Suggested Revisions to Primary and Secondary Compliance Testing Methods and Protocols

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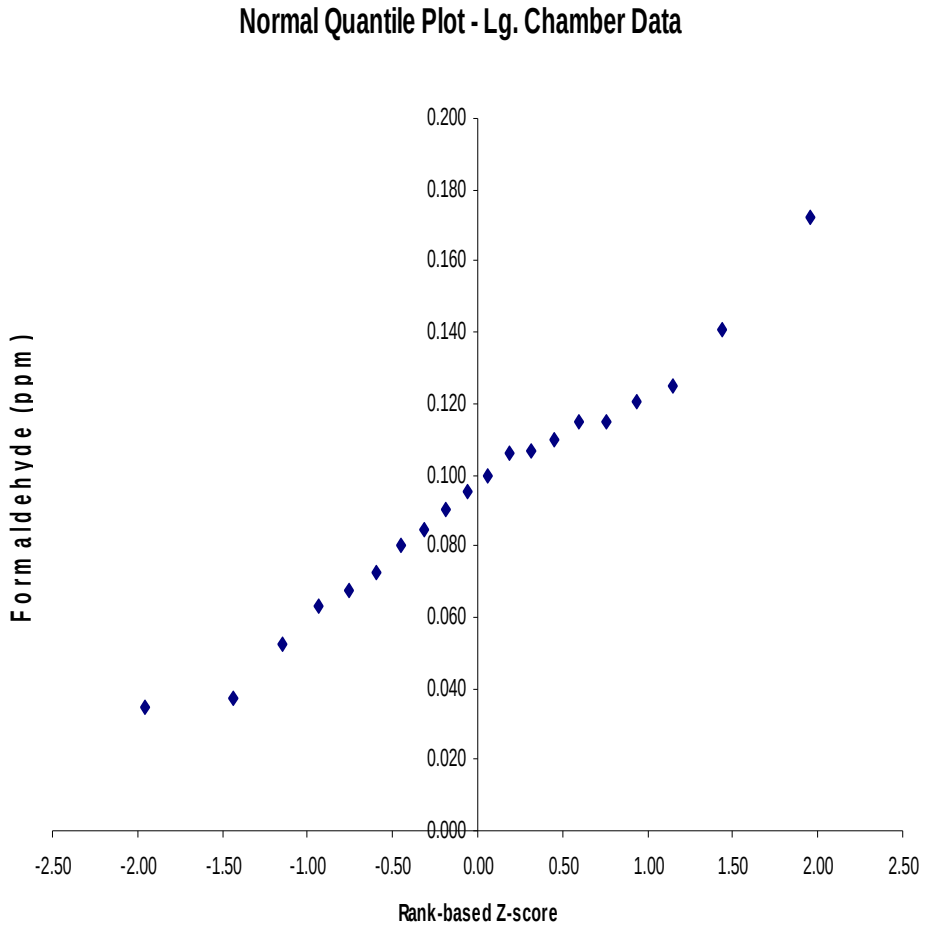
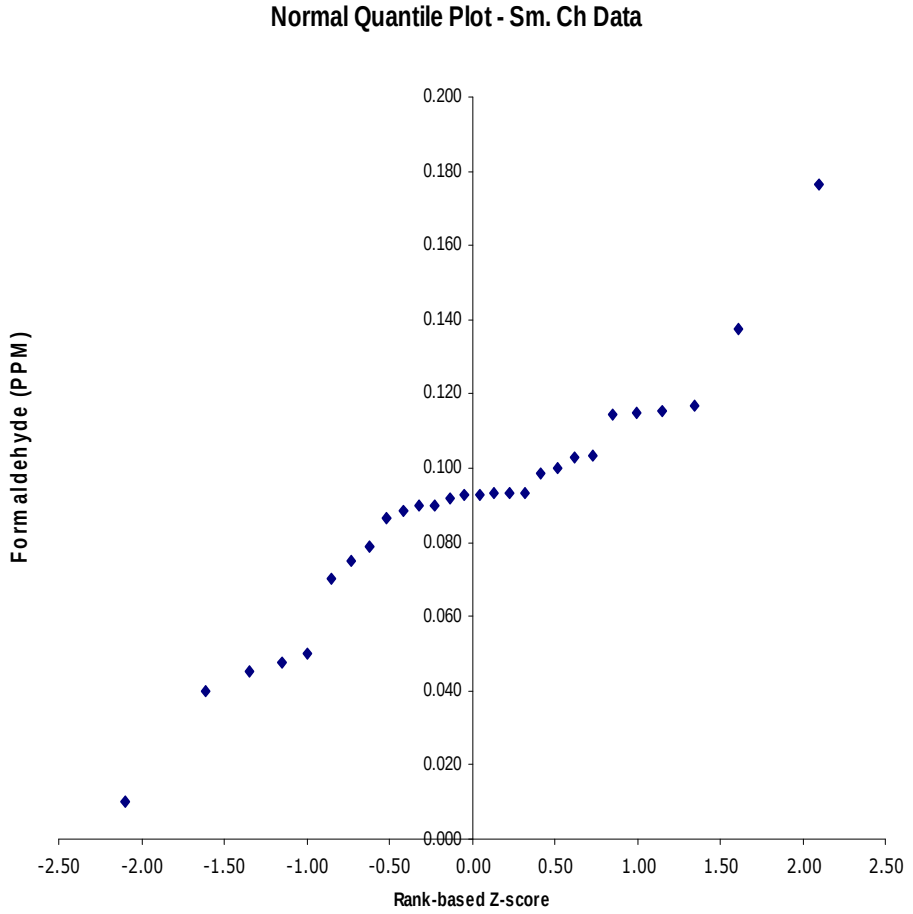
Background

