

## **Assessing Asphalt Ignition Oven Performance and its Impact on the Asphalt Content Test Result**

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## Executive Summary

This report examines the use of the asphalt ignition oven temperature-time series generated during the asphalt content by ignition test method in identifying erroneous test results. The study was undertaken to provide an empirical tool for asphalt laboratory staff in troubleshooting and validating asphalt content test results.

Results of the data analysis show that some variations in testing procedure can be identified through the temperature-time series. In particular, the first tests performed each day are readily discernable from subsequent tests, even after allowing significant oven warm-up time. Variations in sample size or asphalt content are also shown to create differing temperature-time series; however, the difference is not significant enough to identify errors on individual tests, it is only demonstrable across the group averages. Despite the differences in temperature-time data, no conclusive difference in the accuracy of the test results was found.

It is concluded that the monitoring of the temperature-time series may be a valuable tool in identifying systematic and gross errors introduced during the asphalt content by ignition test method. However, while the preliminary results of this study demonstrate that differences do exist, additional testing should be undertaken to assess the reliability of the proposed method under real-world scenarios. Additional trials will also be required to identify any other procedural variations which result in differences in the temperature-time trend.

# 1. Introduction

## 1.1. Background

The asphalt content of hot-mix asphalt (HMA) is a key volumetric property from which other material properties are derived, and is critical to the materials performance, durability, and longevity. It is common practice to monitor the HMA asphalt content during its production to ensure an acceptable product is being produced. Today, the most common method used to determine HMA asphalt content is the ignition method. The practice of determining asphalt content by ignition method was first investigated in 1969 by the National Cooperative Highway Research Project (NCHRP) and further refined in the early to mid 1990's. It is widely favored due to its simplicity, accuracy, and non-reliance on solvents and chemicals (Brown, Murphy, Yu, & Mager, 1995).

There are many potential sources of error during an asphalt content by ignition test. This is particularly true of field asphalt labs where, for the sake of durability, ovens often lack features such as internal weigh scales. The dusty working environments also cause filters to readily clog, which may impact oven performance. In general, these issues can be mitigated by diligent and knowledgeable staff; however, errors which may result from malfunctioning or inconsistent equipment are not always easily detectable. Some examples of these errors may be an oven which becomes starved of oxygen, or insufficient warm up time at the start of a shift. As these variables are not typically tracked on a test-to-test basis, errors resulting from them may go undetected.

Tracking the ignition oven's temperature-time trend during tests may provide an empirical method to identify errors introduced during the ignition process. This data, when collected throughout the duration of a project, may serve as a basis to better validate individual asphalt content test results and assist in the identification of variance in the ignition oven performance.

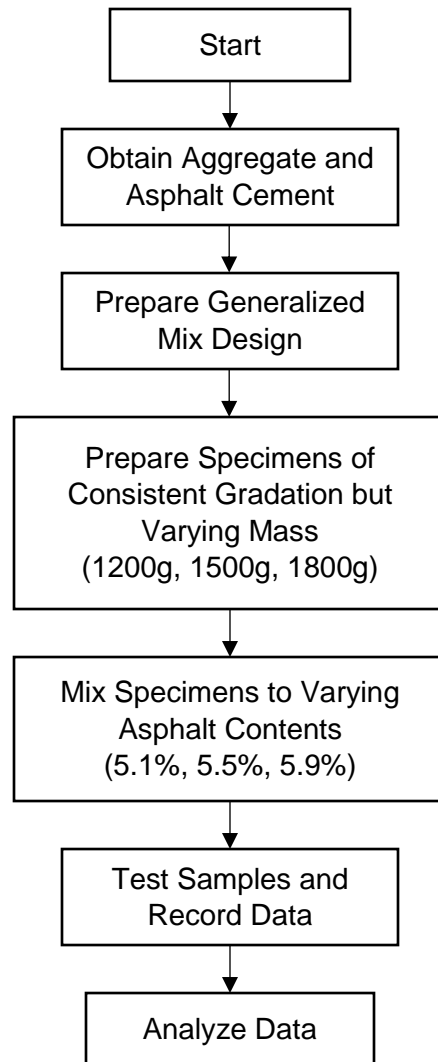
## 1.2. Objective

The experiment was conducted to identify if a predictable trend in the temperature-time curve exists when HMA samples are tested in an ignition oven in a consistent manner, and whether this trend is deviated from under the influence of controlled variations in the HMA properties or testing procedure.

