ISO 17025 Accreditation/Quality Management Systems Panel Discussion

AAFCO 2014 Annual Meeting

Laboratory Methods & Service Committee

Sacramento, CA

July 26, 2014

Association of American Feed Control Officials

ISO/IEC 17025 LABORATORY ACCREDITATION OVERVIEW

Presented by Brenda Snodgrass
Oklahoma Dept. of Agriculture, Food & Forestry
Moderator

Acknowledgement:
Many thanks to Kristi McCallum, Colorado Department of Agriculture for providing her slides for the overview presentation

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What is Accreditation?

- The procedure by which an authoritative body certifies that an entity or person is competent to perform a certain procedure or task
- Laboratories are accredited for specific test methods or calibrations performed
- ILAC (International Laboratory Accreditation Cooperation) sets the standard (example: ISO/IEC 17025). The standard is a set of requirements used to assess quality and competency.
- ILAC developed Mutual Recognition Agreements (MRA) with three Regional Cooperation Bodies. Under the MRA, these Regional Cooperation Bodies must ensure that laboratories accredited to ISO/IEC 17025 comply with the standard.

Information obtained from https://www.ilac.org/

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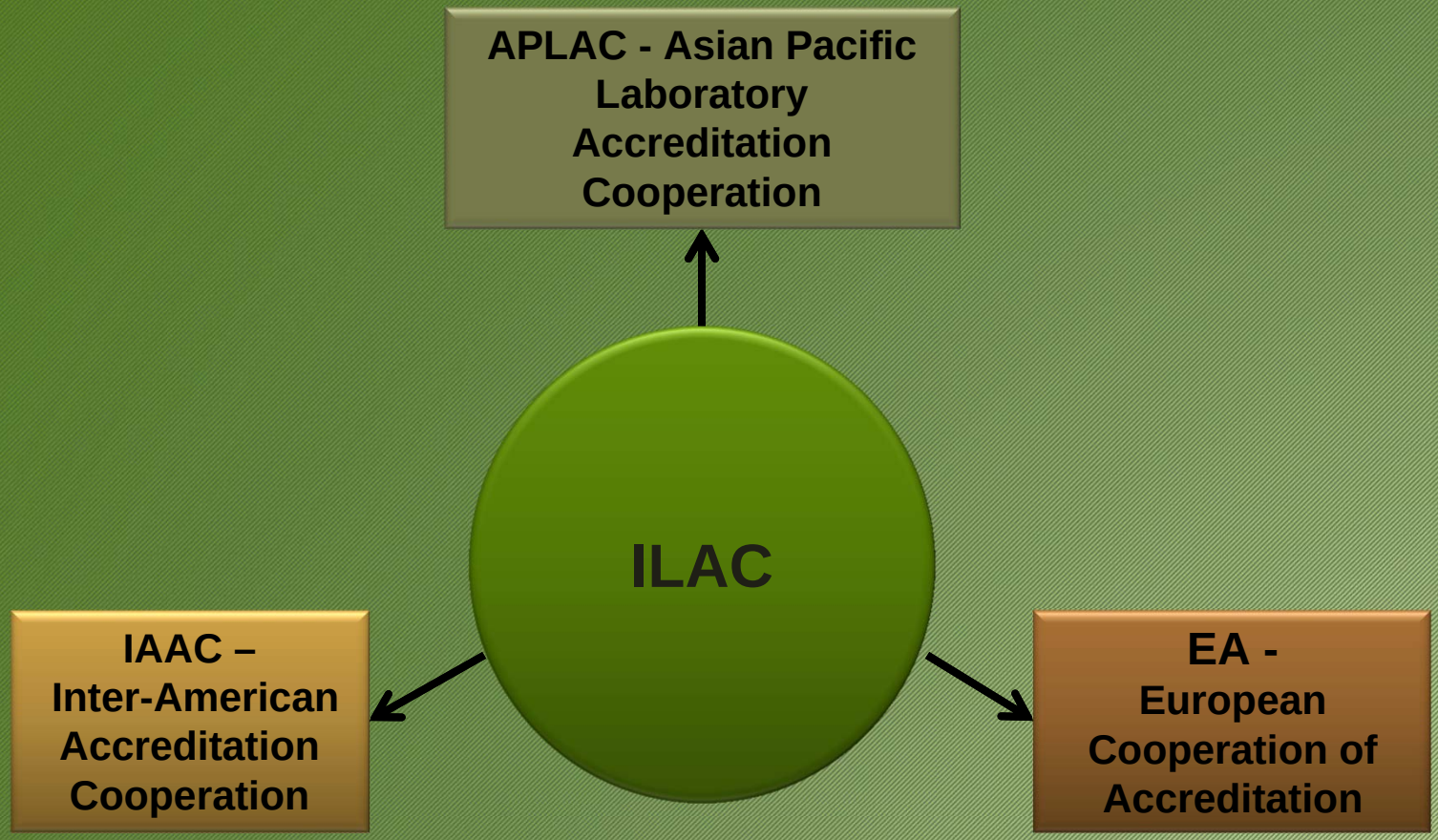
ILAC Mutual Recognition Agreement Regions



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United States Accrediting Bodies

- ANSI-ASQ National Accreditation Board and their associated brands ACLASS, FQS, and ANAB
- The American Association for Laboratory Accreditation (A2LA)
- Laboratory Accreditation Bureau
- Perry Johnson Laboratories
- American Industrial Hygiene Association
- International Accreditation Service
- National Voluntary Laboratory Accreditation Program (NVLAP) – Government agency that only accredits a few narrow disciplines
- American Society of Crime Laboratory Directors-Laboratory Accreditation Board (ASCLD-LAB)

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First things first…..

- **Must have management on board!** Your lab cannot become accredited without the support of senior management
- Must obtain a copy of the ISO/IEC 17025:2005 Standard available from http://www.iso.org
- Very strict copyright laws pertain to all ISO Standards, including checklists which includes verbatim language of the Standards
- Must have a clearly defined Management System in place
- Must have a Quality System Manual in place
- Define the scope of accreditation (what methods are used and fields of testing)

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Quality Management System (QMS)

- Must meet the requirements of ISO/IEC 17025, Section 4.2

- Consists of the Organizational Policies, Quality System Manual and Standard Operating Procedures (SOPs) for all management system policies and procedures and technical procedures
    - Section 4 includes the QMS Management Requirements
    - Section 5 includes the QMS Technical Requirements

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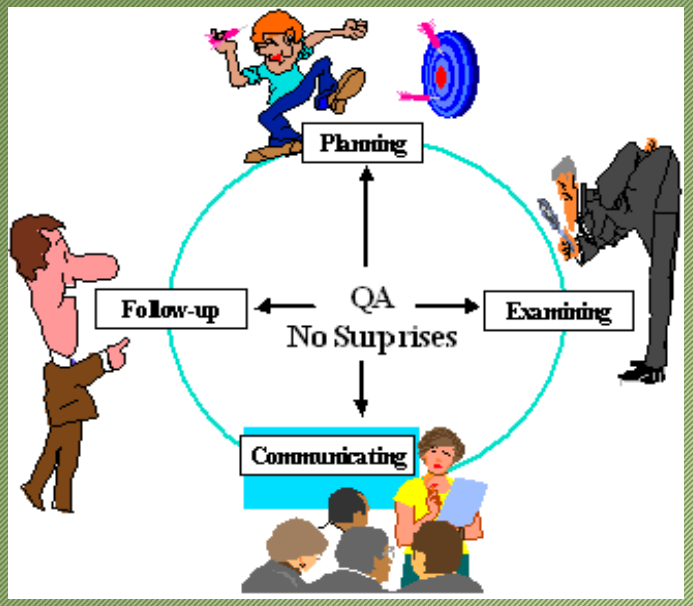
Top 3 Things for a Functioning Management System

- Written policies and procedures

- Implementation

- Audit system to ensure it is working



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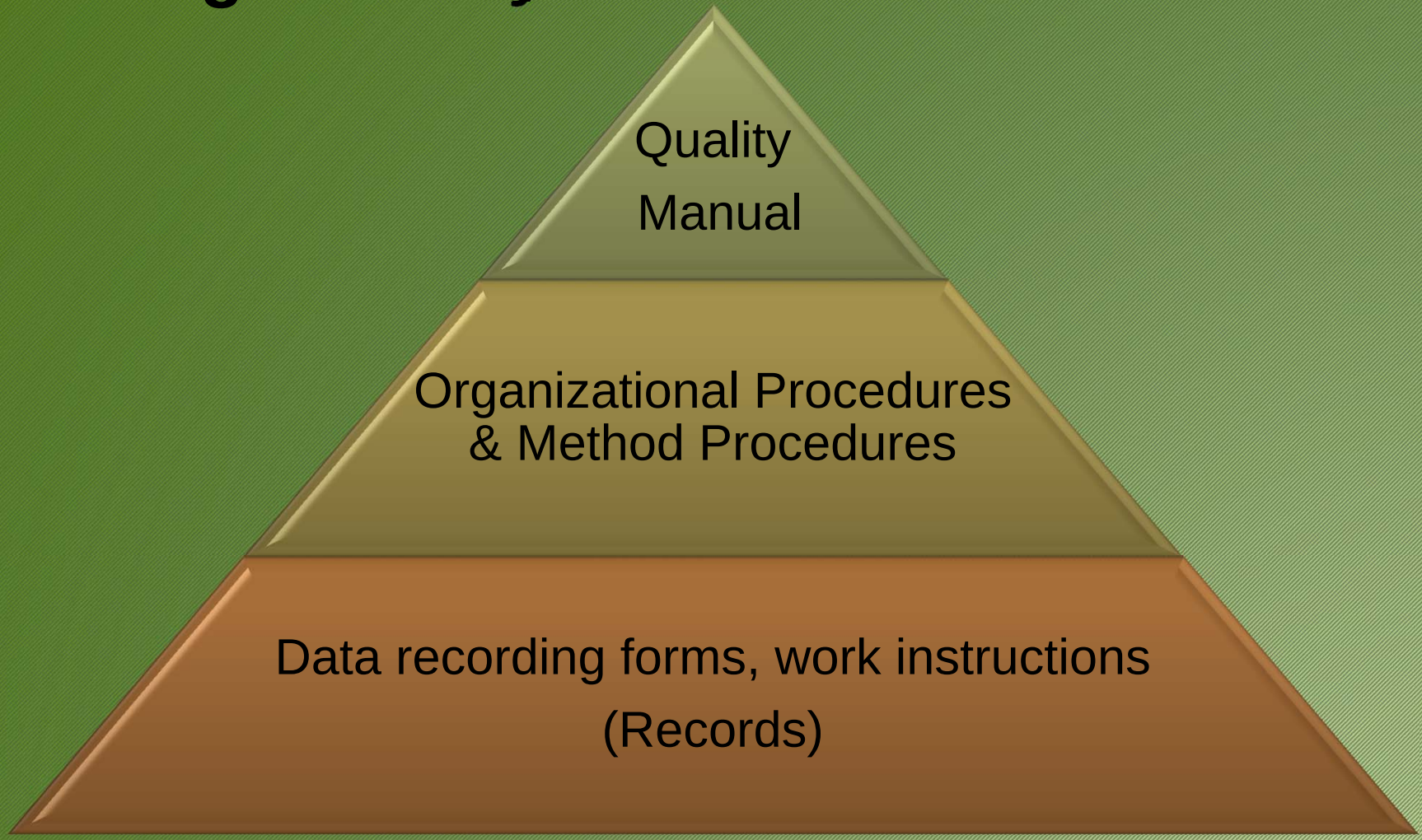
Management System Document Structure



