

# Comparison of the Heat Release Rate from the Mass Loss Calorimeter to the Cone Calorimeter for Wood-Based Materials

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## ABSTRACT

There is a growing demand for material properties to be used as inputs in fire behavior models designed to address building fire safety. This comparative study evaluates using the mass loss calorimeter as an alternative to the cone calorimeter for obtaining heat release rates of wood-based materials. For this study, a modified mass loss calorimeter utilized an improved heat release rates method that added four thermocouples installed on a chimney wall above the conical heater in addition to the standard air thermopile at the chimney top. The cone calorimeter used the standard oxygen consumption method. The MLC results showed agreement between the mass loss calorimeter and the cone calorimeter. Nineteen commercially available wood products were compared and included untreated and fire-retardant-treated particleboards, untreated and fire-retardant-treated medium density fiberboards, untreated high density fiberboards, untreated plywoods, untreated oriented strand boards and untreated flooring and lumber. The use of the MLC could provide a low-cost approach for obtaining material properties.

## INTRODUCTION

Bench-scale tests are commonly used as the first step in obtaining combustion and flammability characteristics of materials. The cone calorimeter (ASTM E1354) and the mass loss calorimeter (ASTM E2102-11) are two standard bench-scale tests used to obtain the time to ignition, heat release rates (HRR), and mass loss rates. The aim of this study was to compare and determine the level of agreement between the two bench-scale tests, with a focus on the heat release rate. The quality and accuracy of the heat release rate measurements are examined.

The cone calorimeter was developed in the 1980s by Dr. Vytenis Babrauskas and is commonly used to determine the heat release rate using the oxygen consumption method by calculating the gas flow rate in the exhaust duct<sup>1</sup>. In the cone calorimeter test, a 100 mm by 100 mm specimen is placed on a load cell with a conical heating element 25 mm above the surface of the specimen. The surface of the specimen is then exposed to an imposed heat flux and spark ignition. The cone calorimeter test at the Forest Products Laboratory (FPL) (Figure 1) has been shown to provide fire performance assessment and fire properties that are indicative of the material fire performance in full-scale fires<sup>2, 3, 4</sup>. The cone calorimeter has been the main test method used at FPL to evaluate the relative flammability of untreated and fire-retardant-treated forest products as reflected in the data for heat release rate and ignition times.<sup>7</sup>

The mass loss calorimeter (MLC) at FPL is from Fire Testing Technology (MLC 2004 model) (Figure 2) and was installed with the optional chimney which was modified to include a thermopile on the exhaust pipe stack to compensate for radiant energy losses to the wall. The MLC uses a truncated conical electric heater that provides a constant heat flux onto a test specimen and, after piloted ignition by the spark igniter, the mass loss and heat release rate are recorded. Similar to the cone calorimeter, the maximum heat flux for the MLC is 100 kW/m<sup>2</sup>, however, 50 kW/m<sup>2</sup> is the value most used and corresponds to impinging flames. The test specimen is 100 mm by 100 mm and wood products of up to 50 mm thick can be used although a thickness of 19 mm is standard practice. The

MLC determines the HRR by either multiplying the mass loss rate by a known effective heat of combustion or via the optional gas convection thermopile method. Both of these HRR techniques are recognized as being less accurate than the oxygen consumption method of the cone calorimeter. This study determines the agreement between the two bench-scale tests.

## MATERIALS AND METHODS

The study was organized as follows: (i) 19 wood based materials were tested in triplicate using both the MLC and the cone calorimeter; (ii) the results of the tests were statistically analyzed and compared to show the agreement between the MLC and the cone calorimeter and potentially justify the MLC's use in lieu of the cone calorimeter. Prior to testing, the samples were stored in a 50% relative humidity room at 21 degrees C for over 30 days or until they reached equilibrium with the environment. All samples were subjected to irradiance at 50 kW/m<sup>2</sup> in a horizontal orientation. The range of wood products in this study included untreated and fire retardant-treated (FRT) particleboards, untreated and FRT medium density fiberboards (MDF), untreated plywood, untreated OSB and untreated flooring and lumber (Table 1).