If a correlation is identified, it may provide a basis for the use of the temperature-time trend as a tool in recognizing erroneous or inconsistent asphalt content results. In this manner, laboratory staff will be better equipped to identify and subsequently rectify issues as they occur. In addition, the temperature-time data can serve as a record of oven performance should the accuracy of test results come into question.

## 2. Methodology

Figure 1: Methodology Flowchart



### 2.1. Sample Preparation

A typical mix design was generated in accordance with the Saskatchewan Ministry of Highways and Infrastructure type 72 Hot-Mix Aggregate specification. Three-way split aggregate was supplied as 5.0 - 12.5 mm crushed coarse, < 5.0 mm crushed fines, and < 5.0 mm screened natural fines. 150/200A grade asphalt cement was supplied from a single source to ensure consistency. The mix design details are outlined in Table 1 on the following page.

**Table 1: Sample Mix Design**

Sieve Size (mm)	Crushed Coarse (35%)	Crushed Fines (32%)	Natural Fines (33%)	Blend
	Cumulative Percent Passing			
16.0	100.0	100.0	100.0	100.0
12.5	92.5	100.0	100.0	97.4
9.0	51.0	99.8	100.0	79.3
5.0	0.0	91.5	98.0	61.6
2.0	0.0	53.5	70.8	40.5
0.900	0.0	35.2	42.1	25.2
0.400	0.0	23.8	12.4	11.7
0.160	0.0	14.2	5.6	6.4
0.071	0.0	10.0	4.8	4.8

To prepare the samples, the supplied aggregates were oven dried, dry sieved into individual specified sieve sizes, then recombined to match the mix design gradation. The aggregate specimens and 150/200A asphalt cement were warmed in an oven to 110°C, then mixed together to known asphalt contents. All samples were prepared in advance, then reheated before testing.

A standard samples size of 1500g was chosen in accordance with ASTM D6307 (*Standard Test Method for Asphalt Content of Asphalt Mixture by Ignition Method*) and practice D140 (*Practice for Sampling Bituminous Materials*). A standard asphalt content of 5.5% was selected to approximate a typical asphalt mix design.

Additional samples at varying asphalt contents and masses were produced to determine the effect these variables may have on the temperature-time trend, if any. Samples were prepared that were 20% larger and 20% smaller than the standard sample size, and samples were prepared that were 0.4% richer and 0.4% drier than the standard asphalt content. These variations are on the extreme end of what is typically encountered in practice and were selected to maximize the observable effects. The samples produced are outlined in Table 2.

**Table 2: Samples Produced**

Sample Count	Sample Mass (g)	Asphalt Content (%)
20	1500	5.5
7	1800	5.5
7	1200	5.5
7	1500	5.1
7	1500	5.9

## 2.2. Ignition Oven

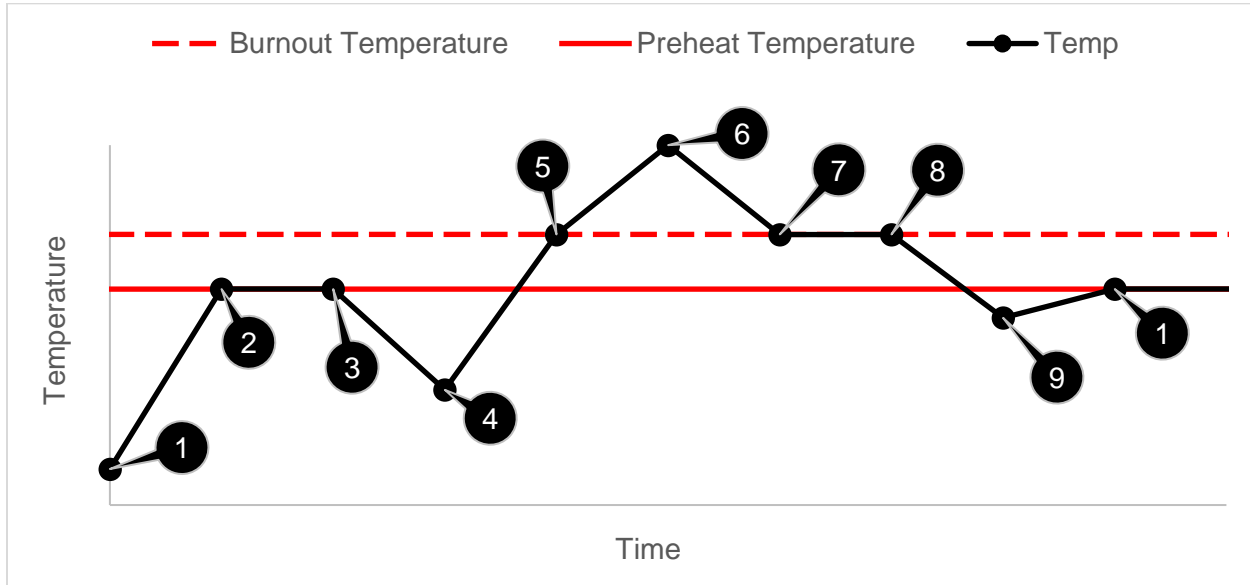
The experiment was performed using a HM-378 Gilson Asphalt Content Furnace which fully meets ASTM and AASHTO standards for asphalt content determination of hot-mix asphalt. This oven is commonly used in mobile laboratories due to its simplicity and durability during transport and is also commonly used in centralized laboratories. Default oven settings were used, with the exception of an increased hold. The increased hold time was chosen to help ensure complete decoking of the specimens. Detailed oven settings can be found in Table 3.

