Development of a Framework to Evaluate Asphalt Binder and Plant Produced Asphalt Mixes for Acceptance in Canada

by

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AUTHOR'S DECLARATION

I hereby declare that I am the sole author of this thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners. I understand that my thesis may be made electronically available to the public.

ABSTRACT

Asphalt binder, sometimes referred to as asphalt cement binder, or asphalt cement, is the binder that holds the aggregates together in asphalt mixes for asphalt concrete pavements. In Canada, asphalt binder properties are accepted in accordance with the American Association of State and Highway Transportation Officials (AASHTO) standards: AASHTO M 320 – Standard Specification for Performance-Graded (PG) Asphalt Binder. For asphalt mixes, the material is accepted based on criteria set on parameters for aggregates, asphalt binder, recycled materials such as reclaimed asphalt pavement (RAP), and volumetric properties such as air voids, voids in mineral aggregate, and voids filled with asphalt. The parameters are set because they have historically provided a good indication of a mixture's probable performance.

As well, there has always been an interest in determining the properties of asphalt binder of in-situ asphalt mixtures, for research or forensic investigation purposes, or as a way of confirming that the correct asphalt binder was utilized during asphalt mix production, to ensure the desired performance is achieved. With the increased use of RAP, many user agencies are also looking for ways to evaluate the properties of the blended asphalt binder (i.e., new asphalt binder and old binder from RAP) since this also has an impact on the asphalt pavement performance.

One option is to conduct mixture performance testing of asphalt mixes. Another option, often selected by users because of its relative simplicity, is to determine the physical properties the recovered asphalt binder from plant produced asphalt mix. Typically, the original and recovered asphalt binders are required to meet the same specification. Although intuitive and relatively simple, using recovered asphalt binder properties – particularly in a specification – is not without some potential concerns.

This research compared the physical properties of original asphalt binder to the properties of the same asphalt binder recovered from asphalt mix after plant production. The asphalt mixes included seven surface course mixes, two of which include 15 percent RAP. The asphalt mixes were produced with most common PG grades of asphalt binder used in Ontario.

The results showed that the significant increase in ash content in the recovered asphalt binder coupled with the difference in oxidation between laboratory aging and plant production produced

rheological properties that show the recovered asphalt was stiffer and less representative of the tank asphalt. There was a statistically significant difference between the tank asphalt properties and the recovered asphalt properties. Additionally, the physical properties of recovered asphalt showed higher variability than the same physical property tests on tank asphalt.

Additionally for asphalt binders, the Superpave PG system simulates aging and its effect on the asphalt properties using two accelerated laboratory conditioning procedures: the Rolling Thin Film Oven (RTFO) test for short-term (production and placement) aging, and the Pressure Aging Vessel (PAV) for longer-term (in-service) aging. However, these aging protocols have been shown to not correlate with actual field aging, posing a challenge for predicting performance.

The Ministry of Transportation Ontario (MTO) conducted a study in 2009 that showed that correlation of the PG system can be improved if field aging can be better replicated in the laboratory. Additionally, the Superpave volumetric properties have been shown to not completely predict the long-term performance of asphalt pavements. Therefore, implementation of suitable performance tests and aging protocols is crucial to predict performance and maintain sustainability in highway infrastructure.

This research also compared the aging that is simulated by the RTFO and PAV aging in the laboratory, to the short-term aging that occurs during asphalt mix production and placement on a job site. The aging was determined with chemical analysis used to determined concentrations of the asphalt fractions (i.e., saturates, aromatics, resins, and asphaltenes).

The concentrations are used to calculate an aging index as an indication of degree of oxidation for laboratory versus field. The outcome of this analysis showed that the level of oxidation offered by RTFO aging of tank asphalt in the laboratory (laboratory short term aging) is less severe and does not simulate the short-term aging obtained in the field through plant production and placement of the hot mix.

Lastly, although the Superpave PG system is an improvement over previous grading systems, the PG system evaluates cracking behavior by only considering properties of asphalt binder and fails to consider the aggregate portion of the asphalt mixes, which makes up about 90 to 95% of the total weight of the asphalt mix. Additionally, new parameters have been researched since the implementation of Superpave that better characterize the oxidative behavior of asphalt binders. In this research, the Delta Tc (ΔT_c) parameter is evaluated for the asphalt binders, along with Illinois Flexibility Index Test, and Asphalt Mix Performance Tester (AMPT) Flow Number for the asphalt mixes.

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As asphalt binders oxidize or age, their ability to relax stresses at low temperatures diminishes. The ΔT_c parameter provides an indication of loss of ductility: when the asphalt binder cannot relax the stresses fast enough to prevent breaking. The ΔT_c of an aged binder is more negative than that of an unaged binder and would be more likely to exhibit non-load related pavement distresses such as: block cracking, raveling, and longitudinal or transverse cracking.

The Flow Number was developed as part of a research sponsored by the Federal Highway Administration (FHWA) and the National Cooperative Highway Research Program (NCHRP). The intent was to develop and validate simple performance tests for permanent deformation and fatigue cracking to be incorporated in the Superpave volumetric mixture design process. The flow number has been correlated to the mixture's rutting resistance, with a higher flow number indicating higher resistance to rutting.

The Illinois Flexibility Index Test (I-FIT) uses semi-circular bending (SCB) specimen geometry to determine the fracture resistance of an asphalt mixture at an intermediate temperature. Generally, higher FI value indicates the better premature cracking resistance of the asphalt mix.

Correlation tables showed that the properties of the tank asphalt binder showed better correlation with the IFIT Flexibility Index and Flow Number of the resultant mixes than with the recovered asphalt binder properties.

These results not only provide an evaluation of the impact comparing values of recovered asphalt to test criteria and variability derived for original asphalt, but it also provides framework for moving toward acceptance of asphalt mixes based on performance testing in Canada.

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Lastly, I would like to thank my family, for always being in my corner. This journey would not have happened without support my parents and my brother always give me whenever I take on new challenges. I am so grateful to them.

DEDICATION

To my parents, Emmanuel, and Veronica. My brother, Kwasi. And to my son, Jeramus Kwasi - I love you more.

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1 INTRODUCTION

1.1 Statement of the Problem

In Canada, asphalt binder properties are accepted based in accordance with the American Association of State and Highway Transportation Officials (AASHTO) standards: AASHTO M 320 – Standard Specification for Performance-Graded (PG) Asphalt Binder. With asphalt mixes, the material is accepted based on criteria set on parameters for aggregates, asphalt cement, recycled materials, and volumetric properties such as air voids, voids in mineral aggregate, and voids filled with asphalt. The parameters are set because they have historically provided a good indication of a mixture's probable performance.

In 2009, the Ministry of Transportation Ontario (MTO) conducted a study to determine how well the PG grading system correlated to field performance. The study concluded that correlation of the PG grading system can be improved if field aging can be better replicated in the lab. (Huber et al., 2012)

Additionally, with the increased use of recycled materials such as reclaimed asphalt pavement (RAP) in asphalt mixes, many user agencies in Canada are looking for ways to evaluate the properties of the resultant asphalt mix, and the properties of the blended asphalt (i.e., new asphalt binder and old asphalt from RAP), since this has an impact on the asphalt pavement performance.

An option often selected by owners to evaluate the blended asphalt, is to conduct solvent extractionrecovery testing on the asphalt and determine the physical properties of the recovered asphalt in accordance with a standard specification. Users often use the same specification by which the asphalt was originally verified. However, there are concerns with the appropriateness of acceptance criteria based on recovered asphalt, since research has shown that the extraction and recovery process can impact the recovered asphalt properties because of contamination from the solvent and/or aggregate fines that affect the recovered asphalt viscosity. (Stroup-Gardiner & Nelson, 2000; Williams et al., 2002)

In fact, the Ministry of Transportation Ontario (MTO) conducted proficiency testing in 2016 with five labs testing identical mix samples, where the results showed that testing variability was generally higher for recovered asphalt compared to original asphalt. (MTO, 2016) Nonetheless, there is an increasing number of public sector agencies adopting recovered asphalt specifications for acceptance. The variability in test results also makes it more challenging to accurately predict pavement performance.

There is a need for industry to understand how the physical properties of original asphalt binder compare and relate to the properties of the same asphalt binder that is extracted and recovered from a plant produced asphalt mix, and to see the impact of comparing values of recovered asphalt binder to test criteria and tolerances derived for unrecovered (original) asphalt binder. There is also a need to understand this for polymer modified asphalt binder, and the impact when the asphalt mixes contain recycled materials.

There is also a need to understand how well the short-term laboratory conditioning protocol in the PG system simulates the short-term ageing that occurs in the production and paving.

Lastly, there is a need to provide a framework for agencies for the acceptance of asphalt binders and mixes using test methods that have been demonstrated to correlate to field performance, to differentiate between good and poor performing asphalt mixes.

1.2 Research Hypothesis

The hypotheses for this research are as follows:

- There will be a statistically significant difference in test results of tank asphalt binder samples and recovered asphalt binder samples.
- The short-term aging of asphalt binders simulated through laboratory aging in the PG system is less severe than the aging experienced during asphalt mix production.
- More reliable tests can be added to the current acceptance tests to better predict asphalt pavement performance.

1.3 Objectives of the Research

The overall objective of this research is to provide a framework to better characterize the asphalt binder and asphalt mixes with RAP with field verified test methods and parameters linked to pavement performance. The framework will be developed through these specific objectives:

- Evaluate the inter-laboratory standard deviation of the test methods utilized for acceptance of asphalt binders in Ontario.
- Compare the oxidative aging that is simulated by the laboratory conditioning, to the short-term aging that occurs in the field.
- Evaluate the asphalt binders and mixes using more recent test methods that have been shown to predict performance.

1.4 Methodology of Study

There are three parts of this research: (1) Comparing testing variability produced when testing tank asphalt binder and recovered asphalt from plant-produced asphalt mixes; (2) Comparing the aging that is simulated through laboratory conditioning, to the short-term aging that occurs in the asphalt binder during mix production; and (3) Evaluating more recent test methods for asphalt binder and asphalt mix that have been demonstrated to field performance. Each part is elaborated below:

1.4.1 Inter-Laboratory Study

The first part of the research is an Inter-Laboratory Study (ILS) to compare testing variability of the results of tank asphalt (shown as Sample 'B' in Figure 1-1) and results of the recovered asphalt from plant-produced asphalt mixes sampled from the paving site (shown as Sample 'D' in Figure 1-1).

The ILS is intended to help compare the reproducibility of testing conducted on tank asphalt and recovered asphalt. Reproducibility concerns the variability between independent test results obtained in different laboratories, each of which has applied the test method to test specimens taken at random from a single quantity of homogenous material obtained for the ILS. (Heyes, 1993)

To conduct the ILS, samples of asphalt binder and asphalt mixes were collected from supplier terminals, hot mix asphalt production plants, and paving sites in southern Ontario and stored by Aecon Materials Engineering. Members of the Ontario Asphalt Pavement Council donated asphalt cement and asphalt mixes to be included in the research.



Figure 1-1: Visual representation of asphalt cement and mix sampling locations

Five industry testing labs volunteered to participate in the ILS. Within three months of sampling the materials, the testing labs received seven sets of asphalt materials that included (1) asphalt binder sampled from the asphalt plant (referred to as tank asphalt); and (2) the respective plant produced asphalt mixes sampled from the paving site.

Participating labs received instructions to determine material properties of the tank asphalt following the appropriate test method identified in Table 1-1, and to follow MTO standards and specifications for extraction and recovery of asphalt from the plant produced asphalt mix (referred to as recovered asphalt) and determine the material properties. It is important to note that the test methods marked with an asterisk (*) in Table 1-1 have not been evaluated through an ILS for recovered asphalt until this research was conducted. In addition, the Extended Bending Beam Rheometer (ExBRR) test and Double Edge Notched Tension (DENT) test are unique to MTO and not utilized by other agencies in Canada, however it also has not been previously evaluated in an ILS and is being utilized by municipalities in Ontario.

Description	Test Method
Quantitative Extraction of Asphalt Cement and Analysis of Extracted	MTO LS 282
Aggregate	
Recovery of Asphalt from Solution by Abson or Rotary Evaporator	MTO LS 284
*Ash Content	MTO LS 227
Grading or Verifying the Performance Grade of an Asphalt Binder	AASHTO R 29
*Multiple Stress Creep Recovery Test of Asphalt Binder Using a	AASHTO T 350
Dynamic Shear Rheometer (DSR)	
*Performance Grade of Physically Aged Asphalt Cement using	MTO LS 308
Extended Bending Beam Rheometer (ExBBR)	
*Asphalt Cement's Resistance to Ductile Failure Using Double Edge	MTO LS 299
Notched Tension (DENT) Test	
*Accelerated Aging of Asphalt using Pressure Aging Vessel Protocols	MTO LS 228

Table 1-1: Test Methods for Interlaboratory Study

* Test methods that have not been previously evaluated through an ILS until this research.

1.4.2 Laboratory Aging versus Field Aging

As part of the ILS, participating labs collected and saved samples of the tank asphalt and recovered asphalt residue at the different stages of laboratory aging. The purpose of this was to address the second part of this research: to compare the aging that is simulated in the lab, to the short-term aging that occurs in the asphalt binder during mix production.

