State of California
California Environmental Protection Agency
Air Resources Board

Results of Interlaboratory Comparison of Composite Wood Product Formaldehyde Emissions Testing by Third Party Certifiers & Contract Laboratories 2014

April 2015

Table of Contents

Summary

The Airborne Toxic Control Measure to Reduce Formaldehyde Emissions from Composite Wood Products (Composite Wood Products ATCM or ATCM, title 17, California Code of Regulations, sections 93120-93120.12) requires third party certifiers (TPCs) and their contract laboratories to participate in an interlaboratory comparison (ILC) during the first year the laboratory is used by a TPC, followed by participation in interlaboratory comparisons every two years. In the fall of 2014, California Air Resources Board (ARB) staff conducted an interlaboratory comparison herein referred to as the 2014 ILC.

The primary objectives for the 2014 ILC were to:

- Evaluate the proficiency of individual laboratories to perform formaldehyde emission testing of composite wood products using either ASTM E 1333 (large chamber) and/or ASTM D 6007 (small chamber)[1] based on z-scores;[2]
- Assess the variability between ASTM E 1333 and ASTM D 6007 formaldehyde test method results for laboratories that operate both methods;
- Evaluate within-laboratory repeatability (precision);
- Identify measurement issues and potential sources of error within individual laboratories and;
- Suggest corrective actions to improve future performance.

There were 43 participants in the 2014 ILC representing TPCs, contract laboratories, and government organizations. Two types of unfinished composite wood products were selected as the test materials for the 2014 ILC: medium density fiberboard (MDF) and particleboard (PB). The assigned value for the test material's formaldehyde concentration was based on the consensus of the participants' results using the "robust mean" calculated in accordance with the recommendations in the IUPAC Technical Report. The target standard deviation was based on the standard deviation of the individual participant's results. For each material, the laboratory proficiency was determined based on the calculated z-scores.

Under the protocol, laboratories were selected for a follow-up evaluation if they had a z-score greater than ±2.0 based on the results from the testing of the MDF and PB materials and if their test results were greater than 0.03 ppm from the robust mean. In addition, follow up evaluations were conducted if the ASTM E 1333 and ASTM D 6007 results for the same test material differed by more than 0.03 parts per million (ppm) and

[1] In this report, all references to ASTM D 6007 or small chamber are referring to the ATCM's secondary test method (a small chamber which has been tested and found to produce equivalent results to the large chamber)

[2] Individual laboratory proficiency is expressed in terms of z-scores in accordance with the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories (IUPAC Technical Report), Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem*., 78(1), 145-196. For both MDF and PB, laboratory proficiency was based on z-scores calculated using the standard deviation of the participants' results.

if within-laboratory precision was more than 0.02 ppm. Based on the criteria outlined above, all 43 laboratories had satisfactory results. All laboratories that conducted both ASTM E 1333 and ASTM D 6007 testing had close agreement between the two test methods, and all laboratories demonstrated satisfactory within-laboratory precision, where individual laboratory's test results were within 0.02 ppm.

I. Introduction

The Composite Wood Products ATCM requires TPCs and their contract laboratories to participate in an ILC during the first year the laboratory is used by a TPC, followed by participation in ILCs every two years. In the past, the California Air Resources Board (ARB) conducted three ILCs, in 2009, 2011-12 and in 2013. In the fall of 2014, ARB staff initiated the 2014 ILC. For the 2014 ILC, two types of composite wood products were used as test materials: MDF and PB. The following sections describe: objectives for the 2014 ILC, participants, program design and testing protocols, approach for using the results to evaluate laboratory proficiency, data analysis, and results for the 2014 ILC.

II. Objectives

Interlaboratory comparison studies are useful in assessing the performance and technical capability of individual laboratories in conducting tests and for monitoring performance over time. An individual laboratory can use the information from an interlaboratory comparison study to improve and/or maintain internal operating procedures, instruments, and the analytical skills of laboratory staff. The primary objectives for the 2014 ILC were to:

- Evaluate the proficiency of individual laboratories to perform formaldehyde emission testing of composite wood products using either ASTM E 1333-02 *Determining Formaldehyde Concentrations in Air and Emission Rates from Wood Products Using a Large Chamber* (ASTM E 1333) and/or ASTM D 6007-02 *Determining Formaldehyde Concentration in Air from Wood Products Using a Small Scale Chamber* (ASTM D 6007).
    - Individual laboratory proficiency was evaluated in terms of z-scores that are based in accordance with the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories (IUPAC Technical Report[3]) using the standard deviation of the participants' results.
- Assess the variability between ASTM E 1333 and ASTM D 6007 formaldehyde test method results for laboratories that were selected to conduct both methods.
- Evaluate within-laboratory repeatability (precision);
- Identify measurement issues and potential sources of error within individual laboratories.
- Suggest corrective actions to improve future performance.

[3] Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem*., 78(1), 145-196.

III. Participants

There were 43 participants in the 2014 ILC, representing TPCs, contract laboratories, and government organizations. The names of the participants are presented in Appendix 1. The ARB Monitoring and Laboratory Division (MLD) and the Indoor Air Quality Lab from the California Department of Public Health (CDPH) comprised the two participating government laboratories. Seven laboratories were subcontractors that provide analytical testing services for their respective TPCs. One laboratory, which is currently not operating as a subcontract laboratory, was allowed to participate in the 2014 ILC. The remaining 33 laboratories were TPCs. It should be noted that although the participant's names have been provided in Appendix 1, all information regarding z-scores, test results, and any follow-up evaluations have been kept confidential through the use of codes. The code for an individual laboratory is known only to ARB staff and the respective laboratory.

For MDF products, 12 of the participants operated both large and small chambers, 10 reported results only using small chambers, and 3 only used large chambers. For PB products, 8 of the participants operated both large and small chambers and reported results using both size chambers, 11 reported results only using small chambers, and 4 only used large chambers.

IV. Study Design

Timeframe

Notifications regarding the ILC and the Protocol for the ILC (included in Appendix 2) were sent to participants on October 28, 2014. The test materials were mailed to each participant during the week of November 10, 2014. The test results from the participants were reported back to ARB staff between late-November 2014 to late-January 2015. The delay in data reporting for several laboratories/TPCs was the result of customs/border protection agencies delays and several temporary (winter holiday) laboratory closures.

Test Materials

Two types of test materials were used for the interlaboratory comparison: 49"x97" x12.0 mm (0.47") MDF panels and 48"x96"x9.5 mm (0.375") PB panels. The MDF panels were marked as meeting the ATCM's Phase 2 emission standard and the PB panels were made with ultra-low emitting formaldehyde based resin (ULEF) and labeled as such.

Bundles of test material were selected from the same batch for both MDF and PB products to minimize sample variability. Several panels (10 panels total) were also randomly selected and emission tested by ARB's MLD to confirm homogeneity. For ease of handling and reduction of shipping costs, each full panel was cut into thirds by ARB staff, yielding three pieces that measured approximately 48"x32".

