Proficiency Testing & Check Samples

ISO/IEC 17025 Section 5.9.1

Presented by Louise Ogden
Minnesota Department of Agriculture

AAFCO Mid-Year Meeting - San Antonio
January 14, 2015

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ISO 17025

Section 5.9 *Ensuring the Quality of Test and Calibration Results*

5.9.1 The laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

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ISO continued

(a) regular use of Certified Reference Materials and/or internal quality control using secondary Reference Materials;

(b) participation in interlaboratory comparison or ***proficiency-testing programmes***;

(c) replicate tests or calibrations using the same or different methods;

(d) retesting or recalibration of retained items;

(e) correlation of results for different characteristics of an item.

Note: The selected methods should be appropriate for the type and volume of work undertaken.

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Requirements in addition to ISO/IEC 17025

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Quality Assurance/Quality Control Guidelines
For Feed Laboratories

supplement to

ISO/IEC 17025:2005
General Requirements for the Competence
of
Testing and Calibration Laboratories

including

AAFCO Accreditation Requirements

2014

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What the AAFCO document says

5.9 Proficiency Testing Samples / AAFCO Check Sample Program

Participating in proficiency testing schemes provides laboratories with an objective means of assessing and demonstrating the reliability of the data they are producing. Although there are several types of proficiency testing schemes, as described in ISO/IEC 17043 "Conformity assessment – General requirements for proficiency testing", they all share a common feature: test results obtained by one testing laboratory are compared with those obtained by one or more other testing laboratories. ***Laboratories are encouraged to participate in the AAFCO Check Sample Program because of its broad inclusion of analytes in feed matrices.***

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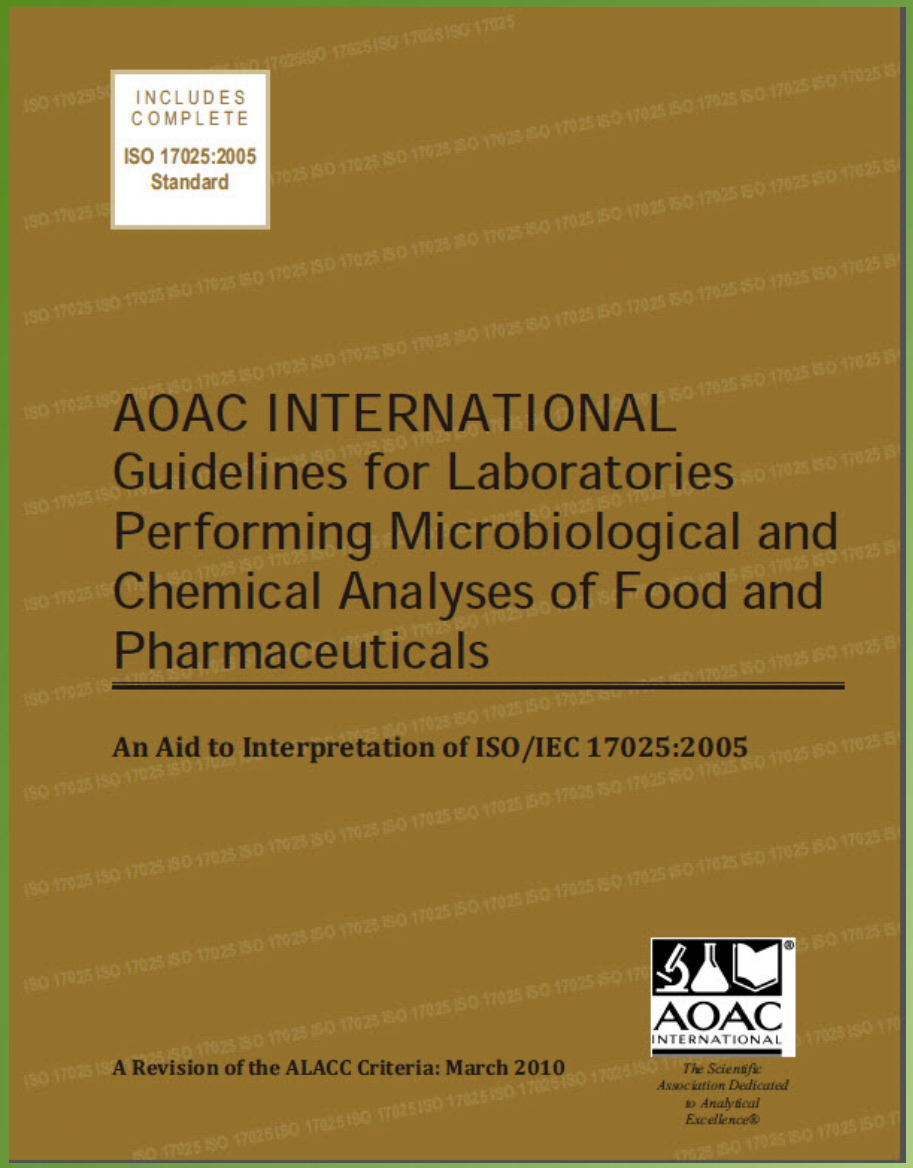
And