Short-term aging of asphalt binder in road construction is aging that occurs during mix production at the asphalt plant, transportation to site and placement of the mix. It is characterized by high temperatures and fast oxidation. Long-term aging refers to aging of the asphalt during the in-service life of a pavement layer. This is a slower oxidation process and includes the effects of ultraviolet radiation. (Anderson, 2007)

Asphalt is an organic material composed of many complex molecules, which can be grouped into four main fractions of increasing polarity: saturates, aromatics, resins, and asphaltenes. (Boysen & Schabron, 2015; Petersen, 2009) Since asphalt is organic in nature, it reacts with atmospheric oxygen, resulting in oxidation or aging of the asphalt. This aging process changes the concentrations of the asphalt fractions resulting in an overall increased concentration of the asphaltenes fraction. Increasing concentrations of asphaltenes during oxidation changes the microstructure, and thus the mechanical behavior of the asphalt over time. (Hofko & Hospodka, 2016)

To simulate aging and its effect on the asphalt properties, two accelerated conditioning procedures are used in the Superpave PG system: the rolling thin film oven (RTFO) test for short-term aging,

intended to simulate aging during production and placement of asphalt mix; and the pressure aging vessel (PAV) for accelerated longer-term aging, which is intended to simulate in-service aging of the asphalt after several years. (Anderson, 2007)

These aging protocols have been shown to not correlate with field aging. (Galal & White, 1997) As such, a chemical analysis is conducted in this study to determine the concentrations of the asphalt fractions at various stages of laboratory conditioning with the RTFO and PAV, then compared with the asphalt fractions of recovered asphalt that is short term aged in the field. The concentrations of the fractions are used to determine an aging index as an indication of degree of aging for laboratory versus field.

1.4.3 Alternative Test Methods

Asphalt mix is susceptible to several types of distresses during its service life, such as fatigue cracking, rutting, and thermal cracking. Typically, majority of these distresses are a result of repeated loading (fatigue) from traffic vehicles in combination with freezing and thawing cycles associated with temperature variations throughout the seasons of the year. The presence of these distresses directly and severely compromises the overall structural and functional performance of the pavement, and consequently diminishes the service life and ride quality of roads.

Among these asphalt pavement distresses, fatigue cracking is the most critical because once it occurs, it may lead to rapid pavement structure deterioration and severely reduced ride quality. Thus, to mitigate this fatigue cracking, it is imperative to explore and characterize the complex fracture mechanics behind crack initiation and propagation in AC mixtures and extract fracture parameters to serve in the selection of better suited mixtures to resist cracking/fracture.

Currently the PG system uses a series of tests through the dynamic shear rheometer (DSR) and bending beam rheometer (BBR) and specifies that a particular asphalt binder must pass these tests at specific temperatures that are dependent upon the specific climatic conditions in the location of the project. In this way, a binder used in Southern Ontario would have different properties than one used in Northern Ontario or the Alaskan tundra. This concept is not new and was used in the penetration and viscosity graded systems, but the relationships between asphalt binder properties and conditions of use are more complete and more precise with the Superpave PG system.

Although the PG system is an improvement over the penetration and viscosity grading systems, the PG systems evaluates cracking behavior by only considering properties of asphalt binder, however,

fails to consider the aggregate portion of the asphalt mixes, which makes up about 90 to 95% of the total weight of the asphalt mix.

To address this, a parameter, and two tests proposed in this research to be added to the current acceptance specifications, to ensure the appropriate performance and the required service life are achieved. The proposed parameter is Delta Tc (ΔT_c), and the tests are Illinois Flexibility Index Test, and Asphalt Mix Performance Tester.

1.4.3.1 Delta Tc

The Delta Tc (ΔT_c) parameter was developed by Mike Anderson as part of a research project involving airfield asphalt pavements, to evaluate the relationship between asphalt binder properties and nonload related cracking. (Blankenship et al., 2010) The study relied on past research that showed some relationship between ductility (related to flexibility) and the durability of an asphalt pavement. Ductility is an asphalt binder's ability to be stretched without breaking. ΔT_c is calculated using values from the Bending Beam Rheometer (BBR) test by subtracting the BBR m-critical temperature from the BBR stiffness-critical temperature:

$\Delta T_{c} = (T_{s\text{-critical}} - T_{m\text{-critical}})$

The critical temperatures, T_{s-critical} and T_{m-critical}, are the temperatures at which the stiffness (S) and mvalue (m) specification requirements are met (i.e., S=300 MPa, m-value=0.300) respectively. They can be determined following ASTM D7643, Standard Practice for Determining the Continuous Grades for PG Graded Asphalt Binders; or AASHTO R29, Standard Practice for Grading or Verifying the Performance Grade (PG) of an Asphalt Binder.

 ΔT_c is intended to provide an indication of loss of ductility: when the asphalt binder cannot relax the stresses fast enough to prevent breaking. As asphalt binders oxidize and age, their ability to relax stresses at low temperatures diminishes. This would be captured in the BBR m-value and result in a higher (less negative) $T_{m-critical}$.

The ΔT_c of an aged binder would be more negative, than that of an unaged binder, and would be more likely to exhibit the non-load related pavement distresses. This has generated a lot of interest in using ΔT_c to characterize asphalt mix containing RAP, due to the contribution of highly oxidized asphalt from these recycled materials.

1.4.3.2 Illinois Flexibility Index Test

The Semi-Circular Bending (SCB) Test was developed as a fracture test to characterize the lowtemperature cracking resistance of asphalt mixtures to differentiate mixtures whose service life might be compromised by cracking. The SCB test method is generally valid for specimens that are tested at temperatures of 10 °C or below. (ASTM, 2020) The University of Illinois modified the SCB procedure, called the Illinois Flexibility Index Test (I-FIT), based on a thorough investigation of test temperatures, loading rates and sample geometry to quantify the cracking potential of asphalt mixtures at intermediate temperatures. (Albritton et al., 1999)

The I-FIT test, pictured in Figure 1-2, quantifies the cracking resistance of asphalt mixtures using a Flexibility Index (FI), which includes the fracture energy and post-peak behavior of asphalt mix. Researchers have found that the I-FIT shows consistent and repeatable trends for changes in asphalt mix design properties, and the FI parameter is shown to provide a greater distinction between mixtures' fracture properties relative to the total fracture energy parameter alone. As such, the I-FIT test is considered ready for implementation in Wisconsin, as a reliable test to identify the cracking potential of asphalt mixtures at intermediate temperatures. (Batioja-Alvarez et al., 2019; Illinois-DOT, 2016; Ling et al., 2017) The I-FIT test is included in this study to characterize the asphalt mix cracking resistance at intermediate temperatures.



Figure 1-2: I-FIT test setup and example plot of results (Illinois-DOT, 2016)

1.4.3.3 Flow Number with Asphalt Mix Performance Test

Flow Number is a parameter determined through the Asphalt Mixture Performance Tester (AMPT), which is designed to measure the engineering properties of asphalt mixes (Figure 1-3). The AMPT applies a frequency sweep of uniaxial, compressive, sinusoidal loading, to obtain the mix stiffness at various temperatures. The data can be used to characterize mix performance with respect to permanent deformation (rutting) through the Flow Number (FN) parameter. (AASHTO, 2015; Witczak et al., 2002)



Figure 1-3: Setup of AMPT for testing asphalt mixes

To determine the flow number is of the asphalt mix, an asphalt sample is subjected to repeated cyclic axial loading, then the cumulative permanent deformation is measured as a function of the number of load cycles. The test is performed at a high pavement temperature representative of the project location and pavement layer depth to evaluate the rutting resistance of the asphalt mixtures. The FN gives the number of load cycles corresponding to the minimum rate of change of permanent axial strain during a repeated load test. (Farcaş, 2012)

1.5 Thesis Organization

The thesis is divided into five chapters. Chapter One introduces the research study. Chapter Two outlines the literature review undertaken to frame the research in the current state of the practice. Chapter Three outlines the methodology for the research. Chapter Four discusses the results of the research, including the findings from the ILS, the chemical analysis comparing laboratory aging and field aging, and results from the alternate test methods proposed for addition to acceptance of asphalt binder and asphalt mixes. Chapter Five summarizes the findings and provides recommendations for future work.

2 LITERATURE REVIEW

This chapter summarizes the relevant literature for testing recovered asphalt, asphalt cement aging in the laboratory versus field, and new test methods and parameters for asphalt binder and asphalt mix that have been correlated to field performance.

2.1 Recovered Asphalt Testing

The process of determining the material properties of a recovered asphalt starts with extracting that asphalt binder from asphalt mix, followed by recovering of the binder for testing. The extraction can be performed through a few procedures; however, some are more commonly used than others. Asphalt binder recovery seems to have been practiced since the early 1900's, as laboratory operators have always desired to determine the effect of plant mixing operation on the physical characteristics of asphalt binder. (Abson, 1933)

The ASTM approved the standard D2172 for extraction of asphalt from asphalt mixtures in 1963. (ASTM, 2017) The standard provided various methods of asphalt extraction from asphalt mixtures, and each method required the use of a reagent such as trichloroethylene (TCE) or methylene chloride, with an extraction equipment, the common ones being: centrifuge (Method A), reflux extractor (Method B), or vacuum extractor (Method C) as illustrated in Figure 2-1.



Figure 2-1: Asphalt cement extractor, from left to right: centrifuge (Method A), reflux extractor (Method B), vacuum extractor (Method C)

In centrifuge extraction (Method A), the asphalt mixture is placed in an extraction bowl with solvent. After the solvent breaks down the mixture, the effluent is drained with a centrifuge that revolves up to a speed of 3600 revolutions/min, until there is no solvent draining from the machine (Figure 2-1). The process is repeated with the addition of more solvent and centrifuging, while the effluent is collected into a beaker.

In reflux extraction (Method B), hot plates heat the solvent to generate solvent vapor that passes through and around the asphalt mixture sample contained in wire mesh cones lined with filter paper. Reflux solvent condensing on a water-cooled condenser, percolates through the asphalt mixture until the solvent flowing from the lower cone is a light straw color.

When the vacuum extractor (Method C) is used, solvent is added to the asphalt mixture and allowed to dissolve in a stainless-steel beaker. The effluent is then poured into the vacuum extractor which uses suction to pull asphalt-solvent slurry through a filtration bed. More solvent is added to the mixture and stirred, then the process is repeated until the solution is a light straw color, and the aggregate is visually clean.

The effluent that is collect through extraction is a mixture of solvent, asphalt binder, and mineral fines from the aggregates used in producing the asphalt mix. The process of collecting the asphalt binder component is referred to as asphalt recovery, conducted through the Abson or the Rotary evaporator recovery methods.

The Abson recovery method was approved by ASTM as a standard in 1961. (ASTM, 2015) The method uses ordinary distillation with the application of heat, modified by bubbling carbon dioxide (CO_2) through the dissolved asphalt (Figure 2-2). The CO_2 provides both a reduction in partial pressure and mechanical agitation, making it easier to remove the solvent at lower temperatures of 300°F to 325°F (149°C to 163°C).



Figure 2-2: Abson recovery method apparatus

Even though Gene Abson, showed through his research that complete solvent removal was possible with his Abson recovery method, many researchers were showing results from their studies where residual solvent was affecting their test results. Studies of asphalt binder recovered from hot mix asphalt samples showed negative hardening when one would expect more hardening due to oxidation. The conclusions from these studies where that the negative hardening was the result of residual solvent in the recovered binders. (Abson & Burton, 1960; Davis, 1983)

The Rotary evaporation method become common for asphalt recovery in the mid-1970's. In this method the extracted effluent is placed in a distillation flask which rotates in a heated oil bath. (Figure 2-3) A partial vacuum is applied to the system along with a flow of nitrogen or CO₂ gas to remove the solvent. (ASTM, 1993)



Figure 2-3: Rotary Evaporator apparatus

More recently, the AASHTO officials approved a standard in 2003 for a new asphalt extraction and recovery method for the Strategic Highway Research Program (SHRP). The standard addressed many of the challenges associated with the procedures at the time. In the SHRP procedure the extraction is performed in a rotating drum to allow more contact of the solvent with the asphalt mixture. The extract is vacuum filtered through a two-stage filtering process to remove most of the aggregate fines before being transferred to a rotary evaporator where the solvent is vacuum distilled. The same research also investigated the centrifuge method but modified it with the use of toluene as a replacement for TCE and added 15 percent ethanol (EtOH) to the solvent in late washes This was referred to it as Modified Method A which was found to provide better asphalt samples that were

more comparable to the asphalt in the original mixture, with "properties that are nearly unaltered during extraction." (Burr et al., 1993)

A survey published in 2017 with government agencies and research laboratories in the United States, Canada and Europe received 40 responses, the majority of which were from United States Departments of Transportation, found that the centrifuge was the most common extraction method used, the rotary evaporator was the most common method of recovery, and TCE was found to be the most common solvent. (Mikhailenko & Baaj, 2017) A follow up literature review published in 2019 found that most commonly used solvents were n-propyl bromide (nPB) and chlorinated solvents, due to their ability to be reusable, however both had reported issues of ineffectiveness as well as major concerns about user safety. (Mikhailenko et al., 2019)

Improvements were also made to the Rotary Evaporator method to enhance its effectiveness during these times. The Rotary Evaporator method of recovery specified the use of TCE, methylene chloride, or nPB as reagents for extraction. Research done by Asphalt Institute used toluene as a primary solvent for extraction in combination with the Rotary Evaporator. The temperature of the oil bath was increased by approximately 10°C since toluene has a higher boiling point, however a benefit to the change was that toluene does not have 1,2-epoxybutane as a stabilizer. This stabilizer can cause problems with asphalts containing acids during the extraction process, which will affect the properties of the recovered binder. Asphalt Institute advanced the procedure changes to ASTM, and it was approved as ASTM D7906 in 2014. (ASTM, 2014)

During the early 2000's, there was also a change in the asphalt industry with an increasing use of polymer modified asphalts to meet the demands for more robust asphalt pavements to withstand increasing traffic loads. To increase resistance to permanent deformation (rutting) and cracking, asphalt is modified with different polymers to enhance its elastic properties. The extraction and recovery procedures and technologies used then, and still today, were developed when the asphalt industry was only using paving grade (unmodified) asphalts. Thus, research investigating the effect of these procedures on the polymer modified asphalts after extraction and recovery is limited.