Table 1  
Materials tested in the cone calorimeter and the mass loss calorimeter

| Material ID | Type of Material <sup>1</sup>                                             |
|-------------|---------------------------------------------------------------------------|
| 1           | FRT Douglas Fir Plywood                                                   |
| 2           | Oak Veneer Plywood                                                        |
| 3           | Douglas Fir Plywood                                                       |
| 4           | FRT Southern Pine Plywood                                                 |
| 5           | Douglas Fir plywood                                                       |
| 6           | Southern Pine plywood                                                     |
| 7           | Particleboard                                                             |
| 8           | Oriented Strandboard (OSB)                                                |
| 9           | Hardboard                                                                 |
| 10          | Redwood Lumber                                                            |
| 11          | White Spruce Lumber                                                       |
| 12          | Waferboard                                                                |
| 13          | Red Oak Flooring                                                          |
| 14          | Stucco on Oriented Strandboard                                            |
| 15          | FRT Medium Density Fiberboard, UF, manufactured in a southern U.S. plant  |
| 16          | FRT Medium Density Fiberboard, NAF, manufactured in a southern U.S. plant |
| 17          | FRT Medium Density Fiberboard, manufactured in a western U.S. plant       |
| 18          | Untreated Medium Density Fiberboard, manufactured in a western U.S. plant |
| 19          | Southern Pine boards                                                      |

<sup>1</sup>“NAF” indicates no urea formaldehyde resin was added to the board while UF indicated urea formaldehyde was present.

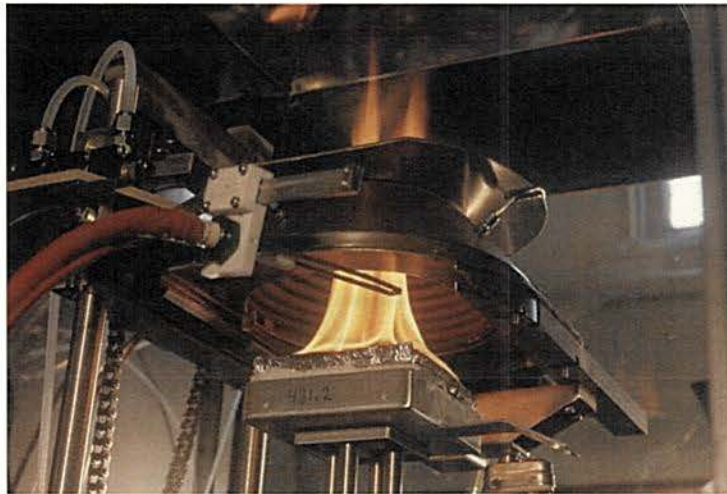
### Cone Calorimeter

The Cone calorimeter procedure and analysis was done according to ASTM E1354 using only the oxygen depletion method. The cone calorimeter test has been shown to provide fire performance assessment and fire properties that is indicative of their material fire performance in full-scale fires.<sup>2</sup> The ASTM E1354 standard for the cone calorimeter sets out to determine the response of materials exposed to controlled levels of radiant heating with or without an ignition source. Radiant heat is the

major cause of fire spread and the cone measures intensity of the peak heat release rate (PHRR); one of the critical factors in predicting the growth rate of fire.

The test apparatus consists of the following components: a conical radiant electric heater; specimen holders; an exhaust gas system with oxygen monitoring and flow measuring instrumentation; an electric ignition spark plug; a data collection and analysis system; and a load cell for measuring specimen mass loss.