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Accrediting Bodies (AB) Requirements

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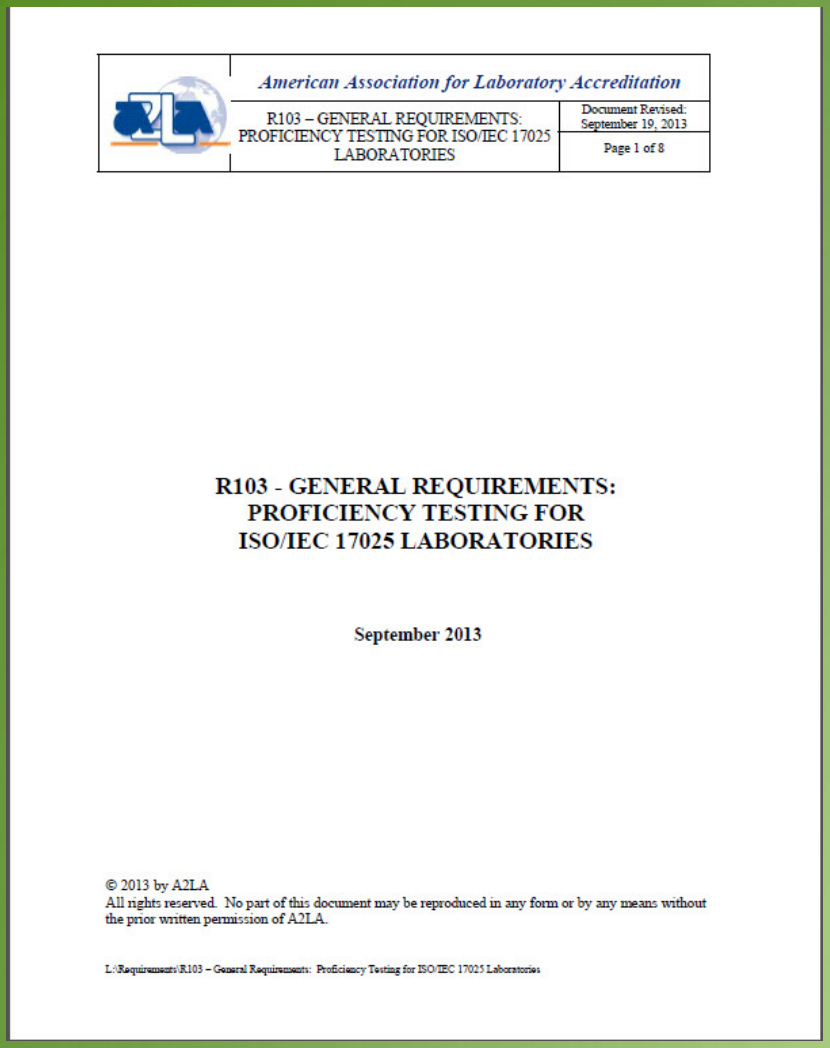
Accrediting Bodies (AB) Requirements: *A2LA*



