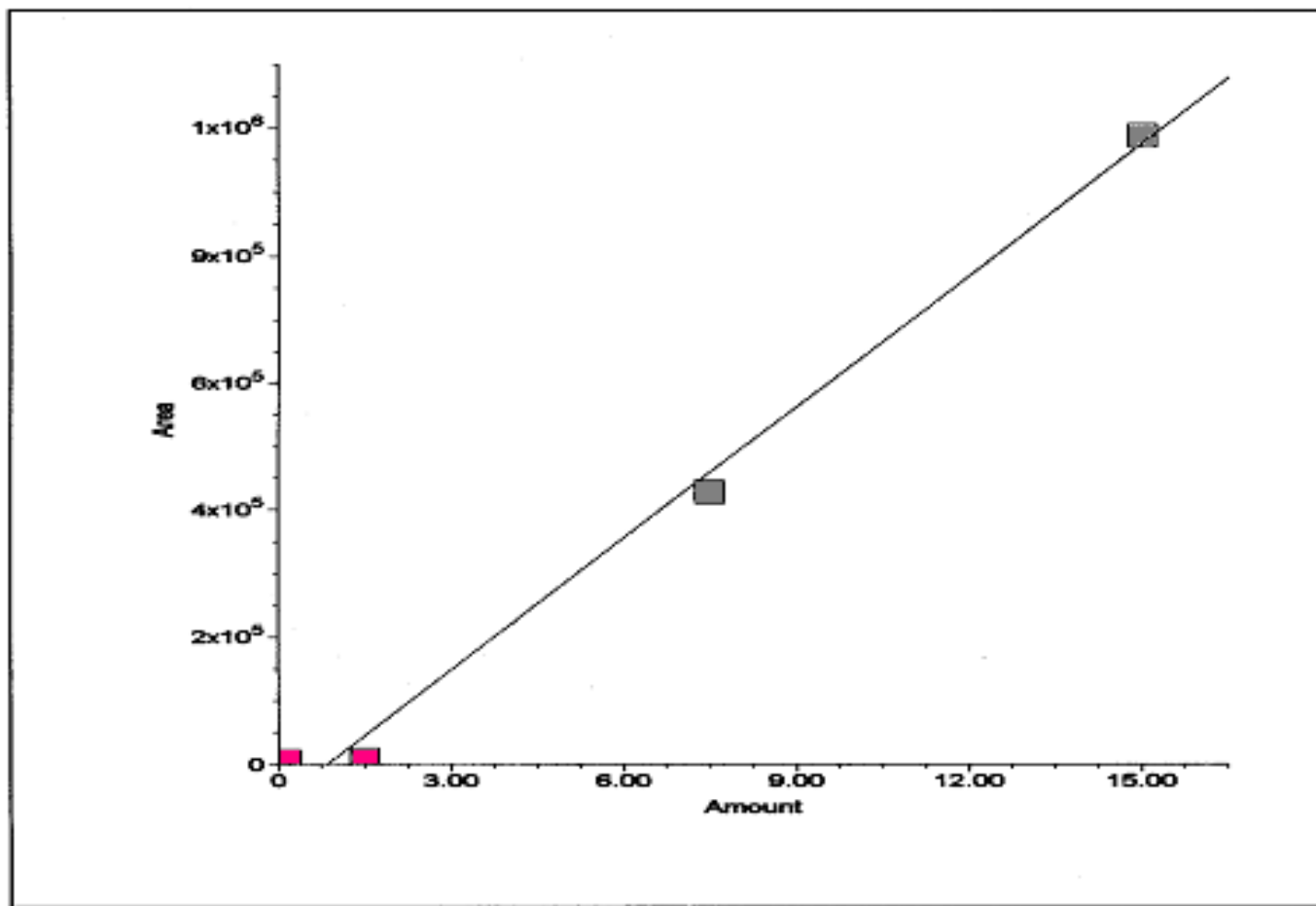
Basics

- Linear Systems
 - Response to concentration
 - $y = mx + b$



Calibration Examples

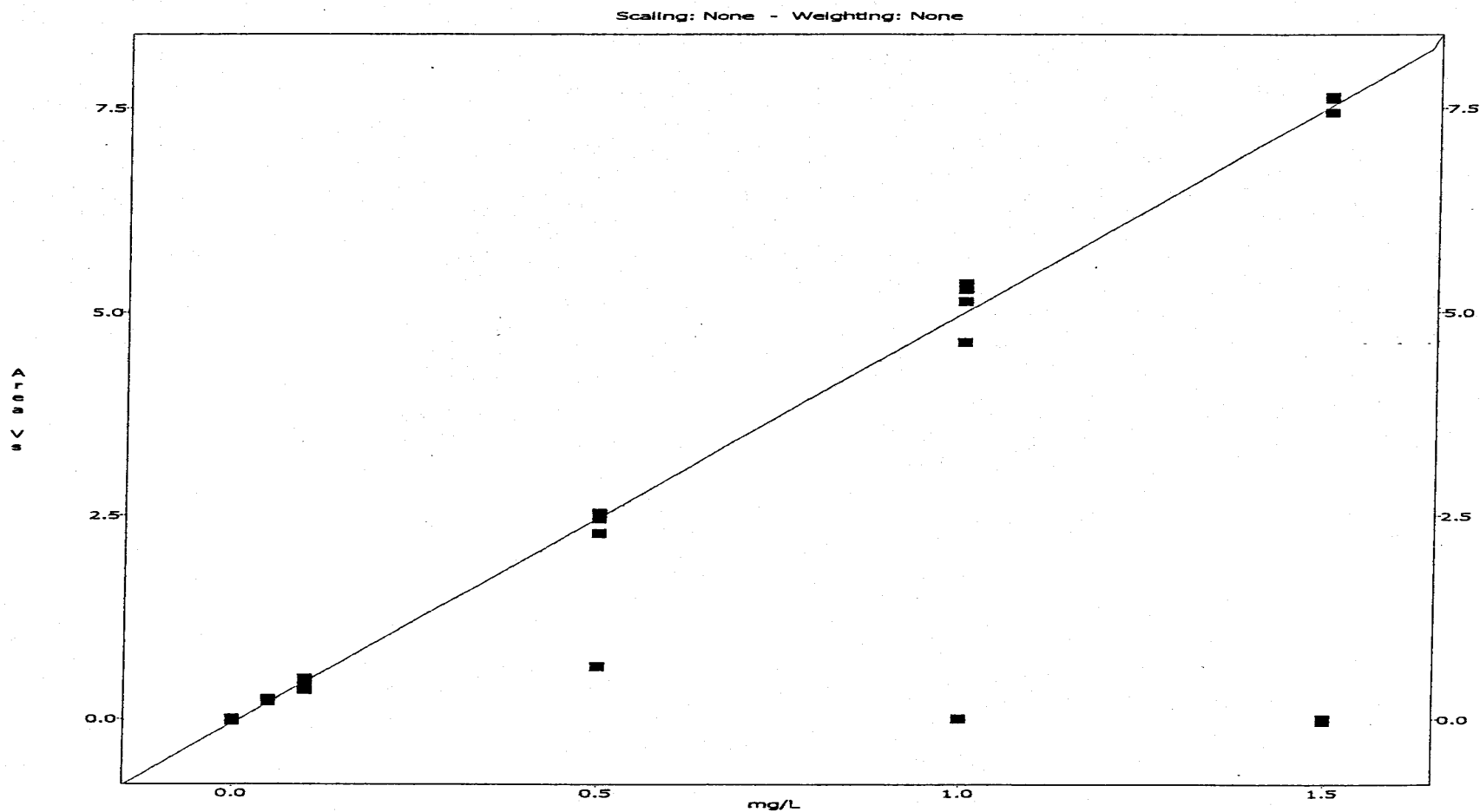
5. Component:Nitrate-N
Standard:External Fit Type:Linear
Origin:Ignore Calibration:Area
 $r^2=0.991983$
Amt= $1.28e-005$ *Resp+0.8488