For the purposes of the 2014 ILC, ARB staff provided each TPC/laboratory with one type of composite wood product, except for the two government laboratories (MLD and CDPH) and one voluntary participant laboratory, who received one of each product types. Based on random selection, half of the participating TPCs/laboratories received MDF samples and the other half received PB panels. TPCs/laboratories that only operate small chambers received one full panel, amounting to three 48"x32" pieces. TPCs/laboratories that operate a large chamber received sufficient test materials to accommodate their reported large chamber sizes (either two or three full panels) amounting to either six or nine 48"x32" pieces. ARB staff requested that TPCs/laboratories that operate both large and small chambers test the provided sample materials in both chambers. ARB staff also requested that following large chamber testing, TPCs/laboratories obtain nine pieces of samples cut from one of their full panels to the appropriate size for their small chamber testing.

Panels were wrapped in a heavy plastic sheeting (6-mil poly sheeting) which fully covered the boards. Waster sheets were added, one on each side to protect the package from damage during transit. The packages were bundled together using heavy duty plastic strapping and shipped via FedEx ground (domestic) and FedEx International Economy (overseas).

2014 ILC Testing Protocol

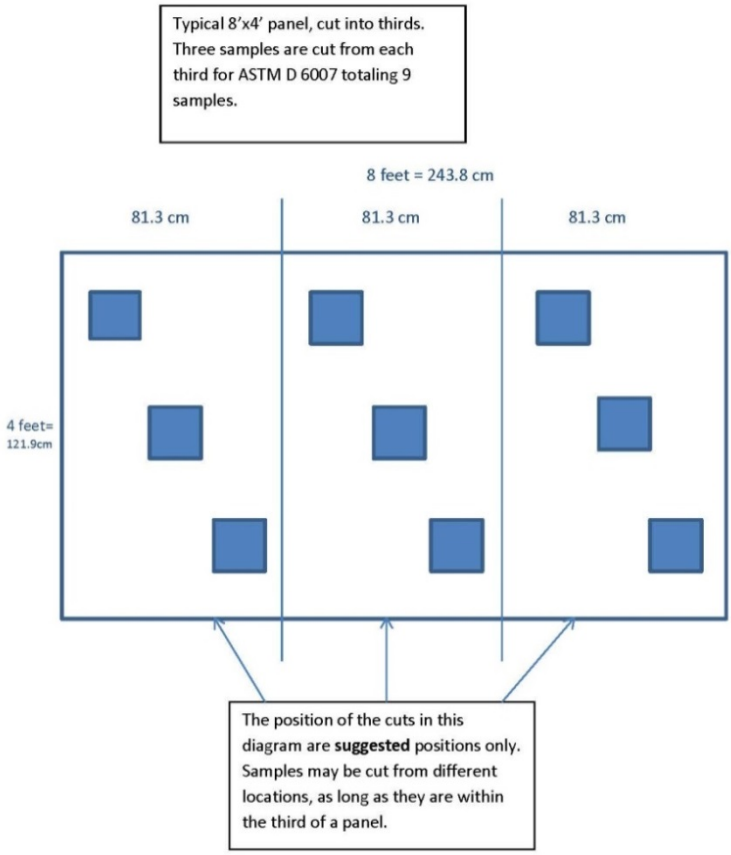
Laboratories were asked to condition and emission test their samples, and to report their results on a data submission sheet by a suggested schedule in an effort to avoid potential decay in formaldehyde emissions prior to testing (for additional detail, please refer to Appendix 2).

ASTM E 1333 – Large Chamber Testing: For ASTM E 1333 testing, each laboratory was directed to prepare samples so that they met the required loading ratio for the chamber, conduct the test according to the ASTM E 1333 requirements, and provide information about testing such as dates, temperature, relative humidity, and any event that might have affected the results of the study. Although section 10.2 of ASTM E 1333 requires that at least two simultaneous air samples be taken, for the purposes of this interlaboratory comparison, all laboratories were required to collect four air samples from their chamber. These four air samples could be collected simultaneously or as sequential sample pairs. The data were entered as results 1a, 1b, 2a, 2b on the data submission sheet provided to each laboratory. The results for MDF and PB were submitted on separate data sheets.

ASTM D 6007 – Small Chamber Testing: For ASTM D 6007 testing, the sampling methodology used is described in section 93120.9(a)(2)(A) of the ATCM. For small chamber testing, all laboratories were directed to condition samples according to the period used to establish equivalence to the primary method (ASTM E 1333). As shown in Figure 1, ASTM D 6007 requires that nine samples are cut from evenly distributed portions across the panel. The nine samples are required to be tested in groups of three samples resulting in three emission test results. For sampling (section 10.2 of ASTM D 6007), duplicate air samples for each of the three chamber tests were

collected and the results were entered as 1a,1b, 2a, 2b, 3a, 3b on the data submission sheet (e.g., 1a is the first result of test #1, 1b is the duplicate result). Each laboratory was directed to conduct the test according to the ASTM D 6007 requirements, and provide information about testing such as dates, temperature, relative humidity, and conditioning time. The results for MDF and PB were submitted on separate data sheets.

Figure 1. Sample Preparation for ASTM D 6007



V. Statistical Evaluation of the Results

Assigned Value

The assigned value (robust mean) for the concentration of formaldehyde associated with the test materials was calculated for each test sample from the mean values of the test results reported using the application of robust statistics according to Section 3.3.2: *Recommended scheme for obtaining a consensus value and its uncertainty, Recommendation 1* in the IUPAC Technical Report.

Performance Indicator and Target Standard Deviation

The performance of an individual laboratory is expressed by a z-score, which is calculated according to equation 1:

$$z_i = \frac{x_i - \bar{X}}{\sigma} \qquad \text{Equation 1}$$

where z_i is the z-score of laboratory "i" for the respective sample; "x_i" the reported formaldehyde content of laboratory "i" for the MDF and PB test sample, expressed as the mean of 4 or 6 determinations (depending on large or small chamber testing); $\bar{X}$ is the assigned value for the respective sample, and "σ" the target standard deviation. For the purposes of this ILC, the target standard deviation (acceptable) was based on the standard deviation of all the participants' results for both MDF and PB product samples.

For the MDF and PB, test sample z-scores were calculated. The laboratory performance was evaluated according to the following limits:

$z \leq \pm 2.0$	Satisfactory
$z > \pm 2.0$	Follow-up Evaluation Required

Additional Criteria for Follow-up Evaluation

Laboratories whose z-score was greater than ±2.0 but their test results were within 0.03 parts per million (ppm) of the robust mean were not considered for follow-up evaluation since the 0.03 ppm variation is within the test method variability. ARB staff also evaluated laboratories that used both the ASTM E 1333 and ASTM D 6007 methods. For these laboratories, follow-up was warranted when the test results for the same test material tested according to ASTM E 1333 and ASTM D 6007 differed by more than 0.03 ppm. This threshold was based on the results from the 2009, 2011/12 and 2013 ILCs. Laboratories were considered for follow-up evaluation if their emission test results showed more than 0.02 ppm difference between their repeat measurements, as well as laboratories with identical measurements. The basis for the 0.02 ppm value was also derived from the previous interlaboratory comparison studies (CARB 2009, 2011/12 and 2013 ILC).