Some studies have been conducted in Europe with differing results. A study conducted in Belgium evaluated three different highly modified binders, prepared with linear and radial SBS polymers, and three different solvents: methylene chloride, TCE, and toluene. They performed rheological tests over a temperature range of -10°C to 90°C and found that some of the polymer modified asphalts lost their

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elasticity after recovery. (Nosler et al., 2008) The researcher confirmed this by looking at the molecular weights of the polymers before and after recovery through gel permeation chromatography (GPC) and found that even by simply dissolving and recovering the polymer modified asphalt, changes occurred to the molecular weight that explained the decreased elasticity noted from the rheological tests. (Nosler et al., 2008)

In another study, the Belgian Road Research Centre researched both the effect of extraction on the binder content as well as the effect of extraction and recovery on the properties of the recovered polymer modified asphalt. They evaluated several solvents and 19 polymer modified binders. They found the results in deviations were within the limits of repeatability for dichloromethane and toluene solvents however, found that TCE resulted in higher changes. (Pierard et al., 2010)

A recent study from Switzerland investigated five polymer modified asphalts available on the Swiss market and four different solvents (toluene, xylene, dichloromethane, tetrachloroethylene). They found that in most cases the properties of the recovered binders showed little difference with respect to penetration and elastic recovery for all solvents, except with dichloromethane. They did not find any correlation between polymer dispersion and rheological properties but did note that the number of polymer-modified asphalt samples included in the project was not sufficient to draw final conclusions. (Hugener & Pittet, 2016) It is evident that more research is needed in this area, especially within North America.

2.2 Measurement Uncertainty

Tests performed on presumably identical materials and in presumably identical circumstances don't always yield identical results, and this is due to unavoidable random errors inherent in every test procedure. (Heyes, 1993) For this reason, it is important when interpreting test data, to take this inherent variability into account. For example, if the difference between a test result and some specified value is within the expected deviation due to random errors, then a real deviation from the specified value has not been demonstrated.

Factors that may contribute to the variability in a test procedure the operator, the equipment used, the calibration of the equipment, and the environment (temperature, humidity, air pollution, etc.). With this reasoning, it is expected that the variability between test results obtained by different operators or with different equipment will usually be greater than between test results obtained by a single operator using the same equipment. (Heyes, 1993)

When reviewing test results the average or mean of the test data shows where most of the data points lie, whereas variability summarizes how far apart the data points are. This is important because the amount of variability determines how well one can generalize results from the sample to the population. Low variability is ideal because it means that one can better predict information about the population based on sample data. High variability means that the values are less consistent, so it's harder to make predictions.

One way to measure variability is by calculating the standard deviation of a data set, which is a measure of how spread apart the data points or test results are from the mean. The standard deviation is also a measure of a test's precision. This means the higher the standard deviation, the more variable the data set is. As such a lower standard deviation is desired. The equation of standard deviation is expressed as follows:

$$SD = \sqrt{\frac{\sum (X - \overline{x})^2}{n - 1}}$$

Where: SD = sample standard deviation Σ = the sum of X = each value/test data \overline{x} = sample mean/average

n = number of values in the sample

It's important to evaluate both the mean and standard deviation because some data sets can have the same mean, but different levels of variability or vice versa. Calculation of both the mean and the variability together gives a more complete picture of the data.

Another way to assess variability is by calculating the coefficient of variation (COV). COV is a normalized measure of the dispersion of a probability distribution in statistics and probability theory. It is calculated as the ratio of the standard deviation to the mean:

$$COV = \frac{SD}{\overline{x}}$$

Where:

SD = sample standard deviation \overline{x} = sample mean/average

COV is commonly used to test the accuracy of a test method and is beneficial for making comparisons between two different data sets. A low COV (less than 10) is desirable, and a higher COV (greater than 30) is considered as a higher rate of dispersion around mean value.

Test standards typically include precision and bias statements where the precision is measured in terms of repeatability and reproducibility. Repeatability refers to the single-operator precision or intra-laboratory precision, and is the precision of test results that are "obtained with the same test method in the same laboratory by the same operator with the same equipment in the shortest practicable period of time using test specimens taken at random from a single quantity of homogenous material." (ASTM C670, 2015) Reproducibility refers to the multi-laboratory precision or interlaboratory precision and is the precision of "test results obtained in different laboratories" using the same test method "on random test units from the same lot of homogeneous material." (ASTM C670, 2015) Both reproducibility and repeatability are calculated as a standard deviation of test results, and they establish upper and lower limits for the precision of a test method.

Bias is also known as systematic error, is defined as the "error inherent in the test method that contributes to the difference between a population mean of test results and the accepted reference or true value." (ASTM E177, 2014) Bias results from any systematic process in which there is a distortion of the measured value from the true value. Since it is the result of a systematic process, any amount of bias in the measurements are predictable and consistent. This contrasts with random error (precision), which is unpredictable and is typically due to interpolation, human error, and other environmental factors. Without precision and bias statements, test standards do not have much merit, this could be due to not having enough laboratory data available to determine precision and bias.

2.3 Aging of Asphalt Cement Aging in the Laboratory versus in the Field

Since asphalt is an organic material, its properties change with time in service through thermal and oxidative aging. Since this change occurs over time, the physical properties of the asphalt cement as determined on the original sample will not be representative of the physical properties of the asphalt at the various stages of the pavement service life. Therefore, for asphalt properties to be more relevant to in-service performance, various conditioning or aging procedures can be utilized:

2.3.1 Natural Weather Aging

One of the ways to age asphalt is through natural weather aging, where the asphalt is directly exposed to the natural environment, while observing the aging phenomenon frequently, as illustrated in Figure 2-4. (Terrenzio et al., 1997) Other methods were researched by Feng et al. (Feng et al., 2017), who placed 50-gram asphalt samples in RTFO pans then placed in humid subtropical monsoon climate for one year to study the aging conditions. They conducted tests on the asphalt samples and documented observations after every three months of exposure.

In another study conducted by Wang et al., the researchers prepared asphalt samples to have a thickness of 0.5 mm. The samples were subjected to subtropical monsoon climate to investigate the aging phenomenon within 8 months. (Feng et al., 2017) It is obvious that these methods, although most representative and relatively easy for application, they require a long time to finish the test, and thus making them impractical for adoption into specifications.



Figure 2-4: Conditions of natural weather aging (Terrenzio et al., 1997)



Figure 2-5: Conditions of natural weather aging (Feng et al., 2017)

2.3.2 Accelerated Weather Aging

Accelerated weather aging is a method developed to solve time consuming problems of natural weather aging methods. It is important to recognize that accelerated weather aging methods need to simulate the condition of natural weather exposure and provide an accurate and efficient aging simulation. (Menapace et al., 2016)

Accelerated aging equipment have been developed to achieve this purpose. The key feature of these machines is the involvement of ultraviolet (UV) that is typically neglected by the traditional methods. UV is a key component of the natural sunlight that has been verified to have significant impact on the aging of the asphalt. (Yu et al., 2018) Typical accelerated weathering machines can be seen in Figure 2-6.



Figure 2-6: Accelerated weather aging devices: (a) Accelerated Weathering Machine; (b) Accelerated weathering tester; (Menapace et al., 2016) (c) UV aging oven (Zhu et al., 2018)

2.3.3 In-Field Aging

In-field aging is the most authentic and straightforward method for testing of asphalt aging. (Rasool et al., 2018) It uses a combination of field and laboratory testing to evaluate field aging of asphalt cement. The samples are obtained from asphalt pavement sections in service for any period, through coring or saw cutting. The asphalt binder is then extracted with solvent and recovered for testing. Field aging is difficult to establish, however, because of several drawbacks, such as destructive, time-consuming testing, traffic disturbance, and multiple uncontrollable variables.

2.3.4 Aging in Superpave Performance Grading System

The Superpave performance grading (PG) system addresses aging through two accelerated conditioning procedures: the rolling thin film oven (RTFO) for short-term aging, and the pressure aging vessel (PAV) for accelerated long-term aging.

Short-term aging refers to the changing of the characteristics during asphalt mix production and placement that happens within several hours. The asphalt mix production process requires high temperatures, which leads to fast oxidative aging of the asphalt binder, with evaporation of the lighter fractions, sometimes referred to as volatiles, from the asphalt binder. (Anderson, 2011)

Long-term aging is caused by the slow oxidation process during service in the field. Atmospheric oxygen and other oxidant gases in the field cause slow aging phenomenon of asphalt in the long term. Over time, with the drastic changing of temperatures, traffic volumes, and other environmental conditions, the asphalt binder contained in the pavement structure becomes stiffer and more brittle, which results in pavement damage such as low-temperature cracking, fatigue cracking, and even worse, permanent structure failure. Oxidative aging is one of the critical factors contributing to asphalt pavements performance. (Boysen & Schabron, 2015; Petersen, 2009)

2.3.4.1 Rolling Thin Film Oven Test

During asphalt mix production, hot asphalt binder is mixed with heated aggregates, and a thin film (typically less than 10μ m) of asphalt coats the aggregates. The thin film of asphalt is exposed to elevated temperatures and air during the mixing process and cause oxidation to occur at a faster rate. (Anderson, 2007) The RTFO test is used to simulate the effect of heat and air on a moving thin film of asphalt. To perform this test, a small sample of asphalt cement in sample bottles are placed in a rotating carriage in an oven for a set time. Hot jet air blows into the sample bottle as it passes the bottom position of the carriage as illustrated in Figure 2-7. This process is considered as the short-term aging, and it represents the aging that occurs during asphalt mix production and placement on the job site. (AASHTO, 2004b)

After RTFO aging in the PG grading system, tests are performed to characterize the asphalt binder's early age performance such as rutting, and/or additional aging is conducted to characterize its long-term in-service performance. (AASHTO, 2012b, 2016)

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Figure 2-7: Rolling Thin Film Test

2.3.4.2 Pressure Aging Vessel

In the PAV, additional aging is achieved by subjecting the RTFO-aged binder to high pressures and temperatures for accelerated aging of the binder. RTFO-aged binder is poured into PAV sample pans and loaded into the sample rack as shown in Figure 2-8. The PAV is designed to provide accelerated aging to simulate the in-service oxidative aging, and "may be used to estimate the physical or chemical properties of asphalt binders 5 to 10 years of in-service aging in the field." (AASHTO, 2012a) It is worth noting that the AASHTO procedure also states that there is no correlation between the aging time and temperature in the PAV and in-service pavement age and temperature. Asphalt Institute manual suggests that the PAV provides accelerated aging of the asphalt, to simulate up to seven years of in-service aging. (Anderson, 2007)



Figure 2-8: Pressure Aging Vessel

Research conducted at Perdue University by Galal and White that compared the relation between PAV aged asphalt cement to asphalt recovered from field cores of eight-year-old pavements, showed that the PAV aged asphalt properties did not correspond to the properties of the asphalt recovered from the field cores. (Galal & White, 1997) Other research has showed the RTFO to have difficulties simulating oxidative aging for highly viscous (i.e., polymer-modified) binders because they do not flow properly in the bottles as they are rotated. (Angius et al., 2018; Yan et al., 2017)

In another study, the Bituminous Section of the Materials Engineering Research Office (MERO) of the MTO carried out field trials starting in 2003 to evaluate how well the standard PG grading system correlated with field performance. The study looked at correlation (R²) values for tested asphalt samples, with respect to transverse cracking. The correlation values are summarized in Table 2-1 for tank asphalt collected during the original construction in 2009, and recovered asphalt extracted from cores taken from the surface mixes in 2008, five years after construction. (Huber et al., 2012)

Test Method	Property Measured	R ² to Thermal Cracking Tank asphalt	R ² to Thermal Cracking Recovered asphalt (5 years)
AASHTO M 320	Low Temperature Grade	0.001	0.81
		(No correlation)	(Strong correlation)
ExBBR	Grade Loss	0.55	0.83
		(Some correlation)	(Strong correlation)
DENT	Ductility	0.17	0.39
		(Poor correlation)	(Poor Correlation)

Table 2-1: Correlation of test for characterizing low temperature properties of Virgin AC

Note: ExBBR is Extended Bending Beam Rheometer Test discussed in Chapter 3. DENT is Double-Edge-Notched Tension Test discussed in Chapter 3.

MTO's study concluded that since properties of the recovered asphalt from field cores showed better correlation to transverse cracking, a pavement distresses associated with low temperature performance of the asphalt binder, the poor correlation of the PG grading system for low temperature properties can be improved if field aging can be better replicated in the lab. (Huber et al., 2012)

In addition to the above-described standard laboratory aging methods, researchers also adopted other methods to study the aging of the asphalt cement through natural weather aging method, accelerated aging devices, and field aging methods. Significant difference of these methods between
the standard laboratory aging methods is the role of sunlight that brings large amount of aging through photo-oxidative aging.

2.4 New Methods for Evaluating Asphalt Binder

In recent years, with the fast emergence of new technologies, studies on asphalt have also expanded from macroscopic physical properties to microstructure. Several novel test methods have been invented in material science and applied to test asphalt materials more accurately. Generally, these new methods can be grouped into three categories, which are: 1) Spectral analysis-based methods, such as Spectrophotometry method (Hou et al., 2018), Fluorescence spectroscopy (Arnold & Shastry, 2015) and Fourier transform infrared spectroscopy (FTIR). (Shi et al., 2015); 2) Microscopy based methods, such as atom force microscopy method (Loeber et al., 1996), fluorescence microscopy method (Ding et al., 2018); 3) Chromatography based methods, such as gel permeation chromatography method (Meng et al., 2020). Table 2-2 is a general summary and descriptions of these new aging test methods.