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Management Procedures Include

- Organization - Organization chart, how your organization is managed; confidentiality and ethics policies & procedures
- Customer - Customer contracts, contract reviews, customer surveys
- Sub-contracting of analyses
- Purchasing
- Non-conforming analysis, complaint handling, corrective/preventive actions
- Internal audits
- Document control
- Management reviews and quality improvement processes

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Technical Procedures Include

- Personnel - training, proficiency assessment
- Building and environmental conditions
- Analytical methods - suitability of method and method validations/verifications
- Measurement uncertainty and traceability
- Equipment - calibration/verification, maintenance
- Sample Handling - receipt, chain-of-custody, handling of test sample, sample storage, sample preparation
- Reporting Results - data reporting, data review, data handling, data storage

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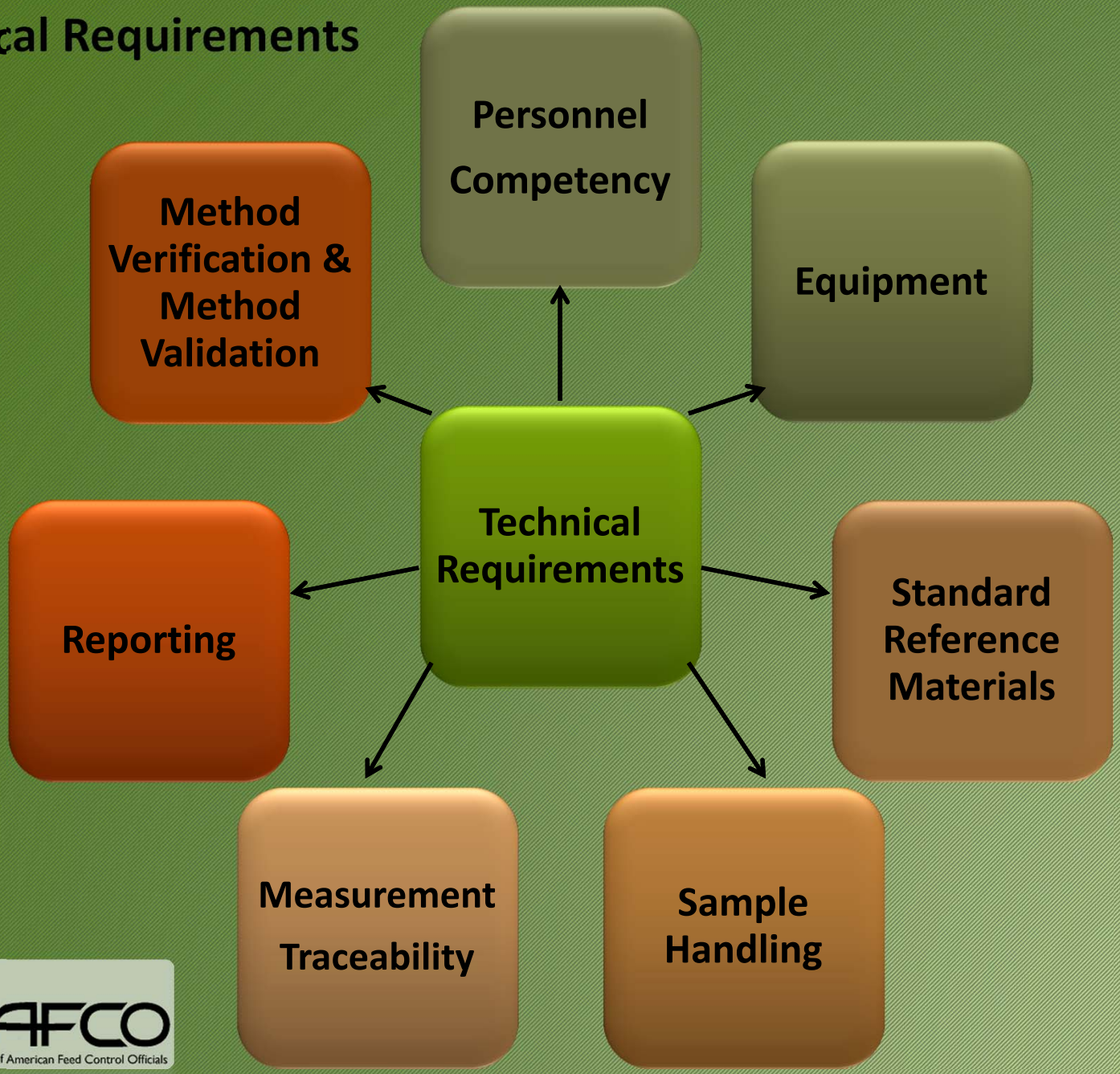
Technical Requirements



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Management Review

Presented by Teresa Grant
North Carolina Department of Agriculture & Consumer Services

Relevant section of the standard

4.2.2 Management System

4.10 Improvement

4.1.1 Corrective Action

4.13.1.1 Control of Records

4.15 Management Reviews

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During the course of an annual Management Review, the following information and/or data is presented for review and discussion as it complies with the ISO/IEC 17025 Section 4.15.1. This information generally includes items as it pertains to our accrediting body, customers, policies, quality management system, and laboratory testing.

- Suitability of policies and procedures
- Reports from managerial and supervisory personnel
- Recent internal audit findings
- Corrective and preventive actions
- Assessments by external bodies
- Results of inter-laboratory comparisons or proficiency tests
- Changes in the volume and type of work
- Customer feedback
- Complaints
- Recommendations for improvement
- Other relevant factors, such as quality control activities, resources and staff training
- Management objectives, goals and action plans for the coming year
- Action Items

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Management Review Agenda

Attendees:

- Changes to type and volume of work; future direction of FDPD Lab –
- Training – plans, goals, objectives – document individually in work plans; matrix documenting ongoing competency and cross-training also supports this.
- Quality Objectives
- Discussion of review topics (these need to include review of audit findings not covered in Sept Management Review meeting – refer to report for specific audit topics to be covered)
  1. Section 4.13 Control of Lab Records
     a. General comments -
     b. Internal audit results – QA
  2. Section 5.4.7 Protection of Electronic Data
     a. General Comments –
     b. Internal audit results – QA
  3. Report on audit of QMS.023.004 – QA
  4. Additional discussions:

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Management Review Agenda (continued)

- Sample Rejections: 2/1/14-4/30/14
- NCRs: 2/1/14-4/30/14
- PARs: 2/1/14-4/30/14
- PTs: 2/1/14-4/30/14
- Status of Action Items from last meeting
- Status of Internal Audits -
- Status of “Annual QMS Tasks” (see end of Management Review Planners)
- Continuous improvement

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Management Review Agenda (continued)

Assignments for Next Meeting:

ISO clause 4.6 (QMS.006)

ISO clauses 4.7, 4.8 (QMS.007)

ISO clause 4.13 (QMS.011)

Related Clause	Action Item	Assigned To	Target Date	Status
***NEW ACTION ITEMS (Q4)* 2014**				
Quality Objective				

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Management Review Opening Comments - Date

➢ Budget

Total spent			
Total available			
Remaining			
Save for repairs, incidental expenses			
Available			

➢Staffing

Vacancies	Status

➢Facility updates

➢LIMS updates

➢Training Dates

Name of class	Trainer	Location	Attendee

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Management Review Preparation Planner

(To be completed by Quality & Managerial Personnel)

Name:_____

Meeting Date: _____

Meeting Preparation

Read the QMS procedure(s) indicated by checkboxes. In addition, read that section of the Quality Manual (FDPD-QMS.001) and the ISO 17025/AOAC Guidelines. Use the Comment Sections below to address suitability and effectiveness for each assigned topic. Use the drop down list at the start of each Comment section to identify the topic.