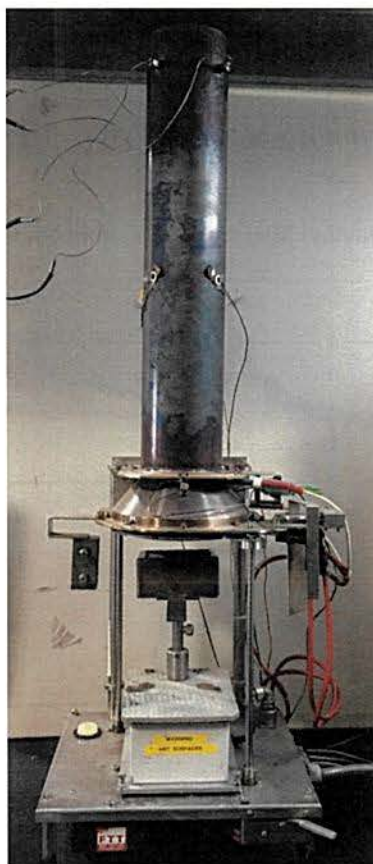
Figure 1  
Cone Calorimeter at FPL



#### Mass Loss Calorimeter

The MLC procedure followed the ASTM E2102-11 test method, but the analysis was modified to improve the HRR results from the published optional HRR method. The MLC was confirmed to have significant systematic HRR errors using the ASTM thermopile method due to thermal radiation heat losses varying between materials with differing soot production, as presented by Dietenberger, et al.<sup>2</sup> An improved HRR method and calculation was developed by constructing another thermopile on the fume stack itself, digitally deconvolving the signal of fume stack thermal response to radiant and convective energy absorption, and combining the resulting processed signal with the ASTM thermopile signal into a composite value,  $T_{cp}$ , described in more detail below and in Dietenberger and Boardman, 2013.<sup>2</sup> Finally, a linear correlation with known HRR of reference materials was established, similar to the ASTM E2102 method but relying heavily on HRR of polymethyl methacrylate (PMMA) and ethylene glycol to establish the calibration constant.<sup>2</sup>

Figure 2  
Mass Loss Calorimeter modified to include thermopile on chimney walls to compensate for radiant energy errors of the in-flow thermopile.



#### ***Determining Mass Loss Rates with Savitzky-Golay Filtering and Exponential Smoothing***

ASTM E2102-11 currently does not specify the numerical procedure for calculating the mass loss rates from the weight cell measurements. In this study, the raw data was captured from the weight cell and thermocouples at 2 Hz and subsequently processed using a macro in an Excel spreadsheet. A Savitzky-Golay filtering algorithm was applied to smooth the data and determine the mass loss rate as the derivative of the smoothed curve. Since certain wood specimens will show an initial sharp peak for the mass loss rate, as measured in the cone calorimeter, it can be a challenge to reproduce the mass loss rate curve with the MLC if the time response of the weight cell differs from the cone weight cell; as was our case. However, our cone has a slow responding oxygen analyzer which means that the time response of the HRR curve can be matched to the MLC given appropriate exponential filtering to match time responses. Therefore, it is possible that the HRR computations may miss the initial HRR peak that should correspond with the initial sharp mass loss rate peak. We applied an exponential smoothing to the mass loss rate curve to match the slower time response of the HRR computations while using deconvolution to speed up the response of the stack thermocouples.

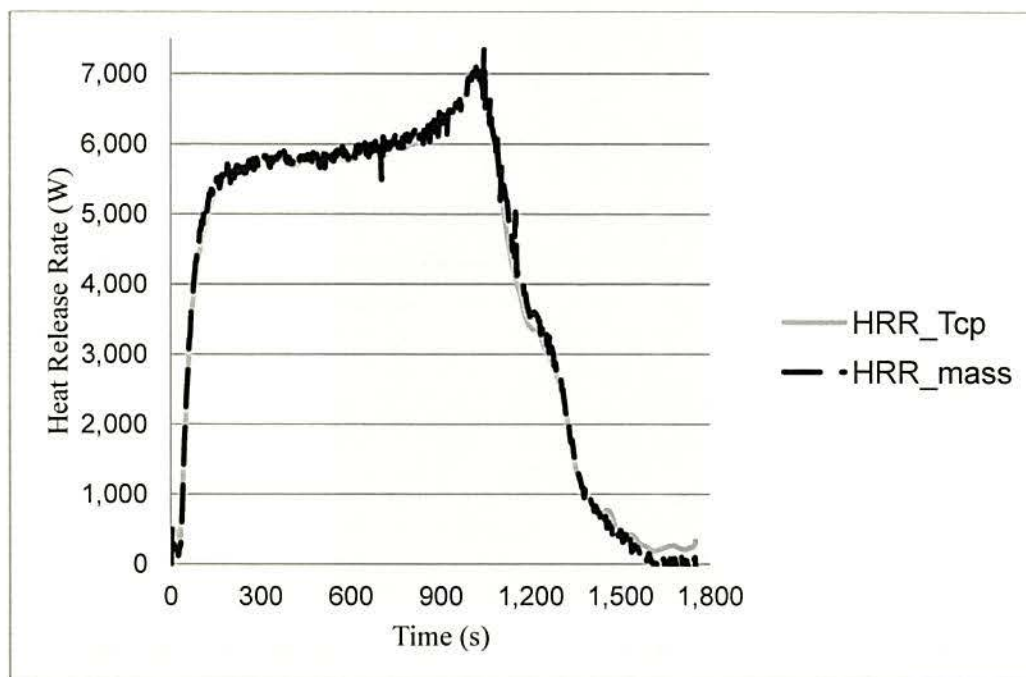
#### ***Calibration of Thermopiles for HRR Measurements***