- CARB Website lists 916 line items for certified mills (6/22/11) and 99 line items for NAF/ULEF Exemptions (6/24/11).
  - There are significant questions about the overall validity of the testing behind these certifications and exemptions. (See Slide 3)
  - Mills and downstream customers concerned with the spread of results among labs.
- The formaldehyde emissions classifications and limitations for the covered composite wood products are performance-based.
  - Most critical at determination of NAF/ULEF Exempt (See Slide 4)
- All labs performing compliance testing must be able to deliver accurate and reproducible results.
  - Differentiate between 0.05 and 0.04 ppm.
  - Should be able to determine concentration to ± 0.005 ppm.
  - Higher quality lab performance must be defined in the regulation, and enforceable.

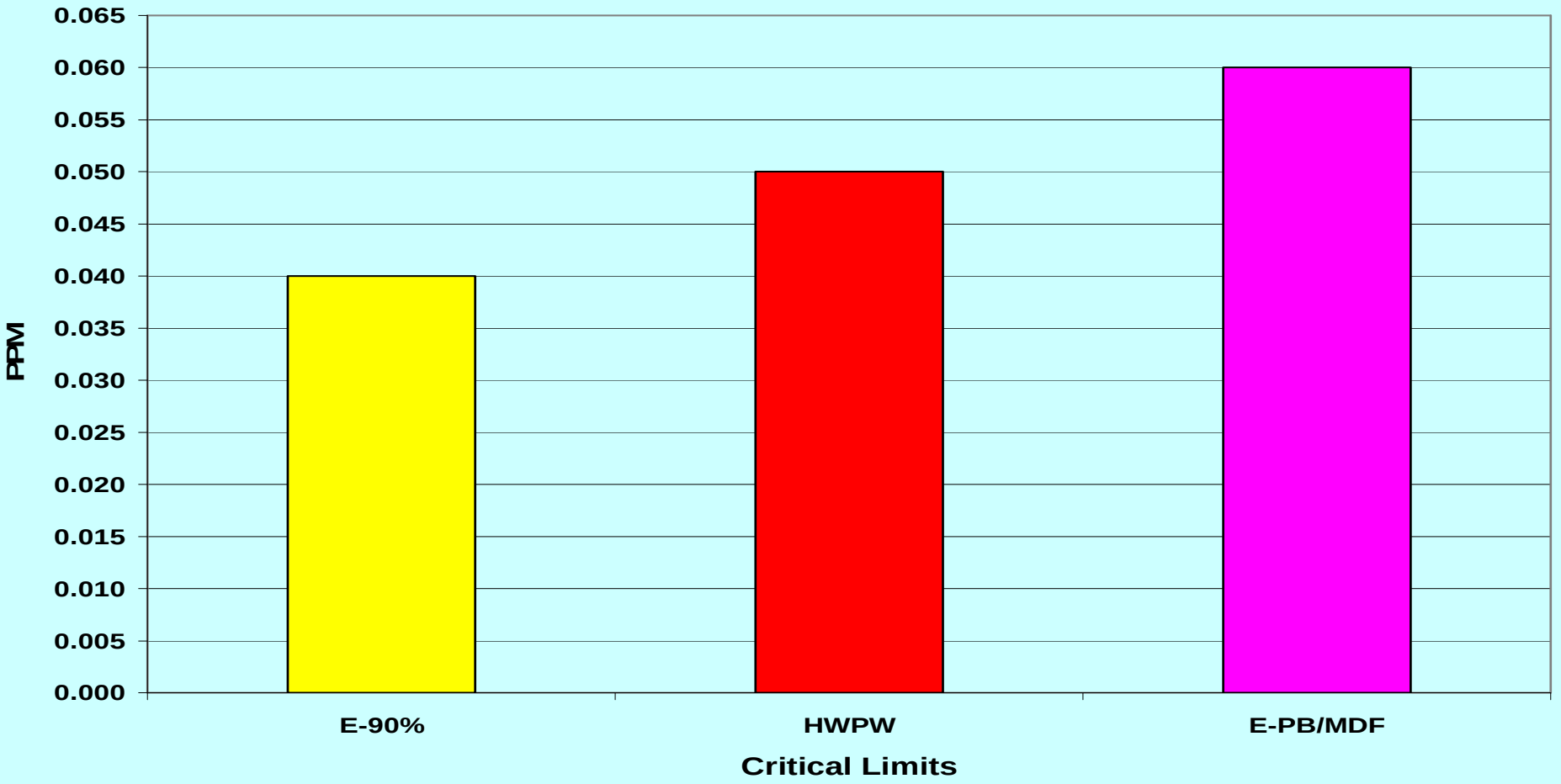
ILCS 1 – Plots of Lab Average/Test Type





23 August 2011

Title 17 CCR § 93120 *et.seq.* – Critical Limits



23 August 2011

Recommended Test Method Changes – (1)

Changes compared to existing test methods. Blanks indicate no change. The "TPC Changes" are for compliance testing by qualified laboratories.		E1333		D6007		