VI. Data Analysis for MDF

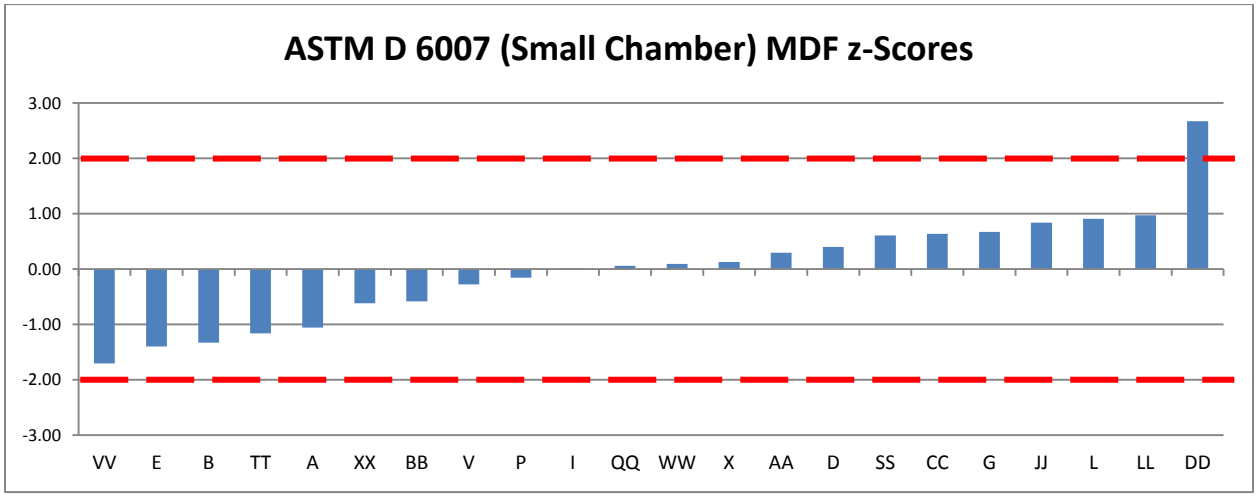
The raw data for each laboratory was entered into an Excel spreadsheet to facilitate the calculations and analysis (MS Office Excel - 2010). Results for laboratories reporting three or more significant figures were rounded to two. Table 1 provides a summary of the assigned consensus value, standard deviation (based on the participants' results) and other relevant data pertaining to the MDF test results. In Appendix 3, a summary of the reported results and z-scores for each laboratory are provided in Table A3-1. Figure 2 below, provides a graphic summary of the z-score results for the ASTM D 6007 and ASTM E 1333 results, respectively. While one laboratory had a z-score >2.0 for both ASTM D 6007 and ASTM E 1333 methods, this laboratory's emission test results were less than 0.02 ppm from the consensus value of 0.04 ppm (robust mean) for both chambers. Thus, follow-up evaluation for this laboratory was not justified.

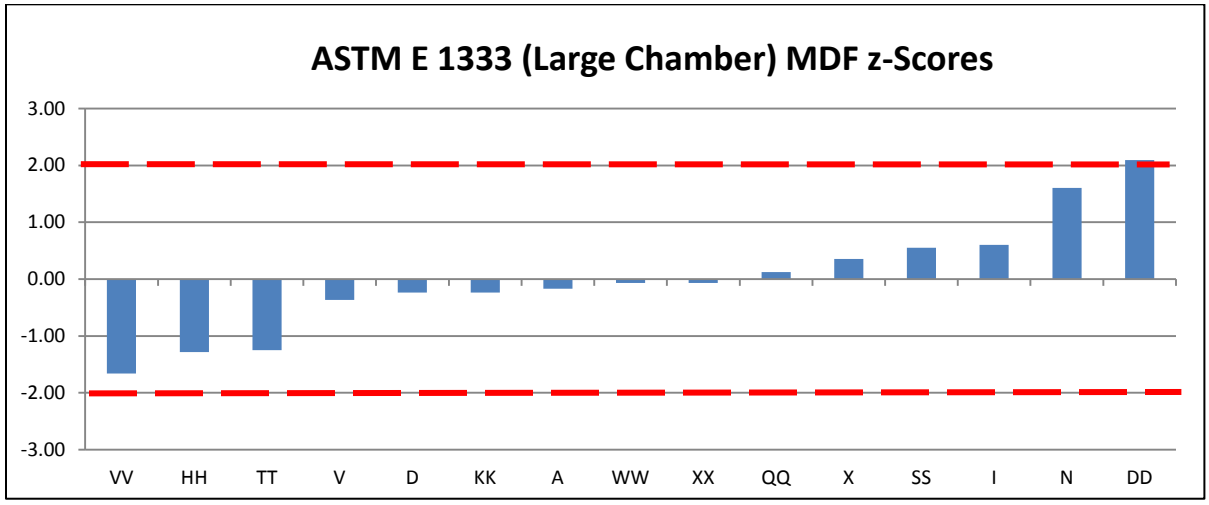
Table 1: ASTM E 1333 and ASTM D 6007 Summary Statistics for MDF

Parameter	ASTM D 6007	ASTM E1333
Number of Laboratories	22**	15*
Assigned Consensus Value ($\bar{X}$)	0.04 ppm	0.04 ppm
Standard Deviation	±0.005	±0.01
Range in Test results	0.03 to 0.05 ppm	0.03 to 0.05 ppm
Number of Laboratories with z > ±2.0 for One or Both Methods	1	

Note:
12 TPCs/laboratories conducted both ASTM E 1333 and D 6007 testing
10 TPCs/laboratories conducted ASTM D 6007 testing only
3 TPCs/laboratories conducted ASTM E 1333 only
* One laboratory provided one additional test result; ** Two laboratories provided one additional test results each