The ASTM E2102 standard method for the linear correlation of the methane flow rate with the stack thermopile temperature produces poor results due to difficulty in measuring the methane flow rate and lack of soot in the exhaust.<sup>2</sup> We choose to calibrate with PMMA and ethylene glycol measuring their mass loss rate and multiplying by the known heat of combustion to determine the HRR. The PMMA

exhaust has more soot, and thus has more radiant heat transfer to the side walls of the chimney. To compensate for this potential error in HRR predictions based on methane calibration, a secondary thermopile was constructed of flattened thermocouples placed between thin ceramic washers and screwed into the chimney wall, as shown in Figure 1. This signal was modified by deconvolution as detailed in Dietenberger and Boardman 2013 to obtain  $T_c$  and then combined with the temperature from the air thermopile,  $T_p$ , to obtain the correlation in equation 1 with results shown for PMMA in Figure 3.

$$HRR = 4.468(T_p + 3.5T_c) - 15.75 \quad [1]$$

Figure 3  
Polymethyl Methacrylate Calibration Curve



### Statistical Analysis

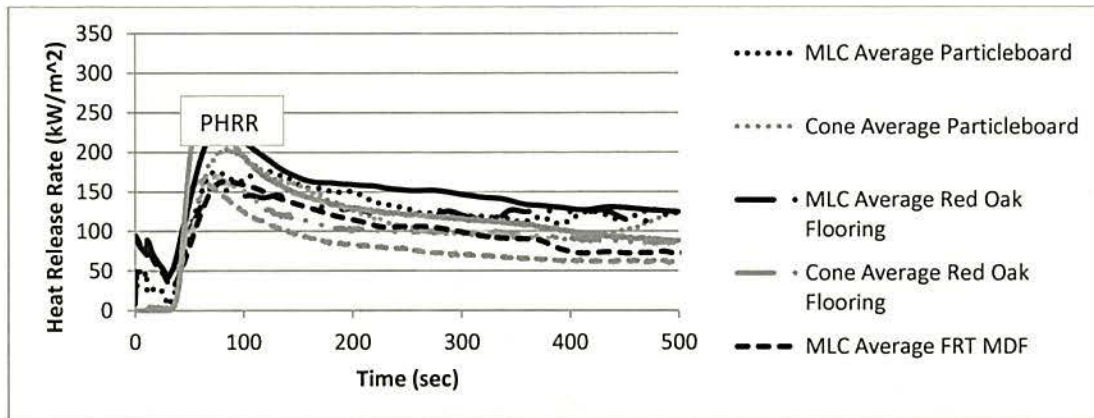
Agreement between the two methods was assessed with scatter plots. One set shows the relationship between the means of the materials as measured by both MLC and the cone calorimeter, and includes both the within material variation (as  $\pm 1SE$ ) and the between material variation and their scatter relative to the line of equality (Figures 5 and 6). A second set examines the agreement between the two methods' average values for each material by plotting the difference in the mean material values for each method versus their average material values for the two methods (Figures 6 and 8). These are Tukey mean difference plots (also known as Bland-Altman plots) and include an estimated bias line and 95% limits of agreement.<sup>6</sup> Under assumptions of normality and homogeneity, approximately 95% of the material differences would be expected to fall between the two lines. The limits are calculated by including estimates of the within material variability, although it should be noted that the measurements in this case are destructive, and there are indications that the within material variation is not homogeneous across materials.

### RESULTS AND DISCUSSION

The primary result from both the cone calorimeter and MLC tests is the curve for heat release rate as a function of time. Figure 4 illustrates the comparison of the average heat release rate curve of

three specimens tested in both the MLC and the cone calorimeter for selected materials. For reporting purposes, these curves were reduced to single numbers via individual results such as the recorded first peak heat release rate (PHRR) and the total heat release (THR).