R103 - GENERAL REQUIREMENTS: PROFICIENCY TESTING FOR ISO/IEC 17025 LABORATORIES

September 2013

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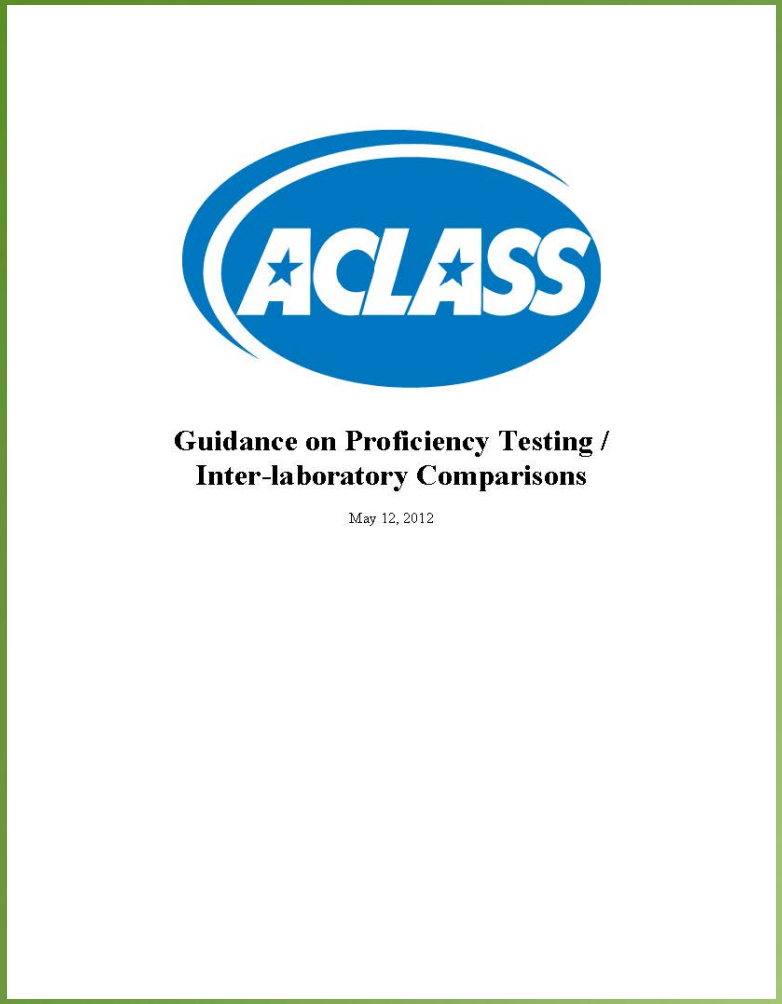
Accrediting Bodies (AB) Requirements: *ACLASS*



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Accrediting Bodies (AB) Requirements: *L-A-B*

Policy		
A-S-B ACCREDITATION SERVICES BUREAU LABORATORY ACCREDITATION BUREAU	Subject: Assuring Quality and Proficiency Testing	Policy 002
	Revision 10 Date: 11/13/14	Page 1 of 7

1. Purpose

1.1. The purpose of the policy is to define the requirements for laboratories and inspection service organizations (Conformity Assessment Bodies; CABs) to assure quality and demonstrate proficiency in order to obtain and maintain accreditation from Accreditation Services Bureau (A-S-B) and its division Laboratory Accreditation Bureau (L-A-B).

2. Scope

2.1. CABs that wish to become accredited and maintain their accreditation are responsible for assuring the quality of calibration, testing and inspection activities by participating in proficiency testing (PT), inter-laboratory comparisons (ILC) or an internal quality assurance (IQA) program that will meet the requirements of A-S-B, L-A-B and the International Laboratory Accreditation Cooperation (ILAC).

From ILAC-P9:2014 - Note: Proficiency testing may be used in some types of inspection where available and justified by the inclusion of testing activities that directly affect and determine the inspection result or when required by law or by regulators. It is, however, recognized that proficiency testing is not a usual and expected element in the accreditation of most types of inspections.

3. References

3.1. ILAC-P9:2014 – ILAC Policy for Participation in Proficiency Testing Activities.

3.2. ISO/IEC 17025:2005 – General Requirements for the Competence of Testing and Calibration Laboratories.

3.3. ISO/IEC 17020:2012 - Conformity assessment — Requirements for the operation of various types of bodies performing inspection.

3.4. ISO/IEC 17043:2010 - Conformity assessment – General requirements for proficiency testing.

4. Terms and Definitions

4.1. Conformity Assessment Body (CAB)

Body that performs conformity assessment services and that can be the object of accreditation.