		Existing	Proposed	Existing	QC	TPC
Conditioning:	Units	Standard	Changes	Standard	Changes	Changes
Time to Condition Target	Day	7		----		7
Time to Condition Target	Hour	----		2		----
Condition Time Range Allowance	Hour	± 3		----		± 3
Condition Time Range Allowance	Min.	----		± 15		----
Temperature Target	°F	75	77	75	77	77
Temperature Range Allowance	℉	± 5	± 3	± 5	± 3	± 3
RH Target	%	50		50		
RH Range Allowance	%	± 5	± 4	± 5	± 4	± 4
Background Formaldehyde	ppm	≤ 0.1	≤ 0.020	≤ 0.1	≤ 0.020	≤ 0.020
Air Changes per Hour past sample	ACH	N/S	≥ 1.00	N/S	≥ 1.00	≥ 1.00
Test Chamber:						
Minimum Chamber Volume	m^3	22		0.02		0.060
Maximum Chamber Volume	m^3	N/S		1		1.000
Air Changes per Hour, Minimum	ACH	0.5	0.500	Q/A based		0.500 **OR**
Air Changes per Hour, Maximum	ACH	0.5	0.500	Q/A based		1.000 ↵
Air Change Allowance	ACH	± 0.05	± 0.010	N/S	± 0.010	± 0.010
Pre-test time in chamber	Hour	18 ± 2		Steady State	MIN 3 air changes	18 ± 2
Temperature Target	°F	77		77		
Temperature Range Allowance	℉	± 2		± 2		
RH Target	%	50		50		
RH Range Allowance	%	± 4		± 4		
Background Formaldehyde	ppm	≤ 0.02	≤ 0.010	≤ 0.02	≤ 0.010	≤ 0.010
Positive pressure required	Pa	≥ 1		N/S	≥ 1	≥ 1

