TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Evaluating Reports/COCs to ensure data is legally defensible

Karen Kuoppala

QA Manager TestAmerica Denver

May 2009

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The lab process

Samples are collected and submitted to the lab

Samples are accepted by the lab

Laboratory logs samples in to their LIMS

Lab prepares samples for analysis

Samples are analyzed

Your project manager generates a report

You interpret your data

Data are Submitted to the end user

A Quality Process

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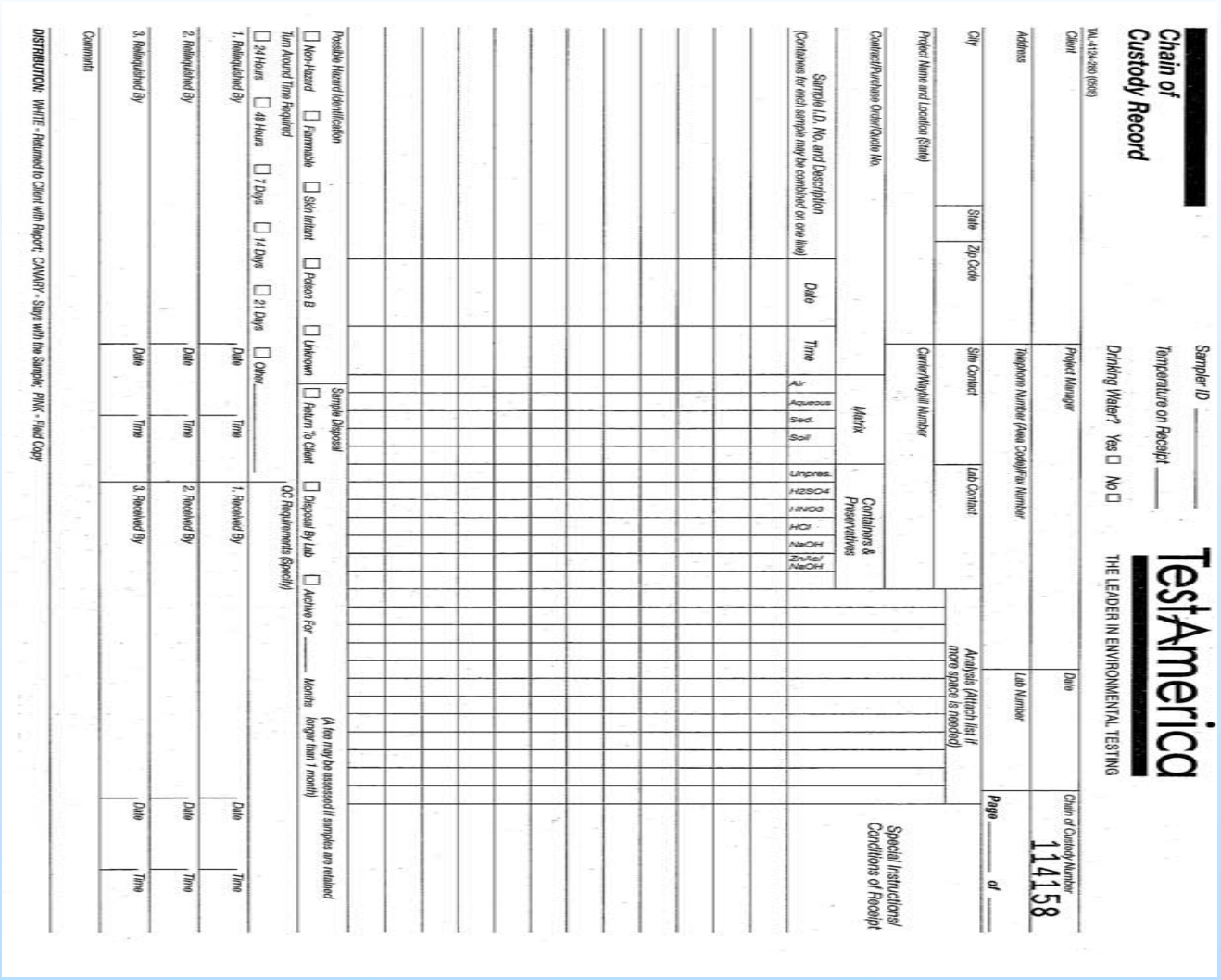
Example Chain of Custody