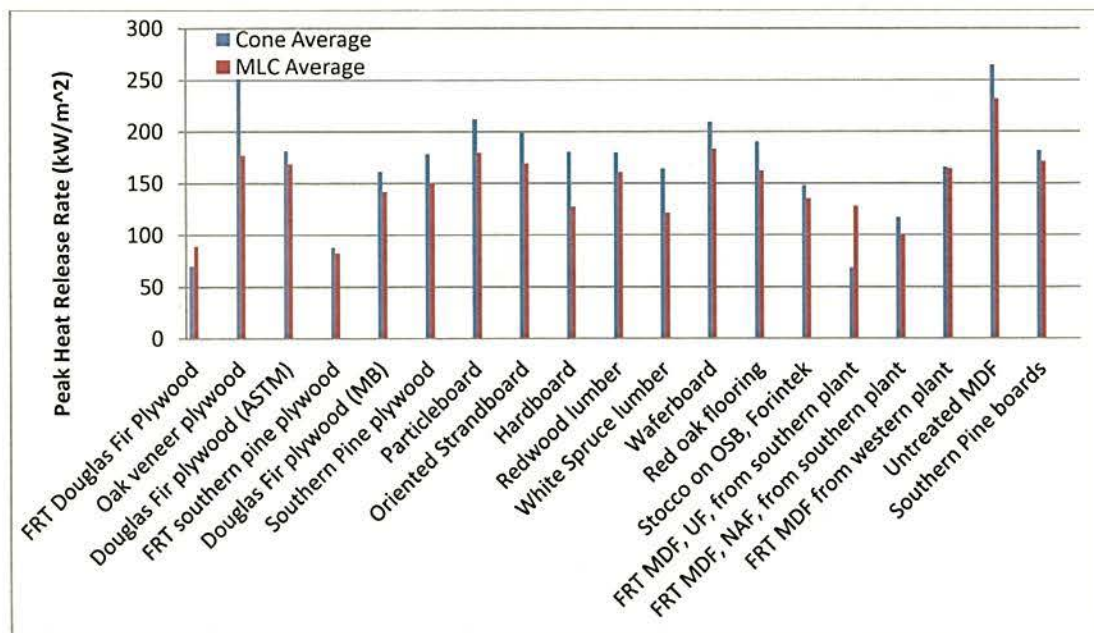
Figure 4  
Average Heat Release Rate Curves for Particle Board, Red Oak Flooring, FRT MDF and Untreated MDF



#### First Peak Heat Release Rate

The HRR measurements result in curves for the HRR as a function of time and generally exhibited a sharp initial peak heat release rate. For wood and wood products, the initial peak is generally accredited to the uncharred wood having a prompt HRR prior to the protective char layer forming as the result of the thermal degradation of the wood.<sup>5</sup> Figure 5 presents the averages first PHRR measured by the cone calorimeter and the MLC.

Figure 5  
Comparison of Cone and MLC Average First Peak Heat Release Rates



Scatterplots (Figures 6 and 7) indicate the level of agreement between the cone and the MLC for the materials in Table 1. For the peak heat release rate, the scatterplots both show that there appears to be a positive bias, with the cone measuring higher values (most differences fall above zero). Regression of the difference values against the mean values also show a positive relationship between the differences and mean values (slope p-value=0.0033), indicating that for larger peak heat release rates the cone value increasingly exceeds the mass loss calorimeter value.

Figure 6

Mean PHRR for each material. The bars extending from each mean pair represent  $\pm 1$  standard error.