Testing methods	Description	Category
Spectrophotometry	An optical test method developed based	Spectral analysis-
	on colorimetric method. Colorimetry is	based methods
	used to determine a specific component	
	content by comparing the depth of the	
	solution colour.	
Fluorescence	Using X-ray Fluorescence Spectroscopy	
Spectroscopy	(XRF) to measure the contents of asphalt	
FTIR Spectroscopy	Method to study the spectra change of the	
	asphalt components before and after	
	aging	
Atomic Force	Use atomic force microscopy to visualize	Microscopy based
Microscopy (AFM)	the components changing of the asphalt	methods
	before and after aging	
Fluorescence	Use fluorescence microscopy to visualize	
Microscopy	the components changing of the bitumen	
	before and after aging	
Gel permeation	Method mainly used for the	Chromatography
chromatography	characterization of the three species of	based methods
	polymer-modified asphalt cements:	
	asphaltenes, maltenes and polymer	

 Table 2-2: Summary of new methods for asphalt aging test

Figure 2-9 is the 3D and 2D AFM images of unaged base (or neat) asphalt, PAV aged base asphalt, unaged polymer modified binder, and PAV aged polymer modified binder. Obvious changes of asphalt components can be observed from these images. Meanwhile, Figure 2-10 shows the fluorescent images of asphalt binder of unaged, RTFO aged and PAV aged samples for polymer modified (i.e., SBS-70 and SBS-90). Figure 2-11 is the fluorescent images of SBS-70 with weather aging times of 0, 3, 6, 9, and 12 months. Significant changes of asphalt characteristics can be observed with the help of these new technologies, thereby more accurate investigations can be conducted.



Figure 2-9: 3D and 2D AFM images: (a) unaged base asphalt; (b) PAV aged base asphalt; (c) unaged SBS modified binder; (d) PAV aged SBS modified binder (Wu et al., 2009)



Figure 2-10: Fluorescent images of asphalt binder: (a) unaged; (b)RTFO; (c) PAV; first row SBS-70; second row: SBS-90 (Zhu et al., 2018)



Figure 2-11: Fluorescent images of SBS-70 with different weather aging times: (a)0 month, (b) 3 months, (c) 6 months, (d) 9 months, (e) 12 months (Zhu et al., 2018)

Additionally, the National Cooperative Research Program (NCHRP) completed projects related to paving grade asphalt binder testing and specifications that are currently in draft stage in review for publication. The first project of relevance is NCHRP 09-59, Relating Asphalt Binder Fatigue Properties to Asphalt Mixture Fatigue Performance. The objectives of this research were to: (1) determine asphalt binder properties that are significant indicators of the fatigue performance of asphalt mixtures; (2) identify or develop one or more practical, implementable binder tests to measure those

properties for use in a performance-related binder purchase specification such as AASHTO M 320 and M 332; (3) propose necessary changes to existing AASHTO specifications to incorporate the identified binder properties and their specification limits; and (4) validate the binder fatigue properties, test(s), and changes to existing and/or proposed AASHTO test methods and specifications with data from field projects and accelerated loading facilities, supplemented with data from laboratory experiments. (Christensen & Tran, 2021)

The second project of relevance is NCHRP 09-60, Addressing Impacts of Changes in Asphalt Binder Formulation and Manufacture on Pavement Performance through Changes in Asphalt Binder Specifications. The objectives of this research were to propose changes to the current performancegraded (PG) asphalt binder specifications, tests, and practices to remedy gaps and shortcomings related to the premature loss of asphalt pavement durability in the form of cracking and raveling. The draft report is recommending the inclusion of Δ Tc in AASHTO M 320 and M 332 for PAV-aged asphalt binder as an added parameter for durability and relaxation. There is a proposed specification criterion established for PAV-aged asphalt binder (standard 20-hour PAV aging). (Planche et al., 2018)

Lastly NCHRP 09-61, Short- and Long-Term Binder Aging Methods to Accurately Reflect Aging in Asphalt Mixtures had the objectives to: (1) develop practical laboratory aging methods to accurately simulate the short-term (from production to placement) and long-term (in-service) aging of asphalt binders; and (2) determine the relationship between different methods of laboratory aging of asphalt binders and the actual aging that occurs during mixture production, transport, and placement as well as during the service life of the pavement structure. (Bonaquist et al., 2021)

2.5 New Methods for Evaluating Asphalt Mixes

With respect to characterization of asphalt mixes, National Cooperative Highway Research Program (NCHRP) 20-07/Task 406 report highlights various concerns and gaps identified by government agencies in using the current volumetric properties approach in the Superpave methodology to predict pavement distresses. In 2015 The Federal Highway Administration (FHWA) formed a Balanced Mix Design (BMD) Task force that defined BMD as Asphalt mix Design using performance tests on appropriately conditioned specimens that address multiple modes of distress. (West et al., 2018)

A survey was conducted to collect data from 50 government agencies, contractors, and consultants on their current design procedures and performance testing to create a framework. The task group proposed three potential preliminary BMD methods to help introduce performance testing to counter distresses in pavements based on the data. The most important distresses identified by agencies were: Thermal Cracking; Reflection Cracking; Fatigue Cracking; Longitudinal Cracking; and Raveling.

Further surveys were conducted in different states on current performance tests in use or intended to use, infrastructure, and willingness to adopt new framework. As per the data collected following three different approaches were suggested as preliminary BMDs based on performance testing:

- 1. Volumetric Design with Performance Verification
- 2. Performance-Modified Volumetric Mix Design
- 3. Performance Design.

Approach 1 - Volumetric Design with Performance Verification. This approach starts with the current Superpave mix design method for determining an optimum asphalt binder content. The mixture is then tested with selected performance tests to assess its resistance to rutting, cracking, and moisture damage at the optimum binder content. If the mix design meets the performance test criteria, the JMF is established and production begins; otherwise, the entire mix design process is repeated using different materials (e.g., aggregates, asphalt binders, recycled materials, and additives) or mix proportions until all the performance criteria are satisfied.

Approach 2 - Performance-Modified Volumetric Mix Design. This approach begins with the Superpave mix design method to establish a preliminary aggregate structure and binder content. The performance test results are then used to adjust either the preliminary binder content or mix component properties or proportions (e.g., aggregates, asphalt binders, recycled materials, and additives) until the performance criteria are satisfied. For this approach, the final design is primarily

focused on meeting performance test criteria and may not be required to meet all the Superpave volumetric criteria.

Approach 3 - Performance Design. This approach establishes and adjusts mixture components and proportions based on performance analysis with limited or no requirements for volumetric properties. Minimum requirements may be set for asphalt binder and aggregate properties. Once the laboratory test results meet the performance criteria, the mixture volumetrics maybe checked for use in production.

Regardless of which approach is utilized, agencies have a list of test methods to select from based on the type of distress to be characterized, rutting, cracking as well as tests for accessing moisture susceptibility. Table 2-3 provides an overview of asphalt mixture performance tests that are commonly used in asphalt research for characterizing rutting and cracking and are being considered for implementation by owner agencies.

Name of Test	Test Method	Description
Asphalt Pavement Analyzer	AASHTO T 340-	The APA tracks a loaded wheel back and
(APA)	10	forth across a pressurized linear hose over an asphalt mixture sample to characterize permanent deformation or rutting of the asphalt mix. A temperature chamber is used to control the test temperature. Rut depths along the wheel path are measured for each wheel pass. The sample is typically loaded for 8,000-wheel passes.
Flow Number Test	AASHTO TO 79- 15	The Flow Number test is conducted by applying repeated haversine axial compressive loads to a cylinder specimen at a specific test temperature. For each load cycle, the recoverable strain and permanent strain are recorded. The flow number used to access the rutting potential of an asphalt mix and is determined as the number of load cycles corresponding to the minimum rate of change of permanent strain (i.e., onset of tertiary flow).

Table 2-3: Example Rutting	& Cracking Performance	Tests for Asphalt Mixes	(West et al., 2018)
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Hamburg Wheel-Tracking Test	AASHTO T 324-	HWT is like the APA in that the asphalt mix
(HWT)	14	is subjected to repetitive applications of wheel loads. However, with the HWT test, two sets of cylinder or slab specimens are submerged in water. Rut depths at different positions along the specimens are recorded for each wheel pass. An advantage of HWT over the APA is that typical result curves from HWT test consist of post-compaction phase, creep phase, and striping phase that can be used to access a mixtures susceptibility to moisture damage.
Direct Tension Cyclic Fatigue	AASHTO TP 107-	As suggested in the name, this test is used
Test	14	to predict the fatigue life of the asphalt mix. Cyclic fatigue damage tests are performed at three different peak-to-peak on- specimen strain levels. The stress and strain results are used to determine the damage characteristic curve of the asphalt mixture as well as to predict the pavement fatigue life.
Flexural Bending Beam Fatigue	AASHTO T 321-4	This test is also used to characterize the
Test	/ ASTM D7469- 10	fatigue behaviour of asphalt mixtures. An asphalt beam specimen is held by four equally spaced clamps and a sinusoidal controlled deflection mode of loading is applied at the two inner clamps. The magnitude of the load applied by the actuator and the deflection measured at the centre of beam is recorded and used to calculate the flexural stiffness, cumulative dissipated energy, and the cycles to failure.
Semi-Circular Bend Test	AASHTO TP-105- 13	This test is used to determine the fracture energy and fracture toughness of an asphalt mix. During the test, a vertical load is applied on the semi-circular specimen at a constant rate of 0.0005 mm/s. The test stops when the load drops below 0.5 kN or when the crack mouth opening displacement gauge range limit is reached, whichever occurs first.

Indirect Tensile Asphalt	N/A	In the IDEAL-CT test, a vertical monotonic
Cracking Test (IDEAL-CT)		load is applied on a cylinder specimen at a constant rate of 50 mm/min. The test is stopped when the load is reduced to 0.1kN. During the test, the crosshead displacement is continuously monitored and recorded. The test parameter CT Index is calculated as a function of total fracture energy and the slope of the post-peak curve at 25 percent
		reduction from the peak load.
Illinois Flexibility Index Test (I-	AASHTO T 124-	In the I-FIT test A 150-mm diameter by 50-
FII)	16	mm thick semi-circular specimen with a 15-
		on the flat surface. The load is applied to the
		curved surface above the notch at a vertical
		rate of 50 mm/min. Load and vertical
		drops below 0.1 kN. Fracture energy is
		calculated from the area beneath the load
2 8 3 9		displacement curve to 0.1 kN. The post-peak
		indicator of the brittle to ductile failure. The
A ANT		flexibility index parameter is calculated by
		multiplying the fracture energy by a scaling
Thermal Stress Restrained	BS FN 12697-	The TSRST test determines the low fracture
Specimen Test	46:2012	temperature of an asphalt mixture when the
		sample is subjected to cooling at a constant
		rate. To perform the test, a beam specimen
		is placed in an environmental chamber with
		The temperature in the chamber is then
		reduced at a specified rate, and the stress in
		the beam is monitored until the beam
		stress. The failure stress and failure
		temperature are then recorded and used to
		access the low temperature performance of
		the asphalt mix.

The inclusion of these tests is a great step forward to address the need in industry to properly characterize asphalt mixes beyond volumetric properties. As stakeholders explore responsible ways of continuing to use RAP in asphalt mixes, and as new additives get introduced, it will be critical to have the knowledge and tools to properly access the properties of asphalt mixes so that performance is not hindered. Furthermore, responsible use of recyclable materials in asphalt pavement is a fundamental design approach not only for limiting the environmental impact of the construction industry, but also for reducing the overall costs of the road infrastructures.

There are few limitations and challenges associated with implementation of these tests. Firstly, there is limited data for agencies to understand which tests accurately characterize the various types of pavement cracking, and have been correlated to field performance, in their local climate. Another challenge for the industry with respect to implementation of performance tests is the equipment cost for the tests. The NCHRP report estimated infrastructure costs range from \$10,000 to \$125,000 for test equipment only. (West et al., 2018) The type of testing selected will dictate the equipment cost. Currently these tests are not specified by Canadian agencies thus most contractors and testing labs do not possess the equipment to conduct majority of the performance testing.

In Ontario, MTO is investing in testing equipment and conducting I-FIT tests on select contract mixes for information and for possible adoption of this test method for asphalt mix designs. MTO has also used AMPT Flow Number to assess rutting performance and included in their specification for assessing Warm Mix Asphalt, which are a group of technologies used to lower production and placement temperatures of asphalt mixes. (Speight, 2016) For that reason, AMTP Flow Number and I-FIT Flexibility index have been included in this research to assess the rutting and cracking performance of collected asphalt mixes respectively.

Furthermore, agencies will need to establish the precision and bias statements for the performance tests they chose to adopt, which will require Round Robin testing and setting up experiments to determine the between lab variability of performance tests. Lastly, it is critical that agencies establish a relationship between the lab produced samples for mix design, and plant-produced field samples for quality assurance. Extensive testing will be required to have enough data to prepare standard deviations and coefficient of variation. The data that has been collected in this research can be a starting point for agencies to reference for future round robin testing.

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2.6 Summary of Knowledge Gaps

Agencies across North America have been experiencing premature fatigue cracking in asphalt pavements, specifically Ontario has been experiencing premature cracking (within two years of construction) in asphalt pavements since before 2004. (Lane, 2015; Lysyk, 2016) Some research and forensic investigations carried out by MTO, and others determined the premature cracking was caused by poor quality of the asphalt binder. (Burke et al., 2011; Hesp et al., 2009; Modi et al., 2016; Wright et al., 2011)

Additionally, with the increased use of recycled materials such as reclaimed asphalt pavement (RAP) in asphalt mixes, many user agencies in Canada are looking for ways to evaluate the properties of the resultant asphalt mix, and the properties of the blended asphalt (i.e., new asphalt binder and old asphalt from RAP), since this has an impact on the asphalt pavement performance.