Recommended Test Method Changes – (2)

Changes compared to existing test methods. Blanks indicate no change. The "TPC Changes" are for compliance testing by qualified laboratories.		E1333		D6007			
		Existing	Proposed	Existing	QC	TPC	
Product Loading Ratios ± 2%						@ 0.5 ACH	@ 1.0 ACH
Hardwood Plywood Wall Paneling	m^2/m^3	0.95		N/A		0.95	1.90
Particleboard Flooring Materials Industrial Particleboard Panels Industrial Hardwood Plywood Panels	m^2/m^3	0.43		N/A		0.43	0.86
Medium Density Fiberboard (MDF)	m^2/m^3	0.26		N/A		0.26	0.52
Particleboard Door Core	m^2/m^3	N/S	0.13	N/A		0.13	0.26
Product Q/A Ratios ± 2%							
Hardwood Plywood Wall Paneling	m/h	N/A		0.526		N/A	
Particleboard Flooring Materials Industrial Particleboard Panels Industrial Hardwood Plywood Panels	m/h	N/A		1.173		N/A	
Medium Density Fiberboard (MDF)	m/h	N/A		1.905		N/A	
Particleboard Door Core	m/h	N/A		3.846		N/A	
Air Sampling & Testing							
Pre-sampling line purge time	min	5		1			
Collection Rate (Liquid Trap MAX)	L/min	1	1.000	1	1.000	1.000	
Collection Rate - DNPH (up to)	L/min	N/A	1.500	N/A	1.500	1.500	
Collection Rate Allowance	L/min	± 0.05	± 0.010	± 0.05	± 0.010	± 0.010	
Collection Time	min	≥ 60		30	≥ 30	≥ 60	
Collection Time Allowance	sec	± 5		± 5			
Simultaneous Samples Collected	number	2		N/A			
Reagent blank vs DI max difference	Absorbance	0.040	0.005	0.100	0.005	0.005	
Required agreement between matched air samples	ppm	≤ 0.03	0.005				
Report							
Emissions, round to	ppm	0.01	0.001	0.01	0.001	0.001	
Emission Factor, round to	$mg/(m^2 \cdot h)$	0.001		N/S		0.001	

Recommendations for TPC labs using E1333 and D6007 methods –(3)