**Table 3: Gilson HM-378 Oven Settings**

Description	Value
Hold Time	25 Minutes*
Main Chamber Preheat Temperature	500°C
Main Chamber Burnout Temperature	538°C
Afterburner Temperature	850°C
Afterburner Fan-On Temperature	850°C

\*Hold time increased from the default value of 20 minutes

**Figure 2: Gilson HM-378 Ignition Sequence**



Adapted from the Gilson HM-378 Operating Manual (Gilson Company, Inc., 2018)

The typical ignition sequence for the Gilson HM-378 is shown in Figure 2. Key points throughout the ignition sequence are described below.

1. The oven is at ambient air temperature before being turned on.
2. The oven preheats to the main chamber preheat temperature.

3. The oven door is opened, heat escapes as the sample is inserted, the oven door is closed, and the test is started (CONTINUE is pressed).
4. The temperature drops until the ambient air inside the chamber is reheated. The temperature begins to climb under the power of the heating elements.
5. The burnout temperature is reached, and the chamber elements are switched off.
6. The temperature peaks.
7. Temperature returns to the specified burnout temperature and the HOLD period begins.
8. Hold time finishes, and the test is complete. The oven door is opened, and the specimen is removed.
9. The oven door is closed.
10. The chamber returns to equilibrium at the preheat temperature.

### 2.3. Testing Procedure

Each sample was prepared and classified into one of the following categories:

**Table 4: Sample Categories**

Group Name	Asphalt Content (%)	Dry Aggregate Mass (g)	Description
BM	5.5	1500	Benchmark samples
FT	5.5	1500	The first tests performed each day
5.9	5.9	1500	'Richer' mix, 0.4% more asphalt content than benchmark
5.1	5.1	1500	'Drier' mix, 0.4% less asphalt content than benchmark
1800	5.5	1800	Proportionally larger sample size
1200	5.5	1200	Proportionally smaller sample size

Samples are tested using the following procedure in accordance with ASTM D6307 Method B standards:

1. Prepared samples are warmed in an oven to  $110 \pm 5^\circ\text{C}$ .
2. The ignition oven is switched on and allowed to warm to  $500^\circ\text{C}$  (approximately 75 minutes).
3. A sample is spread uniformly on the ignition oven basket, weighed, then placed in the oven for testing. The test procedure is started by pressing "CONTINUE" on the Gilson HM-378 Asphalt Content Furnace.
4. The oven temperature, as indicated by the oven's built-in digital readout, and time are recorded throughout the duration of the test. Readings are taken at 10 second intervals.
5. Once a test is completed, the sample is removed from the oven. The sample is cooled to room temperature, and the oven is returns to equilibrium (preheat temperature of  $500 \pm 5^\circ\text{C}$ ).



6. When the sample has reached room temperature, its final weight is obtained, and the dry aggregate is wash sieved. The resultant gradation is used to verify the accuracy of the specimen and to identify any aggregate mass loss.
7. Once the oven has reached equilibrium, the next sample can be tested resuming from step 3.

Variations in the first tests performed each day were identified early on in testing. Testing continued as outlined above with the first tests given their own category. These tests mimic the scenario of an inadequately preheated oven, and to ensure that the oven is sufficiently warmed for subsequent tests.

## 2.4. Analysis

Following ignition, the burnt aggregate from each sample was wash sieved in accordance with ASTM C136 (*Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates*) to confirm their gradations did not vary significantly from the mix design gradation.