An option utilized by owners is to evaluate the blended asphalt, by conducting solvent extractionrecovery testing on the asphalt and determine the physical properties of the recovered asphalt in accordance with a standard specification. Users often apply the same specification by which the original asphalt was verified. However, there are concerns with the appropriateness of acceptance criteria based on recovered asphalt, since research has shown that the extraction and recovery process can impact the recovered asphalt properties because of contamination from the solvent and/or aggregate fines that affect the recovered asphalt viscosity.

As such, there is a need to understand how the physical properties of original asphalt compare or relate to the properties of the same asphalt recovered from a plant produced mix, and to understand the impact of comparing values of recovered asphalt cement to test criteria and tolerances derived for unrecovered (original) asphalt cement.

Additionally, there is a need to understand how accurately the short-term laboratory conditioning protocol in the PG system simulates the short-term ageing that occurs in the production and paving.

Lastly, there is a need to investigate and adopt test methods that characterise plant produced asphalt mix that have been verified through field performance.

3 RESEARCH METHODOLOGY

To address the knowledge gaps identified in Section 2., a research plan is developed to evaluate asphalt binder properties through the life of the asphalt binder from production to construction, to see which methods and parameters determined in the laboratory testing, are effective in relating to performance of asphalt pavements.

This required sampling asphalt binder and asphalt mix at various stages of production and construction as illustrated in Figure 3-1:

Sample 'A' is asphalt binder sampled from asphalt binder supplier's terminal. This serves as the starting point of the asphalt binder's "life" as it relates to this research.

Sample 'B' is the same asphalt binder (i.e., from the same batch/lot number) delivered to the asphalt mix production plant. This sample is obtained from the asphalt binder storage tank. Sample A was stored for backup, if there was suspicion of contamination of Sample B during transport from the supplier's terminal to the asphalt mix production plant. Furthermore, current Ontario standards require original asphalt binder samples be obtained from the asphalt tank at the HMA production facility.

Sample 'C' is plant produced asphalt mix, produced mix with the same asphalt from Sample 'B'. This is collected for conducting performance testing to characterize the asphalt mix.

Sample 'D' is the same plant produced asphalt mix from Sample 'C' collected from the paving site.



Figure 3-1: Visual representation of asphalt cement and mix sampling locations

Invitations were sent to members of the Ontario Road Builders Association (ORBA) to request material to be donated for the research and the materials collected are shown in Table 3-1:

Identification	Asphalt Mix Class	PGAC Grade	RAP Content
1-0708	HL1 / 12.5FC2	70-28	0
2-0809	12.5FC2	70-28	15
3-0915	12.5	58-34	15
4-1003	12.5	58-34	0
6-1006	12.5	58-28	0
7-1010	12.5FC2	64-28	0
8-1031	12.5FC1	58-34	0

Table 3-1: List of asphalt binder and asphalt mix collected for the research

The PGAC Grades collected covered the common PGAC Grades in Ontario. The PG 70-28 for samples 1-0708 and 2-0809 were donated by the same asphalt binder supplier, and the respective asphalt mixes were produced at the same asphalt mix production plant. Both asphalt mixes contained the same aggregates, except for 15% RAP material incorporated in 2-0809. The intent was to compare the properties of 0% RAP asphalt mix and 15% RAP asphalt mix. The same applies to PG 58-34 liquid asphalt for 3-0915 and 4-1003.

3.1 Inter-laboratory Study

Invitations were sent to certified asphalt testing labs, and five labs across in Ontario and one laboratory in the United States (US) agreed to participate in the inter-laboratory study (ILS). Each laboratory received seven sets of Sample B, referred to herein as tank asphalt, and its equivalent plant produced asphalt mix, Sample D, from the paving site (Figure 3-1).

Testing labs were requested to follow the MTO laboratory standards (LS) for all testing the tank asphalt and MTO's procedure for solvent extraction by centrifuge, and recovery by either Abson or Rotavapor. The only exception made was limiting the solvent for extraction to reagent grade trichloroethylene (TCE).

The test methods followed are:

• Ash Content: MTO LS-227 (MTO, 2015)

This test is used to determine the percentage of inorganic materials present in an asphalt or emulsified asphalt residue. The sample is burned away in a crucible and when it is returned to room temperature, it is weighed to compare with the starting weight. Ash content test evaluates the total inorganic content and cannot identify individual percentages of different inorganic materials. There is no precision and bias statement available for this test method.

• PG Asphalt Binder Continuous Grading: AASHTO R29 (AASHTO, 2012b)

This test method is used to determine or verify the performance grade of an asphalt binder. The PG asphalt binder specification relies on testing asphalt binders in conditions that simulate critical stages during the life of the asphalt binder. Tests performed on the original asphalt binder represent the first stage of transport, storage, and handling. The second stage represents the asphalt binder during mix production and construction and is simulated by the aging the asphalt binder using the rolling thin-film oven (RTFO). The RTFO exposes thin asphalt films to heat and air to approximate the aging of the asphalt during mixing and construction. The third stage of aging occurs as the asphalt binder ages over a long period of time as part of the asphalt pavement layer. This stage is simulated in the specification using the PAV. The PAV exposes the asphalt binder samples to heat and pressure to accelerate oxidation and simulate years of in-service aging in pavement. (Anderson, 2011)

Generally, PG is reported by two numbers, which are the average seven-day maximum pavement temperatures (°C) and the minimum pavement design temperature (°C) that are expected to be

experienced. For example, a reported value of PG 58-22 represents that this asphalt binder can be used where the average seven-day maximum pavement temperature is 58°C and the expected minimum pavement temperature is -22°C for the best expected performance to be achieved. There is no precision and bias statement available for this test method.

Multiple Stress Creep Recovery (MSCR) Test: AASHTO T350 (AASHTO, 2014b)

The primary objective of the PG specification was to relate asphalt binder performance criteria to the distress conditions with respect to different climate and traffic loading. The challenge with the PG specification is that it was developed based on a study of neat (unmodified) binders, so it may not properly characterize modified binders. The use of polymer modified asphalt binder increased since the implementation of the Superpave PG system, mainly due to the tremendous growth in the use of polymer modified asphalt in the United States and Canada to meet the demand of heavier truck traffic loading on asphalt pavements.

The MSCR test was developed to monitor the creep and recovery conditions of the asphalt binder to see the tendency of permanent deformation for polymer modified pavements. The test in done based on the dynamic shear rheometer. The RTFO aged binder sample is initially loaded with a 1-second creep load, then removed for 9 seconds of recovery and to see the change of characteristic. The applied load starts from 0.1kPa in the first 10 creep-recovery cycles, then increase to 3.2kPa for another 10 cycles. Figure 3-2 shows the general response of the polymer modified asphalt in the MSCR test.



Figure 3-2: Creep-recovery response of the polymer modified asphalt in MSCR test

The test temperature applied for the MSCR test if decided based on the actual environmental high temperature. Two parameters will be considered from the MSCR test, the nonrecoverable creep compliance at 3.2 kPa ($J_{nr3.2}$) and the MSCR percentage of recovery (MSCR), since they are included in the current Ontario specifications. (MTO, 2014) The $J_{nr3.2}$ parameter has been shown to correlate well with rutting in asphalt pavements in the field. (Dubois et al., 2014; Lv et al., 2019). The precision statement of this test shows that the acceptable coefficient of variation between labs, representing 1s% (one standard deviation) for Average Nonrecoverable Creep Compliance at 3.2 kPa, $J_{nr3.2}$ (kPa-1) is 10.8 percent. The acceptable range of two test results between laboratories, representing s2s% (two standard deviations) for the same parameter is 30.7 percent.

This means for example that if two tests were conducted by two labs and reported Average Nonrecoverable Creep Compliance at 3.2 kPa, $J_{nr3.2}$ (kPa⁻¹) of 1.20 kPa-1 and 1.79 kPa-1 respectively, the average of the two results I 1.495 kPa-1. With an acceptable range of test results between labs for this parameter is 30.7 percent of 1.495 it equates to 0.459 kPa-1. The difference between the two results (1.20 kPa-1 and 1.79 kPa-1) is 0.59 kPa-1, this is greater than 0.459 kPa-1, therefore the results are considered not within the acceptable range. There is no information on bias for this test method because no material having an accepted reference value is available. (AASHTO, 2014a)

Double Edge Notch Tension (DENT) Test: MTO LS-299 (MTO, 2007)

Double Edge Notch Test is developed to measure the fracture resistance of the asphalt binder at intermediate temperature, thereby analyzing the fatigue performance. To perform of the DENT test, an asphalt binder specimen is prepared to have a 30-degree notch on each side as shown in Figure 3-3. The asphalt binder is then placed in a ductility bath to apply a pulling force to each side. The pulling force is then removed to show load-displacement to recovery. By measuring the force-displacement curves of the specimen, the area under the curve can be obtained as Figure 3-3 shows. These areas represent the energy used to deform the asphalt binder specimen. Usually, the specimens are tested under different ligaments lengths between the notches, such as 5, 10, and 15mm.

Usually, the unit value of work is obtained by dividing the total work by the thickness times width between the notches. Then, a linear correlation can be obtained between the unit work and the ligament length. At the same time, DENT can obtain the crack tip opening displacement (CTOD) parameter of the specimen. CTOD is defined as the essential work required to cause the specimen fracture divided by the peak load when the ligament length is 5mm. It is a failure mechanics property that reflects the strain tolerance of a very thin fiber of material in the ductile state under high constraint (such as in between two coarse aggregate particles). The CTOD has been shown to provide a reasonable measure of the ability of a pavement to stretch before failure occurs, and as such the amount of cracking distress should be inversely proportional to CTOD. (Hesp et al., 2014; Paliukaite et al., 2015) Other research showed that the DENT test is not effective in showing a clear relationship between increasing concentration of polymer modification, making the DENT test ineffective in predicting fatigue performance. (Aurilio et al., 2017)



Figure 3-3: DENT test machine (left) and the typical data distribution (right)

• Extended Bending Beam Rheometer (ExBBR) Test: MTO LS-308 (MTO, 2011)

The MTO introduced the Extended Bending Beam Rheometer Test (EBBR) to evaluate the physical hardening of the asphalt binder. Physical hardening of the asphalt binder is a phenomenon that occurs when cooling the asphalt binder. When asphalt binder cools, the molecular movements and vibration decreases, which results in the reduction of the volume of the binder. It has been found that the extent of volume reduction is proportional to the temperature drop. (Wright et al., 2011) In this test, the specimens are prepared and conditioned for 1, 24, and 72 hours in the environment of 10 and 20 degrees higher than the minimum design pavement temperature. Then, these specimens are tested under the condition of 16°C and 20°C higher than the minimum design pavement temperature limiting grades (LLTG) and the grade loss.

3.2 Oxidative Aging and SAR-AD[™]

SAR-AD[™] is an approach to asphalt chemical analysis that was developed by the Western Research Institute (WRI). In practice, the SAR-AD[™] method combines an automatic Asphaltene Determinator (AD) separation with an automatic SAR (Saturates, Aromatics and Resins) separation. This provides a fully integrated SARA (Saturates, Aromatics, Resins and Asphaltenes) separation using milligram sample quantities. (Delfosse et al., 2017) The automated SAR-AD[™] separation was used by WRI to conduct research for the Federal Highway Research Administration (FHWA) the method of separation was found to be highly repeatable, and it provided differences between asphalt binders that allow for correlations between chemical content and physical properties. (Boysen & Schabron, 2015)

The tank asphalt binder samples were aged following RTFO and PAV protocols (AASHTO, 2004a, 2012a) with the addition of an extended aging cycle following MTO's modified procedure for accelerated aging with 40 hours of PAV. (MTO, 2017) Figure 3-4 shows a flowchart of where asphalt samples were collected and their respective aging phases for the tank asphalt: RTFO, 20-hour, and 40-hour PAV aging, for SAR-AD[™] analysis.





Chemical analysis through SAR-AD[™] was used to determine concentrations of the asphalt fractions (saturates, aromatics, resins, and asphaltenes). The concentrations were used to calculate an aging index as an indication of degree of oxidation for laboratory versus field.

SAR-AD[™] analysis for this research was performed in collaboration with Imperial (ExxonMobil), with no interpretation of results.

3.3 Alternative Test Methods for Asphalt Binder and Asphalt Mix Acceptance

3.3.1 Delta Tc

The oxidative behavior of asphalt is one of the critical factors contributing to the performance of asphalt pavements. Pavement distresses associated with oxidized or aged asphalt binders are commonly referred to as non-load related distresses: block cracking, raveling, and longitudinal or transverse cracking.

Block cracking is a series of large (typically one foot or more), rectangular cracks on asphalt pavement surface as illustrated in Figure 3-5. It is caused by shrinkage of the pavement due to temperature cycles and is usually an indication that the asphalt binder has aged or oxidized significantly.



Figure 3-5: Block cracking in asphalt pavement

Raveling is wearing of the pavement surface caused by aggregate particles dislodging due to loss of asphalt binder. This type of pavement distress is usually an indication that the asphalt binder has oxidized and hardened significantly. Longitudinal and transverse cracks are cracks that respectively run parallel or perpendicular to the centerline of the pavement. They can be the result of poor construction or can be reflected from cracks in base layers. These cracks can also be the result of shrinkage in the asphalt pavement due to low temperatures or hardening of the asphalt binder. Overall, the main impact on non-load related distresses in asphalt pavements is the oxidation and subsequent age hardening of the asphalt binder.