Assigned Topics:

4.1 Organization (QMS.002)
4.2 Management
4.3 Document Control (QMS.003)
4.4 Review of Requests, Tenders, Contracts (QMS.004)
4.5 Subcontracting
4.6 Purchasing Services &Supplies (QMS.006)
4.7 Service to the Customer (QMS.007)
4.8 Complaints (QMS.007)
4.9 Control of Nonconforming Testing (QMS.009)
4.10 Improvement (QMS.010)
4.11 Corrective Action (QMS.009)
4.12 Preventive Action (QMS.010)
4.13 Control of Records (QMS.011, QMS.019)
4.14 Internal Audits (QMS.012)
4.15 Management Review (QMS.013)
5.1 General
5.2 Personnel (QMS.014)
5.3 Accommodation & Environmental Conditions & AOAC Appx. B (QMS.015 & subordinates)
5.4 Test Methods & Method Validation
5.4.2 Selection of Methods (QMS.016)
5.4.3-5.4.5 Method Validation (QMS.018)
5.4.6 Estimation of Measurement Uncertainty (QMS.017)
5.4.7 Control of Data (QMS.019, QMS.025.001)
5.5 Equipment & AOAC Appendix A (QMS.020 & subordinates)
5.6 Measurement Traceability (QMS.021 & subordinates)
5.7 Sampling
5.8 Handling of Test Items (QMS.023)
5.9 Ensuring the Quality of Test Results (QMS.024 & subordinates)
5.10 Reporting the Results (QMS.025)
A2LA Advertising Policy (QMS.026)

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Management Review Preparation Planner (Continued)

QA – Record Annual Document Review

Comments Section

Keep in mind the definition of "Effective": The degree to which objectives are achieved and the extent to which targeted problems are solved.

Choose an item

Is the policy both **suitable and effective**? Why/why not?

Is the procedure both **suitable and effective** for our lab? Why/why not?

[Note: this is more than just an audit against ISO or the Quality Manual – does the process in this procedure work here? Is it the right process for our lab?]

Comments/corrections for the DRR list (be specific):

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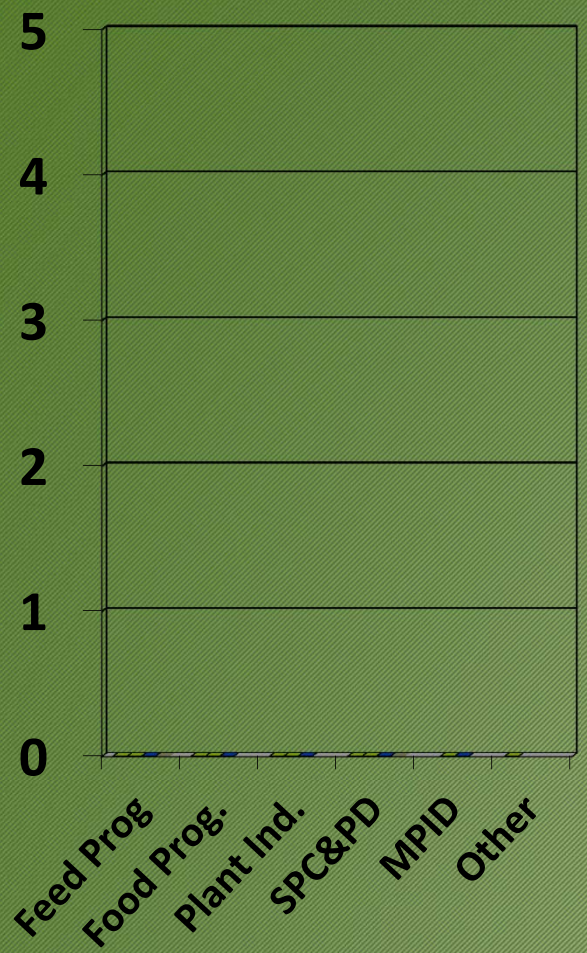
Feedback and Complaint Summary

(2/1/14 – 4/30/14)



- 0 new records in Database
- How many led to NCR? 0
- How many led to PAR? 0
- How many were positive? 0
- Complaints? 0
- Inquiries? 0

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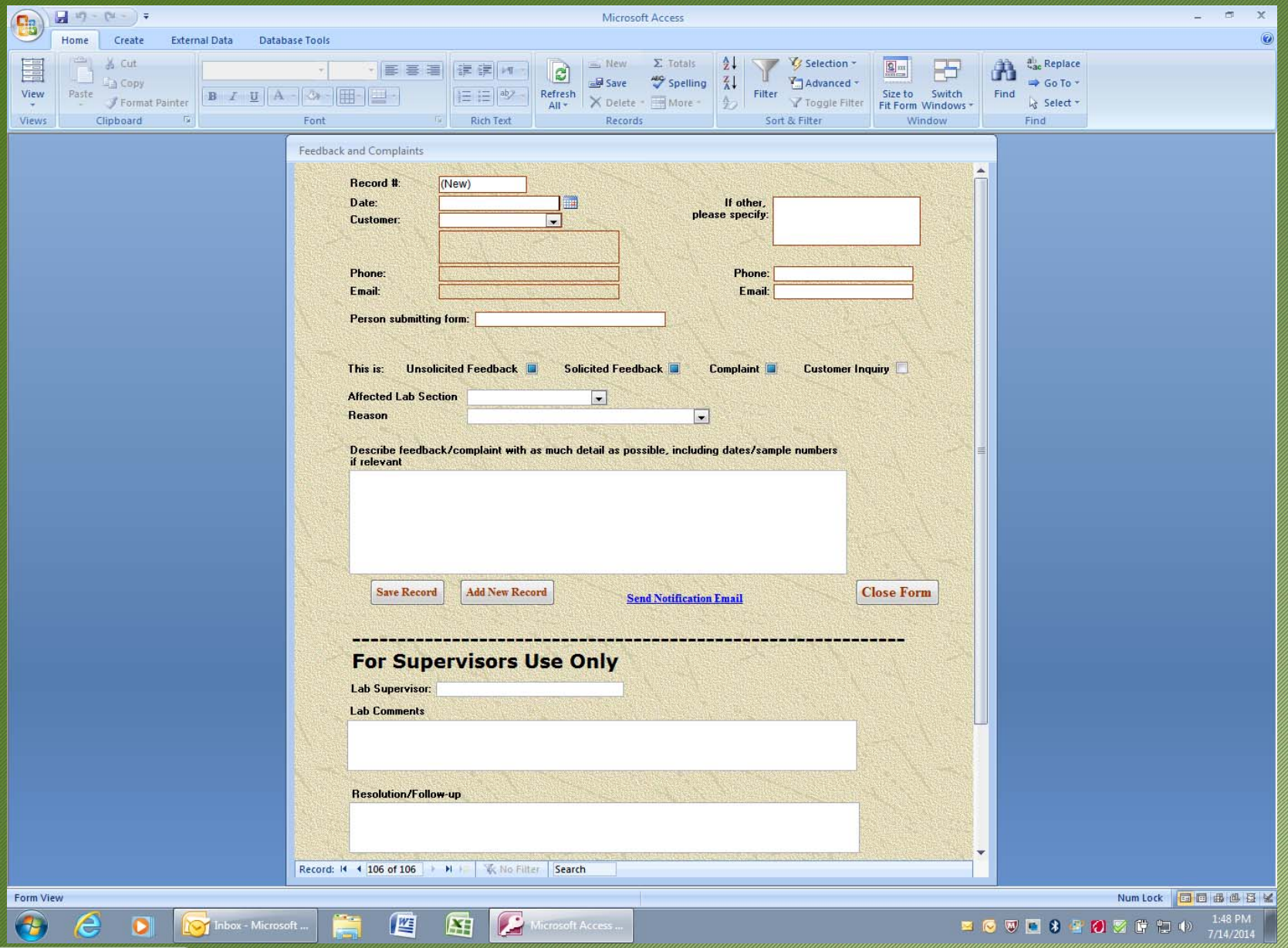

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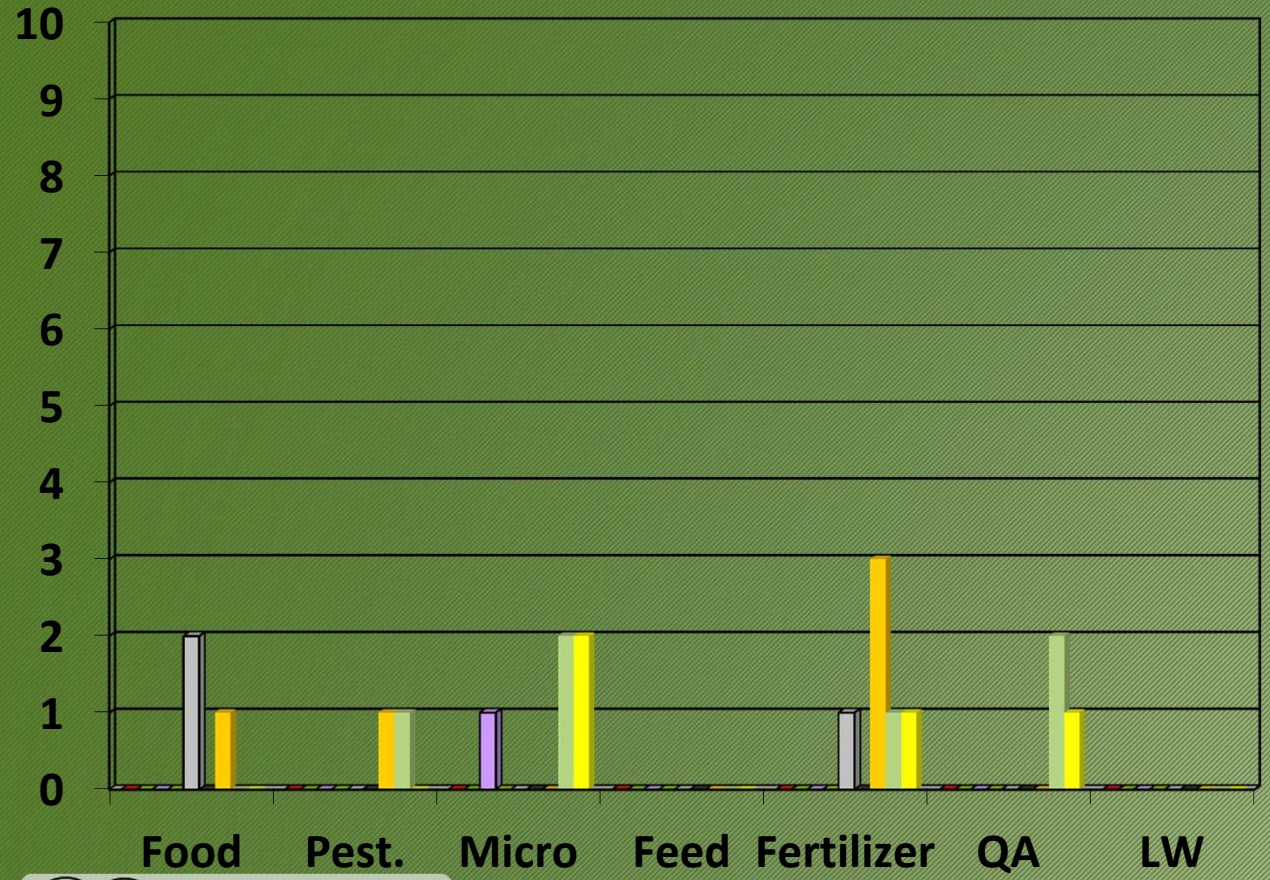
NCRs by Lab