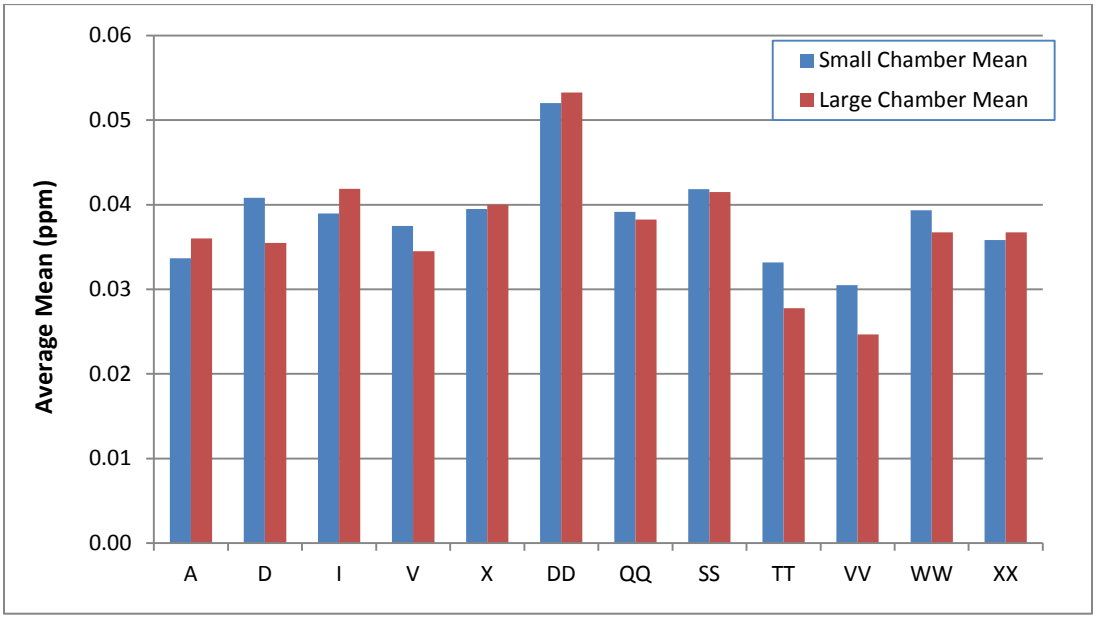
Figure 2: MDF z-Scores





In addition to evaluating z-scores for the MDF test results, ARB staff compared the test results from laboratories that submitted both ASTM E 1333 and ASTM D 6007 method test results for MDF. As shown below in Figure 3, there were 12 laboratories that operated both the ASTM E 1333 and ASTM D 6007 test methods for the MDF sample. Of these, all laboratories test results were within 0.01 ppm, well below the acceptable maximum 0.03 ppm.

Figure 3: Comparison of ASTM E 1333 and ASTM D 6007 MDF Test Results for Individual Laboratories



VII. Data Analysis for PB

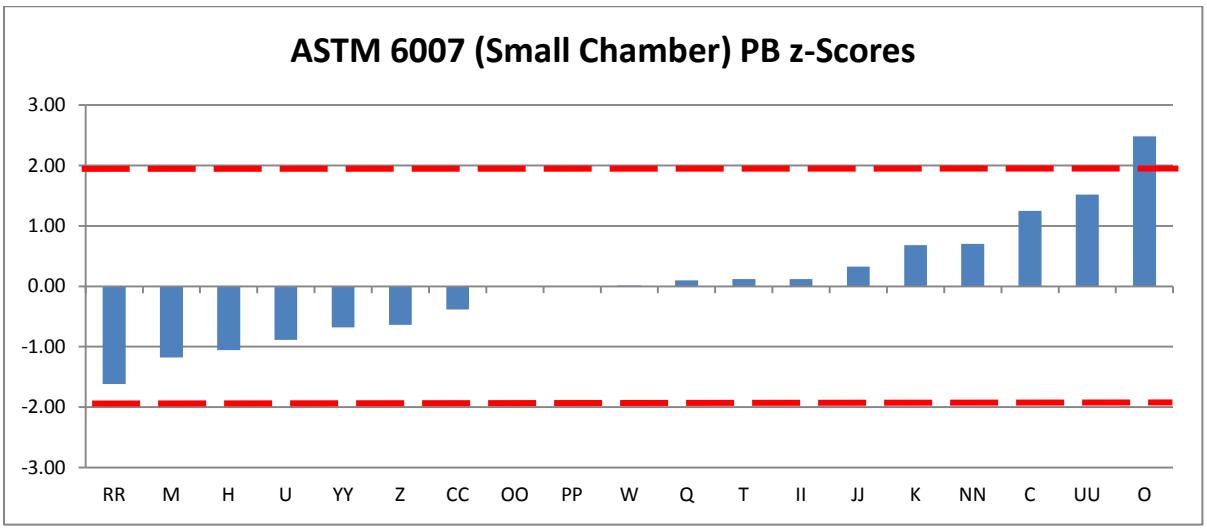
The raw data for each laboratory was entered into an Excel spreadsheet to facilitate the calculations and analysis (MS Office Excel - 2010). Results for laboratories reporting three or more significant figures were rounded to two. Table 2 provides a summary of the assigned consensus value, target standard deviation and other relevant data pertaining to the PB test results. In Appendix 3, a summary of the reported results and z-scores for each laboratory are provided in Table A3-2. Figure 4 below provides a graphic summary of the z-score results for the ASTM D 6007 and ASTM E 1333 results, respectively. While one laboratory had a z-score >2.0 for the ASTM D 6007 methods, this laboratory's emission test result was less than 0.02 ppm from the consensus value (robust mean) of 0.02 ppm for ASTM D 6007 chamber. Thus, follow-up evaluation for this laboratory was not justified.

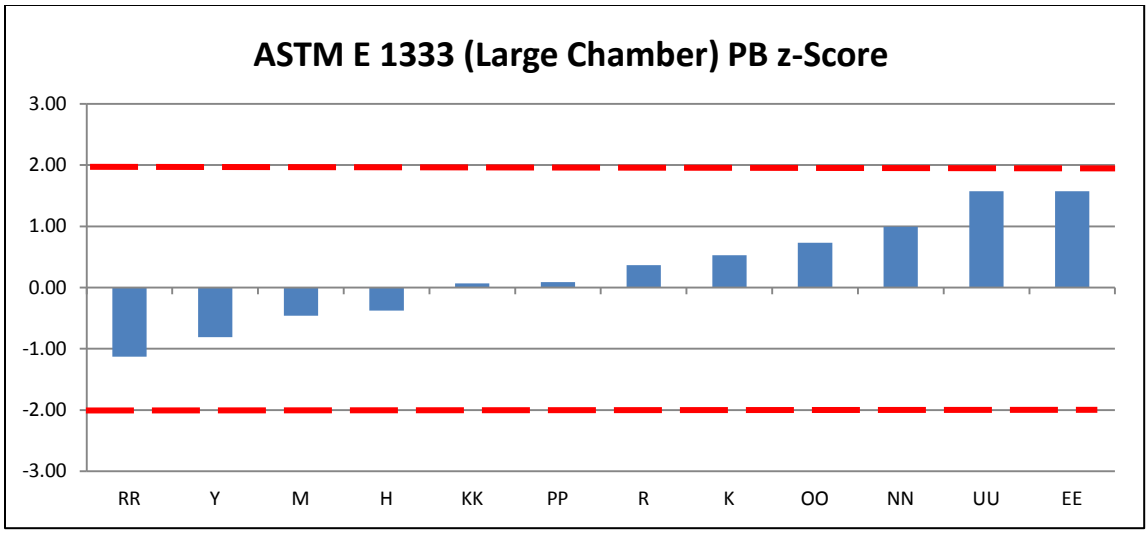
Table 2: ASTM E 1333 and ASTM D 6007 Summary Statistics for PB

Parameter	ASTM D 6007	ASTM E 1333
Number of Laboratories	19	12
Assigned Consensus Value ($\bar{X}$)	0.02 ppm	0.02 ppm
Target Standard Deviation	±0.01 ppm	±0.01 ppm
Range in Test Results	0.01 – 0.04 ppm	0.01 – 0.04 ppm
Number of Laboratories Identified for Follow-up Based on Results from One or Both Methods	1	

Note:
8 TPCs/laboratories conducted both ASTM E 1333 and D 6007 testing
11 TPCs/laboratories conducted ASTM D 6007 testing only
4 TPCs/laboratories conducted ASTM E 1333 only