Researchers in the asphalt binder technical community and user agencies continue seeking physical property parameters that will improve asphalt pavement performance and are constantly investigating cracking index parameters to evaluate the cracking potential of asphalt binders. The idea being that if we identify parameters that can correlate with asphalt flexibility, we can use them to monitor when flexibility reaches a state where corrective action is needed before cracking occurs. One such parameter is Delta Tc (ΔT_c).

 ΔT_c was developed by Mike Anderson as part of a research project involving airfield asphalt pavements, to evaluate the relationship between asphalt binder properties and non-load related cracking. (Blankenship et al., 2010). The study relied on past research that showed some relationship between ductility (related to flexibility) and the durability of an asphalt pavement. Ductility is an asphalt binder's ability to be stretched without breaking. ΔT_c is calculated using values from the Bending Beam Rheometer (BBR) test by subtracting the BBR m-critical temperature from the BBR stiffness-critical temperature:

$\Delta T_{c} = (T_{s-critical} - T_{m-critical})$

The critical temperatures, $T_{s-critical}$ and $T_{m-critical}$, are the temperatures at which the stiffness and m-value specification requirements are met (i.e., S=300 MPa, m-value=0.300) respectively. They can be determined following ASTM D7643, Standard Practice for Determining the Continuous Grades for PG Graded Asphalt Binders; or AASHTO R29, Standard Practice for Grading or Verifying the Performance Grade (PG) of an Asphalt Binder.

In the BBR test, a constant load is applied to an asphalt beam, which is held at a constant temperature. The test temperature is related to a pavement's lowest service temperature. The purpose is to determine how the asphalt beam responds to mechanical stresses at low temperatures. This is important for asphalt pavements because as the surrounding temperatures drop, the pavement contracts (or shrinks), but the asphalt binder contracts to a much larger degree than the aggregates in the pavement. When these stresses exceed the tensile strength of the asphalt mix, a low-temperature crack develops in the pavement.

The Superpave PG system sets a criterion for creep stiffness (S) to minimize the contribution of the asphalt binder to low-temperature cracking: $S \le 300$ MPa after 60 seconds of loading at the appropriate temperature.

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The m-value is the rate at which the asphalt binder stiffness changes over time. A higher m-value is an indication that the stiffness may not increase as quickly when temperature decreases. This means the tensile stresses in the asphalt will be smaller as the contraction occurs, reducing the chances of low-temperature cracking. Therefore, the PG system specifies a minimum m-value of 0.300 after 60 seconds of loading at the appropriate temperature. Asphalt binders that are not too stiff at low temperatures and are able to relax built up stresses are desirable.

 ΔT_c is intended to provide an indication of loss of ductility: when the asphalt binder cannot relax the stresses fast enough to prevent breaking. As asphalt binders oxidize and age, their ability to relax at low temperatures diminishes. This would be captured in the BBR m-value and result in a higher (less negative) $T_{m-critical}$.

The ΔT_c of an aged binder would be more negative, than that of an unaged binder, and would be more likely to exhibit the pavement distresses described earlier: block cracking, raveling, and longitudinal or transverse cracking. This has generated a lot of interest in using ΔT_c to characterize asphalt mix containing (RAP), due to the contribution of highly oxidized asphalt from these recycled materials. ΔT_c can be measured from virgin asphalt and asphalt recovered from the asphalt mix.

3.3.2 Asphalt Mixture Performance Tester (AMPT) Flow Number test

Most state highway agencies have implemented the Superpave volumetric mix design process created during the Strategic Highway Research Program (SHRP) as part of their system for designing asphalt mixtures. However, at the conclusion of SHRP no test was available that provided information on the probable performance of asphalt mixtures designed using Superpave volumetric mix design. The Federal Highway Administration (FHWA) and the National Cooperative Highway Research Program (NCHRP) sponsored research to develop and validate simple performance tests for permanent deformation and fatigue cracking to be incorporated in the volumetric mixture design process. The AMPT shown in Figure 3-6 was developed as part of this research. The AMPT was recommended to be used to conduct three tests to evaluate permanent deformation of asphalt mixtures: dynamic modulus (E*) using the triaxial dynamic modulus test, flow time (Ft) using the triaxial static creep test, and flow number (FN) using the triaxial repeated load test. The flow number has been correlated to the mixture's rutting resistance, with a higher flow number indicating higher resistance to rutting. (Witczak et al., 2002)

To conduct the AMPT tests, a Superpave gyratory compacted asphalt mix is prepared with the size of 150mm diameter by 170 mm height. The specimen is then cored and sawed to the appropriate diameter of 100mm and height of 150mm per for the test. This ensures homogeneity of the test specimen.



Figure 3-6: Photographs of AMPT Equipment by Interlaken Technology Corporation (left) and IPC Global (right)

3.3.3 Illinois Flexibility Index Test (IFIT)

I-FIT test uses semi-circular bending (SCB) specimen geometry to determine the fracture resistance of an asphalt mixture at an intermediate temperature. The provisional standard test method, AASHTO TP-124, "Determining the Fracture Potential of Asphalt Mixtures Using the Semi-Circular Geometry at Intermediate Temperature," calls for 50-mm thick, 150-mm diameter semi-circular specimens to be tested using a three-point bending principle, at the constant displacement rate of 50 mm/min. Figure 3-7 presents a photograph of the I-FIT test arrangement. A 15-mm deep, 1.5-mm wide notch is cut along the specimen's axis of symmetry to force the failure location. Prior to testing, the test specimen is conditioned for two hours in an environmental chamber at 25°C, the standard test temperature.



Figure 3-7: I-FIT Test using Semi-Circular Bending (SCB) specimen geometry

One of the primary outputs of I-FIT is the fracture energy, which represents the energy dissipated by the crack propagation. This parameter is calculated as the area under the load-displacement curve divided by the area of the crack that propagates during testing. The fracture energy is a function of both the strength and ductility of the material, which are related to the peak load and maximum displacement, respectively. Generally, the higher the fracture energy, the better the cracking resistance.

3.4 Summary of Research Methodology

The research methodology was developed to evaluate asphalt binder properties through the life of the asphalt binder from production to construction, to see which test methods and parameters determined in the laboratory testing, are effective in relating to performance of asphalt pavements.

This required sampling asphalt binder and asphalt mix at various stages of production and construction.

The first part of the research involved five labs across in Ontario and one laboratory in the United States (US) participating in an inter-laboratory study (ILS). Each laboratory received seven sets asphalt binder and a corresponding plant produced asphalt mix. The five labs followed the MTO laboratory standards for all testing of the tank asphalt and MTO's procedure for solvent extraction by centrifuge, using reagent grade trichloroethylene (TCE), and recovery by either Abson or Rotavapor.

Chemical analysis through SAR-AD[™] was used to determined concentrations of the asphalt fractions (saturates, aromatics, resins, and asphaltenes). The concentrations are used to calculate an aging index as an indication of degree of oxidation for laboratory versus field.

Lastly, a new parameter, Delta Tc, calculated from current tests was included in the research as it has been shown to correlated with non-load related asphalt pavement distresses, along with two tests, the Illinois Flexibility Index Test, and Asphalt Mix Performance Tester, both of which have been shown to correlate with performance.

4 RESEARCH RESULTS

4.1 Interlaboratory Study

The tank and recovered asphalt binder properties presented in this section are organized in tables for each PGAC grade. The tables show the average values and standard deviations of the measured properties for each PGAC as an evaluation of the ILS. All the individual test results from the individual laboratory are summarized in Appendix A. The sample size is the number of results received from the participating labs for analysis. Asphalt mixes that contained RAP is separated from asphalt mixes with no RAP for PG 70-28 and PG 58-34. Due to limited resources of some of the participating labs, some sample sizes are too low to apply statistical analysis to the results, however the results are provided in the tables for information.

Notes for all tables and charts:

- PG High = Performance Graded High Temperature
- PG Low = Performance Graded Low Temperature
- MSCR Jnr = Multiple Stress Creep Recovery Non-Recoverable Creep Compliance
- LTLG = Low Temperature Limiting Grade
- CTOD = Crack Tip Opening Displacement.
- StDev = Standard Deviation
- COV = Coefficient of Variance/Variation

4.1.1 PGAC 58-28: ILS Results for Tank and Recovered Asphalt

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	0.08	59.75	-34.30	2.19	2.73	-30.20	13.78
Min	0.05	58.90	-35.40	2.02	2.20	-31.20	9.70
Max	0.11	60.60	-33.00	2.37	4.00	-28.90	17.90
StDev	0.022	0.850	0.990	0.127	0.740	0.834	3.011
COV (%)	28.0	1.4	2.9	5.8	27.1	2.8	21.9
Sample Size	4	2	3	4	4	4	4

Table 4-1: PGAC 58-28 Tank Asphalt Properties

Table 4-2: PGAC 58-28 Recovered Asphalt Properties (No RAP)

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	2.57	58.15	-35.53	4.77	5.25	-29.13	8.65
Min	1.32	51.50	-37.80	1.26	3.50	-30.00	1.54
Max	4.11	64.80	-34.10	8.68	8.20	-27.50	14.70
StDev	0.997	6.650	1.621	3.198	1.845	1.156	5.424
COV (%)	38.7	11.4	4.6	67.1	35.1	4.0	62.7
Sample Size	4	2	3	4	4	3	4

Table 4-3: PGAC 58-28 Comparing Variability in Results for Tank and Recovered Asphalt

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
StDev (Tank Asphalt)	0.022	0.850	0.990	0.127	0.740	0.834	3.011
StDev (Rec Asphalt)	0.997	6.650	1.621	3.198	1.845	1.156	5.424
% Change StDev	4431	682	64	2418	149	39	80

Table 4-3 shows that for PG 58-28, depending on the parameter being measured, the standard deviation in test results increased between 39% to 4431% for recovered asphalt binder versus the tank asphalt, with the highest increase in ash content results. The COV of results of recovered asphalt are also higher that tank asphalt. Interesting to note that the COV of Ash Content, Grade loss, and CTOD from tank asphalt properties in Table 4-1 are higher than for all the other asphalt properties. This suggests higher variability in test results even before recovering the binder from a plant produced mix.

4.1.2 PGAC 64-28: ILS Results for Tank and Recovered Asphalt

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	0.08	65.40	-35.30	0.29	3.48	-30.40	14.03
Min	0.04	64.90	-37.10	0.19	3.10	-31.40	6.40
Max	0.12	65.90	-33.30	0.36	3.90	-29.20	21.20
StDev	0.030	0.500	1.558	0.064	0.334	0.797	5.324
COV (%)	39.1	0.8	4.4	22.2	9.6	2.6	38.0
Sample Size	4	2	3	4	4	4	4

Table 4-4: PGAC 64-28 Tank Asphalt Properties

Table 4-5: PGAC 64-28 Recovered Asphalt Properties (No RAP)

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	4.97	76.25	-31.63	0.38	5.53	-25.08	6.70
Min	1.68	69.90	-32.20	0.05	3.90	-28.70	4.90
Max	7.79	82.60	-31.20	0.79	9.70	-20.50	8.90
StDev	2.355	6.350	0.420	0.266	2.417	2.963	1.626
COV (%)	47.4	8.3	1.3	70.0	43.7	11.8	24.3
Sample Size	4	2	3	4	4	4	4

Table 4-6: PGAC 64-28 Comparing Variability in Results for Tank and Recovered Asphalt

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
StDev (Tank Asphalt)	0.030	0.500	1.558	0.064	0.334	0.797	5.324
StDev (Rec Asphalt)	2.355	6.350	0.420	0.266	2.417	2.963	1.626
% Change StDev	7750	1170	73	316	624	271	69

Table 4-6 shows that for PG 64-28, the standard deviation increased for all parameters except for PG Low and CTOD. The increase in standard deviation for the other properties ranged from 271% to 7750% for the recovered asphalt samples depending on the measured parameter, with ash content having the highest increase in standard deviation. Although the standard deviation decreases for CTOD of recovered asphalt, it is also important to note the difference in the average result of CTOD: 14.0mm and 6.7mm for tank and recovered asphalt respectively. The impact of this difference becomes evident in the next section when the results are compared to MTO's asphalt acceptance

criteria. The same is noted for the average PG Low values of -35.30 versus -31.63 from tank asphalt and recovered asphalt respectively.

4.1.3 PGAC 58-34: ILS Results of Tank and Recovered Asphalt

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	0.25	62.84	-37.43	0.62	3.33	-33.11	25.45
Min	0.07	61.60	-39.90	0.38	1.50	-36.80	15.80
Max	0.65	65.00	-35.40	0.95	4.70	-27.80	37.40
StDev	0.204	1.049	1.497	0.182	1.008	2.433	6.419
COV (%)	83.1	1.7	4.0	29.2	30.3	7.3	25.2
Sample Size	12	8	10	12	12	12	12

Table 4-7: PGAC 58-34 Tank Asphalt Properties

Table 4-7 shows higher sample sizes because it includes test results of tank asphalt used for both 3-0915 and 4-1004. As previously discussed, the same PG 58-34 asphalt was used in the production of both 0% RAP asphalt, 4-1003, with recovered asphalt results in Table 4-8, and 15% RAP asphalt, 3-0915, with recovered asphalt results in Table 4-9.