Chain of Custody Record

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Sampler ID _____
Temperature on Receipt ____
Drinking Water? Yes ☐ No ☐

TAL-4124-260 (0308)

Client | Project Manager | Date | Chain of Custody Number 114158

Address | Telephone Number (Area Code)/Fax Number | Lab Number | Page ____ of ____

City | State | Zip Code | Site Contact | Lab Contact | Analysis (Attach list if more space is needed)

Project Name and Location (State) | Carrier/Waybill Number

Contract/Purchase Order/Quote No. | Matrix | Containers & Preservatives | Special Instructions/ Conditions of Receipt

Sample I.D. No. and Description (Containers for each sample may be combined on one line) | Date | Time | Air | Aqueous | Sed. | Solid | Unpres. | H2SO4 | HNO3 | HCl | NaOH | NaOH/ZnAc | NH4OH

Possible Hazard Identification | Sample Disposal | (A fee may be assessed if samples are retained longer than 1 month)

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown | ☐ Return To Client ☐ Disposal By Lab ☐ Archive For ____ Months

Turn Around Time Required | QC Requirements (Specify)

☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other _____

1. Relinquished By | Date | Time | 1. Received By | Date | Time

2. Relinquished By | Date | Time | 2. Received By | Date | Time

3. Relinquished By | Date | Time | 3. Received By | Date | Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

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Samples are accepted by the lab

- NELAC has established requirements for lab acceptance of samples.
- The lab must have and abide by an acceptance policy that contains the required NELAC elements.
- Failure to comply with this requirement could result in the loss of accreditation through NELAC.
- Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.
- Approval to proceed with samples not meeting the acceptance policy requirements should be obtained from the client.

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Sample Acceptance Policy

- Cooler seals intact;
- A COC filled out completely;
- Samples must be properly labeled;
- Proper sample containers with adequate volume for the analysis and necessary QC;
- Samples must be preserved according to the requirements of the requested analytical method;
- Sample holding times must be adhered to;
- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- The project manager will be notified if any sample is received in damaged condition.

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Example Sample Receiving Checklist

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Sample Receiving Checklist

Lot #:_____ Date/Time Received:_____

Company Name & Sampling Site:_____

PM to Complete This Section: *Yes* *No* *Yes* *No*
Residual chlorine check required: ☐ ☐ Quarantined : ☐ ☐

Quote #:

Special Instructions:

Time Zone:
• EDT/EST • CDT/CST • MDT/MST • PDT/PST • OTHER

Unpacking Checks:

Cooler #(s): _____ _____ _____ _____ _____ _____ _____ _____

Temperatures (°C): _____ _____ _____ _____ _____ _____ _____ _____

N/A	*Yes*	*No*		*Initials*
☐	☐	☐	1. Cooler seals intact? (N/A if hand delivered) If no, document on CUR.	_____
☐	☐	☐	2. Coolers scanned for radiation. Is the reading ≤ to background levels? Yes: ____ No:_____	
	☐	☐	3. Chain of custody present? If no, document on CUR.	
	☐	☐	4. Bottles broken and/or are leaking? If yes, document on CUR.	
	☐	☐	5. Multiphasic samples obvious? If yes, document on CUR.	
	☐	☐	6. Proper container & preservatives used? (ref. Attachment D of SOP# DV-QA-0003) If no, document on CUR.	
☐	☐	☐	7. pH of all samples checked and meet requirements? If no, document on CUR.	
	☐	☐	8. Sufficient volume provided for all analysis requested? (ref. Attachment D of SOP# DV-QA-0003) If no, document on CUR, and contact PM before proceeding.	
	☐	☐	9. Did chain of custody agree with labels ID and samples received? If no, document on CUR.	
☐	☐	☐	10. Were VOA samples without headspace? If no, document on CUR.	
☐	☐	☐	11. Were VOA vials preserved? Preservative ☐HCl ☐4±2°C ☐Sodium Thiosulfate ☐ Ascorbic Acid	
	☐	☐	12. Did samples require preservation with sodium thiosulfate?	
☐	☐	☐	13. If yes to #11, did the samples contain residual chlorine? If yes, document on CUR.	
☐	☐	☐	14. Sediment present in dissolved/filtered bottles? If yes, document on CUR.	
☐	☐	☐	15. Is sufficient volume provided for client requested MS, MSD or matrix duplicates? If no, document on CUR, and contact PM before proceeding.	
	☐	☐	16. Receipt date(s) > 48 hours past the collection date(s)? If yes, notify PA/PM.	
	☐	☐	17. Are analyses with short holding times requested?	
	☐	☐	18. Was a quick Turn Around (TAT) requested?	