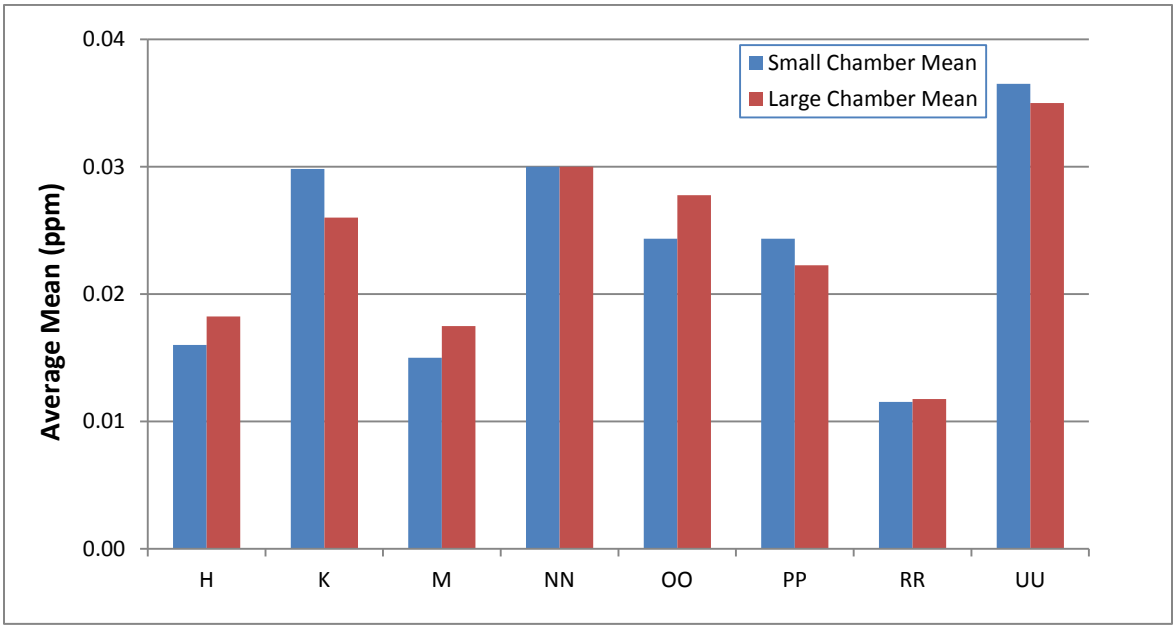
Figure 4: PB z-Scores





Similar to the analysis of MDF test results, ARB staff compared the test results from laboratories that submitted both ASTM E 1333 and ASTM D 6007 test results for PB. As shown below in Figure 5, of the eight laboratories that conducted both the ASTM E 1333 and ASTM D 6007 test methods for the PB sample, all laboratories test results were within 0.01 ppm, well below the acceptable maximum 0.03 ppm.

Figure 5: Comparison of ASTM E 1333 and ASTM D 6007 PB Test Results for Individual Laboratories



VIII. Within-laboratory Analysis

ARB staff also evaluated within-laboratory repeatability (precision) for both MDF and PB test results. Of the six data points submitted by laboratories for ASTM D 6007, paired test results were evaluated (e.g., 1a/1b, 2a/2b, 3a/3b) for repeatability. For ASTM E 1333, of the four data points submitted by laboratories, data were individually assessed as laboratories had the option of collecting four individual air samples simultaneously or sequentially collecting two sample pairs.

Laboratories were considered for follow-up evaluation if their emission test results showed more than 0.02 ppm difference between their repeat measurements, as well as laboratories with identical measurements. The basis for the 0.02 ppm value was derived from the previous interlaboratory comparison studies (CARB 2009, 2011/12 and 2013 ILC). Such occurrences may indicate within-laboratory imprecision, which may be due to rounding reported test values, an indication of insensitive measurement or resolution, or other measurement issues. For both MDF and PB products, repeat measurements were all within less than 0.02 ppm for all participating laboratories, likely due to the low emission levels for both products.

IX. Discussion

The 2014 ILC results showed minimal variability among the participating TPCs/laboratories test results using ASTM D 6007 and ASTM E 1333 method tests. ARB's MLD emission test results for the randomly selected MDF and PB products (10 panels) were all shown to be homogeneous for each product type. While one laboratory was identified as having a z-score >2.0 for MDF and one laboratory as having a z-score >2.0 for PB products, follow-up evaluation was not warranted, since the variation from the consensus mean for both laboratories were within an acceptable range (less than 0.03 ppm, which accounts for the variability of the test methods). The slightly higher z-scores for the two laboratories may have been attributed to sample heterogeneity, test method variability, differences in laboratory methods, and variations within the analytical methods used.

Other than the two cases discussed above, all laboratories had satisfactory z-scores which indicated proficiency in conducting ASTM D 6007 and/or ASTM E 1333 testing and overall improvement of precision and accuracy of their chamber testing compared to past ARB ILCs. Of the laboratories that tested the MDF and PB samples using both the ASTM E 1333 and ASTM D 6007, all participating laboratories test methods had close agreement and the test results were within 0.01 ppm. Repeatability seeks to address the variability between independent test results that are obtained within a single laboratory. All participating laboratories' repeat measurements using both ASTM D 6007 and ASTM E 1333 methods showed consistent testing of a maximum deviation of 0.02 ppm within test results.

For the 2014 ILC, ARB staff requested participants using ASTM D 6007 test chambers to provide photos of their sample preparation, conditioning and small chamber testing. The photos provided valuable insight for ARB staff to better understand individual

TPCs/labs testing processes. ARB staff made several observations concerning sample preparation and emission testing based on the photos provided. ARB staff followed up with those TPCs/labs whose photos indicated deviations from the ILC protocol and suggested corrective actions.

Appendix 1

List of 2014 ILC Participants

Table A1: List of 2014 ILC Participants

TPC/Contract Laboratory Name	Location
Composite Panel Association (CPA)	United States
Benchmark Holdings	United States
PFS Corporation	United States
Fraunhofer-Institut for Wood Research (WKI)	Germany
AsureQuality	New Zealand
PT Mutuagung Lestari (MUTU Certification)	Indonesia
Timberco (TECO)	United States
Hardwood Plywood and Veneer Association (HPVA)	United States
Entwicklungs- und Pruflabor Holztechnologie GmbH (EPH)	Germany
Holzforschung Austria (HFA)	Austria
SP Technical Research Institute	Sweden
SGS-Hong Kong	China
Asociacion de Investigacion y Desarollo en la Industria del Mueble y Afines (AIDIMA)	Spain
CATAS	Italy
MPA Eberswalde Materialprufanstalt Brandenburg GmbH (MPA)	Germany
Instytut Technologii Drewna (ITD)	Poland
SGS-CSTC Guangzhou Branch	China
Dancert Danish Technological Instutue (DTI)	Denmark
Vyzkumny a Vyvojovy Ustav Drevarsky (VVUD)	Czeck Republic
Laboratorio Prevenzione Incendi (LAPI)	Italy
NTA Incorporated	United States
SGS-Taiwan	China
SGS-CSTC Standards Technical Services Co. Shanghai	China
TUV Rheinland-Shenzhen	China
Centre Technique de L'Industrie du Bois-Technisch Centrum der Hountnijverheid (CTIB)	Belgium
Intertek-Shanghai	China
Intertek-Hong Kong	China
Intertek-Shenzhen	China
TUV Rheinland-Hong Kong	China
TUV Rheinland-Germany	Germany