NO3/NO2 High

| Lvl | Area | mg/L | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Replic STD | Replic % RSD | Residual 1st Poly |
|-----|---------|------|---------|---------|--------|---------|---------|---------------|-----------------|----------------------|
| 1 | 7446844 | 1.50 | 7446844 | 7640317 | 8582 | -23553 | 7617180 | 4150014.8 | 91.5 | 1.1 |
| 2 | 5138770 | 1.00 | 5138770 | 5283882 | 8461 | 5357485 | 4638801 | 2296292.0 | 56.2 | -2.7 |
| 3 | 2463948 | 0.50 | 2463948 | 2531248 | 648727 | 2532188 | 2286252 | 813298.5 | 38.9 | 0.6 |
| 4 | 354901 | 0.10 | 354901 | 397555 | 396226 | 438138 | 500404 | 54927.3 | 13.2 | 20.3 |
| 5 | 222958 | 0.05 | 222958 | 236342 | 240669 | 220855 | 249279 | 12022.0 | 5.1 | -7.2 |
| 6 | -138 | 0.00 | -138 | -20601 | -12640 | -8455 | 7763 | 10997.7 | -161.4 | |

1st Order Poly
 Conc = 1.979e-007 Area + 9.480e-003
 r = 0.9996



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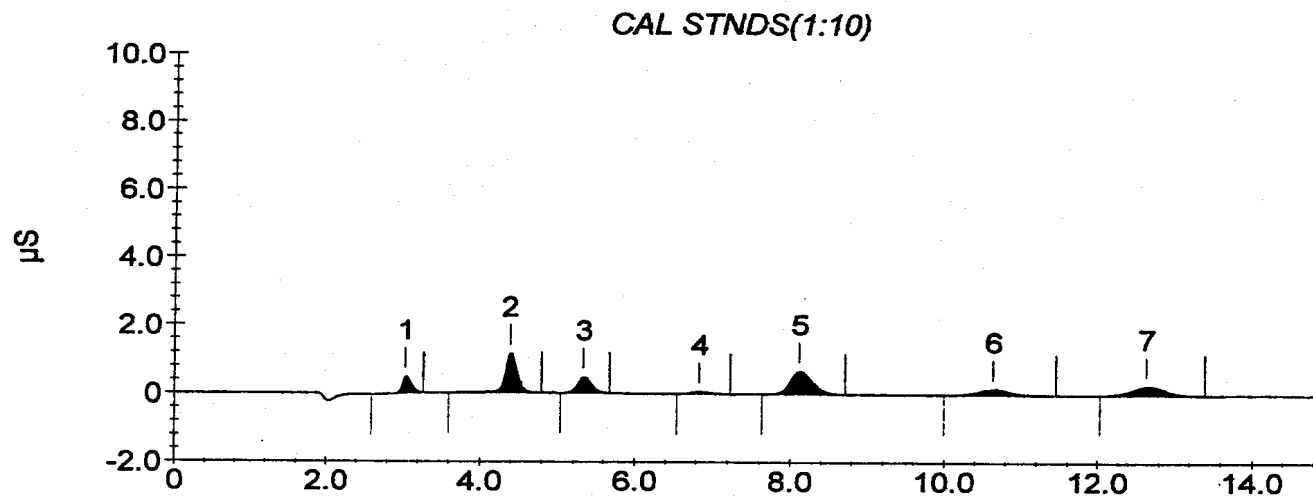
Calculating Back Into the Curve

| Std./Sample | Response | Conc. |
|------------------|----------|--------|
| 1 | 0.002 | 0.460 |
| 5 | 0.033 | 4.503 |
| 10 | 0.073 | 9.719 |
| 20 | 0.158 | 20.804 |
| 30 | 0.239 | 31.368 |
| 60 | 0.452 | 59.145 |
| SO4 CNTR | 0.240 | 31.493 |
| SO4 CCV | 0.072 | 9.566 |
| SO4 71890 (1/10) | 0.028 | 3.830 |

Calibration Response

Peak Information : Found Components

| Peak # | Retention Time | Component Name | Cal Response (Previous) | Cal Response (Measured) | Cal Response (New) |
|--------|----------------|----------------|-------------------------|-------------------------|--------------------|
| 1 | 3.03 | Fluoride | 90310 | 42690 | 66500 |
| 2 | 4.38 | Chloride | 233688 | 133615 | 183651 |
| 3 | 5.33 | Nitrite-N | 128330 | 62159 | 95245 |
| 4 | 6.83 | Bromide | 23596 | 12946 | 18271 |
| 5 | 8.12 | Nitrate-N | 236292 | 136425 | 186358 |
| 6 | 10.63 | Phosphate-P | 68789 | 53275 | 61032 |
| 7 | 12.63 | Sulfate | 117416 | 81020 | 99218 |