\QA\Edit\FORMS\Sample Receiving\Sample Receiving Checklist 9-2-08

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Example Sample Receiving Checklist

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Sample Receiving Checklist

Lot #_____

Login Checks: *Initials* _____

N/A	Yes	No		
	☐	☐	19. Sufficient volume provided for all analysis requested? (ref. Attachment D of SOP# DV-QA-0003)	If no, document on CUR, and contact PM before proceeding.
☐	☐	☐	20. Is sufficient volume provided for client requested MS, MSD or matrix duplicates? If no, document on CUR, and contact PM before proceeding.	
	☐	☐	21. Did the chain of custody includes "received by" and "relinquished" by signatures, dates, and times?	
☐	☐	☐	22. Were special log in instructions read and followed?	
☐	☐	☐	23. Were AFCEE metals logged for refrigerated storage?	
	☐	☐	24. Were tests logged checked against the COC? Which samples were confirmed? _____	
☐	☐	☐	25. Was a Rush form completed for quick TAT?	
☐	☐	☐	26. Was a Short Hold form completed for any short holds?	
	☐	☐	27. Were special archiving instructions indicated in the General Comments? If so, what were they?	

_____

Labeling and Storage Checks: *Initials* _____

☐	☐	☐	28. Was the subcontract COC signed and sent with samples to bottle prep?
	☐	☐	29. Were sample labels double-checked by a second person?
☐	☐	☐	30. Were sample bottles and COC double checked for dissolved/filtered metals by a second person?
	☐	☐	31. Did the sample ID, Date, and Time from label match what was logged?
☐	☐	☐	32. Were stickers for special archiving instructions affixed to each box? See #27
☐	☐	☐	33. Were AFCEE metals stored refrigerated?

Document any problems or discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR).

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Laboratory logs samples into the LIMS

- Samples are evaluated for compliance with program requirements. For example:
  - Special preservation (e.g. drinking water samples, AFCEEE)
  - Additional radiation screening
- Special instructions are noted. For example:
  - Rush samples
  - Short holding times
  - Rapidly expiring samples

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Example Condition Upon Receipt Anomaly Report

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Condition Upon Receipt Anomaly Report (CUR)

Lot No : _____ Date/Time: _____
Client : _____ Initiated by: _____
Affected Samples COC#_____

Client ID	Lab ID	Analyses Requested

CONDITION/ANOMALY/VARIANCE (CHECK ALL THAT APPLY):

❑COOLERS
- ❑Received, No Chain of Custody (COC)
- ❑Not Received but COC(s) Available
- ❑Leaking
- ❑Other:

❑ TEMPERATURE (greater than 6° C)
- ❑Cooler Temp _____
- ❑Temperature Blank _____

❑CONTAINERS
- ❑Leaking
- ❑Broken
- ❑Extra
- ❑Without Labels
- ❑VOA Vials with Headspace _____mm
- ❑Other:

❑CUSTODY SEALS (COOLER(S)/CONTAINER(S)
- ❑None
- ❑ Not Intact
- ❑Other:

❑CHAIN OF CUSTODY (COCs)
- ❑Not relinquished by Client; No date/time Relinq.
- ❑ Incomplete Information
- ❑Other:

❑ CONTAINER LABELS
- ❑Not the same ID/info as in COC
- ❑ Incomplete

❑ID ❑COLLECTION ❑Time ❑Date ❑PRESERVATIVE

- ❑ Markings/Info smeared or illegible
- ❑Torn
- ❑Other:

❑ SAMPLES
- ❑Samples NOT RECEIVED but listed on COC ——
- ❑Samples received but NOT LISTED on COC
- ❑Logged based on Label Information
- ❑Logged based on info from other samples on COC
- ❑ Logged according to Work Plan
- ❑Logged on HOLD UNTIL FURTHER NOTICE
- ❑ Other: _____

❑will be noted on COC Client to send samples with new COC
❑ Trip Blank received, not on COC, _____vials received
❑ Mislabeled as to tests, preservatives, etc.
❑Holding time expired
❑ Improper container used
❑Not preserved / Improper preservative used
❑ Improper pH _____ Lab to preserve sample
❑ Insufficient quantities for analysis

Comments: _____

Corrective Action:
- ❑ Client Informed: verbally on: _____By: _____: In writing on: _____ By: _____
- ❑ Sample(s) processed "as is". _____
- ❑ Sample(s) on hold until: _____ If released, notify: _____

Sample Control Supervisor Review: _____ Date: _____
Project Management Review: _____ Date: _____
SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

L:\QA\Edit\Forms\Sample Receiving\ Condition Upon Receipt Report 5/30/04 version

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Laboratory logs samples into the LIMS con't

- Samples are assigned unique identifiers – internal lab COC begins here
- Containers are placed in designated locations for proper storage
- Paperwork is given to your PM to insure proper login
- Samples now appear on lab/PM backlogs

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Example Internal Chain of Custody

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Sample Transfer Audit Report

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4955 Yarrow Street
Arvada, CO 80002