(2/1/14 - 4/30/14)

	Total NCRs	Audit	Self Report
Food	3	0	2
Pest	2	1	1
Micro	5	0	5
Feed	3	0	3
Fert.	6	0	3
QA	3	0	2
LW	3	3	0



Total =include # already open; Audit/Self Report = # initiated during this time period

- Incor. Data
- Supplies
- Eqp
- Contam.
- QC
- Customer
- PT
- Doc

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PARs
(2/1/14 - 4/30/14)

Section	# Total PARs submitted	Topics	Record numbers
Fert	2	Xxxxxxx yyyyyyy	14-133; 14-136
Food	1	zzzzzzzzz	14-139
Micro	1	aaaaaaaa	14-129
Pest Res	0		
Feed/Forage	2	Bbbbbbb cccccccc	14-134; 14-138
QA	1	ddddddd	14-135
LW	2	Eeeeeeee fffffffffffffff	14-128, 14-131,

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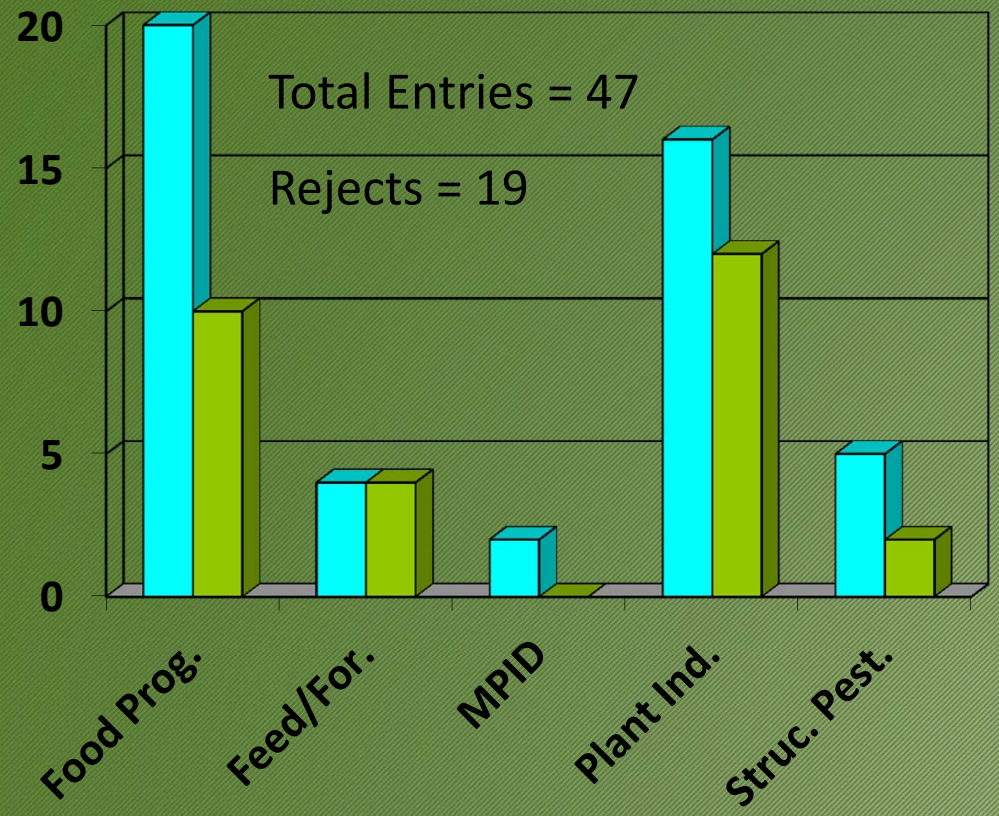
Sample Disposition Records

Totals by Customer

(2/1/14 – 4/30/14)



Reason for Entry	# Entries for that Reason
Sample # or # subs does not match transcript	1
Leaking/ spilling	6
Insufficient amount	7
Security seal broken	2
Other	31

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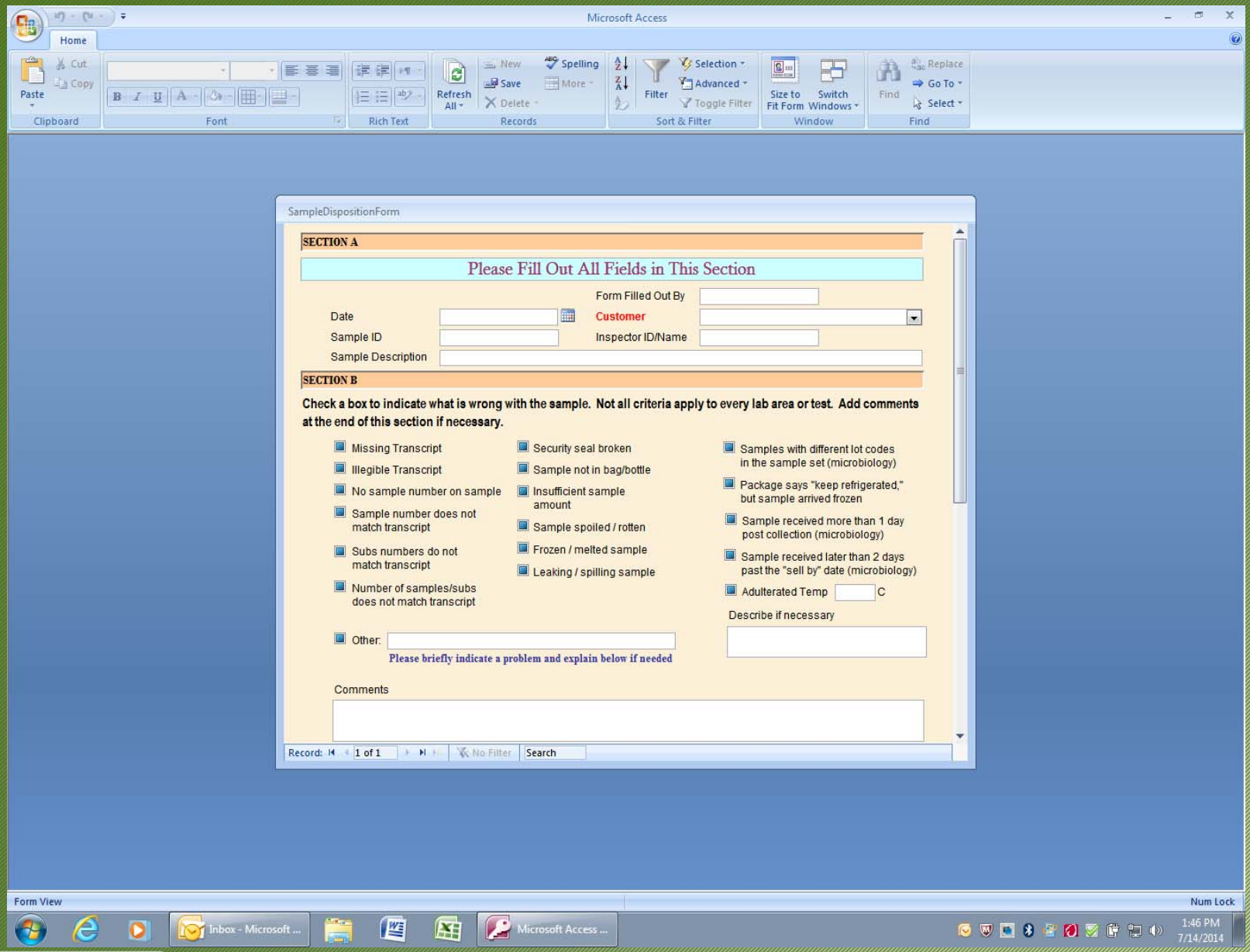

