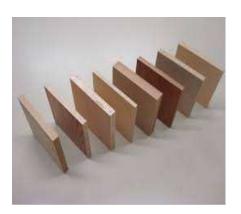
# State of California California Environmental Protection Agency Air Resources Board

# Results of Interlaboratory Comparison of Composite Wood Products Formaldehyde Emissions Testing by Third Party Certifiers & Contract Laboratories 2013







October 2013

### **Table of Contents**

Cont	<u>ents</u>		<u>Page</u>
Sum	mary		1
l.	Introduction	on	3
II.	Objectives	;	3
III.	Participant	ts	4
IV.	Study Desi	ign	4
٧.	Statistical	Evaluation of the Results	7
VI.	Data Analy	sis for tMDF	8
VII.	Data Analy	sis for PB	10
VIII.	Follow-up	Evaluations	12
IX.	Discussion	n	12
Арре	endices		
A	ppendix 1	List of 2013 ILC Participants	
A	ppendix 2	Notification Letter and Protocol for Interlaborator Comparison of Composite Wood Products Third Certifiers	•
Α	ppendix 3	Reported Results	

#### 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 spring of 2013, California Air Resources Board (ARB) staff conducted an interlaboratory comparison herein referred to as the 2013 ILC.

The primary objectives for the 2013 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) or ASTM D 6007 (small chamber) based on z-scores;<sup>1</sup>
- Assess the variability between ASTM E 1333 and ASTM D 6007 formaldehyde test method results for laboratories that conduct both methods;
- Identify measurement issues and potential sources of error within individual laboratories; and
- Suggest corrective actions to improve future performance.

There were 44 participants in the 2013 ILC representing TPCs, contract laboratories, and government organizations. Two types of unfinished composite wood products were selected as the test materials for the 2013 ILC: thin medium density fiberboard (tMDF) and shop-grade 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 (acceptable standard deviation) for proficiency assessment was based on a fitness-for-purpose criterion for the PB product. Due to the very low formaldehyde emissions associated with the tMDF product, the target standard deviation was based on the standard deviation of the participants' results. For each material, the laboratory proficiency was determined based on the calculated z-scores.

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 PB and tMDF materials. 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).

\_

<sup>&</sup>lt;sup>1</sup> Individual laboratory proficiency for PB 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 tMDF, laboratory proficiency was based on z-scores calculated using the standard deviation of the participants' results.

Based on the criteria outlined above, 34 laboratories had satisfactory z-scores and 10 laboratories were identified for follow-up evaluations. Follow-up evaluations consist of conducting an additional round of testing of materials obtained from one of the TPC/laboratory's certified mills or from a distributor, in the event a given TPCs/laboratory did not have mills certified under them. In either case, the test materials are similar to the samples selected for the initial interlaboratory comparison. For the follow-up evaluation, the laboratory is to test the material and submit a duplicate sample to ARB staff for testing by ARB's Monitoring and Laboratory Division (MLD). The follow-up evaluations are currently underway. The outcome of the follow-up evaluation will be provided individually to the laboratories later this year.

#### 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. The first ILC was conducted in 2009, shortly after the ATCM went into effect and the second ILC took place in the 2011-2012 timeframe. In the spring of 2013, California Air Resources Board (ARB) staff initiated the 2013 ILC. For the 2013 ILC, two types of composite wood products were used as test materials: tMDF and PB. In the following sections, ARB staff describes: objectives for the 2013 ILC, participants, program design and testing protocols, approach for using the results to evaluate laboratory proficiency, data analysis, and results for the 2013 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 2013 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) 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 on a fitness-for-purpose criterion in accordance with the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories (IUPAC Technical Report<sup>2</sup>) or 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.
- Identify measurement issues and potential sources of error within individual laboratories.
- Suggest corrective actions to improve future performance.