Spectrometry Data Review

- Calibration
- Verification
- Quality control data
- Support information



Chromatography Data Review

- Retention Time
- Background
- Integration Parameters and Practices
- Selection



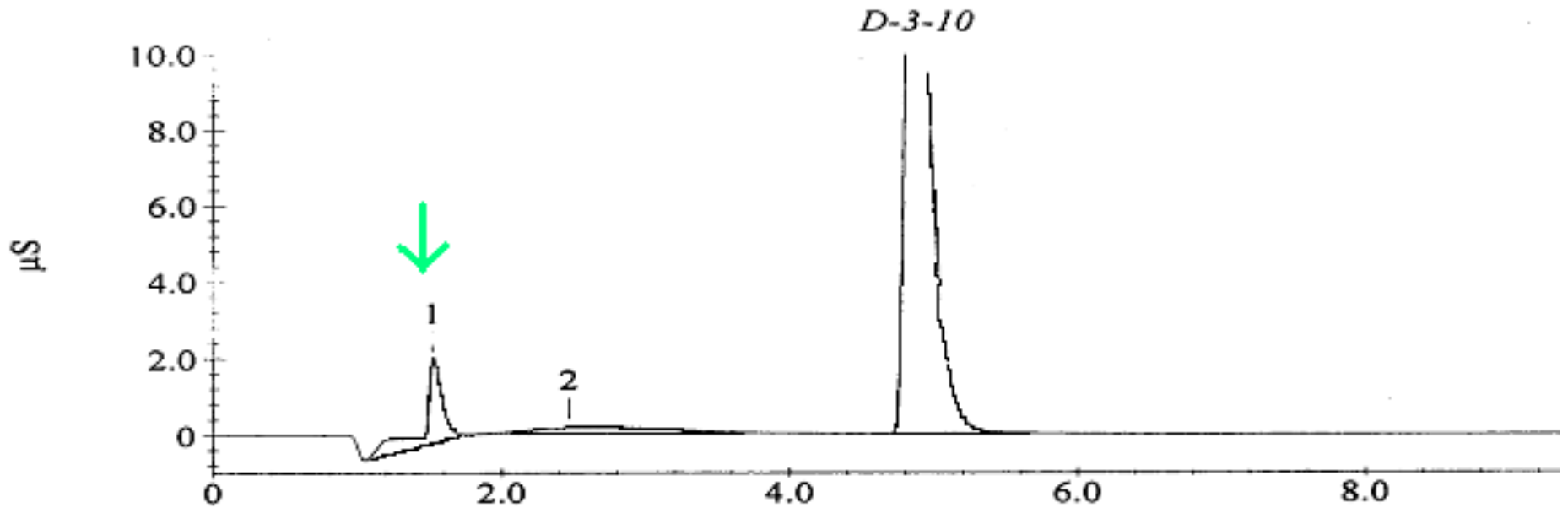
Integration

- Integration is often set by peak start to peak end for area, keeping in mind those compounds that have peak tailing
- Integration of peak heights
- Threshold is set correctly
- Also look for extended or shortened integrations, this requires the analyst zooming in

Integration Issues

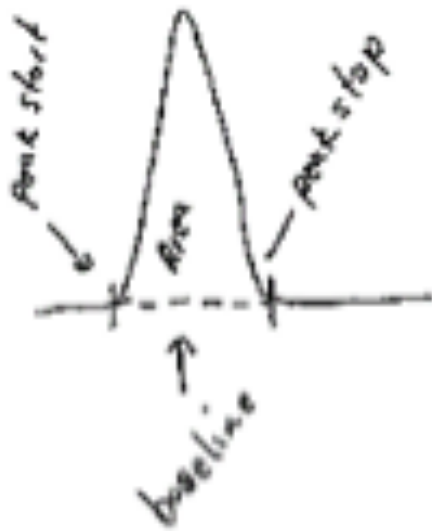
Peak Information : All Peaks

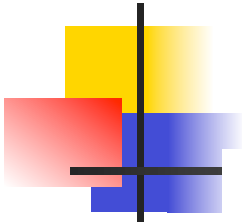
| Peak # | Component Name | Retention Time | Amount (PPM) | Peak Area |
|--------|----------------|----------------|--------------|-----------|
| 1 | CHLORIDE | 1.52 | 83.59 | 202168 |
| 2 | | 2.47 | 0.00 | 114525 |
| 3 | SULFATE | 4.85 | 911.56 | 1936986 |