Laboratorio Tecnologico per la Qualita (CosMob)	Italy
TUV Rheinland-Shanghai	China
Ośrodek Badawczo – Rozwojowy Przemysłu Płyt Drewnopochodnych sp. z.o.o.(OBRPPD)	Poland
Contract Laboratories	Location
Berkeley Analytical	United States
Nanjing Wood-based Panels Testing Center (Nanjing Forestry)	China
Advanced Testing Services (ATS)	United States
Beijing Quality Supervision & Inspection Station of Wood Furniture (BQSISWF)	China
FP Innovations	Canada
Shanghai Hongjun Science and Technology Co., Ltd. (SHST)	China
Forest Research Institute Malaysia (FRIM)	Malaysia
MUTU-China*	China
Government Laboratories	Location
ARB-Monitoring and Laboratory Division (MLD)	United States
California Department of Public Health (CDPH)	United States
Several TPCs not listed here relied on their contract laboratory(ies) to participate in the ILC * MUTU-China is a laboratory under MUTU Certification providing laboratory services in China	

Appendix 2

Notification Letter and Protocol for Interlaboratory Comparison of Composite Wood Product Third Party Certifiers

Air Resources Board

Mary D. Nichols, Chairman
1001 I Street • P.O. Box 2815
Sacramento, California 95812 • www.arb.ca.gov

Matthew Rodriquez
Secretary for
Environmental Protection

Edmund G. Brown Jr.
Governor

October 28, 2014

Subject: 2014 Air Resources Board Interlaboratory Comparison

Dear Third Party Certifiers and Contract Laboratories:

This letter is to provide you information regarding the upcoming Air Resources Board (ARB) interlaboratory comparison (2014 ILC) of third party certifiers and contract laboratories, and to provide you with the protocol for the 2014 ILC. We request your participation, pursuant to the Airborne Toxic Control Measure to Reduce Formaldehyde Emissions from Composite Wood Products ("ATCM," title 17, California Code of Regulations, sections 93120-93120.12).

The ARB staff will initiate the 2014 ILC during November 2014. The tentative schedule is included in the attached protocol.

For the purposes of the 2014 ILC, ARB staff will provide each TPCs/laboratory with one type of composite wood product: particleboard (PB) or medium density fiberboard (MDF). Based on random selection, half of the participating TPCs/laboratories will be receiving PB samples and the other half will be receiving MDF panels.

TPCs/laboratories that only operate small chambers, will be receiving one full 4'x8'panel, which will be cut into thirds (48"x32"). TPCs/laboratories that operate a large chamber will be receiving sufficient test material to accommodate their reported chamber sizes. ARB staff requests that TPCs/laboratories that operate both large and small chambers test the provided sample materials in both chambers. ARB staff requests that once the large chamber testing is concluded, TPCs/laboratories would use one full panel to obtain nine pieces to the appropriate size for their small chamber testing. TPCs/laboratories are to perform formaldehyde emission testing of composite wood products per the enclosed protocol, using either ASTM E 1333 (large chamber) or ASTM D 6007 (small chamber, deemed equivalent to the primary method) depending on which test chamber size they operate, or both chambers (if applicable).

ARB staff intends to evaluate individual laboratory formaldehyde emission testing proficiency in terms of z-scores that are based on a fitness-for-purpose criterion in accordance with the International Harmonized Protocol for the Proficiency Testing of

Analytical Chemistry Laboratories (IUPAC Technical Report, Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, 78(1), 145-196). For the purposes of the ILC, ARB staff will find third party certifiers and/or contract laboratories proficient when their z-scores are less than or equal to ±2.0. Statistical outliers will be evaluated (z-scores of more than ±2.0) and may be required to participate in follow-up testing or be subject to further examination to evaluate their testing practices and to assist them in improving their proficiency.

As a condition for participating in the 2014 ILC, ARB staff requires participating contract laboratories to release their test results to the third party certifier(s) to which they are under contract to provide testing services.

Enclosed with this letter is the 2014 ILC protocol, which provides additional details on implementation and expectations for the 2014 ILC. If you have any questions regarding the protocol, please contact Ms. Angela Csondes at (916) 445-4448 or via email at acsondes@arb.ca.gov, or Mr. Lynn Baker at (916) 324-6997 or via email at lbaker@arb.ca.gov.

Thank you in advance for your cooperation and participation.

Sincerely,

Daniel E. Donohoue

Daniel E. Donohoue, Chief
Emissions Assessment Branch
Transportation and Toxics Division

Enclosure

cc: Ms. Angela Csondes
Air Pollution Specialist
Emissions Assessment Branch

Mr. Lynn Baker
Staff Air Pollution Specialist
Emissions Assessment Branch

Protocol for Interlaboratory Comparison of Composite Wood Product Third Party Certifiers

State Of California
California Environmental Protection Agency
Air Resources Board

October, 2014

Purpose: This interlaboratory comparison will fulfill the requirement specified in Appendix 3, section (b)(1)(F) of the Airborne Toxic Control Measure to Reduce Formaldehyde Emissions from Composite Wood Products ("ATCM," title 17 California Code of Regulations, sections 93120-93120.12, see http://www.arb.ca.gov/regact/2007/compwood07/fro-final.pdf). Data from this interlaboratory comparison will be used to assess Third Party Certifier (TPC) emissions testing capabilities in the Air Resources Board's (ARB) on-going administration of the TPC program.

Additional Considerations - Please be sure to note the following:

- Do not discard test materials. Immediately after testing, please wrap your materials similarly as to how you received them. Please hold onto test material(s) until you receive notification that the ILC is completed or further instructions are provided. ARB staff may request that you retest your materials.
- Waster sheets can be discarded (outer cover panels that protect sample materials during shipping).
- Do not store samples in the freezer.

Materials: For the purposes of this study, 4'x8' medium density fiberboard (MDF) panels and 4'x8' particleboard (PB) panels will be used as the material tested to gather emissions data for the comparison. For ease of handling and reduction of shipping costs, the 4'x8' panels will be cut into thirds, yielding pieces that measure approximately 48"x32". For this study:

- Half of the participating TPCs/laboratories will be receiving MDF panels and the other participants will be receiving PB panels.

- ARB staff requests that TPCs/laboratories that operate both large and small chambers test the provided sample materials in both chambers. TPCs/laboratories that operate both large and small chambers should test their panels in their large chamber first; then cut specimens from the same sample materials to the appropriate size to accommodate their small chamber for testing.

- TPCs/laboratories that operate large chambers only will be receiving enough panels to accommodate their large chambers (suitable for the proper loading rate for the size of their large chambers). (Note: some cutting may be needed so that the test material corresponds to the loading ratio for each large chamber.) TPCs and laboratories that only operate the small chamber will receive one single panel, which totals three 48”x32” pieces.

Shipping: The panel pieces will be stacked and wrapped in 6-mil poly sheeting. Waster sheets, used to protect the test material, will be placed on the exterior of the poly wrapped bundle and subsequently bound together (the waster sheets may be discarded). In order to minimize the time of exposure to large variations in temperature, each sample will be shipped via FedEx express carrier. Immediately upon receipt of the samples, the laboratory should store the wrapped boards in a room with a controlled environment.