<sup>&</sup>lt;sup>2</sup> 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 44 participants in the 2013 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. Nine laboratories were subcontractors that provide analytical testing services for their respective TPCs. The remaining 33 laboratories were comprised of third party certifiers. It should be noted that although the participant's names have been provided in Appendix 1, all information regarding z-scores, test results, follow-up evaluations and the like 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 PB products, 11 of the participants operated both large and small chambers and reported results using both ASTM E 1333 and ASTM D 6007, 26 reported results only using ASTM D 6007 (small chambers), and seven only used ASTM E 1333 (large chambers). For tMDF products, 10 of the participants operated both large and small chambers, 26 reported results only using small chambers, and eight only used large chambers. It should be noted that one laboratory had problems with their test chambers and their test results were not included in the data analysis. This laboratory was required to participate in a follow-up evaluation.

#### IV. Study Design

#### <u>Timeframe</u>

Notifications regarding the ILC and the Protocol for the ILC (included in Appendix 2) were sent to participants on April 17, 2013. The test materials were mailed to each participant during the week of April 22-25, 2013. The test results from the participants were reported back to ARB staff between early-May to late-August, 2013. The delay in data reporting for several laboratories/TPCs was the result of customs/border protection agencies delays and damage to shipment. ARB staff provided the laboratories with their results via email during August. Follow-up evaluations began in September 2013.

#### **Test Materials**

Two types of test materials where used for the interlaboratory comparison: 4'x8' 6.4 mm (0.25") tMDF panels and 4'x8' 9.5 mm (0.375") PB panels. The tMDF panels were marked as meeting the ATCM's no-added formaldehyde (NAF) exemption and the PB the Phase 2 emission standard. Bundles of test material were selected from the same batch for both tMDF and PB products to minimize sample variability. Several panels were also randomly selected and emission tested by ARB's MLD to confirm homogeneity. For ease of handling and reduction of shipping costs, each 4'x8' panel was cut into thirds by ARB staff, yielding three pieces that measured approximately 48"x32." The majority of laboratories that operate both large and small chambers

received three full tMDF and PB panels, which amounted to nine 48"x32" pieces for each product type. Several laboratories that operate larger size chambers received up to four or five panels. Laboratories operating only a small chamber typically received a single panel, amounting to three 48"x32" pieces. 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 entire package was also wrapped in stretch wrap and shipped via FedEx ground (domestic) and FedEx International Economy (overseas).

Due to budget constraints, ARB staff did not provide all laboratories that operate both a large chamber and a small chamber with materials for testing in their large chambers. Only a selected number of laboratories that operate both a large and small chamber were required to test using both chambers. These laboratories received sufficient test materials to test in both their small and large chambers and were selected based on the volume of composite wood product certification services that they provide. Laboratories that operate both small and large chambers that were not selected to test in both chambers were provided one panel of each test material for testing in their small chamber. Laboratories that only operate a large or small chamber received test material to accommodate their particular chamber size.

#### 2013 ILC Testing Protocol

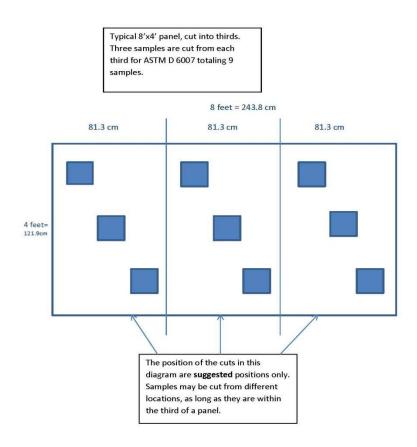
Laboratories were asked to condition and emission test their samples, and to report their results on a data submission sheet by a suggested schedule in an effort to avoid potential decay in formaldehyde emissions prior to testing (for additional detail, please refer to Appendix 2).

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 tMDF and PB were submitted on separate data sheets.

<u>ASTM D 6007</u> – 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 tMDF 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:

$$z_i = \frac{x_i - \overline{X}}{\sigma}$$
 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 tMDF 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 " $\sigma$ " the target standard deviation. For the purposes of this ILC, the target standard deviation (acceptable) was based on a fixed target of ±20% for PB product sample. This target standard deviation is a measure of the interlaboratory variation that, in the judgment of ARB staff, could be expected from the participants given the concentration of formaldehyde in the test samples. It is important to note that this target standard deviation ( $\sigma$ ) used to calculate z-scores was selected by ARB staff and is not the standard deviation of the participants' results. However, due to the extremely low emissions for the tMDF sample materials, the target standard deviation for tMDF was based on the standard deviation of all the participants' results.