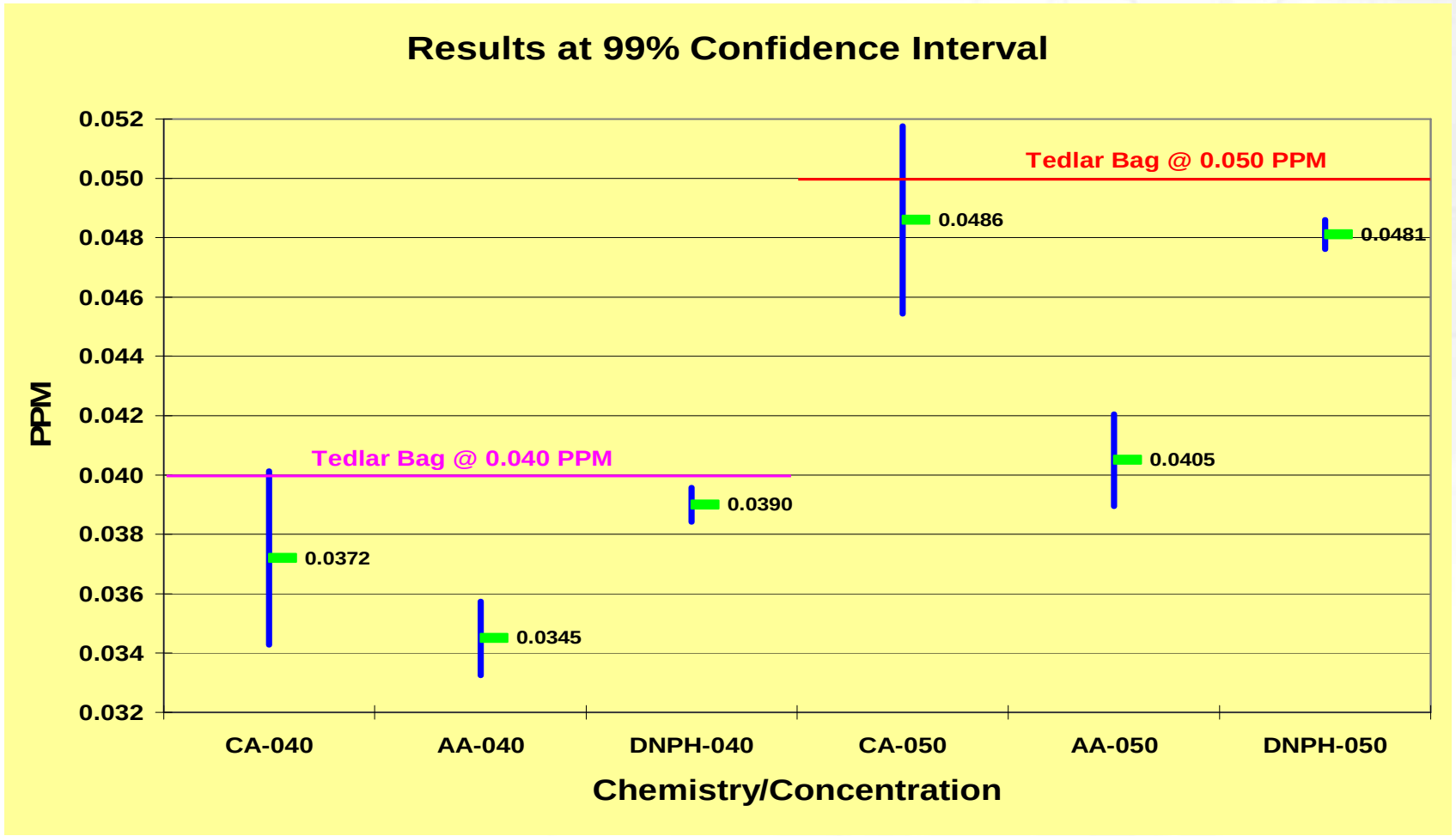
- All laboratories should participate in a proficiency demonstration under the "Tedlar Bag Protocol", and show that they are capable of achieving results that are within $\pm$ 0.005 ppm of the standard air concentration.
  - Base solutions for standard air concentrations to be supplied as unknowns suitable for the above protocol, by the controlling agency/designee.
- Require that all test results (E1333 and D6007): [Also see slide 12.]
  - Be reported to the third decimal place.
  - Have an in-lab standard deviation of $\leq$ 0.010 ppm, and
  - There also needs to be a reasonable accuracy requirement for interlaboratory comparison study results. Perhaps this may also be in terms of a standard deviation of $\leq$ 0.010 ppm.

[Note: More clarity and detail will be provided separately.]

Recommendations for TPC labs using E1333 and D6007 methods –(4)

- All flow measurement by devices calibrated to NIST standards.
  - Use of mass flow controllers recommended.
  - Eliminates need for barometric pressure correction.
  - Easily data logged to corrected (STP) volume collection.
  - Significant improvement over conventional dry gas meter measurement for air change per hour/ air volume.
  - Removes ambiguity from allowing use of ‘bubble tubes’ as calibration device for sample volume collection equipment.
  - With data logging, eliminates need to use calibrated stopwatch to calculate collected volume. Also help eliminate calculation errors.

Is the "Tedlar Protocol Feasible? Collection/Analytical Methods Study



Large and Small Scale Test - Equality

- Current Equivalency demonstration requirement allows too much variation, and does not allow for alternative small scale testing that is statistically equal to large scale results.
- Requirement to test in two different ranges is becoming very difficult, as most products today are right around 0.07 ppm and lower.
- See recap of 53 sets of equivalency tests below, plus statistical analyses.