Asphalt content correction factors (also known as oven corrections) were established for each sample by subtracting the known (mixed) asphalt content from the tested asphalt content:

$$\Delta AC = AC_{measured} - AC_{known}$$

Once the oven corrections were determined, a method of statistical analysis was utilized to assess if any meaningful difference in the test means existed between groups. A one-way Analysis of Variance (ANOVA) test was selected based on a single factor (oven correction) being considered through two or more levels (sample group). The ANOVA test establishes a null hypothesis in which all means are statistically equal. In the case of an acceptance of this null hypothesis, the test results are significant, and all means can be considered equivalent. However, when rejected, the ANOVA test simply states that at least one group mean is different than the rest. Therefore, the Tukey HSD post-hoc test was performed to conduct a pairwise comparison of all samples and determine where these significant differences may exist.

The temperature-time trends were then analyzed to determine if obvious differences existed between the sample groups and, if there was found to be a difference, what its significance may be. A smoothing algorithm (3-point moving average) was applied to smooth out artifacts in the temperature readings resulting from the 10 second recording frequency and precision of the oven's digital readout. The average and standard deviation of the smoothed temperatures, for each group, was calculated at each 10 second time interval to produce group-specific temperature and standard deviation time series.

### 3. Results and Analysis

Of the 48 specimens tested, two (2) were excluded as a result of gradation variability and one (1) was excluded as a result of testing procedure error. The gradation variability is believed to be a result of improper batching; the error was identified during testing procedure and the sample was discarded.

Of the 1500 g 5.5% asphalt content samples, eleven (11) were used to ensure the oven was adequately heated and are categorized as FT. The remaining nine (9) samples were burnt normally and are categorized as BM. The total sample set is outlined in Table 5.

**Table 5: Samples Tested**

Group Name	Original Sample Count	Adjusted Sample Count
BM	9	9
FT	11	11
5.9	7	7
5.1	7	6
1800	7	6
1200	7	6

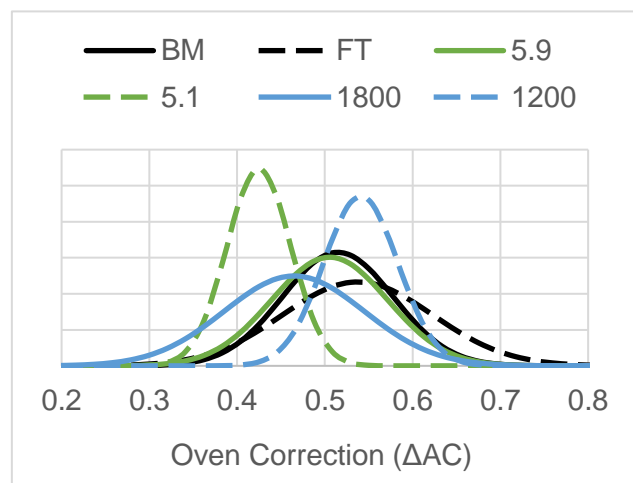
#### 3.1. Asphalt Content Variability

The variance between the measured asphalt content and the known asphalt content, also known as the oven correction, was determined for each sample. These results are summarized in Table 6 below. It can be seen that the FT category has the largest oven correction and standard deviation. While this may be coincidental, it was decided to exclude the FT tests from further analysis within this section.

**Table 6: Oven Correction Analysis**

Group Name	Mean Oven Correction	Standard Deviation
BM	0.51	0.06
FT	0.54	0.09
5.9	0.51	0.07
5.1	0.42	0.04
1800	0.47	0.08
1200	0.54	0.04