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

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

#### Additional Criteria for Follow-up Evaluation

In addition to following up with laboratories having z scores greater than ±2.0, 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 13333 and ASTM D 6007 differed by more than 0.03 parts per million. This threshold was based on the results from the 2009 and 2011/12 ILC. 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 study (CARB 2009 ILC).

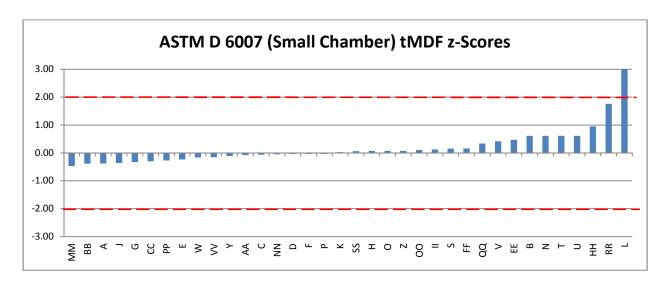
#### VI. Data Analysis for tMDF

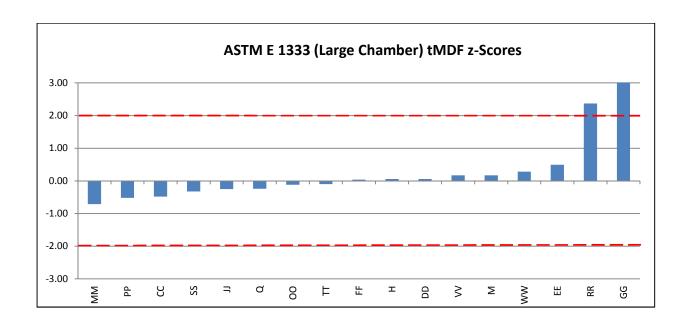
The raw data for each laboratory was entered into an Excel spreadsheet to facilitate the calculations and analysis (MS Office Excel - 2010). Results for laboratories reporting three or more significant figures were rounded to two. Table 1 provides a summary of the assigned consensus value, standard deviation (based on the participants' results) and other relevant data pertaining to the tMDF 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 E 1333 and ASTM D 6007 results, respectively. As will be discussed later, ARB staff conducted follow-up evaluations with 3 laboratories that had z-scores > ±2.0 for ASTM E 1333 and/or the ASTM D 6007 method to investigate potential areas for improvement.

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

Parameter	ASTM E 1333	ASTM D 6007
Number of Laboratories	7	26
Assigned Consensus Value (X)	0.01 ppm	0.01 ppm
Standard Deviation	±0.02	±0.02
Range in Test results	0.00 to 0.05 ppm	0.00 to 0.11 ppm
Number of Laboratories with		
z > ±2.0 for One or Both		3
Methods		

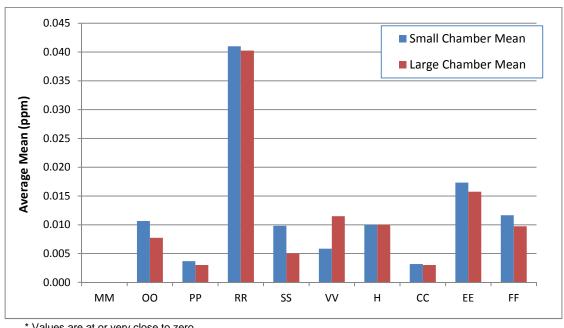
Figure 2: tMDF z-Scores





In addition to evaluating z-scores for the tMDF test results, ARB staff compared the test results from laboratories that submitted both ASTM E 1333 and ASTM D 6007 test results for tMDF. As shown below in Figure 3, there were 10 laboratories that operated both the ASTM E 1333 and ASTM D 6007 test methods for the tMDF sample. Of these, all laboratories test results were within 0.03 ppm.