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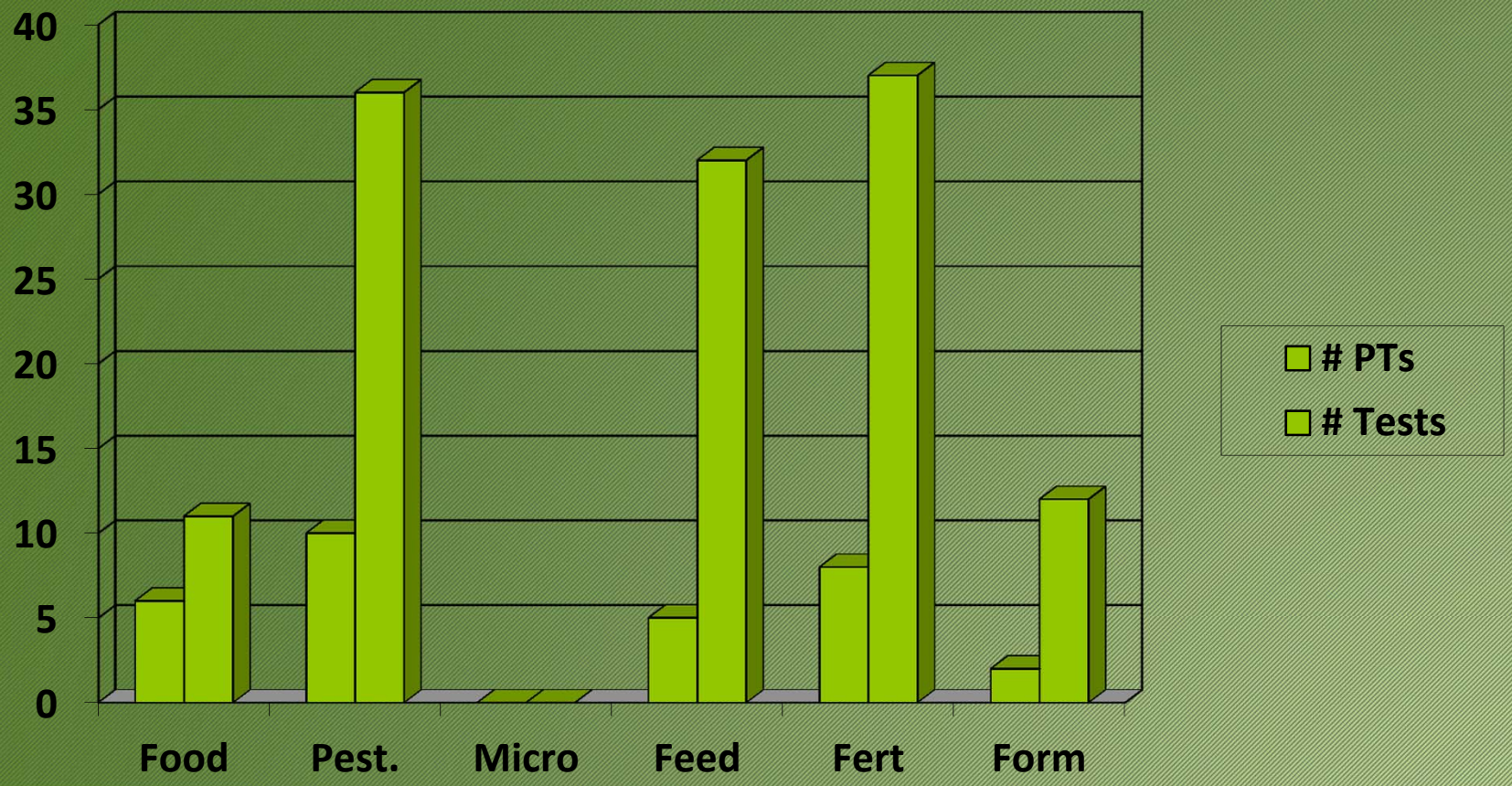
PT Summary

(2/1/14 – 4/30/14) Results back from Provider)

[Analytes counted multiple times if they appeared in multiple PTs]



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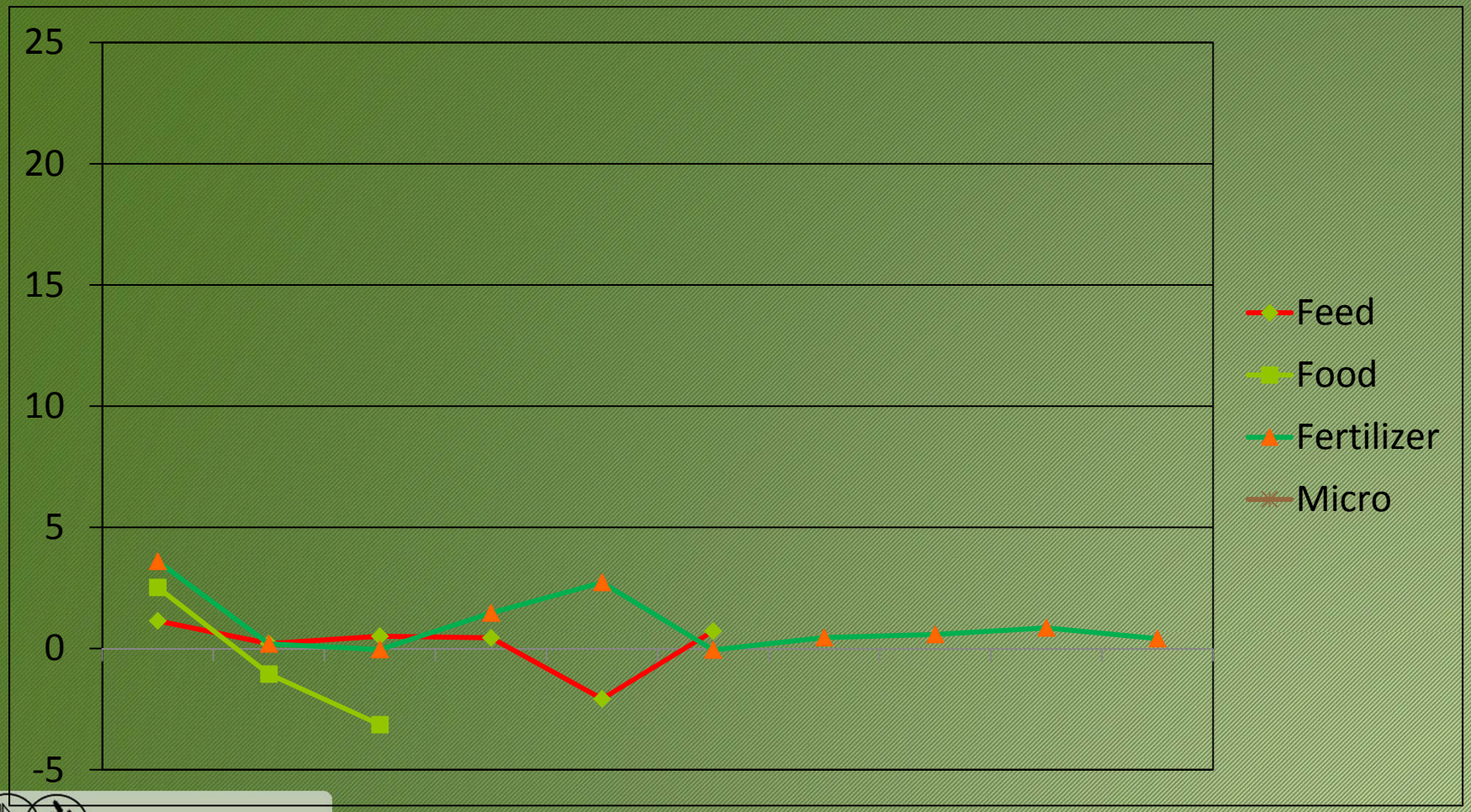
Z-scores by Lab Section

(2/1/14 – 4/30/14)



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Feed Program- Laboratory Customer Meeting
Date, 2014

Attendance:

Email distribution:

1] BSE Grant

- Sample Collection & Testing –
- AAFCO meeting

2] Contracts – reviewed every two years – Feed March 2014

3] LIMS Reports –

4] Receiving –

•Sample Dispositions (from the *lab's* database) 1/17/14-2/9/14 none to report

SD #	Date	Sample #	Sample Descr.	Problem	Tested? Y/N

5] Other Sample Issues –

Additional Discussion:

Action Items and Assignments for next meeting:

Complete updates to contracts during meeting.

Action item	Assigned to	Due date	Completed

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	Technician
KRR	ISO 17025 Accreditation Maintenance and Continuous Improvement [QMS Documentation, Non Conformities, Corrective Actions, Preventive Actions, Audits]
G	Support continuous improvement by identifying at least 1 idea [includes ideas passed along from supervisor or other lab staff]. Examples of ideas include: ♦ improve the quality management system; ♦ improve understanding or interpretation of quality documentation; ♦ submit and develop preventive action [PAR] suggestions ♦ maximize efficient and effective use of personnel, equipment or lab space by optimizing use of personnel work hours, increasing run time on equipment, altering lab space, moving equipment; ♦ minimize testing turnaround time through identifying, investigating and addressing the cause of bottlenecks, delays or disruption to work flow; ♦ improve support to customer; ♦ reduce cost of testing [consumables, chemicals, standards, kits, glassware, optimized batching, etc] ♦ reduce cost of equipment maintenance ♦ improve safety
VG	Support continuous improvement by assisting Supervisor or assigned team with planning and/or implementing section ideas.
O	VG plus Support continuous improvement by identifying at least 2 ideas [includes ideas passed along from supervisor or other lab staff] and assisting Supervisor or assigned team with planning and/or implementing section ideas.