**Figure 3: Oven Correction Normal Distributions**



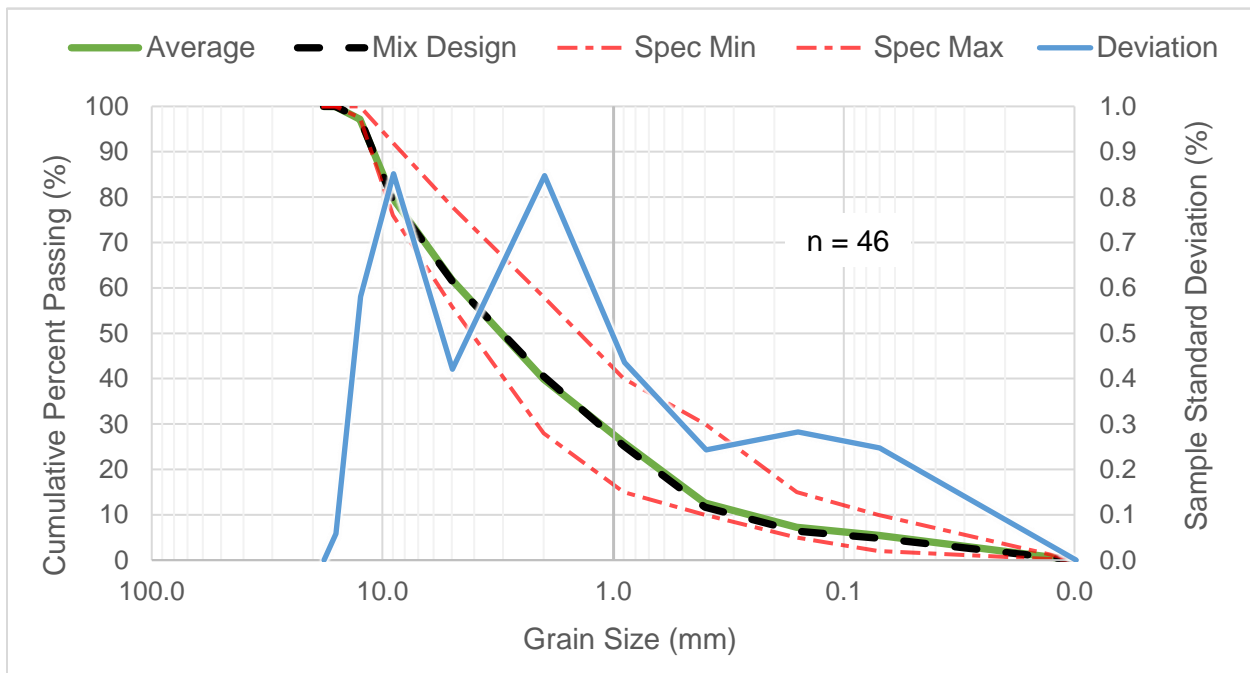
A one-way ANOVA was conducted to compare the effect of the varying HMA properties on the asphalt oven correction. There was found to be a significant effect on the oven correction at a confidence interval ( $\alpha$ ) of 0.05 for the 5 conditions [ $F(0.05,0.07) = 4.62$   $p = 0.005$ ]. Post hoc comparisons using the Tukey HSD test indicate that the mean oven correction for the low asphalt group (category 5.1) was significantly different than all other groups except for the increased mass group (category 1800). Additionally, the low mass group (category 1200) was significantly different than both the 5.1 and 1800 categories. The normal distributions for these groups can be seen in Figure 3.

This analysis suggests that by varying the asphalt content or sample size, the oven corrections also vary. This is an unexpected result, and further testing is suggested.

### 3.2. Gradation Variability

Once burnt, wash sieves were performed on all samples to verify their gradations. The gradation curves for all samples were compared to each other to assess their precision and accuracy to the mix design values. The mean and standard deviations for each sieve was calculated and plotted against the mix design gradation curve. The analysis indicates that the samples were mixed to design and there should be no significant interference from gradation variability on the asphalt test result or the temperature-time trends. The results of this analysis are represented in Figure 4 below.