Table 4-8: PGAC 58-34 Recovered Asphalt Properties (No RAP)

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	2.95	67.43	-37.56	0.70	4.91	-32.21	12.12
Min	1.10	66.90	-39.05	0.23	2.70	-34.90	7.00
Max	6.70	68.50	-36.00	1.90	6.40	-28.10	20.80
StDev	1.645	0.653	1.149	0.536	1.117	2.154	3.969
COV (%)	55.7	1.0	3.1	76.8	22.7	6.7	32.7
Sample Size	8	4	6	7	8	8	8

Table 4-9: PGAC 58-34 Recovered Asphalt Properties (15% RAP)

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	2.44	70.90	-33.53	0.63	6.55	-23.43	4.47
Min	1.68	68.00	-36.40	0.19	4.60	-28.10	-0.14
Max	2.99	75.50	-29.70	1.32	8.00	-18.00	8.20
StDev	0.495	2.992	2.819	0.422	1.230	3.786	3.214
COV (%)	20.3	4.2	8.4	67.3	18.8	16.2	72.0
Sample Size	4	4	3	4	4	4	4

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
StDev (Tank Asphalt)	0.204	1.049	1.497	0.182	1.008	2.433	6.419
StDev (Rec Asphalt – No RAP)	1.645	0.653	1.149	0.536	1.117	2.154	3.969
StDev (Rec Asphalt – 15% RAP)	0.495	2.992	2.819	0.422	1.230	3.786	3.214
% Change StDev (No RAP)	706	37	23	194	10	11	38
% Change StDev (15% RAP)	142	185	88	131	22	14	50

Table 4-10: PGAC 58-34 Comparing Variability in Results for Tank and Recovered Asphalt

Table 4-10 shows that when PG 58-34 asphalt was recovered from a 0% RAP asphalt, the variability in material properties increased for some of the parameters and decreased for others compared to the tank asphalt. It is worth noting that the standard deviation of CTOD decreases for recovered asphalt and this could be due to the increase in ash content in the recovered material which makes the material stiffer in general and less susceptible to testing variability in this test, and not an indication that the recovered binder is more representative of the material. This phenomenon is also observed in Table 4-10 showing a lower standard deviation when RAP is included in the mix, which would also cause the material to be stiffer and less representative of the original asphalt binder. Lastly, the difference in the CTOD average values for tank asphalt, 0% RAP recovered asphalt, and 15% RAP recovered asphalt: 25.5mm, 12.1mm, and 4.5mm respectively.

4.1.4 PGAC 70-28: ILS Results for Tank and Recovered Asphalt

Table 4-11: PGAC 70-28	Tank Asphalt	Properties
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Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	0.07	72.38	-35.50	0.09	4.09	-29.69	12.51
Min	0.04	70.30	-36.70	0.04	2.40	-32.80	4.70
Max	0.10	75.70	-34.80	0.13	6.10	-27.90	23.10
StDev	0.019	1.364	0.540	0.033	1.137	1.409	6.174
COV (%)	27.7	1.9	1.5	38.0	27.8	4.7	49.3
Sample Size	8	10	10	10	8	8	8

Table 4-11 includes test results of tank asphalt used for both 1-0708 and 2-0809. The same PG 70-28 asphalt was used in the production of both 0% RAP asphalt, 1-0708, with recovered asphalt results in Table 4-12, and 15% RAP asphalt, 2-0809, with recovered asphalt results in Table 4-13.

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	2.70	75.24	-34.65	0.07	4.03	-28.50	9.28
Min	2.09	72.10	-35.80	0.04	3.50	-29.60	7.10
Max	3.63	77.80	-32.80	0.12	4.80	-27.10	11.02
StDev	0.588	2.077	1.126	0.029	0.521	0.903	1.403
COV (%)	21.8	2.8	3.2	40.6	13.0	3.2	15.1
Sample Size	4	5	4	5	4	4	4

Table 4-12: PGAC 70-28 Recovered Asphalt Properties (No RAP)

Table 4-13: PGAC 70-28 Recovered Asphalt (15% RAP)

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	3.33	82.04	-27.11	0.09	5.53	-21.47	4.03
Min	2.29	77.40	-35.40	0.08	2.30	-25.20	2.50
Max	4.17	93.00	-11.60	0.11	9.70	-16.00	6.10
StDev	0.812	5.633	9.161	0.013	3.092	3.951	1.517
COV (%)	24.4	6.9	33.8	14.8	55.9	18.4	37.6
Sample Size	4	5	4	4	3	3	3

Table 4-14: PGAC 70-28 Comparing Variability in Results for Tank and Recovered Asphalt

Material Property	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa ⁻¹)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
StDev (Tank Asphalt)	0.019	1.364	0.540	0.033	1.137	1.409	6.174
StDev (Rec Asphalt – No RAP)	0.588	2.077	1.126	0.029	0.521	0.903	1.403
StDev (Rec Asphalt – 15% RAP)	0.812	5.633	9.161	0.013	3.092	3.951	1.517
% Change StDev (No RAP)	2994	52	109	12	54	35	77
% Change StDev (15% RAP)	4174	313	1596	60	172	180	75

These final set of tables show the results for PG 70-28, which is a polymer modified. Table 4-14 shows that when the recovered asphalt included 15% RAP, the standard deviation was significantly higher for ash content, PG High, PG Low, Grade Loss, and LTLG. It is noted that the properties that showed a decrease in standard deviation such as MSCR Jnr and CTOD showed higher COV values, even in the tank asphalt results which is important when comparing results to specifications for acceptance.

4.1.5 Comparison of Tank and Recovered Asphalt Properties to Specification

The preceding tables showed that testing variability determined through standard deviation and COV calculations are generally higher for recovered asphalt compared to tank asphalt. However, for certain parameters the standard deviation decreased, however the COV which takes the average value into account, was high. It is also important to illustrate the impact of testing recovered asphalt for acceptance by comparing the results to specifications and acceptance criteria developed based on tank asphalt binder. Most recovered asphalt specifications in Ontario apply the same acceptance criteria and tolerances to recovered asphalt as they do for tank asphalt. The following graphs show the test results of tank and recovered asphalt on the 2019 MTO specification (MTO, 2014) for acceptance of original asphalt as an example. The purpose of this exercise is to determine the likelihood of acceptance or rejection of asphalt as some owners use the same acceptance criteria for both tank and recovered asphalt. The error bars show the average and standard deviation of the test results usphalt. The error bars show the average and standard deviation of the test results by participating labs.



PG Grades and RAP Content



Figure 4-1 shows that all recovered asphalt samples exceeded the minimum ash content requirement of less than 0.6%. In the ash content test, a sample of asphalt is incinerated at 600°C, until constant

mass is achieved. The final weight corresponds to the number of inorganic materials present in the asphalt binder. The difference in ash contents between the tank and recovered asphalt suggests the recovered asphalts have aggregate fines present in the samples, which were not completely filtered during the extraction process. Aggregate fines in the recovered asphalt increases the asphalt stiffness, which will impact the measured properties. (Burr et al., 1993; Rahbar-Rastgar Daniel, J.S., Reinke, G., 2017) If the recovered asphalt is contaminated with aggregate fines, and all other factors remain unchanged, this will produce higher PG high temperature as illustrated in Figure 4-2, Figure 4-3, and Figure 4-4.



Figure 4-2: Comparing Performance Grade (PG) High Temperature for PG 58-YY to Specification



PG Grade and RAP Content

Figure 4-3: Comparing Performance Grade (PG) High Temperature for 64-YY to Specification



Figure 4-4: Comparing Performance Grade (PG) High Temperature for 70-YY to Specification

Similarly, if the recovered asphalt is contaminated with aggregate fines, and all other factors remain unchanged, this will produce higher (or less negative) PG low temperature properties in Figure 4-5 and Figure 4-6, as well as in the LTLG properties from the ExBBR test, as illustrated in Figure 4-7 and Figure 4-8.



Figure 4-5: Comparing Performance Grade (PG) Low Temperature for XX-28 to Specification







PG Grades and RAP Content

Figure 4-7: Comparing Tank and Recovered Asphalt Low Temperature Limiting Grade (LTLG) from Extended Bending Beam Rheometer (ExBBR) Test for PG XX-28 to Specification





Figure 4-8: Comparing Tank and Recovered Asphalt Low Temperature Limiting Grade (LTLG) from Extended Bending Beam Rheometer (ExBBR) Test for PG XX-34 to Specification

In this ILS variables such as the quantity of aggregate fines left after extraction, the possibility of residual solvent in the recovered asphalt, differences in laboratory aging of tank asphalt versus field aging through production, have an impact on the recovered asphalt properties. While aggregate fines in the recovered asphalt can increase stiffness of the recovered sample, residual solvent can cause a negative hardening of the recovered asphalt. (Burr et al., 1993; McDaniel & Anderson, 2001)

With respect to other properties measured such as Grade Loss from ExBBR test, Non-Recoverable Creep Compliance from the MSCR test, and the CTOD parameter from the DENT test, at the time of this research there was no literature on ILS conducted for recovered asphalt. Nonetheless the results in Figure 4-9 show that the Grade Loss was generally higher for recovered asphalt than the tank asphalt. This could be a result of the higher ash content in the recovered asphalt and/or differences in oxidation in laboratory and plant production.



Figure 4-9: Comparing Tank and Recovered Asphalt Grade Loss from Extended Bending Beam Rheometer (ExBBR) Test to Specification

The MSCR test is intended to characterize rutting resistance asphalt and provide information on quality of polymer modification. No comparisons could be made with the data in Figure 4-10 on the impact of ash content on the Non-Recoverable Creep Compliance of the recovered asphalt. Some research has shown that some polymer modified asphalt binder lose their elasticity when recovered from solvents such as methylene chloride, TCE, and toluene. (Nosler et al., 2008; Pierard et al., 2010) TCE has been documented to affect the polymer network in recovered asphalt and produce results with higher variability. (Pierard et al., 2010) This was confirmed by looking at the molecular weights of the polymers before and after recovery through gel permeation chromatography. Simply dissolving and recovering the polymer modified asphalt, changes occurred to the molecular weight which resulted in decreased elasticity noted from the rheological tests. The results from the samples in this research are shown in Figure 4-10.



Figure 4-10: Comparing Tank and Recovered Asphalt Non-Recoverable Creep Compliance from Multiple Stress Creep Recovery (MSCR) Test to Specification

It is important to include that during the screening process for the SAR-AD analysis, to ensure none of the samples were contaminated with solvent prior to SAR-AD, some of the samples were identified to be contaminated with TCE. The contaminated samples have been identified in the results are also impacted by the quality of recovery process such as the need to ensure complete removal of solvent from the recovered sample. Highlighted in red in Table 4-11 are samples that were identified to be contaminated TCE and the impact on material properties from the MSCR test. These samples were not excluded from the analysis since the intent of the research is the capture the current state of practice and the reality of the risk associated with recovered asphalt binder testing.



Figure 4-11: Results highlighted in red show samples contaminated with TCE or Toluene based on GCD analysis for information purposes.
With respect to the CTOD from the DENT test, a ductility test conducted at intermediate temperature, Figure 4-12 and Figure 4-13 show that the results from the recovered asphalt are generally lower than the tank asphalt. This could be the result of a stiffer recovered asphalt either through oxidation or because of high ash content, or both.



PG Grades and RAP Content

Figure 4-12: Comparing Tank and Recovered Asphalt Critical Tip Opening Displacement (CTOD) from Double Edge Notched Tension (DENT) Test for PG XX-28 to Specification



PG Grades and RAP Content

Figure 4-13: Comparing Tank and Recovered Asphalt Critical Tip Opening Displacement (CTOD) from Double Edge Notched Tension (DENT) Test for PG XX-34 to Specification

4.1.6 Summary of ILS Results

An independent samples t test was conducted to compare the difference between the means of the two groups with the assumption that the two groups are drawn from the same population. The logic in the analysis is that all the asphalt binder samples and mixes went through the same testing protocols, with one group asphalt binder samples having gone through extraction and recovery process prior to testing.

The t test will confirm whether there is a statistically significant difference between the means of these two groups (tank asphalt versus recovered asphalt). The formula for the t test is as follows:

 $t = \frac{Mean \ difference \ between \ the \ two \ groups}{Standrd \ error \ of \ the \ difference \ between \ the \ means}$

The t test is used to test the hypothesis that there is no statistically significant difference in the means of the two groups: tank asphalt and recovered asphalt. The null hypothesis symbolically is:

$$H_o: \mu_{tank} = \mu_{recovered}$$

The significance level is 5 percent (p value 0.05), this means that there is a 5 percent chance that the difference between means is due to random variation. A p value of less than 0.05 means that if there is less than 5% chance a result in the sample occurred by chance, then we are willing to draw the conclusion that the results are statistically significant and reject the null hypothesis that there will be no difference between the tank asphalt results and recovered asphalt results.

PGAC						p values	5		
Grade	RAP	Mix ID	Ash	PG High	PG	MSCR	Grade	LTLG	СТОД
					LOW	Jnr	LOSS		
58-34	0	8-1031	0.02	0.11	0.65	0.45	0.25	0.97	0.01
58-34	0	4-1003	0.01	0.04	0.86	0.72	0.48	0.08	0.00
58-34	15	3-0915	0.00	0.00	0.09	0.94	0.40	0.02	0.00
58-28	0	6-1006	0.00	0.83	0.41	0.21	0.28	0.28	0.24
64-28	0	7-1010	0.01	0.23	0.03	0.59	0.57	0.02	0.06
70-28	0	1-0708	0.00	0.03	0.26	0.40	0.58	0.26	0.20
70-28	15	2-0809	0.00	0.04	0.11	0.76	0.89	0.02	0.04

Table 4-15: Summary of p values from ILS (Tank Asphalt vs. Recovered Asphalt)

The p values summarized in Table 4-15 show that for the samples in this research:

- There is a statistically significant difference in test results for tank and recovered asphalt when testing for ash content for all PGAC grades. This information was not available to Canadian agencies until this research was completed, especially for plant-produced mixes.
- There is a statistically significant difference in test results for tank and recovered asphalt when testing the PG high temperature grade for PG 58-34 and 70-28, and as well when there is RAP incorporated in the asphalt mix. This information with RAP was not available to the Canadian asphalt industry until this research was completed.
- There is no difference in test results when testing PG low temperature of tank and recovered asphalt, except for PGAC 64-28. This information was not available to the asphalt industry until this research was completed, especially for plant-produced mixes.
- There is no difference in test results when testing the MSCR Jnr and Grade Loss of the tank and recovered asphalt.