4.2. Inter-laboratory Comparison (ILC)

Organization, performance and evaluation of measurements or tests on the same or similar items by two or more laboratories / inspection service organizations in accordance with predetermined conditions.

4.3. Internal Quality Assurance (IQA)

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So what are you supposed to do?

Determine if there is a suitable PT available.

- ✓ ***Analyte***
- ✓ ***Matrix***

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Proficiency Testing Providers

Food/Feed Proficiency Testing Materials by Source

Accredited	Accrediting Body	Accreditation Certificate Number	Valid To Date	Source (click for link) Do not edit this column	Frequency	Matrices	Analytes
Yes	A2LA	1782.01	7/31/2017	AOAC	Quarterly	Potatoes, Mashed	**Microbiology** Qualitative Quantitative
					Quarterly	Meat, ground	**Microbiology** Qualitative
					Quarterly	Meat Ground/Processed	**Microbiology** Qualitative
					Quarterly	Meat, Ground	**Chemistry** Nutritional Labeling
					Quarterly	Cheese, Processed	**Chemistry** Nutritional Labeling
					3/year	Fruits/vegetables	**Chemistry** Pesticide Residue
					2/year	Eggs, Liquid	**Microbiology** Qualitative
No	n/a	n/a	n/a	AOCS	Quarterly	Nuts, seeds, milk	**Chemistry** Aflatoxins
					Quarterly	Nuts, seeds, corn meal	**Chemistry** Aflatoxins
					Quarterly	Meats, eggs, cheese	**Chemistry** Cholesterol
					Quarterly	Cottonseed	**Chemistry** FFA, Foreign matter, Moisture, nitrogen, oil
					Quaterly	Feed, Animal	**Microscopy** Ingredient Identification
					Quarterly	Meal, Fish	**Chemistry** Protein, moisure, ash, oil
					Quaterly	Meal, Corn	**Chemistry** Fumonisins
					Quarterly	Oils, various	**Chemistry** Fatty Acid profile
					Quarterly	Flour, Soy	**Microbiological** GMO
					Quarterly	Oil Seeds, Mixed	**Chemistry** Foreign matter, Moisture, nitrogen, etc.
					Quarterly	Soybean	**Chemistry** NIR Nutritional labeling
					Quarterly	Soybean meal	**Chemistry** NIR Nutritional labeling
					Quarterly	Various, dry and liquid	**Chemistry** Fat, FAME, protein, vitamins
					Quarterly	Meals, various oilseeds	**Chemistry** Nutritional labelling
					Quarterly	Peanuts	**Chemistry** Nutritional labelling
					Quarterly	Soybeans	**Chemistry** Nutritional labelling
No	n/a	n/a	n/a	AACC	Monthly	Hard Wheat Flour	**Chemistry** Moisture, ash, protein, falling number, wet gluten gluten index
					Bimonthly	Hard Wheat Flour	**Chemistry**

Found on AAFCO.ORG\Laboratory – Resources for ISO 17025:2005 Accreditation

So what are you supposed to do?

Is the provider accredited to ISO/IEC 17043?

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American Association for Laboratory Accreditation

SCOPE OF ACCREDITATION TO ISO/IEC 17043:2010

AOAC INTERNATIONAL
2275 Research Blvd, Suite 300
Rockville, MD 20850-3250
Arlene Fox Phone: 301 924 7077 x143

PROFICIENCY TESTING PROVIDER

Valid To: July 31, 2017 Certificate Number: 1782.01

In recognition of the successful completion of the A2LA evaluation process, this proficiency testing provider has been found to meet the ISO/IEC 17043:2010, "Conformity assessment-General Requirements for Proficiency testing". Therefore, in recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this organization to provide proficiency testing samples in the following analyte/matrix combinations:

Program Name	Frequency	Sample/Artifact Types
M01 - Standard Microbiology	Quarterly:	Matrix: Mashed Potatoes Organisms: Qualitative: Salmonella Species, Listeria Species, E. coli O157:H7; Quantitative: Coagulase Positive Staphylococcus, Coliform, E. coli, Yeast and Mold, Aerobic Plate Count, B. cereus, Enterobacteriaeae
M02 - Pathogen-Free Microbiology	Quarterly:	Matrix: Mashed Potatoes Organisms: Quantitative: Coliform, E. coli, Yeast and Mold, Aerobic Plate Count
M03 - Meat Microbiology 1	Quarterly:	Matrix: Ground Meat Organisms: Qualitative: Salmonella Species
M04 - Meat Microbiology 2	Quarterly:	Matrix: Ground Meat Organisms: Qualitative: E. coli O157:H7
M05 - Meat Microbiology 3	Quarterly:	Matrix: Processed Meat Organisms: Qualitative: Listeria monocytogenes

5301 Buckeystown Pike, Suite 350 | Frederick, Maryland 21704-8373 | Phone: 301 644 3248 | Fax: 301 662 2974 | www.A2LA.org

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American Association for Laboratory Accreditation

SCOPE OF ACCREDITATION TO ISO/IEC GUIDE 34:2009

AOCS
2710 S. Boulder Drive
Urbana, IL 61802-7190
Dr. Richard Cantrill Phone: 217 693 4830

REFERENCE MATERIALS PRODUCER

Valid To: May 31, 2015 Certificate Number: 3438.01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this Reference Material Producer for the production of certified reference materials and reference materials of the following categories:

Category of Reference Material Certified Reference Material A4.4 Plant Materials	Test Analysis Measurement	Measurement Technique
Seed Non-Modified Canola GT73/RT73 Canola **Powder** Non-Modified Cotton MON1445 Cotton MON531 Cotton MON15985-7 Cotton MON88913 Cotton Non-Modified Maize MON88017 Maize MON87460 Maize MON89034 Maize GA21 Maize MIR604 Maize MIR162 Maize Non-Modified Potato EH92-527-1 Potato Non-Modified Soybean BPS-CV127-9 Soybean MON89788 Soybean MON87701 Soybean MON87705 Soybean Non-Modified Sugarbeet H7-1 Sugarbeet	Purity	Real-time PCR

5301 Buckeystown Pike, Suite 350 | Frederick, Maryland 21704-8373 | Phone: 301 644 3248 | Fax: 301 662 2974 | www.A2LA.org

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Road blocks that you can run into

- PT has to shipped from outside the country and there are restrictions for importing the sample matrix.
- You can find the analyte of interest but at not in matrix or at the wrong concentration (ppm and you need ppb)
- Issues with PT provider – can't meet needs

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DOCUMENT the ISSUES

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Internal Checks:
when an external PT or Check Sample are not available or is determined to be unacceptable for your program.

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1) Use a Certified Reference Material and/or internal quality control using secondary Reference Materials
2) Participate in a round robin.
3) Perform a comparison with another method.
4) **Perform an inter-laboratory comparison.**

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Internal PT and Inter-laboratory comparison

Minnesota State Fish Consumption Advisory

- Mercury
- PCB in Fish 1254/1260
- We added both of these method to our scope.