LotID	ClientSampID	ContainerID	EventID	ClientName	ClientCd	Quote	TranferType	TransferTime	UserName	StorageLoc	Drum/NewLoc	ContType
D9A270231-001	Metals	K6C48-001	103023	TestAmerica Denver	280	81988	Login	01/27/2009 16:24	Fayard, Maria	146	NA	
D9A270231-001	Metals	K6C48-001	103578	TestAmerica Denver	280	81988	Checkout	02/02/2009 15:33	HARRE, JON	146	NA	
D9A270231-001	Metals	K6C48-001	103640	TestAmerica Denver	280	81988	Return to Storage	02/03/2009 12:05	WILLMS, JAY	146	NA	
D9A270231-001	Metals	K6C48-001	103684	TestAmerica Denver	280	81988	Checkout	02/03/2009 15:33	HARRE, JON	146	NA	
D9A270231-001	Metals	K6C48-001	103692	TestAmerica Denver	280	81988	Return to Storage	02/03/2009 16:23	HARRE, JON	146	NA	
D9A270231-001	Metals	K6C48-001	103754	TestAmerica Denver	280	81988	Checkout	02/04/2009 11:27	WILLMS, JAY	146	NA	
D9A270231-002	Cyanide	K6C5N-001	103023	TestAmerica Denver	280	81988	Login	01/27/2009 16:24	Fayard, Maria	146	NA	
D9A270231-002	Cyanide	K6C5N-001	104235	TestAmerica Denver	280	81988	Checkout	02/09/2009 14:30	Bloom, Kevin	146	NA	
D9A270231-002	Cyanide	K6C5N-001	104258	TestAmerica Denver	280	81988	Return to Storage	02/09/2009 17:38	Bloom, Kevin	146	NA	
D9A270231-002	Cyanide	K6C5N-001	110369	TestAmerica Denver	280	81988	Relocate	04/08/2009 13:09	Chavez, Lawrence	146	AQ21	
D9A270231-003	Cyanide,Amenable	K6C5Q-001	103023	TestAmerica Denver	280	81988	Login	01/27/2009 16:24	Fayard, Maria	146	NA	
D9A270231-003	Cyanide,Amenable	K6C5Q-001	103392	TestAmerica Denver	280	81988	Checkout	01/30/2009 12:16	Lambert, Sarah	146	NA	
D9A270231-004	Anions (PT-AN-SOIL)	K6C5R-001	103023	TestAmerica Denver	280	81988	Login	01/27/2009 16:24	Fayard, Maria	146	NA	
D9A270231-004	Anions (PT-AN-SOIL)	K6C5R-001	104662	TestAmerica Denver	280	81988	Checkout	02/12/2009 10:49	Kudla, Ewa	146	NA	
D9A270231-004	Anions (PT-AN-SOIL)	K6C5R-001	104773	TestAmerica Denver	280	81988	Return to Storage	02/12/2009 21:13	Phan, Thu	146	NA	
D9A270231-004	Anions (PT-AN-SOIL)	K6C5R-001	110369	TestAmerica Denver	280	81988	Relocate	04/08/2009 13:09	Chavez, Lawrence	146	AQ21	
D9A270231-005	Nutrients	K6C5V-001	103023	TestAmerica Denver	280	81988	Login	01/27/2009 16:24	Fayard, Maria	146	NA	
D9A270231-005	Nutrients	K6C5V-001	103578	TestAmerica Denver	280	81988	Checkout	02/02/2009 15:33	HARRE, JON	146	NA	
D9A270231-005	Nutrients	K6C5V-001	103640	TestAmerica Denver	280	81988	Return to Storage	02/03/2009 12:05	WILLMS, JAY	146	NA	
D9A270231-005	Nutrients	K6C5V-001	103683	TestAmerica Denver	280	81988	Checkout	02/03/2009 15:23	Wolff, Brett	146	NA	
D9A270231-005	Nutrients	K6C5V-001	103702	TestAmerica Denver	280	81988	Return to Storage	02/03/2009 17:05	Wolff, Brett	146	NA	
D9A270231-005	Nutrients	K6C5V-001	104042	TestAmerica Denver	280	81988	Checkout	02/06/2009 11:30	Wolff, Brett	146	NA	
D9A270231-005	Nutrients	K6C5V-001	104123	TestAmerica Denver	280	81988	Return to Storage	02/06/2009 16:09	Wolff, Brett	146	NA	
D9A270231-005	Nutrients	K6C5V-001	104279	TestAmerica Denver	280	81988	Checkout	02/10/2009 07:55	Gilbert, Bryan	146	NA	
D9A270231-005	Nutrients	K6C5V-001	104344	TestAmerica Denver	280	81988	Return to Storage	02/10/2009 13:39	Gilbert, Bryan	146	NA	
D9A270231-005	Nutrients	K6C5V-001	110369	TestAmerica Denver	280	81988	Relocate	04/08/2009 13:09	Chavez, Lawrence	146	AQ21	
D9A270231-005	Nutrients	K6C5V-002	103034	TestAmerica Denver	280	81988	Login	01/27/2009 16:58	Fayard, Maria	146	NA	
D9A270231-005	Nutrients	K6C5V-002	103602	TestAmerica Denver	280	81988	Checkout	02/03/2009 07:55	Gilbert, Bryan	146	NA	
D9A270231-005	Nutrients	K6C5V-002	103677	TestAmerica Denver	280	81988	Return to Storage	02/03/2009 15:01	Fisher, Elizabeth	146	NA	
D9A270231-005	Nutrients	K6C5V-002	110369	TestAmerica Denver	280	81988	Relocate	04/08/2009 13:09	Chavez, Lawrence	146	AQ21	
D9A270231-006	Flash Point	K6C52-001	103023	TestAmerica Denver	280	81988	Login	01/27/2009 16:24	Fayard, Maria	146	NA	
D9A270231-006	Flash Point	K6C52-001	103233	TestAmerica Denver	280	81988	Checkout	01/29/2009 11:40	Elkin, David	146	NA	
D9A270231-006	Flash Point	K6C52-001	103324	TestAmerica Denver	280	81988	Return to Storage	01/29/2009 16:34	Elkin, David	146	NA	
D9A270231-006	Flash Point	K6C52-001	110369	TestAmerica Denver	280	81988	Relocate	04/08/2009 13:09	Chavez, Lawrence	146	AQ21	
D9A270231-007	Corrosivity	K6C53-001	103023	TestAmerica Denver	280	81988	Login	01/27/2009 16:24	Fayard, Maria	146	NA	
D9A270231-007	Corrosivity	K6C53-001	104188	TestAmerica Denver	280	81988	Checkout	02/09/2009 10:03	Peterson, Braden	146	NA	
D9A270231-007	Corrosivity	K6C53-001	104260	TestAmerica Denver	280	81988	Return to Storage	02/09/2009 17:50	Peterson, Braden	146	NA	