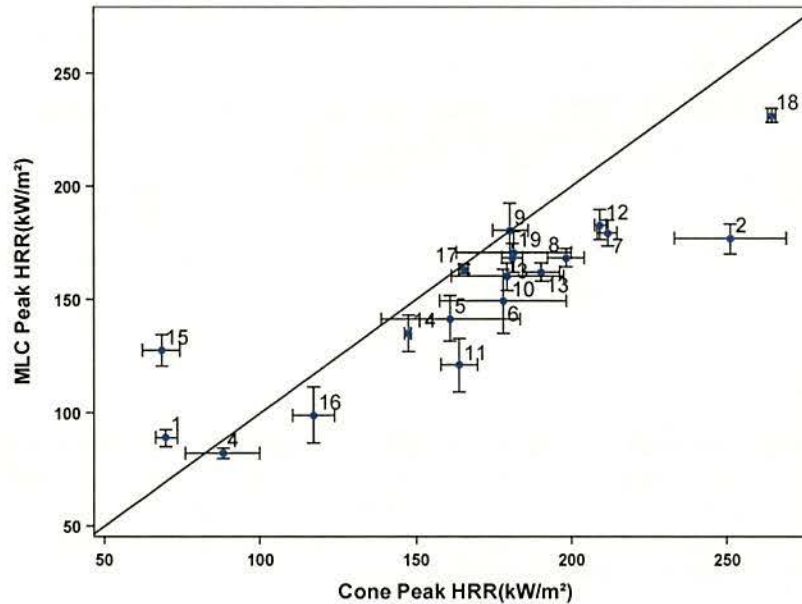
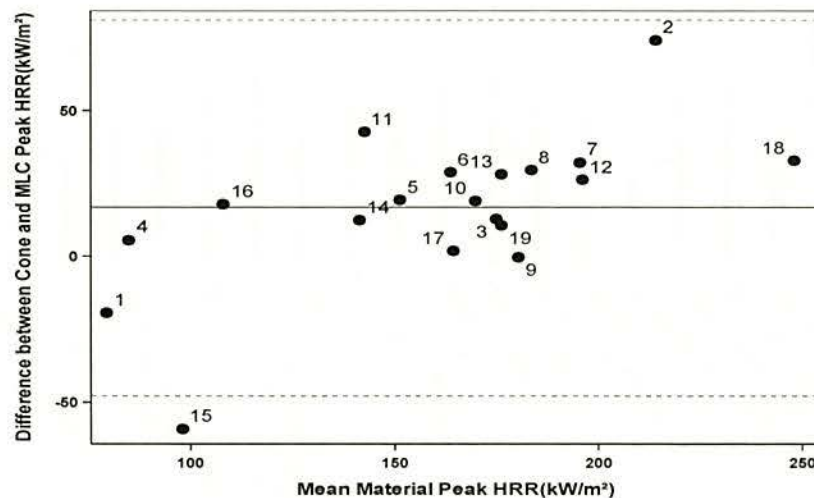


Figure 7

Mean difference plots for determining agreement between the cone and the MLC for PHRR. The solid line is the estimated bias between the methods, and the dashed lines represent the 95% limits of agreement which should enclose about 95% of the differences.

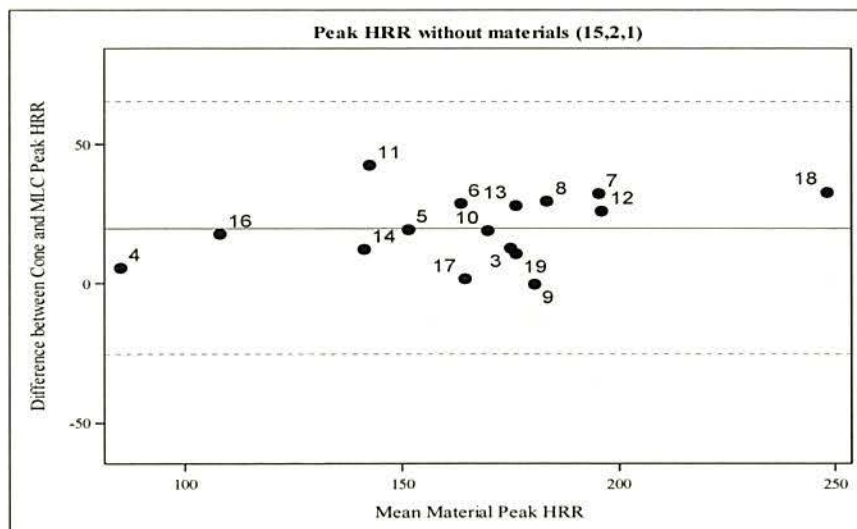


Excluding certain materials improves the limits of agreement between the two methods for peak heat release rate (~30% reduction in estimated standard deviation of the differences). The outliers for the

PHRR are FRT MDF UF (15), oak veneer plywood (2), and FRT Douglas fir plywood (1). Two of the outliers are FRT treated and this may be part of the reason for a larger variation in performance between the two methods. Fire retardant treatment of the wood products reduces the initial HRR, reduces the mass loss rate, increases the residual mass fraction, resulting in lower average effective heat of combustion values, and usually results in longer ignition times.<sup>5</sup> Further fine-tuning of the system and calibration of the mass calorimeter method to the cone will be necessary to obtain agreement for all wood-based materials.