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



<sup>\*</sup> Values are at or very close to zero

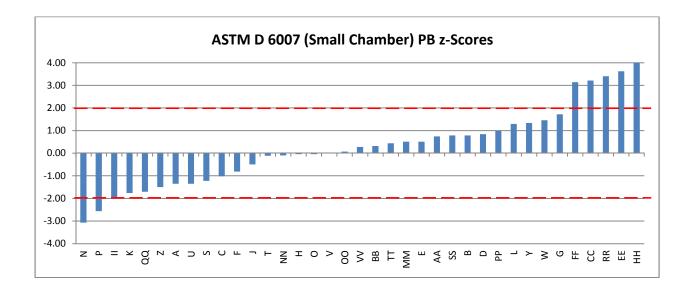
#### VII. Data Analysis for PB

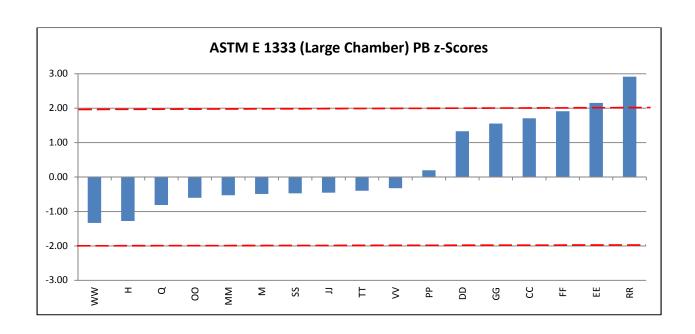
The raw data for each laboratory was entered into an Excel spreadsheet to facilitate the calculations and analysis (MS Office Excel - 2010). Results for laboratories reporting three or more significant figures were rounded to two. Table 2 provides a summary of the assigned consensus value, target standard deviation (based on a fit for purpose criterion) 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 E 1333 and ASTM D 6007 results, respectively. As will be discussed later, ARB staff conducted follow-up evaluations with 7 laboratories that had z-scores > ±2.0 for ASTM E 1333 and/or the ASTM D 6007 method to investigate potential areas for improvement. Please note that in some cases, the laboratory was also subject to follow-up evaluations based on their tMDF test results.

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

Parameter	ASTM E 1333	ASTM D 6007
Number of Laboratories	6	26
Assigned Consensus Value	0.07 ppm	0.06 ppm
Target Standard Deviation	±0.01 ppm	±0.01 ppm
Range in Test Results	0.05 – 0.11 ppm	0.02 – 0.11 ppm
Number of Laboratories Identified for Follow-up		
Based on Results from One or Both Methods	7	7

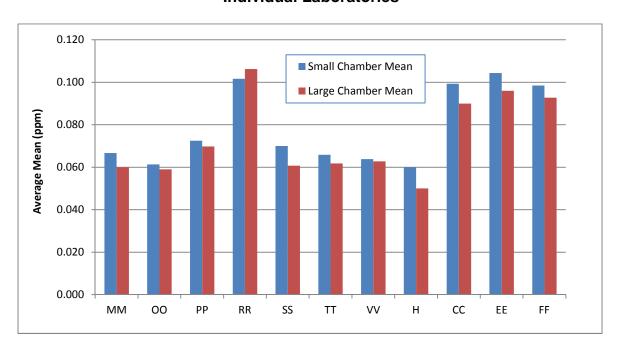
Figure 4: PB z-Scores





Similar to the analysis of tMDF 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 11 laboratories that conducted both the ASTM E 1333 and ASTM D 6007 test methods for the PB sample, all laboratories test results were within 0.03 ppm.

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



While not a primary objective for this ILC, ARB staff also evaluated within-laboratory repeatability (precision) for both tMDF 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 study (CARB 2009 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 tMDF products, repeat measurements were within 0.01 ppm for all participating laboratories, likely due to the low emission levels of this product. For PB product, all laboratories' repeat measurements were within 0.02 ppm. Several laboratories reported identical emission test results for tMDF and PB products using ASTM D 6007 and ASTM E 1333.