Sample labeling: Prior to cutting, each 48”x32” piece will be labeled by ARB staff with an alphanumeric code so that pieces from a common panel are easily identified. For example, the three pieces from MDF panel #1 will be labeled M-1a, M-1b, and M-1c respectively. PB would be labeled similarly as P-1a, P-1b and P-1c.

Sample Testing: We would like all laboratories to initiate their large and small chamber conditioning around the same time. This would be early November 2014 (see Table 1), and testing would commence the following week. Each laboratory should report the date of conditioning on the data submission sheet that will be provided electronically as an Excel worksheet. A hard copy example of the data submission sheet is attached to this protocol. If possible, small chamber testing should also be performed during the period of early to mid-November. We understand that samples will arrive at their destinations at different times due to international shipping and due to shipping delays (e.g., packages may be held for a period of time in customs). If it is not possible to follow this schedule, we ask that conditioning commence not more than two weeks from receipt of the samples.

Table 1: 2014 ARB Interlaboratory Comparison Study Timeline

Task	Responsible Party	Timeline*
Test Sample Preparation	ARB Staff	Early November 2014
Test Sample Shipment to Third Party Certifiers and Laboratories	ARB Staff	Early November 2014
Sample Conditioning	Third Party Certifiers/Contract Laboratories	Mid-November 2014
Emission Testing		Mid to late-November 2014
Report Results to ARB		Early December 2014
Data Analysis	ARB Staff	December 2014
Release of Results	ARB Staff	January/February 2015

*The above dates may shift one to two weeks and you will be notified via email of any changes.
Methodology: For all testing, laboratories must adhere to the following:

1. **Primary Method** - Each laboratory using the primary method is responsible for preparing the test material so that it meets the required loading ratio for the large chamber used as specified in ASTM E 1333. For primary method testing, laboratories must document the requirements of ASTM E 1333, and provide the required information about testing such as: dates, temperature, relative humidity, background formaldehyde concentration, and any significant event that might affect the results of the study. Section 10.2 of the ASTM method requires that at least two simultaneous air samples be taken. For the purposes of the interlaboratory comparison, laboratories should collect **four air samples** from their chamber. These can be collected simultaneously, or sequentially (i.e., two samples collected during a one-hour period, followed by two additional samples collected during a subsequent one-hour period). Data should be entered as results 1a, 1b, 2a, 2b on the electronic data submission form. Please supply all of the information requested on the electronic data submission form.

2. **Secondary Method** - Each laboratory is responsible for preparing the samples to the appropriate dimensions to be consistent with the flow to area (Q/A) ratio for the small chamber used, as specified in ASTM D 6007. For secondary method testing, the sampling methodology described in section 93120.9(a)(2)(A) of the ATCM shall be used. Additionally, samples must be conditioned according to the period used to establish equivalence to the primary method. To reiterate, the secondary method requires that nine samples be taken from evenly distributed portions across the panel. The nine samples are to be tested in groups of three samples, which will result in three emission test results (please see Figure 1 at the end of the protocol). For sampling (section 10.2 of ASTM D 6007), laboratories should collect **duplicate air samples** for each of the three chamber tests. These can be collected simultaneously, or sequentially (i.e., samples collected during consecutive 30-minute sampling periods) and should be entered as results 1a,1b, 2a, 2b, 3a, 3b on the data submission sheet (Attachment). Each lab must document the requirements of ASTM D 6007, and provide information about testing such as: dates, temperature, relative humidity, background formaldehyde concentrations, conditioning time, and any significant event that might affect the results of the study. Please supply all of the information requested on the electronic data submission form.

Immediately after testing, each lab shall wrap the chamber samples in plastic and store them in an environmentally controlled room until the data are analyzed and the interlaboratory comparison is concluded. In some instances, it may be necessary to request that a laboratory re-test or ship the samples to another testing location.

Results: We ask that your test results be submitted to Angela Csondes at acsondes@arb.ca.gov no later than two weeks from the conclusion of testing. Please report the primary and/or secondary test report information requested on the electronic

data submission form and report the following emissions results on the Test Data section of the form:

1. Analytical method standard curve.
2. Primary method results.
3. Secondary method results.
    a. Values for individual chamber tests (i.e. duplicate results for each of the three samples).

Upon receipt of the data from all of the participating laboratories, ARB will summarize the results. All laboratories will be assigned an anonymous identifier known only to ARB and the laboratory. ARB will release the results so that each laboratory can see how they compared to other participants, without disclosing the names of the participants.

For the purposes of the ILC, ARB staff will find third party certifiers and/or contract laboratories proficient when their z-scores are less than or equal to ±2.0. Statistical outliers will be evaluated (z-scores of more than ±2.0) and may be required to participate in follow-up testing or be subject to further examination to evaluate their testing practices and to assist them in improving proficiency.

Contact Information: For any questions about this study, please contact Angela Csondes (916-445-4448) or at: acsondes@arb.ca.gov, or Lynn Baker (916-324-6997) or at: lbaker@arb.ca.gov.

Participating Laboratories:
Air Resources Board – Monitoring and Laboratory Division
All ARB approved Third Party Certifiers
All ARB approved Contract Laboratories

Attachments

1. Interlaboratory Comparison Data Submission Sheet
   (will be provided to TPCs and contract laboratories electronically as well)
2. Figure 1 - Sample Preparation for Secondary Method Testing

Data Submission Sheet

Laboratory Information

TPC#

Laboratory /TPC Name	
Date	
Address	
Phone	
Chemist/Contact	
Dates of testing	
Comments:	

Secondary Method Test Report

Configuration: **Standard face and back**

Test #: **1**

Sample Information		
Date sample received		
Type of comp wood		
Date of manufacture	N/A	
Sample shipped/stored		
Comments:		

Chamber Information		Units
Test chamber volume (m^3)		m^3
Chamber Q/A ratio		m/h
# specimens in chamber		
# exposed sample surfaces		
Analytical method used		

Conditioning of Samples		Units
Conditioning background HCHO		ppm
Conditioning time		hours
Temperature range		^oC
Humidity range		%
Inlet air flow		m^3/h
Sample dimensions		mmxmm
Total exposed area		m^2
Testing of Samples		**Units**
Testing background HCHO		ppm
Temperature range		^oC
Relative humidity range		%
Inlet air flow		m^3/h

Secondary Method Test Report

Configuration: **Standard face and back**

Test #: **2**

Sample Information		
Date sample received		
Type of comp wood		
Date of manufacture	N/A	
Sample shipped/stored		
Comments:		

Chamber Information		Units
Test chamber volume (m^3)		m^3
Chamber Q/A ratio		m/h
# specimens in chamber		
# exposed sample surfaces		
Analytical method used		

Conditioning of Samples		Units
Conditioning background HCHO		ppm
Conditioning time		hours
Temperature range		^oC
Humidity range		%
Inlet air flow		m^3/h
Sample dimensions		mmxmm
Total exposed area		m^2
Testing of Samples		**Units**
Testing background HCHO		ppm
Temperature range		^oC
Relative humidity range		%
Inlet air flow		m^3/h