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	Technician
KRR	Training
G	Complete training as required per FDPD-QMS.014 Training and Competence, attend scheduled training sessions, complete reading assignments per Document Transmittal Notices [DTN] from QA, maintain personal training database and complete required Demonstrations of Competence. No more than 2 incidences of not attending scheduled training sessions, completing reading assignments, maintaining training matrix, completing Demonstration of Competence or completing QMS or lab section training requirements. Attended other training as assigned, such as vendor training. After training opportunities / classes are completed, must submit a brief report describing the take-away points to supervisor

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KRR	Training (continued)
VG	Good level plus conducts training of others as assigned by supervisor or QA. No more than 1 incidence of not completing QMS or lab section training requirements. ***Proactively seeks one technical training opportunities from the following*** -vendor classes or webinars association, government or other sponsor classes -online training - reads a book - reads 2 or more articles = 1 training [all above options must be approved by Supervisor] After training is completed, must submit a brief report describing the take-away points to supervisor

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KRR	Training (continued)
O	Very Good level plus volunteers to train others or develop sections for the lab section training manual or other example of proactive contribution to lab training program. No incidences of not completing QMS or lab section training requirements. ***Proactively seeks two technical training opportunities from the following*** -vendor classes or webinars -association, government or other sponsor classes -online training - reads a book - reads 2 or more articles = 1 training [all above options must be approved by Supervisor] After training is completed, must submit a brief report describing the take-away points to supervisor.

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Validation and Verification of Analytical Methods

Presented by Kristi McCallum
Colorado Department of Agriculture

COLORADO
Department of Agriculture
Inspection & Consumer Services Division

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Relevant section of the standard

5.4 Method Validation

What is Validation?

Defined in ISO/IEC 17025:2005 Section 5.4.1

"The confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled."

IN ENGLISH PLEASE!

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- Defined performance characteristics
- Must compare to a reference method
- Statistical evaluation is performed to show equivalence to a reference method

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When is a Validation Required?

- New method is developed
- Modifications are made to an existing method/official method
- Extension of scope (i.e., additional matrices not evaluated, changes in the intended use)
- Significant changes in instrument parameters, reagents, time, temperature, etc.
- A change in technology/instrumentation

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What is Verification?

- Demonstration that a previously validated method can meet the analytical requirements (i.e., precision, accuracy, interferences, matrices, analyst) and suitability in YOUR lab
- Fit for use – an established method will meet customer or laboratory requirements for detection limits, sample types, etc.
- Validation has already been performed and the method is well established

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When is Verification Required

- Each analysis in your laboratory for which you did not validate
- Can you run a specific method in **your** lab with your equipment, your analysts, your reagents, etc.
- Can your lab meet the performance criteria of the method?
- Remember, if you change/alter/deviate from a validated method, you must **validate** that the change you made is equivalent in performance to the originally validated method!
- FDA Guidance on in-house verification of a validated method

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Performance Characteristic	Validation	Verification
Ruggedness (usually performed prior to beginning validation)	Yes	No
Selectivity	Yes	No unless matrices differ
Robustness/Matrix Effects/cross-sensitivity	Yes	No unless matrices differ
Limit of Detection (LOD)	Yes	Yes – can you meet listed
Limit of Quantitation (LOQ)	Yes	Yes – can you meet listed
Analytical Range	Yes	Yes – can you meet listed
Linearity	Yes	No
Accuracy (Using a CRM)	Yes	Yes
Precision	Yes	Yes
Repeatability	Yes	Yes - once
Reproducibility	Yes	Yes
Measurement Uncertainty	Yes	Yes/No

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Resources and Guidance

- **How to Meet ISO17025 Requirements for Method Verification, AOAC International**, *www.aoac.org/imis15.../alacc_guide_2008.pdf*
- ***Guide to Method Validation for Quantitative Analysis in Chemical Testing Laboratories,*** *www.inab.ie/media/PS15.pdf*
- ***Guidelines for Validation and Verification of Quantitative and Qualitative Test Methods, National Association of Testing Authorities (NATA)*** *http://www.nata.com.au/nata/accreditation-publication/nata-accreditation-guidance-and-information/category/50-nata-tech-notes-info-papers*
- ***Harmonized Guidelines for Single-Laboratory Method Validation of Chemical Test Methods, IUPAC Technical Report*** *http://www.iupac.org/publications/pac/74/5/0835/*
- ***FERN-ADM.0008.00 FERN Validation Guidelines for FERN Chemical, Microbiological and Radiological Methods, Original, 06/22/10.***
- ***AOAC International Method Validation Programs Manual*** *http://www.aoac.org/iMIS15_Prod/AOAC/Publications/Guidelines/AOAC_Member/Pubs/Guides/Guidelines.aspx?hkey=965b2306-1083-404c-b41a-bf159216a610*

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Reference Materials
Reference Standards
Measurement Traceability

ISO 17025

Presented by Louise Ogden

Minnesota Department of Agriculture

Relevant sections of the standard

4.6 Purchasing and Supplies

5.4 Test and Calibration Methods and Method Validation

5.6 Measurement Traceability

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Definitions

- Measurement Traceability
- Reference Standard – shall be calibrated by a body that can provide traceability
- Reference Materials – shall where possible be traceable to SI units of measurement or to a Certified Reference Material
- SI units – International System of Units

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Traceability

- Unbroken chain of comparisons
- Uncertainty of Measurement
- Documentation
- Competency
- Reference to SI Units
- Calibration intervals

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Examples of items to trace *IF* quality critical to the method

- Thermometers
- Temperature Monitoring Systems
- Timers
- Chemicals
- Media
- Pipettes – mechanical, multi-channel, electronic
- Volumetric (non-Class A) flasks, burettes, pipettes
- Balances / Weight sets
- Centrifuge
- Microscopes
- Safety Cabinets
- DI/RO Water

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Example Forms

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Autotrace Prep Worksheet

LC/MS/MS Method: LAB-MTH-0131

Extraction Batches: _____

Autotraces: Brenda, Patience, Grasshopper, Hal and Katniss

Sample	Weight (g)	Ext. #	Turbo Vap #

Date Weighed: _____ **Init:** _____ **Balance ID:** _____

- Sample weights (+ or -) 0.30 grams
- Reagent Water Bottle Lot #: _____

Date Spiked: _____ **Init:** _____ **Pipette ID:** _____

- 100 ul of Surrogate(+) Mix C (Metazachlor): MDA2097C
- 100 ul of PDP2013(+) Mix C: MDA2097B
- 100 ul of Surrogate(-) Mix C (Metazachlor ESA): MDA2088E
- 100 ul of PDP2013(-) Mix C: MDA2095K

Date Extracted: _____ **Init:** _____

- Ext.#: Autotrace Extractor's First Letter and Cell Position

Envi-Carb SPE Tube Lot#: _____

N-Evap #: _____

Acrodisc Syringe Filter Lot#: _____

Final Volume = 1.0ml (30% Methanol/70% Water)

Dilution Solution: _____

Comments: _____

_____

_____

Sheet Reviewed by Analyst: _____ Date: _____

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Salmonella Control Sheet

Date Started:

	Stock Culture Week:		Lot #	Date/Initials	Sterility Reaction/Initials	*C. freundii* ATCC# 43864 Reaction/Initials	*S.* Abaetetuba ATCC# 35640 Reaction/Initials
Pre-enrichment	LB						
	900 mL BPW						
	50 mL BPW						
	UPB						
	BG H_2O	DI H_2O					
		1% BG Dye					
	Other:						
Selective Enrichment	TT						
	I_2-KI						
	0.1% BG Dye						
	RV						
Screening	Low Load M-broth						
	High Load M-broth						
	VIDAS SLM						
	BHI						
	Salmonella BAX						
	Lysis Buffer						
	Other:						
Culture and Confirmation	HE						
	XLD						
	BS						
	BGS						
	Other:						
	TSI						
	LIA						
	Urea						
	Vitek 2						
	0.45% Saline						
	Other:						

Sample#:_____

_____

Notes:_____

Document Number: LAB-FORM-0128 Revision: 04 Effective Date: 30 Dec 2013 12:00 AM

Salmonella Control Sheet

Balances		
8033181106	8033401006	0134B0132021663P
Samples:	Samples:	Samples:
N1031123103102P	8032391074	
Samples:	Samples:	

Materials and Equipment			
Material	**Lot Number**	**Material**	**Lot Number**
Sterile Scalpel(s)		Disposable Sterile Needles	
		20E Pipette Tips	
Sterile Spoons		20P Pipette Tips	
Sterile Forks		50U Pipette Tips	
		200 Pipette Tips	
Sterile Tray(s)		RF-L300F Pipette Tips	
		Sterile Vitek Tubes	
Blender Blades		Other:	
		Other:	
Filtered Stomacher Bags		Other:	
Bird Rinse Bags		Other:	
Sterile Control Bottles		Other:	
1mL Pipettes		**Equipment**	**ID Number(s)**
2mL Pipettes		Steam Box	Steam Box #1
10mL Pipettes		Heat & Go	
1.5mL Microcentrifuge Tubes		Micropipettes	
BAX Lysis (cluster) Tubes		Multichannel Pipette	
BAX Lysis (cluster) Caps		Timer(s)	
Disposable Sterile Loops		Heater Blocks	
Sterile Cotton/Polyester Tipped Applicators		Other:	

Date Finished:_____ Reviewed by:_____ Review Date:_____

Materials:

Reference Materials, Equipment, and Supplies	Expiration	Comments
95% EtOH Glycerol:		
32% EtOH:		
Cottonseed Oil:		
Ascorbic Acid:		
50% KOH:		
Stock Standard No.:		
LCS No.:		
Matrix Blank :		
MeOH for Instrument Blank:		
MeOH for mobile phase:		
SPE Cartridge Lot No:		
HPLC Column:		
Balance No.:		
Auto Pipettes No.:		
Dispensing Device No.:		

Reagent Preparation:

Hydrolysis Solvent – 10 % glycerol in (95% ethanol / 5% DI water)
- In a 1-liter flask add 855 mL of ethanol and 45 mL of DI water, mix, add 100mLs of glycerin or glycerol to the ethanol/water
- Fill out SLL002A and label the reagent bottle with corresponding number, transfer reagent to reagent bottle

Dilution Solution - 32% EtOH: 68% DI-water
- In a 2-liter volumetric flask, add 640 mL of ethanol, bring to volume with DI water
- Fill out SLL002A and label the reagent bottle with corresponding number, transfer reagent to reagent bottle

Standard and Sample Preparation: **Date:**_____

Procedure Step	Initials	Comments
Remove standard from freezer, bring to room temperature		
Sample Worksheet printed from Biochemistry System		
Add 10 mL stock standard into a actinic 250 mL Florence flask		
Weigh 10 g of LCS into and record weight on page 2		
Weigh 10 g of Matrix Blank into and record weight on page 2		
Weigh 10 g of ground sample or 10 mL liquid sample (depending on units in guarantee) and record the weight on Page 2		
Add 150 mg ascorbic acid to each flask		
Add 2 mL cotton seed oil to each flask		

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Sample or RM Number	Guarantee	Solids or Liquids* Weight (g)	Liquids* Amount (mL)	Amount Extracted
Std:	N/A			
LCS:				
Blank:	N/A			

* If the sample is liquid and the guarantee is a volume (i.e. IU/mL, IU/oz, IU/tsp., etc.), add 10 mL of sample. Record the volume.