Combined Low and Intermediate Ranges

E1333 LSCT ppm	D6007 3 Avg ppm	3 Avg Diff. ppm	D6007 Single ppm	Single Diff. ppm
Average		0.005		0.000
Std. Dev.		0.008		0.010
Equiv. Value		0.012		0.009
Max Equiv. Value		≤ 0.026		≤ 0.026

Regression Analysis: Large Chamber E1333 ppm versus Small Chamber D6007 Single Sample (S1) ppm

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The regression equation is
LC-ppm = 0.00499 + 0.964 SC(1)-ppm


Predictor       Coef   SE Coef      T      P
Constant   0.004994  0.002811   1.78  0.082
SC(1)-ppm   0.96399   0.03965  24.31  0.000


S = 0.0100127   R-Sq = 92.1%   R-Sq(adj) = 91.9%


Analysis of Variance

Source          DF        SS        MS       F      P
Regression       1  0.059261  0.059261  591.11  0.000
Residual Error  51  0.005113  0.000100
Total           52  0.064374
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23 August 2011

D6007 Equivalency versus Equality

- Current equivalency demonstration requirement allows too much variation, and the CARB regulation does not allow for alternative small scale testing that is statistically equal to large scale chamber results.
  - Requirement to test in two different ranges is becoming very difficult, as most products today are right around 0.07 ppm and lower.
  - Appropriate single sample small chamber tests are statistically suitable as an alternate secondary method, in place of the “three sets of three” currently required by CARB.
- Repeatability and reproducibility of results should be limited to a standard deviation $\leq$ 0.010 ppm.
  - For both E1333 and D6007 testing of matched samples.

Are In-lab and Interlab Requirements Feasible?

	Individual Test Results			
Test	Lab 1	Lab 2	Lab 3	
1	0.100	0.122	0.110	
2	0.098	0.121	0.110	
3	0.119	0.119	0.120	
4	0.110	0.121	0.120	
5	0.117	0.118	0.110	
6	0.117	0.117	0.120	
7	0.117	0.117	0.120	
8	0.116	0.116	0.120	
9	0.109	0.111	0.110	
10	0.111	0.113	0.110	
				GRAND
Average	0.111	0.118	0.115	0.115
S. D.	0.007	0.004	0.005	0.006

	Inter-laboratory Deviation			
Test	Lab 1	Lab 2	Lab 3	
1	-0.015	0.007	-0.005	
2	-0.017	0.006	-0.005	
3	0.004	0.004	0.005	
4	-0.005	0.006	0.005	
5	0.002	0.003	-0.005	
6	0.002	0.002	0.005	
7	0.002	0.002	0.005	
8	0.001	0.001	0.005	
9	-0.006	-0.004	-0.005	
10	-0.004	-0.002	-0.005	
				GRAND
Average	-0.003	0.003	0.000	0.000
S. D.	0.007	0.004	0.005	0.006

	In-Lab Deviation			
Test	Lab 1	Lab 2	Lab 3	
1	-0.011	0.004	-0.005	
2	-0.013	0.003	-0.005	
3	0.008	0.001	0.005	
4	-0.001	0.003	0.005	
5	0.006	0.000	-0.005	
6	0.006	-0.001	0.005	
7	0.006	-0.001	0.005	
8	0.005	-0.002	0.005	
9	-0.002	-0.007	-0.005	
10	0.000	-0.005	-0.005	
				GRAND
Average	0.000	0.000	0.000	0.000
S. D.	0.007	0.004	0.005	0.005

Safe Harbor

- Incent importers to self-test the goods they are bringing in, by offering a safe harbor for their appropriate due diligence.
  - Testing to be done by an approved laboratory that meets suggested accuracy and precision requirements.
  - Structure so that it lowers financial risk of importers.
  - Importers using this can then effectively enforce the regulation through their purchasing power, which transcends regulatory jurisdictional boundaries on a global basis.
  - Reduce burden on regulatory enforcement agencies.
- Details will require much additional input from importers, and others along the chain of commerce.
- Deconstructive testing method must be finalized and shared with all concerned parties.
  - Protocol for deconstruction
  - Clear definition of what range of results are acceptable to demonstrate compliance.