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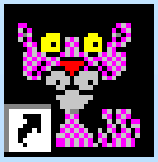
Lab prepares samples for analysis

- Special client and/or program requirements are communicated to lab
- Samples are assigned to a QC batch
- Any method or program required QC samples generated to monitor preparation efficiency are created
- All anomalies occurring with the sample preparation are communicated to the analytical staff and the PM



Clouseau.lnk

Example Non-Conformance

Clouseau
Nonconformance Memo

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NCM #: 04-0152523
NCM Initiated By: Katherine Abbott
Date Opened: 02/10/2009
Date Closed:

Nonconformance: Miscellaneous
Subcategory: Other

Classification: **Anomaly**
Status: **PMQA**
Production Area: Organic Preparation
Tests: 8321A
Lot #'s (Sample #'s): D9A270231 (19),
QC Batches: 9041166,

Problem Description / Root Cause

Name	Date	Description
Katherine Abbott	02/10/2009	Per the PT instructions, this sample was extracted in a clear vial as opposed to an amber vial. The sample will be covered during extraction to prevent light from being a faction in the extraction process.

Corrective Action

Name	Date	Corrective Action
Katherine Abbott	02/10/2009	

Client Notification Summary

Client	Project Manager	Notified	Response	How Notified	Note
	Response	Response Note			

Quality Assurance Verification

Verified By	Due Date	Status	Notes
		This section not yet completed by QA.	

Approval History

Date Approved	Approved By	Position

Date Printed: 4/16/2009

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Samples are analyzed

- Analysis of samples initiates the generation of another set of QC analyses as required by the method.

- Calibration standards, instrument blanks, tuning standards, reporting limit standards, DDT breakdown standards and retention time window standards are all examples.

- The purpose of QC analyses at this step are to evaluate additional precision and bias that is introduced into the data by instrument drift and/or performance.

- Another aspect of instrument calibration is the analysis of a second source standard. This is to verify the accuracy of standards used for quantitation.

- All anomalies are documented for the PM.

Your project manager generates a report

Components of a report NELAC 5.5.10

- **At a minimum, the standard laboratory report shall contain the following information**:
  - A report title (e.g. Analytical Report For Samples) with a “sample results” column header.
  - The report cover page is printed on company letterhead, which includes the laboratory name, address and telephone number.
  - A unique identification of the report (e.g. lot number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.
- **Note:** The total number of pages is indicated at the front of each report.
  - A copy of the chain of custody (COC).
    - Any COCs involved with Subcontracting are included.
    - Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (eg. Sampling information).
  - The name and address of client and a project name/number, if applicable.
  - Client project manager or other contact
  - Description and unambiguous identification of the tested sample(s) including the client identification code.

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Your project manager generates a report con't

- Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation
- or analysis if the required holding time for either activity is less than or equal to 72 hours.
  - Date reported or date of revision, if applicable.
  - Method of analysis including method code (EPA, Standard Methods, etc).
  - Reporting limits.
    - Method detection limits (if requested)
  - Definition of Data qualifiers and reporting acronyms (e.g. ND).
  - Sample results.
  - QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.
  - Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 26.2.4 – Item 3 regarding additional addenda).
  - A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.
  - A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.
  - A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are
  - appointed by the Lab Director.

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Your project manager generates a report con't

- When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons
- and/or justification if they do not. For Example:
- "The results included in this report have been reviewed for compliance with the laboratory QA/QC plan and meet all requirements of
- NELAC. All data have been found to be compliant with laboratory protocol and any exceptions are noted below. "
- Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.
- When Soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.
- Appropriate laboratory certification number for the state of origin of the sample, if applicable.
- If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on
- the report "partial report", and that a complete report will follow once all of the work has been completed.
- Any out of network subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor.
- All in-network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

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You interpret your data

- Evaluating your data should be a straight forward task if:
  - ~ The lab has appropriately documented anomalies
  - ~ You have a basic understanding of the QC elements in your data package
  - ~ You and the lab have appropriately selected methods that meet your project objectives

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You review your data
Project Narrative

- The project narrative should document all anomalies that occurred during receiving, sample preparation, and analysis.
- This should be a valuable resource when evaluating your data.
- Unless you do full validation, this may be your only reference to evaluate bias that occurred during analysis due to instrument drift.
- All deviations from the method and laboratory standard procedures should be documented in the narrative.