Sample Hydrolysis: **Date:**_____

Procedure Step	Initials	Comments
Add 45 mL of hydrolysis solution to each flask		
Add 10 mL KOH solution to each flask		
Add a small stir bar to each flask		
If necessary, place a small amount of stopcock grease on the flask attachment site of each reflux port		
Place the sample(s) on reflux apparatus, turn on magnetic stirrer and heating element for each sample. Turn on the water to the reflux columns		
Reflux the sample(s) w/ stirring for a min. of 60 minutes from the onset of boil		
After refluxing, turn off the heating element to each sample. Allow the sample(s) to stir and cool. Remove flask(s) from the reflux apparatus when cool. Turn off the water to the reflux columns		
Transfer the intermediate standard to a 250 mL volumetric actinic flask, bring to volume with dilution solution, add stir bar, stir for a min. of 10 minutes		
Add 140 mL of dilution solution to each flask (except the standard) and stir each flask for a minimum of 10 minutes		
Stopper each flask and place each flask in the refrigerator (2- 8°C) overnight		

Sample SPE: **Date:**_____

Procedure	Initials	Comments
Remove the sample(s) and the intermediate standard from the refrigerator and warm to room temperature		
Extract three standard volumes :500µl, 750µl, and 1000µl		
Label the SPE cartridges, 2 mL collection vials, and amber HPLC vials with sample and standard numbers		
Place the SPE cartridges on the vacuum manifold		
Determine the amount of each sample to be aliquoted: 2 mL aliquot for samples guaranteed ≤ 15,000 IU/lb 1 mL aliquot for samples guaranteed > 15,000 IU/lb and ≤ 125,000 IU/lb 500 ul aliquot for samples > 125,000 IU/lb and ≤500,000IUlb Samples Above 500,000 refer to ED181		
Record Aliquot(s) on Page 2		
Add 2 mL of acetone to each SPE cartridge/column. Draw 1 mL through, let stand 1 minute and then dry		
Add 2 mL of 2-propanol to each SPE cartridge/column and then dry		
Add 2 mL Methanol to each SPE cartridge/column. Draw some through, let soak one minute		
Add 5 mL of water to each column when the Methanol is 1-2mm above disk. ****Do not let columns go dry**		
Pipette the pre-determined aliquot of standard / sample onto each SPE cartridge when the water is 1-2 mm above disk. ****Do not let columns go dry**		
Wash each SPE cartridge/column with 4 mL of dilution solution		
Use the vacuum to dry each SPE cartridge. Ensure that columns are completely dry		
Insert the pre-numbered collection tube beneath each corresponding SPE cartridge ensuring that the numbers on the tubes match the number on the cartridges		
Pipette 1 mL of Methanol onto each SPE cartridge and elute into tubes		
After the 1 mL of methanol has collected in the tube(s), transfer to the corresponding pre-numbered, dark HPLC vials ensuring that the numbers on the tubes match the number on the vials **use pre-slit caps Perform additional dilutions if necessary		
Perform standard dilutions and record below		

Intermediate Concentration	SPE Aliquot	Additional Dilution	Working Standard Concentration

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Biochemistry Laboratory Form: Vitamin A Preparation and Extraction Number: FFM023A Version: 03	Procedure No.: FF-METH-023 Revision: 02

Instrument Analysis: **Date:**_____

Procedure	Initials	Comments
Turn on the HPLC and rinse the Vitamin A column with methanol for a minimum 15 minutes until the system is equilibrated and the baseline is stabilized		
Load the vitamin A method and allow method/column to equilibrate and stabilize		
After the instrument has stabilized, enter or select the standards preparation injection sequence		
Place the standards and samples in the auto-sampler tray		
Perform one injection for each of the standards and the blank to allow the instrument to stabilize the baseline		
Analyze the data from the preparation injections to determine if the preparation injections are acceptable. If the preparation injections are acceptable, enter the standards and sample into the analysis sequence		
Set-up Sequence per SOP		
Verify sample vial positions against the sample sequence log		
When the analysis run is complete and the data appears acceptable, the column shall be rinsed for a minimum of one hour with methanol		
After the minimum one hour rinse, the instrument shall be shut down following the instrument shutdown procedure		

Data Analysis: **Date:**_____

Procedure	Initials	Comments
Data processed, evaluated, calculated, and summarized on form FFM023B		
$R^2 \geq 0.995$		
Blank intensity < the lowest standard		
End Standard control charted and in acceptable range?		
LCS in control chart and in acceptable range?		
Correct reporting unit		
Flag samples outside of 30% the related sample guarantee as failing		
Calculate %RPD (if necessary)		
Enter data into Biochemistry System		

Uncertainty of Measurement – Estimation & Reporting

Presented by Aaron Price
Canadian Food Inspection Agency

Relevant sections of the standard

Section 5 – Technical Requirements

5.4.6 Estimation of Uncertainty of Measurement

- 5.4.6.2 Testing laboratories shall have and shall apply procedures for estimating uncertainty of measurement.

5.10 Reporting the Results

- 5.10.3.1 c) where applicable, a statement on the estimated uncertainty of measurement;

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Definition and Requirements

- *"a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand."*
  International Vocabulary of basic and general terms in Metrology. ISO, Geneva, (1993). (ISBN 92-67-10175-1)

- An uncertainty estimate yields information as to the quality of the analytical result and hence the test method used.

- Measurement uncertainty values allow comparison of analytical results within and between laboratories or with specification and regulatory limits.

- It should be noted that ISO 17025 does not recommend one approach over another. Any approach that uses statistically valid methodology and yields a reasonable estimate is as valid as another approach.

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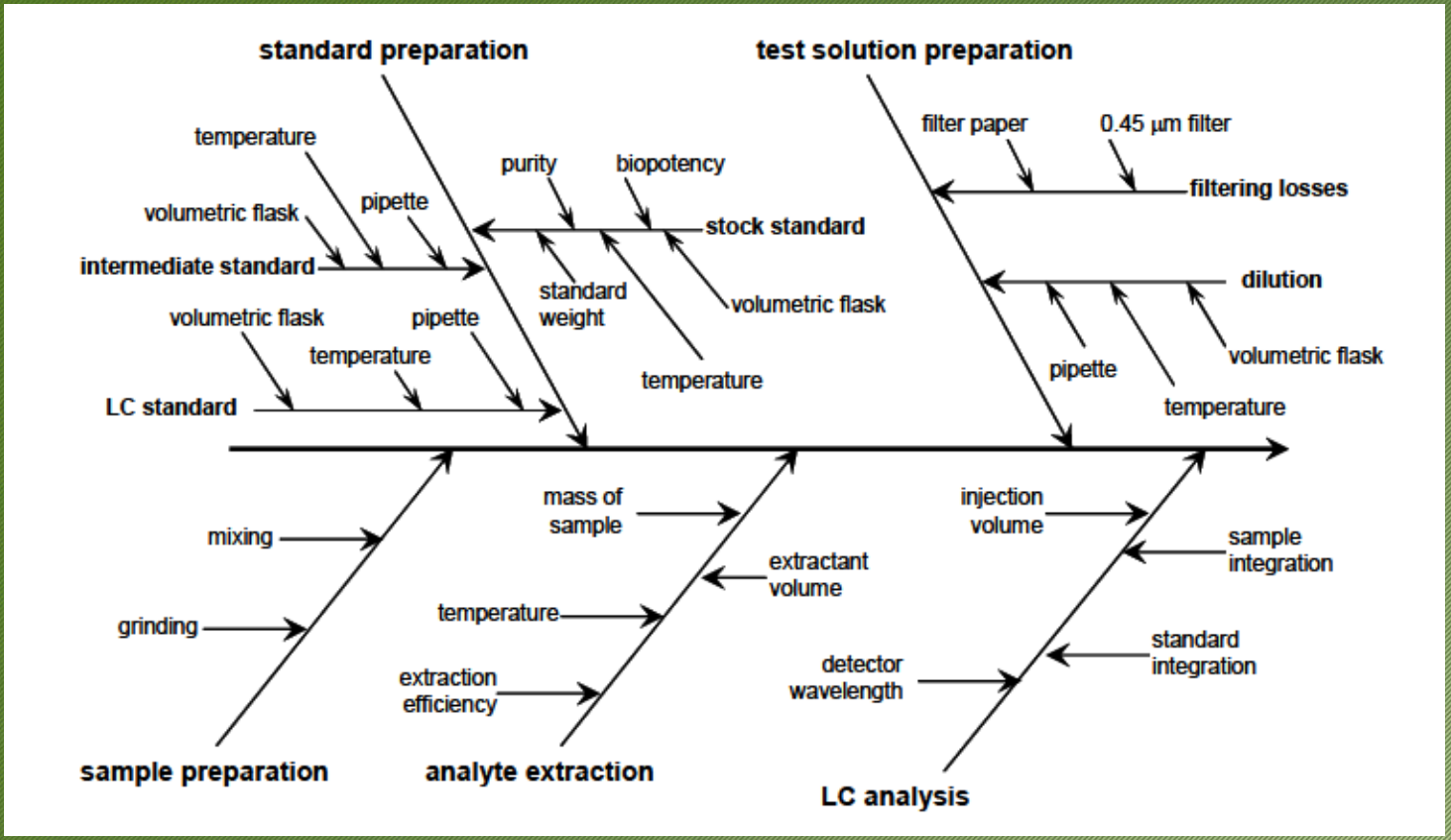
Example Sources of Uncertainty



- Attempt to identify significant uncertainty components
- Typically, components < 1/3 of largest uncertainty component are insignificant
- Cumulative effect of several small components should not be ignored though

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What You're Trying to Accomplish

Two main ways forward:

- **Bottom Up** (Guide to the Expression of Uncertainty in Measurement – GUM)
- **Top Down** (Eurachem – Quantifying Uncertainty in Analytical Measurement, and most others)

For the Top Down method:

- Grouped uncertainty components and remaining uncertainty components are quantified using existing data,
- Typically this includes precision (usually random, various distributions), bias (usually systematic) and any other uncertainty sources (eg. sample processing).