Figure 4: Sample Gradations

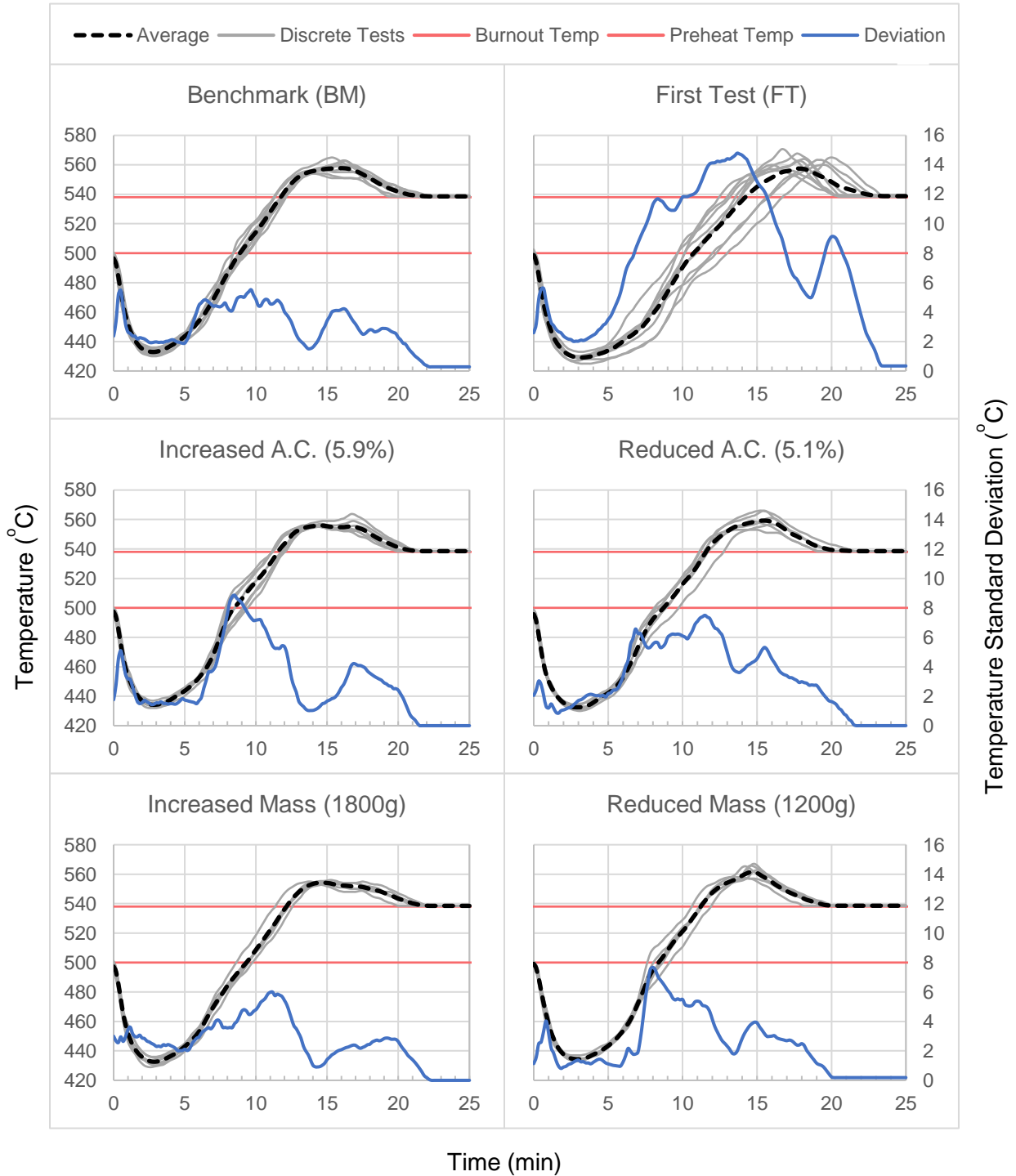


On average, our gradation is finer below the 900  $\mu\text{m}$  sieve and coarser above the 900  $\mu\text{m}$  sieve. This is a result of not washing the dried aggregate when dry sieving it in the preparation of the specimens. Fine material ( $< 160 \mu\text{m}$ ) clings to the coarser material, increasing its mass. After ignition and upon washing, this fine material is accounted for properly.

### 3.3. Temperature-Time Analysis

The discrete temperature-time series, time series averages, and time series standard deviations were plotted for each category to identify trends and assess their variabilities, shown below:

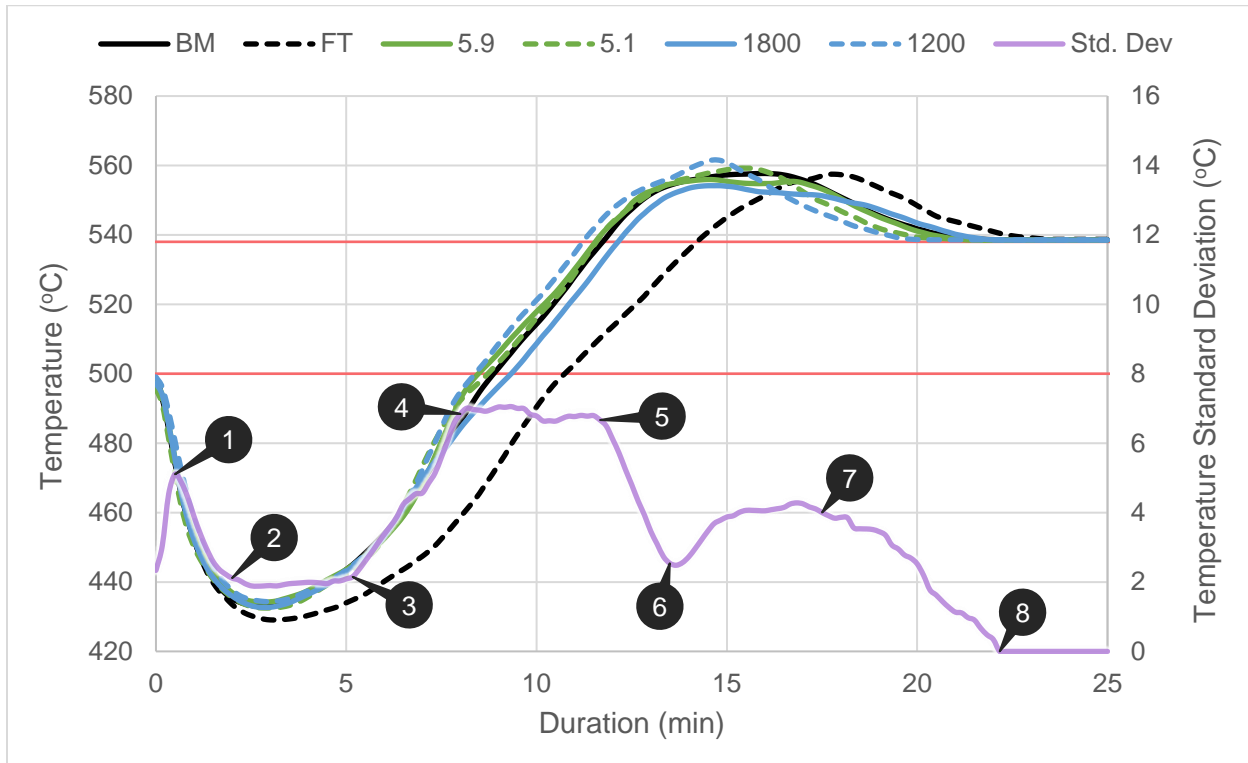
**Figure 5: Categorized Temperature-Time Series Data**



These plots demonstrate that there is a consistent temperature-time trend across the sample sets, which conforms to the predicted trend outlined in the Gilson HM-378 oven manual. The discrete sample time series maintain a reasonably tight fit to their respective group averages. However, there is insufficient variance between group averages to make the temperature-time series data a useful tool in distinguishing one category from another.

The exception to the above is the FT sample set, which experiences the largest variability. In our testing we found that it can take up to three (3) hours of preheating for the oven to produce a temperature-time series comparable to the trends shown in other groups. When the ignition oven is opened, the preheated air within the chamber spills out of the oven; if the bricks lining the chamber have not been preheated thoroughly, it takes longer to reheat the air within the chamber upon closing the door and beginning the test. As a result, there is a noticeable lag between the FT group and the other groups. This lag is directly dependant upon the allotted preheat duration. This behavior is clearly visible in Figure 6.

**Figure 6: Average Temperature-Time Series Data**



Of note is the standard deviation of the temperatures at each time interval. Plotting this time series reveals where the temperature-time series deviate from one another. This time series follows a similar pattern across all datasets and has the following points of interest:

1. The initial peak is an artifact of the oven hold temperature and oven precision. ASTM standards require a precision of  $\pm 5^{\circ}\text{C}$ , therefore the oven at equilibrium is always fluctuating between  $495 - 505^{\circ}\text{C}$ . It is likely that most of the fluctuation is present in the air as it takes

longer for the bricks to heat and cool. This hot air is released upon opening the chamber door, and the standard deviation decreases.