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Example Project Narrative

CASE NARRATIVE
D8K250192

With exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. All laboratory quality control samples analyzed in conjunction with the samples in this project were within established control limits, with any exceptions noted.

The test results presented in this report relate only to the samples in this report and meet all requirements of NELAC, and any exceptions are noted. This report shall not be reproduced, except in full, without written permission from the laboratory.

A project-specific lower acceptable recovery limit of 30% for all QC samples and surrogates has been designated for analytical work performed under the 2008 QAPP for client ABC, Inc. NPL Site. This lower limit is used for this project, rather than historically generated lower recovery limits. All recoveries in this report are above the 30% minimum threshold.

Sample Receiving

Nine samples plus one set of MS/MSD samples were received under chain of custody on November 25, 2008. The samples were received at temperatures of 2.1°C, 2.8°C, 2.9°C, 2.2°C, 1.3°C and 3.7°C. All sample containers were received in acceptable condition.

GC/MS Semivolatiles, Method SW846 8270C SIM

All sample holding times were met.

Samples SLP10FEED-112408 and SLP10FEEDD-112408 were analyzed at two different dilutions to obtain all target analytes within the calibration range. Reporting limits were adjusted accordingly. Surrogate recoveries could not be calculated for the analyses performed at a 4x dilution, because the extracts were diluted beyond the ability to quantitate recoveries.

Surrogate Chrysene-d12 was recovered below the lower control limit in samples SLP4T-112408, SLP4FEED-112408, SLP6-112408 and SLP10T-112408. The samples were reanalyzed with similar results. Re-extraction was not possible due to insufficient remaining sample volume.

The MS/MSD associated with QC batch 8335025 was performed using sample SLP10FEED-112408, as requested. MS/MSD exhibited 17 of the 44 Matrix Spike compound recoveries outside the control limits. MS/MSD exhibited 31 of the 44 Matrix Spike Duplicate compound recoveries and two of the three surrogate recoveries outside the control limits. The MS/MSD exhibited 31 of the 44 Relative Percent Difference (RPD) data outside the control limits. The MS/MSD exhibited percent recoveries and/or relative percent difference data outside the control limits for Acenaphthene, Acenaphthylene, Acridine, Anthracene, Benzo(a)anthracene, Benzo(b)fluoranthene, Benzo(k)fluoranthene 7H-Dibenzo[c,g]carbazole, Dibenz(a,h)acridine, Dibenz(a,j)acridine, 2,3-Benzofuran, Benzo(ghi)perylene, Dibenzo(a,e)pyrene, Dibenzo(a,i)pyrene, Dibenzo(a,h)pyrene, Dibenzo(a,l),pyrene, Benzo(a)pyrene, 7,12-Dimethylbenz(a)anthracene, 2,6-Dimethylnaphthalene, Benzo(e)pyrene, Benzo(b)thiophene, 3-Methylcholanthrene, 6-Methylchrysene, 1-Methylphenanthrene, Biphenyl, Carbazole, 2,3,5-Trimethylnaphthalene, Chrysene, Dibenzo(a,h)anthracene, Dibenzofuran, Dibenzothiophene, 2,3-Dihydroindene, Fluoranthene, Fluorene, Indene, Indeno(1,2,3-cd)pyrene, Indole, 2-Methylnaphthalene, 1-Methylnaphthalene, Naphthalene, Perylene, Phenanthrene, Pyrene, Quinoline, Fluorene d-10 and Chrysene-d12. Details of the specific analyte recoveries can found in the Matrix Spike Sample Evaluation and Data Reports.