Figure 8

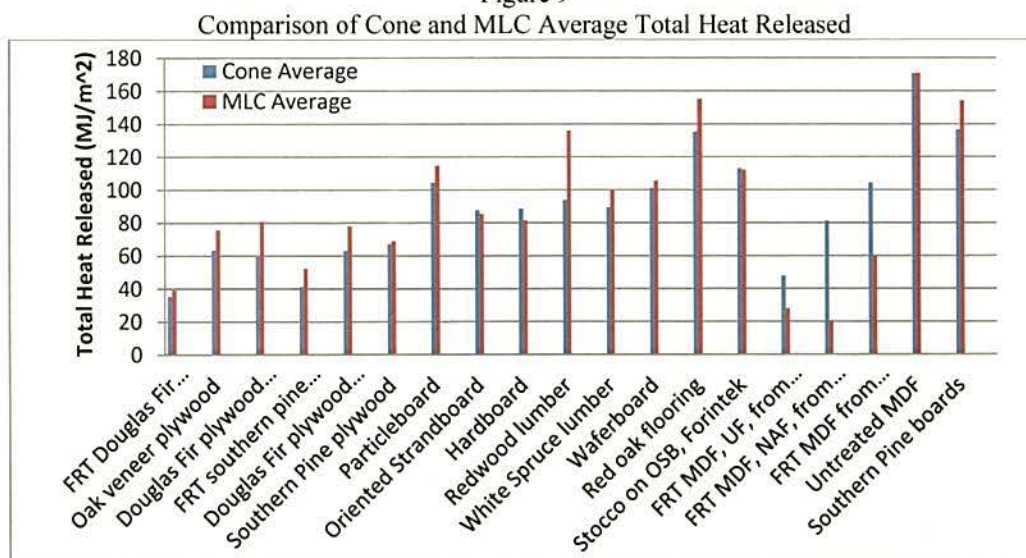
Mean difference plots for determining agreement between the cone and the MLC for PHRR with Materials 15, 2, and 1 excluded.



### Total Heat Release Rate

The total heat released is the heat release rate compiled over time. Figure 9 presents the total heat released per material, averaged from the triplicates, as measured by both the cone and the MLC.

Figure 9



Scatterplots (Figures 10 and 11) indicate the level of agreement between the cone and the MLC for the materials in Table 1. Both illustrate that for most materials, the total heat release is in relative agreement, with a bias of near zero and test of deviation from agreement not significant ( $p$ -value=0.1328). However, the limits of agreement can be greatly tightened if materials 16, 17 and 18 are excluded with a 36.5% reduction in the estimated standard deviation for the differences (Figure 12).

Figure 10

Mean THR for each material. The bars extending from each mean pair represent  $\pm 1$  standard error.

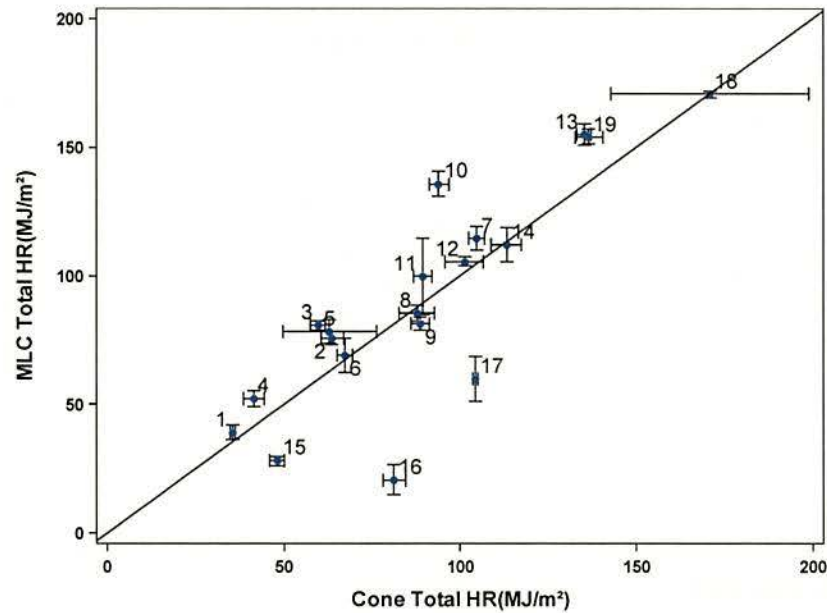


Figure 11

Mean difference plots for determining agreement between the cone and the MLC for THR. The solid line is the estimated bias between the methods, and the dashed lines represent the 95% limits of agreement which should enclose about 95% of the differences.