#### VIII. Follow-up Evaluations

ARB staff is currently conducting follow-up evaluations with ten laboratories (nine outlier laboratories and one laboratory required to retest). As described earlier, follow-up evaluations were conducted if a laboratory's tMDF and/or PB z-score was > ±2.0 or if there was more than a 0.03 ppm difference between ASTM E 1333 and ASTM D 6007 test results for the same test material. Follow-up evaluations consist of conducting an additional round of testing of materials obtained from one of the TPC/laboratory's certified mills or from a distributor, in the event a given TPCs/laboratory does not have mills certified under them. In either case the test materials are similar to the samples selected for the initial interlaboratory comparison. For the follow-up evaluation, the laboratory is to test the material and submit a duplicate sample to ARB staff for testing by ARB's MLD. The results of the follow-up evaluation will be provided to individual outlier laboratories later this year as the follow-up testing is scheduled during the September-November, 2013 timeframe.

#### IX. Discussion

The majority of the laboratories had satisfactory z-scores which indicated proficiency in conducting ASTM E 1333 and/or ASTM D 6007 testing. Of the laboratories that tested the tMDF and PB samples using both the ASTM E 1333 and ASTM D 6007, all participating laboratories test methods had close agreement. Nine laboratories had unsatisfactory z-scores and ARB staff is in the process of conducting the follow-up evaluations with these laboratories and the one laboratory that was not able to complete

the initial round of testing due to chamber operational issues to identify factors that may have led to their initial poor performance. ARB staff will share the outcome and additional observations individually with these laboratories later this year.

# Appendix 1

**List of 2013 ILC Participants** 

**Table A1: List of 2013 ILC Participants** 

TDO/Ossetss at Labouration	Landing
TPC/Contract Laboratory Name	Location
Name	
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	United States
(HPVA) Entwicklungs- und Pruflabor Holztechnologie	Germany
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
FCBA	France
MPA Eberswalde Materialprufanstalt	C = ****
Brandenburg GmbH (MPA)	Germany Poland
Instytut Technologii Drewna (ITD) SGS-CSTC Guangzhou Branch	China
Dancert Danish Technological Instutue (DTI)	Denmark
Vyzkumny a Vyvojovy Ustav Drevarsky	Czeck Republic
(VVUD)	Ozeck Nepublic
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
Berkeley Analytical	United States
Nanjing Wood-based Panels Testing Center (Nanjing Forestry) ATS	China United States
Beijing Quality Supervision & Inspection Station of Wood Furniture (BQSISWF)	China
FP Innovations	Canada
Zhejiang Forestry Product Test Station	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 labora * MUTU-China is a laboratory under MUTU Certification p	

## Appendix 2

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



Matthew Rodriquez

Secretary for

Environmental Protection

#### **Air Resources Board**

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



April 16, 2013

#### Subject: 2013 Air Resources Board Interlaboratory Comparison

Dear Third Party Certifiers and Contract Laboratories:

This letter is to provide you information regarding an upcoming Air Resources Board's (ARB) interlaboratory comparison (2013 ILC) of third party certifiers and contract laboratories, and to provide you with the protocol for the 2013 ILC.

As a reminder, the Airborne Toxic Control Measure to Reduce Formaldehyde Emissions from Composite Wood Products ("ATCM," title 17, California Code of Regulations, sections 93120-93120.12) requires ARB-approved Third Party Certifiers and their laboratories or contract laboratories to participate in an ILC every two years. The requirement to participate is found in Appendix 3 of the ATCM "Requirements for Third Party Certifiers of Composite Wood Products." (see http://www.arb.ca.gov/regact/2007/compwood07/fro-final.pdf).

To assist in fulfilling this requirement, the ARB staff will conduct the 2013 ILC during the April-May 2013 timeframe. The tentative schedule is set forth in Table 1 below.

#### Table 1: 2013 ARB Interlaboratory Comparison Timeline

Task	Responsible Party	Timeline*
Test Sample Preparation	ARB Staff	Mid-April
Test Sample Shipment to Third Party Certifiers and Laboratories	ARB Staff	Late-April
Sample Conditioning	Third Party Certifiers/Contract	Late-April
Emission Testing	Laboratories	Early to mid-May
Report Results to ARB		Late-May
Data Analysis	ARB Staff	June
Release of Results	ARB Staff	July/August

<sup>\*</sup>The above dates may shift one to two weeks and you will be notified via email of any changes.

The energy challenge facing California is real. Every Californian needs to take immediate action to reduce energy consumption. For a list of simple ways you can reduce demand and cut your energy costs, see our website: http://www.arb.ca.gov.