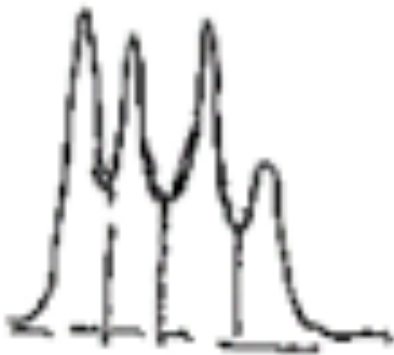
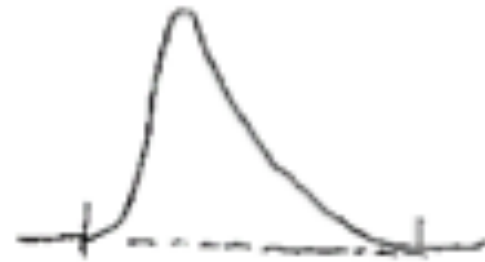
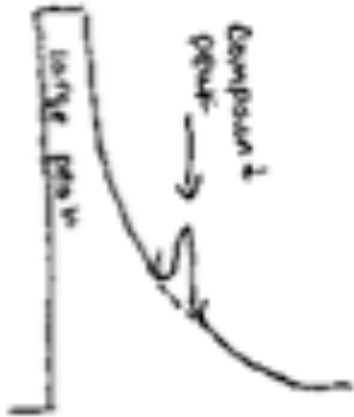