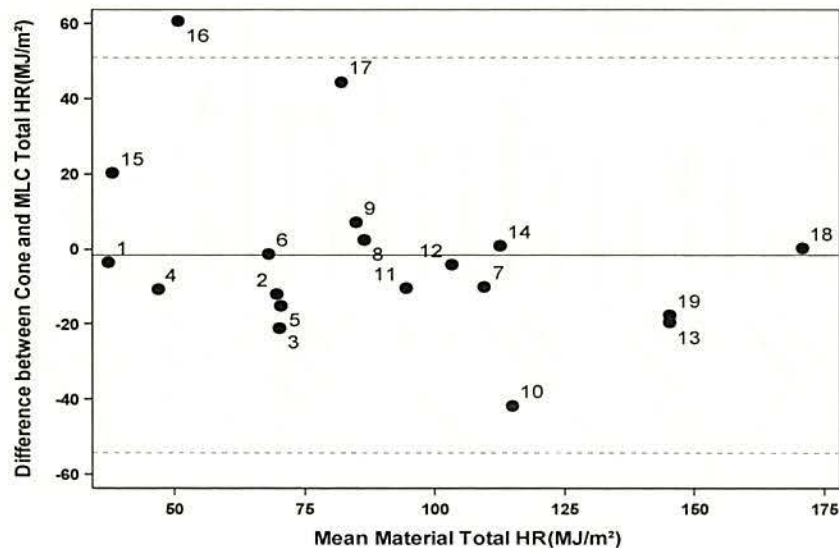
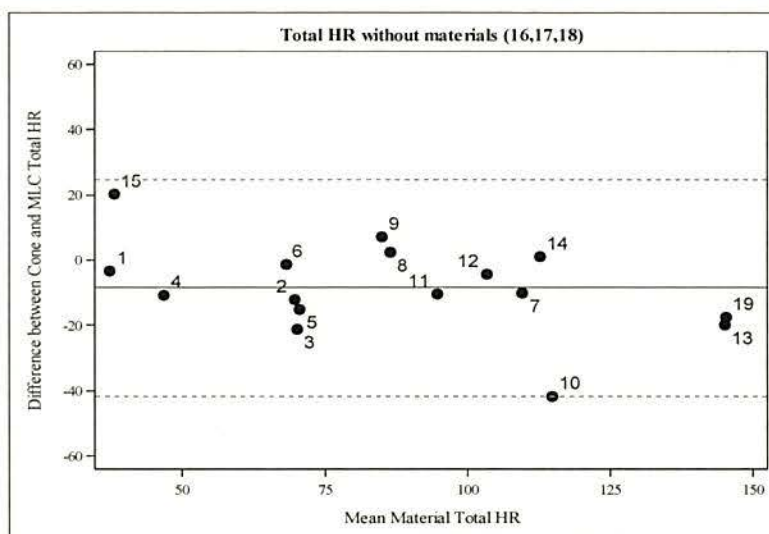


Figure 12  
Mean difference plots for determining agreement between the cone and the MLC for total heat released excluding materials 16, 17 and 18.



## CONCLUSION

The primary objective of this study was to determine the level of agreement for heat release rate measured by the cone calorimeter and the mass loss calorimeter for wood and wood-based products. It is widely accepted that the cone calorimeter, with the oxygen consumption method, is more accurate for obtaining the heat release rates than the MLC which uses the thermopile method. The MLC at FPL was calibrated using the thermopiles with the heat release rates of ethylene glycol and PMMA and provided for reasonable values for the heat of combustion of both untreated and treated wood and has several improvements beyond the standard test method that would be required to obtain a more accurate HRR. In addition to the thermopile at the top of the chimney, an additional thermopile on the chimney wall is used to successfully compensate for radiant heat losses. Based on the test results of the nineteen materials, the mass loss calorimeter is an economical alternative to the cone calorimeter. By using the modified gas convection thermopile method, the MLC provides sensible predictions of the heat release rates measured by the cone calorimeter for wood materials.

## ACKNOWLEDGEMENTS

The authors thank Patricia Lebow for her assistance and statistical skills.

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<sup>6</sup> Bland, JM, Altman, DG 1999, 'Measuring agreement in method comparison studies', *Statistical Methods in Medical Research* vol. 8, pp. 135-160.

<sup>7</sup> <http://www.fpl.fs.fed.us/products/products/cone/introduction.php>

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