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The Impact of Blank Results

A brief description of lab QC samples and their purpose:

- **Method and or instrument blank: Lab blanks are used for two purposes:**
  1. **To determine the possibility of a false positive or negative**
  2. **To determine the possibility of bias to reported results**

  Examples:

Analyte	Reporting Limit	Blank Result	Sample Result	Impact to data
Arsenic	8 ppb	- 12 (ppb)	< 8 ppb	Possible false negative, low bias to reported results
Methylene chloride	10 ppb	7J (ppb)	15 ppb	Possible false positive, high bias to reported result
Bis(2-ethylhexyl)phthalate	10 ppb	56 (ppb)	18 ppb	Probable false positive, high bias to reported results, RL cannot be supported

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The Impact of Surrogate Recoveries

- **Surrogates are compounds that are added to organic analyses to monitor extraction efficiency. They are selected based on their similarity to the target analytes of interest.**
- **The recoveries provide information on a sample by sample basis and can be a very useful tool.**

Examples:

Surrogate Compound	Sample Recovery	Blank Recovery	Impact to reported results
Decachlorobiphenyl	102%	89%	Indicates good extraction efficiency, no bias
Nitrobenzene-d5	126%	78%	If the other surrogates are in control, may only indicate a chromatographic and/or spectral interference. Need more information to determine possible bias.
Dichlorobenzene-d4	4%	36%	Indicates problems with lab extraction. Low bias and reporting limits are not defensible.

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The Impact of Laboratory Spike Recoveries

- **Laboratory Control Spike/Spike duplicates are used to monitor extraction efficiency and insure the lab process is in control.**
- **Most labs will prepare these in duplicate in the event that there is insufficient volume provided by the client to analyze a MS/MSD pair, providing precision data for the batch.**
- **NELAC requires the lab to spike every target analyte at least once every two years.**

NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed Marginal Exceedences
>90	5
71 - 90	4
51 – 70	3
31 – 50	2
11-30	1
<11	0

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The Impact of Matrix Spike Recoveries

- **Collection of sufficient sample to perform matrix spikes is always an issue for the lab.**

- **These samples can provide useful information about additional bias that may be introduced by the sample matrix.**

- **Typically the lab will not control on the MS/MSD unless there is an obvious lab error (e.g. – spike solution not added)**
- **Methods often require the lab to perform matrix spikes for every matrix type.**

- **Because labs don't generally have all the information needed to classify matrix types, we make the assumption that all water samples can be represented by a single MS/MSD.**

- **The information gathered from matrix spike data can be used not only to determine bias in your samples, but also whether or not laboratory method detection limits and reporting limits are valid for your samples.**

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Initial Calibration Failures

- **Methods offer multiple calibration options.**
  - ~ **Average response/calibration factors.**
  - ~ **Linear regressions with multiple weighting options ($1/x$,$1/x^2$).**
  - ~ **Quadratic fits.**
- **Analysis should not begin before the lab achieves an acceptable initial calibration.**

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Second Source Calibration Verification Failures

- This is a NELAC requirement and has differing criteria under different programs.
- It can be very difficult for the lab to obtain matching standards from differing vendors, especially for some of the Appendix IX parameters, or known poor performers.
- Any failures of the second source ICV should be documented in the project narrative.

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Continuing Calibration Failures

- Given the large number of parameters analyzed for some of the organic methods, failures can occur in the CCV.
- Failures should be documented in the project narrative.
- Failures to CCVs for methods where the CCV brackets samples can be caused by the sample matrix. In this case, the lab should substantiate that the samples are causing the problem.
- The lab should comment on the impact of the failure to the reported results.
  - For example: There is no impact when there is a high bias to a CCV standard with associated samples that are non-detect.

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Data are submitted to the end users

- **If everyone has done their homework, this should go smoothly.**
- **The lab has alerted you to any issues, so there are no big surprises.**
- **You have reviewed your data and addressed any significant issues up front.**
- **Any issues that you were unsure about have already been discussed with the end user's of the data.**

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Summary

- **Communication with the laboratory is critical to a successful program**

- Share the project DQOs with the lab prior to project start.
- Alert the laboratory to reporting limit needs prior to analysis.
- Be available. A surprising number of clients are difficult to contact. If you are doing field work, give the lab an alternate contact.

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QUESTIONS ?