Secondary Method Test Report

Configuration: **Standard face and back**

Test #: **3**

Sample Information		
Date sample received		
Type of comp wood		
Date of manufacture	N/A	
Sample shipped/stored		
Comments:		

Chamber Information		Units
Test chamber volume (m^3)		m^3
Chamber Q/A ratio		m/h
# specimens in chamber		
# exposed sample surfaces		
Analytical method used		

Conditioning of Samples		Units
Conditioning background HCHO		ppm
Conditioning time		hours
Temperature range		^oC
Humidity range		%
Inlet air flow		m^3/h
Sample dimensions		mmxmm
Total exposed area		m^2
Testing of Samples		**Units**
Testing background HCHO		ppm
Temperature range		^oC
Relative humidity range		%
Inlet air flow		m^3/h

Primary Method Test Report

Standard face and back

Sample Information	
Date sample received	
Type of comp wood	
Date of manufacture	N/A
Sample shipped/stored	
Comments:	

Chamber Information		Units
Test chamber volume		m^3
Chamber loading ratio		m^2/m^3
# specimens in chamber		
# exposed sample surfaces		
Analytical method used		

Conditioning of Samples		Units
Conditioning background HCHO		ppm
Conditioning time		hours
Temperature range		^oC
Humidity range		%
Inlet air flow		m^3/h
Sample dimensions		mmxmm
Total exposed area		m^2

Testing of Samples		Units
Testing background HCHO		ppm
Temperature range		^oC
Relative Humidity range		%
Average air change rate		AC/h
Air sampling rate and Time		L/min;min
Inlet air flow		m^3/h

Test Data

Standard Curve

	Conc (ug/ml)	Area (or Abs)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
Slope		
Int		
R^2		

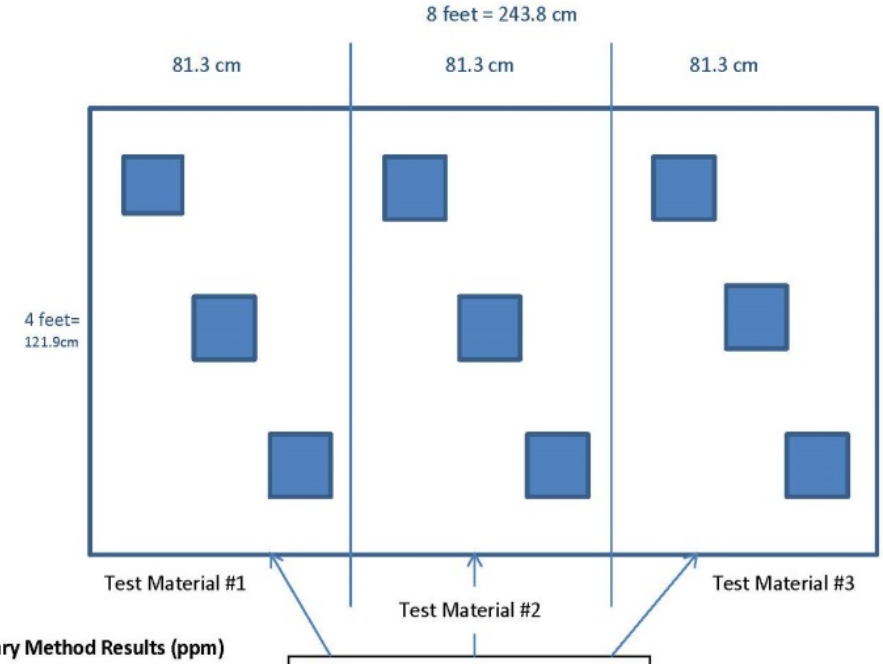
Test #	Primary Method Results (ppm)
1a	
1b	
2a	
2b	
Mean	
SE	

	Secondary Method Results (ppm)
1a	
1b	
2a	
2b	
3a	
3b	
Mean	
SE	

Are Results Background Subtracted?

Sample Preparation for Secondary Method Testing (ASTM D 6007)

Typical 8'x4' panel, cut into thirds. Three samples are cut from each third for ASTM D 6007 totaling 9 specimens. Each set of three samples are to be tested together in the small chamber to provide one test result.



Secondary Method Results (ppm)

Test #1 1a
1b
Test #2 2a
2b
Test #3 3a
3b

Duplicate test results

The position of the cuts in this diagram are **suggested** positions only. Samples may be cut from different locations, as long as they are within the third of a panel.

Appendix 3

Reported Results

Table A3-1: Reported Results and z-Scores for MDF*

	Small Chamber (ASTM D 6007)		Large Chamber (ASTM E 1333)	
Lab ID	**Reported Result (ppm)**	**z-Score**	**Reported Result (ppm)**	**z-Score**
A	0.03	-1.06	0.04	-0.17
B	0.03	-1.33		
C				
D	0.04	0.40	0.04	-0.23
E	0.03	-1.40		
G	0.04	0.67		
H				
I	0.04	0.02	0.04	0.60
K				
L	0.04	0.91		
M				
N			0.05	1.60
O				
P	0.04	-0.16		
Q				
R				
T				
U				
V	0.04	-0.28	0.03	-0.37
W				
X	0.04	0.13	0.04	0.36
Y				
Z				
AA	0.04	0.30		
BB	0.04	-0.58		
CC	0.04	0.64		
DD	0.05	2.67	0.05	2.09
EE				
HH			0.03	-1.28
II				
JJ	0.04	0.84		
KK			0.04	-0.23
LL	0.04	0.97		
NN				
OO				
PP				
QQ	0.04	0.06	0.04	0.13
RR				
SS	0.04	0.60	0.04	0.55
TT	0.03	-1.16	0.03	-1.25
UU				
VV	0.03	-1.70	0.02	-1.66
WW	0.04	0.09	0.04	-0.07
XX	0.04	-0.62	0.04	-0.07
YY				

*z-score differences can be attributed from rounding from 3 significant figures to 2 significant figures.

Table A3-2: Reported Results and z-Scores for PB*

	Small Chamber (ASTM D 6007)		Large Chamber (ASTM E 1333)	
Lab ID	**Reported Result (ppm)**	**z-Score**	**Reported Result (ppm)**	**z-Score**
A				
B				
C	0.03	1.25		
D				
E				
G				
H	0.02	-1.05	0.02	-0.37
I				
K	0.03	0.68	0.03	0.53
L				
M	0.02	-1.18	0.02	-0.46
N				
O	0.04	2.48		
P				
Q	0.03	0.10		
R			0.02	0.36
T	0.03	0.12		
U	0.02	-0.89		
V				
W	0.02	0.01		
X				
Y			0.01	-0.81
Z	0.02	-0.64		
AA				
BB				
CC	0.02	-0.38		
DD				
EE			0.04	1.58
HH				
II	0.03	0.12		
JJ	0.03	0.33		
KK			0.02	0.07
LL				
NN	0.03	0.70	0.03	0.99
OO	0.02	-0.01	0.03	0.73
PP	0.02	-0.01	0.02	0.09
QQ				
RR	0.01	-1.62	0.01	-1.13
SS				
TT				
UU	0.04	1.52	0.04	1.58
VV				
WW				
XX				
YY	0.02	-0.68		

*z-score differences can be attributed from rounding from 3 significant figures to 2 significant figures.