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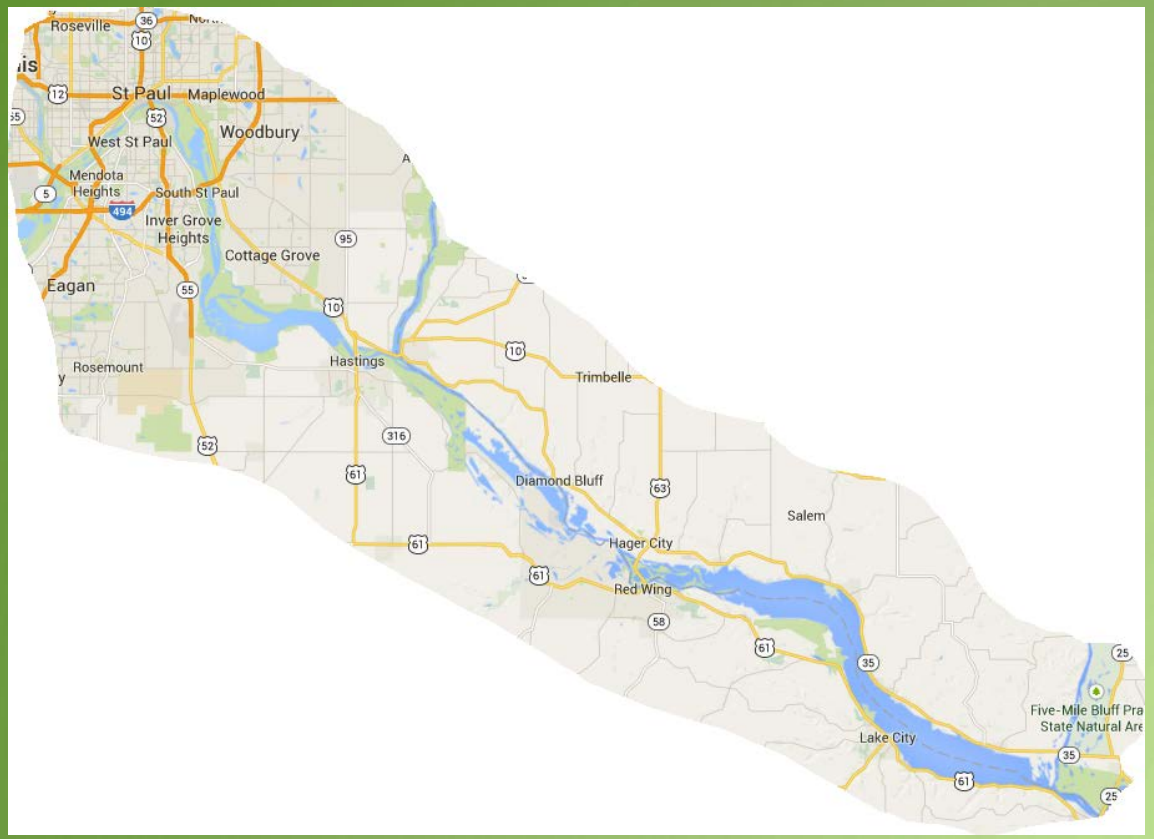
Internal PT and Inter-laboratory comparison

NEMO – 14 pound carp caught in the Mississippi River between Saint Paul and Lake Pepin in 2011.



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Internal PT and Inter-laboratory comparison

MN DNR prepped:

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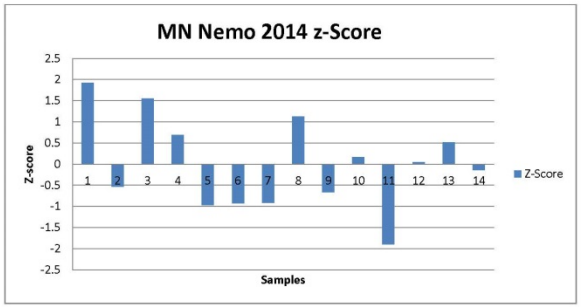
Example Report for this PT

May 6, 2014

Yoko Johnson and Louise Ogden

Summary of NEMO 2014 Internal Check Sample – PCB in Carp

MN MDA LSD Results		
Table: 2014 Data		
Sample	Result	Z-Score
FY14-1	221.2	1.924682
FY14-2	174.2	-0.535
FY14-3	214.2	1.558268
FY14-4	197.7	0.695102
FY14-5	165.9	-0.96842
FY14-6	166.7	-0.92863
FY14-7	167.0	-0.91188
FY14-8	205.9	1.126424
FY14-9	171.8	-0.6601
FY14-10	187.6	0.169037
FY14-11	148.2	-1.89544
FY14-12	185.4	0.050214
FY14-13	194.3	0.5187
FY14-14	181.7	-0.14294



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Internal PT and Inter-laboratory comparison

Our lab (MDA) did additional prep of the samples.

We analyzed many sub-samples.

We approached labs in the Food Emergency Response Network to run a comparison.

Mississippi State Chemical Lab in 2013.

Two more FERN labs have been added for 2015.