California Environmental Protection Agency

Printed on Recycled Paper

Third Party Certifiers and Contract Laboratories April 16, 2013 Page 2

ARB staff will provide you with two types of composite wood products for the 2013 ILC testing: particleboard and medium density fiberboard. 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. Due to budget constraints, for the 2013 ILC ARB staff will not provide all laboratories that operate both a large chamber and a small chamber with materials for testing in their large chambers. Only a selected number of laboratories that operate both a large and small chamber will be required to test using both chambers. These laboratories will be receiving sufficient test material to test in both their small and large chambers and were selected based on the volume of composite wood product certification services that they provide. If you operate both a large and small chamber and are not selected, you will only receive one panel of each test material for testing in your small chamber. Laboratories that only operate a large or a small chamber will be receiving enough test material for their particular chamber size.

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 2013 ILC, ARB staff will require participating contract laboratories to release their test results to the third party certifier(s) to which they are under contract to provide testing services.

Third Party Certifiers and Contract Laboratories April 16, 2013 Page 3

Enclosed with this letter is the 2013 ILC protocol, which provides additional details on implementation and expectations for the 2013 ILC. If you have any questions regarding the protocol, need clarification, or would like a hardcopy mailed to you, please contact Ms. Angela Csondes at (916) 445-4448 or via email at <a href="mailto:acsondes@arb.ca.gov">acsondes@arb.ca.gov</a>, or Mr. Lynn Baker at (916) 324-6997 or via email at <a href="mailto:lbaker@arb.ca.gov">lbaker@arb.ca.gov</a>.

Thank you in advance for your cooperation.

Sincerely,

Daniel E. Donohoue, Chief Emissions Assessment Branch Stationary Source Division

Air Resources Board

Enclosure

CC:

Ms. Angela Csondes Air Pollution Specialist

**Emissions Assessment Branch** 

Mr. Lynn Baker Staff Air Pollution Specialist Emissions Assessment Branch

#### **April**, 2013

**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 <a href="http://www.arb.ca.gov/regact/2007/compwood07/fro-final.pdf">http://www.arb.ca.gov/regact/2007/compwood07/fro-final.pdf</a>). 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:

- Immediately after testing your samples, please wrap your materials similarly as
  to how you received them. Do not discard sample material(s) until you receive
  notification that the ILC is completed or further instructions are provided.
- · Waster sheets can be discarded.
- Do not store samples in the freezer.

To assist your laboratory in preparing for the 2013 ILC, we suggest you review the findings from the 2011-12 ILC that identified the operational/test procedures that were potential contributors to unfavorable test results in the 2011-12 ILC. Ensuring that your laboratory does not have these issues may help you achieve optimal test results.

**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". TPCs and laboratories that operate large chambers and TPCs/laboratories that are selected to test using both small and large chambers will receive samples to accommodate the loading rate for the size of their large chambers. TPCs and laboratories operating the small chamber only will receive two single panels (one MDF and one PB), which totals six 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 economy carrier. Immediately upon receipt of the samples, the laboratory should store the wrapped boards in a room with a controlled environment.

**Sample preparation**: 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 MDF-1a, MDF-1b, and MDF-1c respectively. PB would be labeled similarly as PB-1a, PB-1b and PB-1c. Typically, laboratories testing by the primary method should set aside nine 48"x32"

pieces per product and laboratories testing by their secondary method should set aside three 48"x32" pieces for testing in the secondary method for each product. The exact amount of sample material will vary by laboratory depending on the volume of their large chamber and size of samples used in the small chamber.

Sample Testing: We would like all laboratories to initiate their large and small chamber conditioning around the same time. This would be late-April to early-May 2013 (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 this week. 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: 2013 ARB Interlaboratory Comparison Study Timeline** 

Task	Responsible Party	Timeline*
Test Sample Preparation	ARB Staff	Mid-April
Test Sample Shipment to Third Party Certifiers and Laboratories	ARB Staff	Late-April
Sample Conditioning	Third Party Certifiers/Contract	Late-April
Emission Testing	Laboratories	Early to mid-May
Report Results to ARB		Late-May
Data Analysis	ARB Staff	June
Release of Results	ARB Staff	July/August

<sup>\*</sup>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 samples so that they meet 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. 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 <a href="mailto:acsondes@arb.ca.gov">acsondes@arb.ca.gov</a> 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 ARB and 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: <a href="mailto:acsondes@arb.ca.gov">acsondes@arb.ca.gov</a>, or Lynn Baker (916-324-6997) or at: <a href="mailto:baker@arb.ca.gov">baker@arb.ca.gov</a>.