Peak Integration



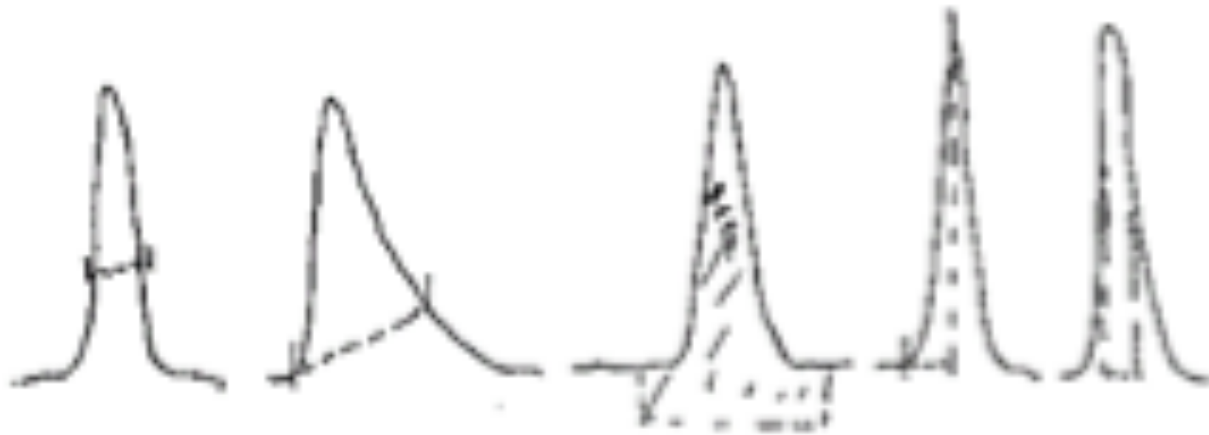


Proper Integrations





Improper Integrations



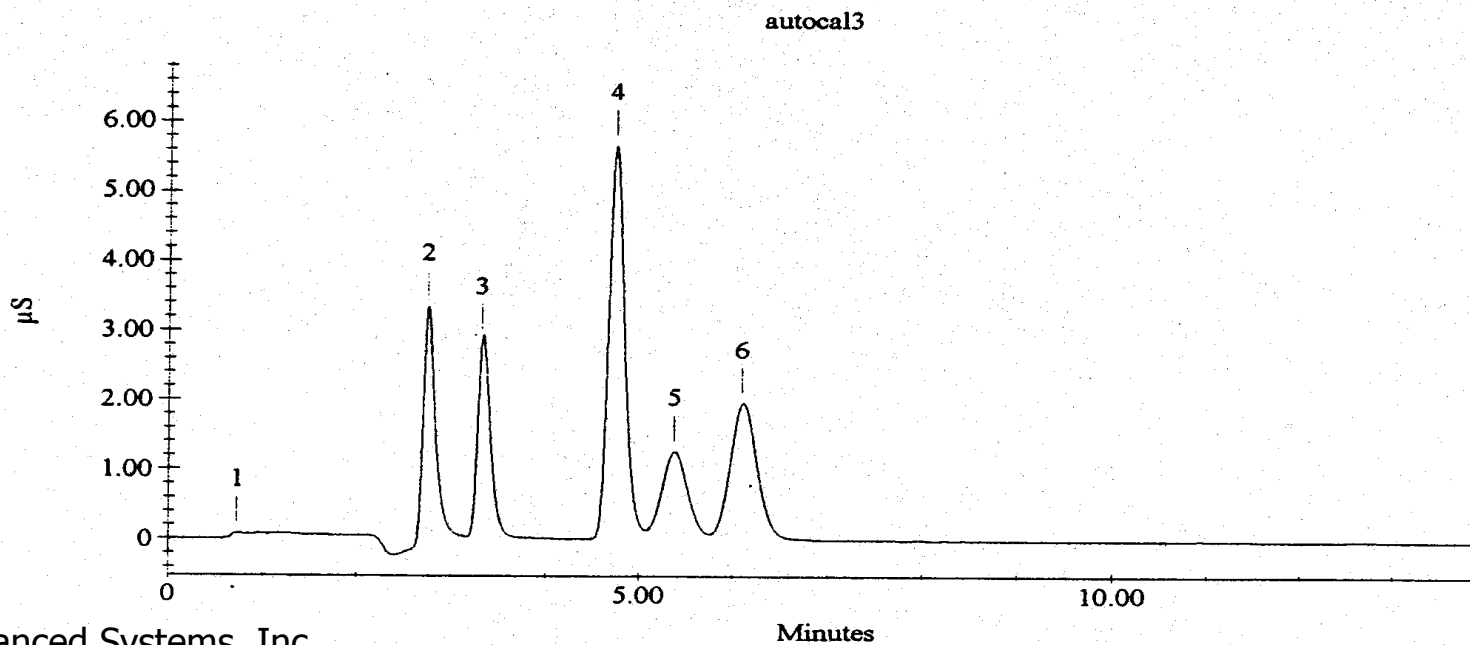


Baseline Resolution

- Ensure that baselines are being printed
- Ensure that unnecessary information is omitted from chromatography
- Ensure that threshold settings are correct

Baseline Example

| Peak Information : All Peaks | | | | | | |
|------------------------------|---------------------|----------------|-----------------------|-----------------------|------------------|-------------------------|
| Peak Number | Peak Retention Time | Component Name | Cal Response Previous | Cal Response Measured | Cal Response New | Component Amount (mg/l) |
| 1 | 0.72 | | | | | 0.00 |
| 2 | 2.75 | fluoride | | 287725 | 287725 | 2.50 |
| 3 | 3.32 | chloride | | 263402 | 263402 | 2.50 |
| 4 | 4.73 | nitrate | | 640208 | 640208 | 2.50 |
| 5 | 5.37 | phosphate | | 199096 | 199096 | 2.50 |
| 6 | 6.08 | sulfate | | 359108 | 359108 | 5.00 |





Run Times & Retention Times

- Shorter run times can cause compounds (peaks) to fall off the column or be mislabeled
- Retention time shifts can cause peaks to be mislabeled