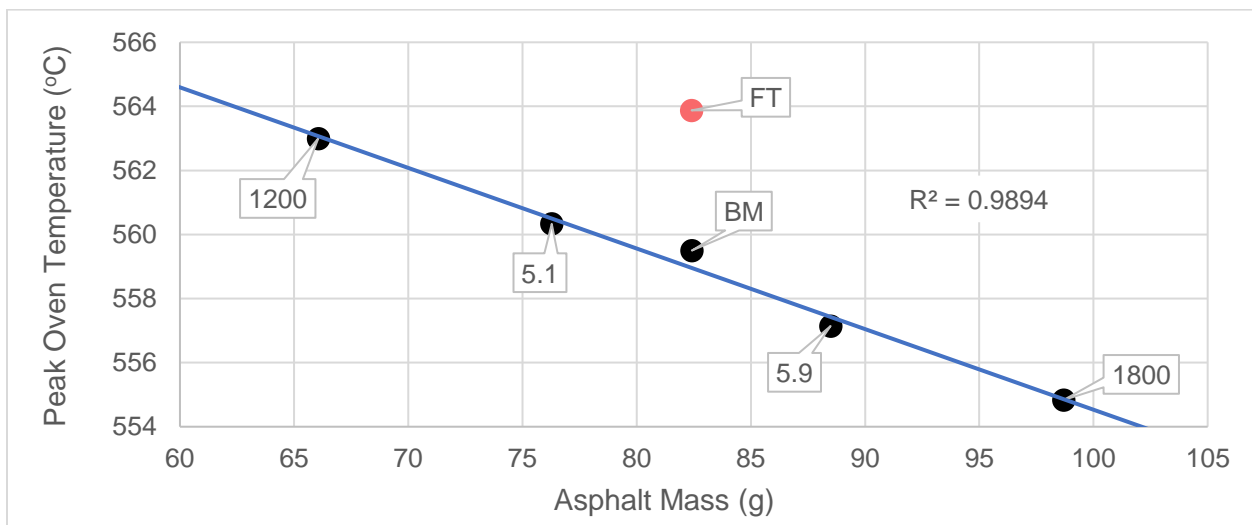
2. The deviations equalize as the temperature reaches the low point and begins to climb.
3. The temperature begins to rise along with the variation. Asphalt may begin smoldering and burning around this point, accounting for the increase in deviation.
4. All samples have ignited by this point. The oven temperature continues to rise at a constant rate (constant slope) amongst all samples, therefore the deviation levels out.
5. The test phase begins, and the oven elements turn off. The temperatures begin to converge around their peaks.
6. The peak temperature is reached, the samples have converged and begin to diverge as they burn off their remaining oil and their temperatures drop.
7. The samples cool and the deviation converges to zero (0) as the test ends.
8. The test is complete, all samples have returned to the burnout temperature.

While not as clear, the essence of this trend is also visible in the individual group plots. It is likely that as the sample count increases, the standard deviation time series will converge towards this pattern. Once again, the exception is the FT group of samples, as the allowed preheat duration has been shown to be fundamental in achieving a consistent temperature-time series.

Also of interest is the distinct differences between the sample groups on average. Looking closer at the average time series in Figure 6 above, we can see that samples of smaller mass ignite sooner than samples of larger mass. The 1200 g samples got hotter quicker and peaked at a higher temperature, on average, than their 1800 g counterparts. Similarly, the BM, 5.9, and 5.1 sample sets, all of which are 1500 g, fall in between the 1800 g and 1200 g datasets.

Another temperature related phenomena is demonstrable by plotting the average peak temperatures vs the total mass of asphalt present in the samples, as seen in Figure 7. This implies that the peak temperature may be influenced by the total asphalt mass present during testing.

**Figure 7: Mass of Asphalt vs. Peak Oven Temperature**



These two observations give credence to the hypothesis that properties of the samples composition may be identifiable by monitoring the ignition oven temperature-time data. However, the trends are only discernible on the group averages. Therefore, caution should be used when using the above observations to make statements about discrete test results.

## 4. Conclusion

The temperature-time series produced by samples tested following the current accepted methods were sufficiently uniform to allow for the identification of variations in testing procedure. Namely, tests performed each day under inadequate oven preheat conditions (i.e. FT category) were readily discernible in the temperature-time series. It is plausible that other systematic or gross errors may be identifiable in this same manner, however further testing is required. Future studies may investigate the following scenarios where inconsistencies could be identified through the temperature-time series:

- Failure to provide sufficient oven preheat time. Our experiment found that it can take up to three (3) hours of preheating to produce a temperature-time series of an oven at equilibrium.
- Not allowing the oven to return to the preheat temperature, such as in cases where you are performing multiple tests back to back.
- An ignition oven starved of oxygen, such as in the case of clogged vents or negative pressures in the oven chamber from lab ventilation fans.
- Failure to initiate the testing procedure, such as in the case the lab technician places the sample in the oven but forgets to press "CONTINUE".

It has also been demonstrated that, on average, the temperature-time series and peak oven temperatures are a function of the mass of asphalt present in a sample, and by extension the sample size and asphalt content. However, while these trends are readily observable on the group averages, there is too much variability within groups to use this relationship in predicting discrete test results. Therefore, making any concrete conclusions towards a discrete sample's true asphalt content based upon the temperature-time series and sample mass is not suggested.

Until further research is performed on the effect of varying gradations and asphalt grades, temperature-time series data should be constrained to individual mix designs.

In conclusion, monitoring of the temperature-time series is a valuable tool to identify systematic and gross errors introduced during the asphalt content by ignition test method. Most agencies currently lack a test-to-test empirical method of tracking ignition oven performance, and as such there is risk that errors may be introduced in the burn process and go undetected. Therefore, we propose a best practice of recording the ignition oven temperature-time series data to aid in identifying erroneous results.

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