A good estimate will:

- Be as representative as possible,
- Ensure concentration ranges and different sample types covered,
- Make sure method performed over a range of conditions under which it might reasonably be expected to be used.

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Steps to Estimate Uncertainty

- Step 1: Specify the Measurand
- Step 2: Identify the Uncertainty Sources
- Step 3: Quantify the Uncertainty Components
  - QC, PT, validation data can be used
  - Equations depend on type of data used
  - components in standard deviation (standard uncertainty)
- Step 4: Calculate the Combined Standard Uncertainty

$$u_c(y) = \sqrt{u(p)^2 + u(q)^2 + \ldots} \quad \text{OR} \quad u_c(y) = y\sqrt{\left(\frac{u(p)}{p}\right)^2 + \left(\frac{u(q)}{q}\right)^2 + \cdots}$$

- Step 5: Calculate the Expanded Uncertainty
  - coverage factor (k), usually 2 (95% confidence)

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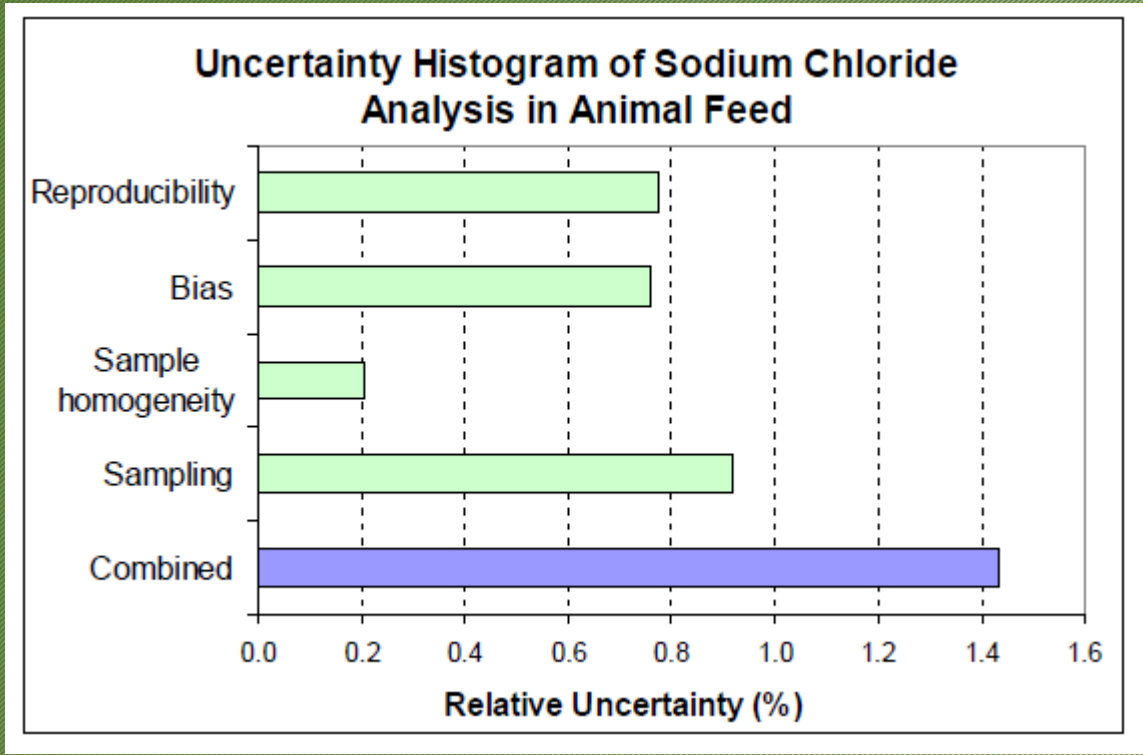
Reporting Uncertainty

Result: y +/- U(y) (% or units)

This means that the true value is within U(y) (% or units) of the measured result, 95%* of the time

*Provided a coverage factor at 95% confidence was used



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Tips and Helpful Hints

- Use the top down approach, probably already have enough data,
- A robust single laboratory validation should contain enough data for a strong uncertainty estimate,
- Extra studies may be required to examine uncertainties due to sample processing, homogeneity, standard preparation, etc,
- Can also use QC samples run with each batch, also PT samples can be used,
- Be careful not to take into account analyst errors or changes in the samples over time,
- Be aware of certain basic statistical principles, eg. square root of the sum of the squares,
- Uncertainty estimates don't need to be updated if nothing has changed,
- Be careful of the uncertainty down at the method's limit of detection/quantitation (it is likely larger!),
- Don't forget to add the coverage factor, and don't be surprised by very large uncertainties.

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Useful References

- Guide to the Expression of Uncertainty in Measurement (GUM), ISO, Geneva, 1993.
- Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, Third edition, (2012).
- AAFCO QA/QC Guidelines for Feed Laboratories, 3rd edition, 2014.
- VAM Project 3.2.1 Development and Harmonisation of Measurement Uncertainty Principles, Part (d): Protocol for uncertainty evaluation from validation data.
- Eurachem/CITAC guide: Measurement Uncertainty Arising from Sampling: A guide to methods and approaches. EURACHEM, (2007).
- ISO 5725: 1994 (Parts 1-4 and 6): Accuracy (trueness and precision) of measurement methods and results. ISO, Geneva (1994).
- ISO 21748:2010: Guide to the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation. ISO, Geneva (2010).
- NORDTEST Technical Report 537: Handbook for calculation of measurement uncertainty in environmental laboratories. NORDTEST 2003.
- P19 – CALA Measurement Uncertainty Policy, Revision 1.10 – May 2010.
- A2LA, Guide for the Estimation of Measurement Uncertainty in Testing, Rev. 1.8.

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Training Competency – Documentation & Assessment

Presented by Yvonne Salfinger
Association of Food & Drug Officials - Consultant
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Relevant Sections of the Standard

Section 5 - Technical Requirements

5.2 Personnel

- 5.2.1 Ensuring competence of qualified staff based on education, training, experience and skills
- 5.2.2 Training policies and procedures & evaluation of effectiveness
- 5.2.5 Maintaining records of authorized staff for competence, qualifications, experience, and training, as well as authorizations.

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Personnel

- The laboratory ***selects*** employees with the defined knowledge, skills and abilities to perform their duties in a competent manner.
- ***Training plan developed*** (includes a standardized checklist of required new employee training).
- Adequate supervision is provided for employees undergoing training.
- Personnel are initially ***authorized*** by their supervisor to perform tests, perform internal audits, and operate equipment. Records of this authorization are maintained. This includes contracted or temporary staff! Competency is assessed periodically.
- Even **supervisors were initially *authorized*** (I used annually updated CVs as proof of competency) and any supervisory/leadership training that was attended during the past year.

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Training and Competency Verification

- All training must be documented
  - Personnel must be trained on all management system policies and procedures as well as the Quality System Manual prior to analytical method training.
  - Must have written training procedures
  - Must have system to verify competency (Training sign-off, quizzes, demonstration of competence , PT samples, etc..)
  - Training documents are looked at heavily by the auditor!

- Three Phase Training System for analytical methods (i.e. test procedures) is an often used successful approach
  - Phase I – Trainee observes trainer
  - Phase II - Trainee performs procedure under supervision of trainer
  - Phase III – Trainee performs procedure independently and passes a PT or known reference material.

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Training Records

- Hard copy records
  - Definitely need to organize
    - We kept rosters for groups by date in a notebook
    - We kept rosters for individuals in files
    - Standardized required training titles-same each year
    - Multiple SOP trainings-had one roster, attached training list
- Database records (i.e. Microsoft Access)
  - Steep learning curve to initially set up (not nice) but allowed us to produce
    - Individual training record
    - Training course attendees
    - Reports for Department (# trained/trainings in-house, external, etc.)

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Competency Records

- Used spreadsheets with most success
- Initial authorization with a form, then tracking after that with spreadsheet
- Have to regularly review spreadsheet, or have a system in place to periodically reassess competency per your SOPs (one suggestion, at annual review)
- Initially, some were considered competent based on past PT performance, etc.
- New staff trained by senior staff-sometimes, that in itself is difficult

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Tips, Challenges and Benefits

- Staff not used to submitting external training and had to follow up a lot (suggest assigning staff that does travel to ask for this as well-harder in a large facility)
- Hard to trap laggards-usually this was the nonconformance
- Hard to track multiple trainings on a single day
- Internal training was captured well
- Used form for “Read and Understood” (minor changes), as well as specific wording needed for things such as Select Agent training.

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