#### **Participating Laboratories:**

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

# Appendix 3

**Reported Results** 

Table A3-1: Reported Results and z-Scores for tMDF

	Small Chamber (ASTM D 6007)		Large Chamber (ASTM E 1333)	
	Reported Result		Reported Result	31.II. <u>2.1000</u> ,
Lab ID	(ppm)	z-Score	(ppm)	z-Score
MM	0.00	-0.47	0.00	-0.71
BB	0.00	-0.39		
Α	0.00	-0.38		
J	0.00	-0.36		
G	0.00	-0.33		
CC	0.00	-0.30	0.00	-0.48
PP	0.00	-0.27	0.00	-0.52
Е	0.00	-0.24		
W	0.01	-0.16		
VV	0.01	-0.16	0.01	0.17
Υ	0.01	-0.11		
AA	0.01	-0.07		
С	0.01	-0.06		
NN	0.01	-0.05		
D	0.01	-0.04		
F	0.01	-0.04		
Р	0.01	-0.04		
K	0.01	0.03		
SS	0.01	0.06	0.01	-0.33
Н	0.01	0.07	0.01	0.06
0	0.01	0.07		
Z	0.01	0.07		
00	0.01	0.11	0.01	-0.12
П	0.01	0.13		
S	0.01	0.15		
FF	0.01	0.16	0.01	0.04
QQ	0.02	0.34		
V	0.02	0.42		
EE	0.02	0.47	0.02	0.50
В	0.02	0.62		
N	0.02	0.62		
Т	0.02	0.62		
U	0.02	0.62		
HH	0.03	0.95		
RR	0.03	1.76	0.04	2.37
L	0.04	5.50	0.04	2.01
JJ	0.11	5.50	0.01	-0.25
Q			0.01	-0.24
TT			0.01	-0.10
DD			0.01	0.06
M			0.01	0.17
WW			0.01	0.29
GG			0.05	3.12
		l .		

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

	Small Chamber (ASTM D 6007)		Large Chamber (ASTM E 1333)	
	Reported Result	Reported Result FFP		FFP
Lab ID	(ppm)	z-Score	(ppm)	z-Score
N	0.02	-3.10		
Р	0.03	-2.56		
K	0.04	-1.76		
II	0.04	-1.94		
QQ	0.04	-1.71		
Z	0.04	-1.50		
Α	0.04	-1.35		
U	0.04	-1.35		
S	0.05	-1.23		
С	0.05	-1.02		
F	0.05	-0.81		
J	0.05	-0.50		
Т	0.06	-0.11		
NN	0.06	-0.10		
Н	0.06	-0.04	0.05	-1.28
0	0.06	-0.04		
V	0.06	0.02		
00	0.06	0.07	0.06	-0.61
VV	0.06	0.28	0.06	-0.33
BB	0.06	0.32		
TT	0.07	0.44	0.06	-0.40
MM	0.07	0.52	0.06	-0.53
E	0.07	0.51		
AA	0.07	0.75		
SS	0.07	0.80	0.06	-0.47
В	0.07	0.79		
D	0.07	0.84		
PP	0.07	1.00	0.07	0.20
L	0.08	1.30		
Υ	0.08	1.34		
W	0.08	1.46		
G	0.08	1.72		
FF			0.00	1 01
	0.10	3.14	0.09	1.91
CC	0.10	3.21	0.09	1.71
RR	0.10	3.41	0.11	2.92
EE	0.10	3.62	0.10	2.15
HH	0.11	4.09	0.55	
GG			0.09	1.56
DD			0.09	1.33
M			0.06	-0.49
WW			0.05	-1.33
JJ			0.06	-0.46
Q			0.06	-0.81