Example

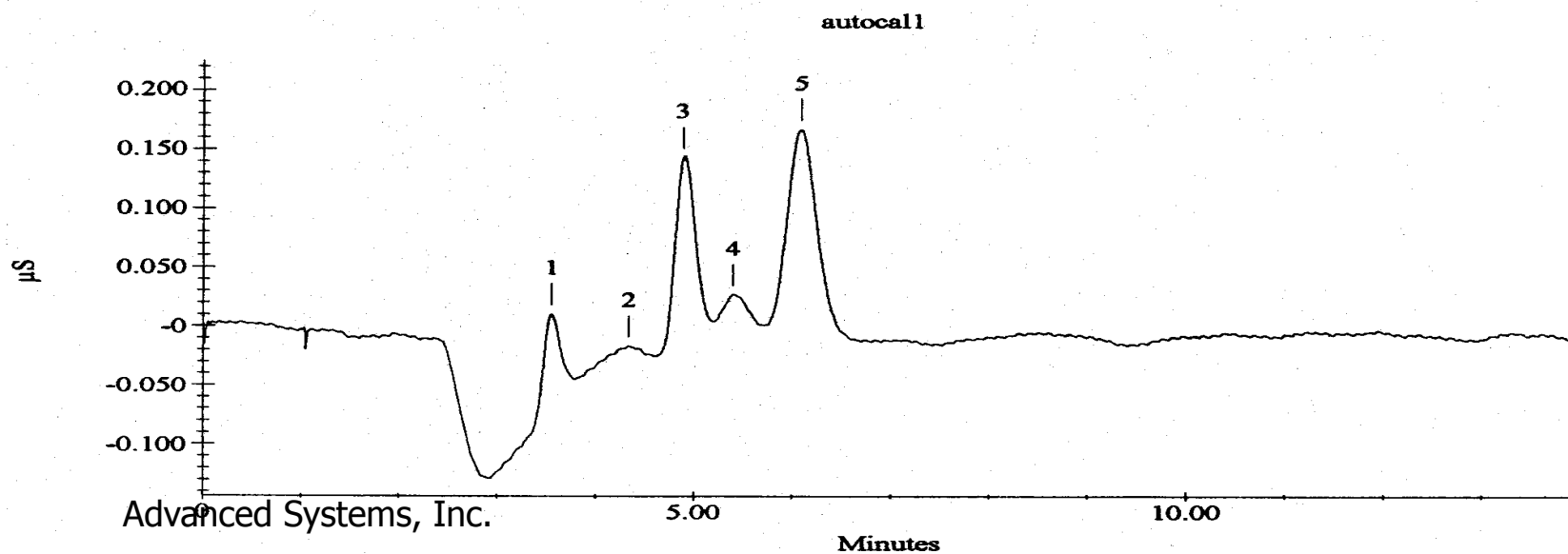
Calibration Update Report

Sample Name : autocal1
Date Time Updated : 3/15/04 10:33:59 AM
Calibration Date : 3/15/04 10:33:59 AM
Injection Number : 2
System Name : I.C.

System Operator
Column Type :
Module ID : 22 02 d5
Moduleware Version : 01.02
Data File Name : C:\PEAKNET\DATA\031504_002.DXD

Peak Information : All Peaks

| Peak Number | Peak Retention Time | Component Name | Cal Response Previous | Cal Response Measured | Cal Response New | Component Amount (mg/l) |
|-------------|---------------------|----------------|-----------------------|-----------------------|------------------|-------------------------|
| 1 | 3.55 | chloride | | 5249 | 5249 | 0.10 |
| 2 | 4.33 | | | | | 0.00 |
| 3 | 4.88 | nitrate | | 20964 | 20964 | 0.10 |
| 4 | 5.40 | phosphate | | 3490 | 3490 | 0.10 |
| 5 | 6.08 | sulfate | | 35206 | 35206 | 0.50 |





Method Files

- Method files tell stories also
 - When information is not adding up correctly it is a good idea to review the method file
 - Look for standard concentrations to be the same
 - Ensure that all standards were actually used in the curve
 - Check retention times listed in the method



Data Transfer

- Report review
- Manual transfer
- Electronic transfer

How does the data get from the instrument to the report?



Corrective Action

- Handling Failures (NELAC 5.4.10.6)
 - QA Officer
 - Analyst
 - Lab Management

If the lab deals
with data problems
correctly -

data integrity is
maintained !



Corrective Action

- Corrective action activities include
 - Departures from policies and procedures
 - Quality control failures
 - Documenting when things need to be changed
- Corrective action is not just a correction
 - Permanent fix to problem – does not reoccur
- Corrective action is documented throughout the process



Summary

- Develop schedule for auditing electronic records
 - Spreadsheets
 - Computer acquiring or calculating data
- Review do's and don'ts
 - Document observations
- Perform Corrective Action, if necessary



Thank you

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