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1) Use a Certified Reference Material and/or internal quality control using secondary Reference Materials
2) Participate in a round robin.
3) Perform a comparison with another method.
4) Perform an inter-laboratory comparison.
5) **Internal Check Samples**

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Example Multi-Analyte Report for an internal check sample

Ultra Scientific Custom Standard			Nov-13	Apr-14	Nov-14						
GCMS	Reported Value	True Value	Recovery	LC+	Reported Value	True Value	Recovery	LC-	Reported Value	True Value	Recovery
Analyte				**Analyte**				**Analyte**			
Acetochlor				Acetamiprid	109	120	90.8%	2,4,5-T			
Alachlor				Aldicarb Sulfone	37	75	49.3%	2,4,5-TP			
Atrazine				Aldicarb Sulfoxide				2,4-D			
Benfluralin	71.6	100	71.6%	Azoxystrobin				2,4-DB			
Bifenthrin				Bensulfuron Methyl				Acetochlor ESA	98.4	120	82.0%
Chlorothalonil	123	201	61.2%	Boscalid	171	180	95.0%	Acetochlor OXA			
Chlorpyrifos				Bromacil				Alachlor ESA	136	120	113.3%
Clomazone		50.1		Carbaryl				Alachlor OXA			
Cyfluthrin				Carbenzadim				Bentazon			
Deethylatrazine				Carbofuran				Clopyralid			
Deisopropylatrazine				Chlorantraniliprole	184	200	92.0%	Dicamba			
Diazinon				Chlorimuron Ethyl				Dichlorprop			
Diazinon Oxon				Chlorpyrifos Oxon				Dimethenamid ESA	16.6	20	83.0%
Dichlobenil	24.3	30	81.0%	Clothianidin				Dimethenamid OXA			
Dichlorvos				Cyanazine				Flufenacet OXA	20.9	25.1	83.3%
Dimethenamid	45.2	60.1	75.2%	DEDI Atrazine				Isoxaflutole Deg	63.2	added to parent	
Dimethenamid		60		Dicrotophos	127	120	105.8%	MCPA			
Dimethoate				Difenoconazole				MCPB			
Disulfoton				Dinotefuran	124	100	124.0%	MCPP			
EPTC				Disulfoton Sulfone				Mesotrione	166	250	66.4%
Esfenvalerate				Diuron				Mesotrione	142	201	70.6%
Ethafluralin				Flumetsulam	188	181	103.9%	Metolachlor ESA			
Ethofumesate		175		Flutriafol	36.3	40	90.8%	Metolachlor OXA	25.3	35.1	72.1%
Fonofos				Halosulfuron Methyl				Picloram			
Lambda Cyhalothrin				Hexazinone				Propachlor ESA			
Malathion				Hydroxyatrazine				Propachlor OXA			
Methoxychlor		20.1		Imazamethabenz Acid				Tembotrione			
Metolachlor				Imazamethabenz Methyl				Triclopyr			
Metribuzin				Imazamox	18.7	27	69.3%				

Metribuzin DA				Imazapic							
Metribuzin DADK				Imazapyr							
Metribuzin DK				Imazaquin							
Oxadiazon		250		Imazethapyr	20.7	25	82.8%				
Parathion Methyl				Imazethapyr	14.6	20	73.0%				
Pendimethalin	290	300	96.7%	Imidacloprid							
Phorate				Isoxaflutole	76.7	160	add deg	Isoxaflutole + Isoxaflutole Deg	139.9	160	87.4%
Prometon				Linuron							
Propachlor				Metalaxyl							
Propazine				Metsulfuron Methyl							
Simazine				Myclobutanil							
Tebupirimphos	74	90	82.2%	Neburon							
Terbufos				Nicosulfuron							
Triallate				Norflurazon	33.8	45	75.1%				
Trifluralin				Norflurazon Desmethyl							
zeta-Cypermethrin				Oxydemeton Methyl							
				Parathion-methyl Oxon							
				Prometryn							
				Propiconazole							
				Pyraclostrobin	45.8	100	45.8%				
				Pyroxasulfone	166	180	92.2%				
				Saflufenacil	51.5	75	68.7%				
				Saflufenacil	38.9	45.1	86.3%				
				Siduron							
				Sulfometuron Methyl							
				Tebuconazole							
				Tetraconazole							
				Thiamethoxam							
				Thifensulfuron Methyl							
				Thiobencarb							
				Triasulfuron	Ultra could not add it to mix because of stability issues						

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Re-Purpose PT/Check Samples

a) Can be used as a laboratory control sample (LCS)

b) Can be used for testing competency of analytical staff

c) Can be used in verification/validation of methods

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THE END