- There is a statistically significant difference in test results for LTLG for PGAC 64-28. As well as and for PGAC 58-28 and 70-28 when 15 percent RAP is incorporated. This information was not available to Canadian agencies until this research was completed.
- There is a statistically significant difference in test results for CTOD when RAP is incorporated in the mix and recovered for testing. This information was not available to Canadian agencies until this research was completed.

Comparing the test results from the various labs with the 2019 MTO asphalt specification shows the following:

- 100% of tank asphalt samples passed ash content requirement, while none of recovered asphalt samples passed.
- In the ExBBR test:
 - 96% of tank asphalt samples passed the grade loss requirement; 70% of recovered asphalt samples passed.
 - 94% of tank asphalt samples passed the XX-28 LTLG requirement; 43% of recovered asphalt samples passed.
 - 33% of tanks asphalt samples passed the XX-34 LTLG requirement; 17% of recovered asphalt samples passed.
- In the DENT test:
 - 67% of tank asphalt samples of PG 64-28 and PG 70-28 passed the CTOD minimum requirement; only 9% of recovered asphalt samples passed.
 - 100% of tank asphalt samples of PG 58-34 passed the minimum CTOD requirement; only 17% of recovered asphalt passed.
- In a GCD analysis that was used to screen samples contaminated with TCE prior to SAR-AD analysis, it was noted that some of the samples were contaminated TCE and therefore were not used for SAR-AD, however the test results were included in ILS analysis to capture the current state of practice and risk associated with using recovered asphalt binder test results for acceptance.

4.2 Oxidative Aging of Asphalt Cement

4.2.1 Gas Chromatographic Distillation

GCD analysis was performed on the recovered asphalt samples to determine the presence of residual solvents (e.g., Trichloroethylene (TCE), Toluene (Tol.)) in the recovered asphalt binders from plant produced hot mix. An example of how a sample contaminated with TCE would be revealed through GCD analysis is shown in Figure 4-14.

Prior to sample analysis, a mixture of TCE and toluene was injected into the GC and retention times were recorded. TCE and toluene eluted out of the GC column at around 1.2 and 1.9 min respectively (shown in bracket in Figure 4-14). The contaminant type was assigned by screening the GCD chromatogram for a peak with a retention time that corresponded to TCE, or toluene as shown for sample Y4-1-708D-RTFO. As a precautionary measure, the contaminated samples were not included for subsequent SAR-AD[™] analysis. None of the samples were found to be contaminated with toluene.



Figure 4-14: Example of GCD analysis for a recovered binder that is contaminated with TCE.

4.2.2 SAR-AD™

SAR-AD[™] is an approach to asphalt chemical analysis that was developed by the Western Research Institute (WRI). In practice, the SAR-AD[™] method combines an automatic Asphaltene Determinator (AD) separation with an automatic SAR (Saturates, Aromatics and Resins) separation. This provides a fully integrated rapid SARA (Saturates, Aromatics, Resins and Asphaltenes) separation using milligram sample quantities. (Delfosse et al., 2017) The automated SAR-AD[™] separation was used by WRI to conduct research for the Federal Highway Research Administration (FHWA) the method of separation was found to be highly repeatable, and it provided differences between asphalt binders that allow for correlations between chemical content and physical properties. (Boysen & Schabron, 2015) More research is needed to determine these correlations, and FHWA produced a technical brief document with additional test results that show research being done to explore how separation profiles from SAR-AD can be useful in binder formulation through blending, rejuvenation, and modification as well as prediction of physical performance. However, more research is needed to further develop and validate any correlations. (Youtcheff, 2016)

4.2.3 Results and Discussion

The SAR-AD[™] results are summarized in Table 4-16 through Table 4-19 for the four sets of samples analyzed in the research. As previously indicated in the Contributions section of this thesis, testing was conducted by Imperial (ExxonMobil) with no interpretation of test results. Also, due the limited time and resources of the external laboratory, only one sample was analyzed for each asphalt binder and as such testing variability cannot be demonstrated for this analysis. Nonetheless, the results serve as good information to show the impact of aging on asphalt binder and the differences of laboratory aging versus plant aging.

The results of tank samples show that as the asphalt aging progressed through the RTFO and PAV, the asphaltene content in the binders increased (especially toluene asphaltenes), and the aromatic content decreased. This was the case for an unmodified neat asphalt PG 58-28, and a polymer modified PG 70-28. Through the oxidation process, molecules from the resin fraction become asphaltenes, and the aromatics fraction oxidize to become resins.

			Maltenes			Asphaltene	S	
Sample ID	Age	Saturates	Aromatics	Resins	CyC6	Toluene	CH2:Cl2:	AAIR
							MeOH	
1-0708-B-1	Original	25.6%	45.1%	14.4%	3.5%	11.1%	0.3%	0.77
1-0708-B-2	RTFO	26.1%	43.2%	15.9%	3.3%	11.3%	0.2%	0.71
1-0708-B-3	20hr PAV	29.7%	34.0%	16.2%	3.3%	16.5%	0.3%	1.02
1-0708-B-4	40hr PAV	29.6%	31.4%	15.6%	3.4%	19.6%	0.3%	1.26
1-0708-D-1	Rec Plant	24.4%	41.7%	14.1%	3.4%	16.0%	0.5%	1.13
	Mix							

Table 4-16: Asphalt Composition of Sample 1-0708: PG 78-28, 0% RAP Mix

Note: Sample ID with 'B' represents liquid asphalt cement that is laboratory conditioned through RTFO and two different PAV times (i.e., 20 hours and 40 hours). Sample ID with 'D' represents recovered asphalt binder from plant mix which was produced with the same asphalt binder as Sample 'B'.

			Maltenes			Asphalte	nes	
Sample ID	Age	Saturates	Aromatics	Resins	CyC6	Toluene	CH2:Cl2:	AAIR
							MeOH	
2-0809-B-1	Original	25.1%	47.4%	13.8%	3.7%	9.7%	0.3%	0.71
2-0809-B-2	RTFO	24.9%	45.4%	13.7%	3.9%	11.8%	0.3%	0.86
2-0809-B-3	20hr PAV	24.8%	40.2%	14.2%	3.5%	16.8%	0.4%	1.19
2-0809-B-4	40hr PAV	24.6%	37.3%	13.9%	3.6%	20.0%	0.6%	1.44
2-0809-D-1	Rec Plant	22.4%	39.0%	15.1%	3.9%	19.2%	0.4%	1.27
	Mix							
2-0809-C1	RAP	16.2%	37.5%	19.2%	4.0%	22.5%	0.6%	1.17

Table 4-17: Asphalt Composition of Sample 2-0809: PG70-28, 15% RAP Mix

Note: Sample ID with 'B' represents liquid asphalt cement that is laboratory conditioned through RTFO and two different PAV times (i.e., 20 hours and 40 hours). Sample ID with 'D' represents recovered asphalt binder from plant mix which was produced with the same asphalt binder as Sample 'B'. Sample ID with 'C' represents asphalt recovered from the RAP material used as 15% of the asphalt mix.

			Maltenes			Asphaltene	S	
Sample ID	Age	Saturates	Aromatics	Resin	СуС	Toluene	CH2:Cl2:	AAIR
				S	6		MeOH	
4-1003-B-1	Original	27.5%	46.2%	14.0%	3.8%	8.1%	0.4%	0.58
4-1003-B-2	RTFO	26.3%	43.6%	14.6%	4.1%	11.2%	0.3%	0.77
4-1003-B-3	20hr PAV	26.9%	39.8%	14.7%	3.6%	14.8%	0.3%	1.01
4-1003-B-4	40hr PAV	27.5%	37.3%	13.7%	3.6%	17.5%	0.4%	1.28
4-1003-D-1	Rec Plant	27.6%	41.3%	13.1%	3.6%	13.8%	0.6%	1.06
	Mix							

Table 4-18: Asphalt Composition of Sample 4-1003: PG 58-34, 0% RAP Mix

Note: Sample ID with 'B' represents liquid asphalt cement that is laboratory conditioned through RTFO and two different PAV times (i.e., 20 hours and 40 hours). Sample ID with 'D' represents recovered asphalt binder from plant mix which was produced with the same asphalt binder as Sample 'B'.

			Maltenes			Aspha	tenes	
Sample ID	Age	Saturates	Aromatics	Resins	CyC6	Toluene	CH2:Cl2:	AAIR
							MeOH	
6-1006-B-1	Original	24.6%	48.8%	15.1%	3.4%	7.9%	0.2%	0.53
6-1006-B-2	RTFO	26.1%	44.3%	15.2%	3.6%	10.5%	0.3%	0.69
6-1006-B-3	20hr PAV	24.9%	41.1%	15.3%	3.6%	14.8%	0.3%	0.97
6-1006-B-4	40hr PAV	24.4%	38.5%	15.4%	3.3%	18.1%	0.4%	1.17
6-1006-D-1	Rec Plant	24.9%	43.4%	14.5%	3.6%	13.0%	0.4%	0.90
	Mix							

Table 4-19: Asphalt Composition of Sample 6-1006: PG 58-28, 0% RAP Mix

Note: Sample ID with 'B' represents liquid asphalt cement that is laboratory conditioned through RTFO and two different PAV times (i.e., 20 hours and 40 hours). Sample ID with 'D' represents recovered asphalt binder from plant mix which was produced with the same asphalt binder as Sample 'B'.

Separation profiles from the SAR-AD[™] are valuable for understanding material properties since the chemical composition of a material impacts its microstructure, and thus the mechanical behavior of the asphalt binder. SAR-AD[™] profiles have been explored for the possibility of developing indicators that correlate with binder performance. One of these indicators is the Absorbance Aging Index Ratio (AAIR) which is the ratio of the toluene soluble asphaltenes to the resins 500 nm peak areas. (Boysen & Schabron, 2015)

$$AAIR = \frac{Toluene Soluble Asphaltenes}{Resins}$$

Building on this, AAIR was calculated for all the samples based on the changes in the concentrations of the SARA fractions. AAIR was calculated for tank samples and recovered samples for comparison. AAIR values are included in the last columns of the preceding tables: Table 4-16 through Table 4-19. AAIR values of tank and recovered asphalt were further analyzed with Bland Altman Plots to determine how laboratory aging and field short term aging compared with respect to degree of oxidation provided by both methods.

4.2.4 Bland Altman Plot of Absorbance Aging Index Ratio

Bland Altman Plot is a tool used to measure agreement between methods. In other words: how does the aging index of laboratory aged asphalt compare with the aging index of the plant aged asphalt recovered from a mix? To answer this question, differences and average AAIR values were plotted, where:

Difference = AAIR (recovered asphalt) – AAIR (tank asphalt)

The plots generated for the SAR-AD[™] results are shown in Figure 4-15 to Figure 4-17. In these graphs, asphalt mixes that contained RAP are highlighted which show the effect of the older, oxidized asphalt from the RAP material on the resultant SARA concentrations of the blended asphalt binder. It is expected, and shown through the graphs, that the mixes containing RAP will produce a higher difference in AAIR due to the forced blending caused by the extraction and recovery process.



Figure 4-15: Bland Altman Plot: Recovered Asphalt from plant mix (short term aged) versus Tank Asphalt (laboratory RTFO aged)



Figure 4-16: Bland Altman Plot: Recovered Asphalt from plant mix (short term aged) versus Tank Asphalt (laboratory RTFO + 20hr PAV-aged)



Figure 4-17: Bland Altman Plot: Recovered Asphalt from plant mix (short term aged) versus Tank Asphalt (laboratory RTFO + 40hr PAV-aged)

With respect to Bland Altman plot analysis, smaller differences (i.e., Bias is closer to zero) suggests that the two methods are producing similar results. In other words, smaller differences suggest the degree of aging/oxidation simulated in the laboratory and produced in the field are similar. This is illustrated by plotting all the points on a single graph in Figure 4-18 to show which pair produced the smallest difference in results.



Figure 4-18: Bland Altman Plot: Recovered Asphalt (field short term aged) versus Tank Asphalt (lab-aged) Figure 4-18 shows that for the samples used in this study, AAIR of laboratory aged RTFO + 20 hours

PAV produced the smallest difference in AAIR. This suggest that the level of oxidation offered by RTFO aging of tank asphalt in the laboratory (laboratory short term aging) is less severe and does not simulate the short-term aging obtained in the field through plant production and placement of the hot mix.

Some of the reasons that can be noted for the differences in aging between laboratory and field include higher and more variable temperatures in the asphalt plant during asphalt mix production than is simulated in an asphalt laboratory. This is the inherent nature of testing samples in a controlled setting in a laboratory versus the natural fluctuations that are expected during bigger scale plant production. Another reason for the difference could be due to thinner asphalt films coating the aggregates in the asphalt plant during production, with more surface area, which exposes more of the asphalt molecules to the higher production temperatures relative to the asphalt films in the RTFO or PAV in the laboratory. This is not to suggest that the laboratory aging is flawed, but to give an appreciation for all the factors that need to be considered when evaluating test data from plant produced asphalt mixes, especially after asphalt binder is recovered from the plant produced mixed.

4.3 Alternative Test Methods for Asphalt Binder and Mix Acceptance

4.3.1 Delta Tc (Δ Tc)

The oxidative behavior of asphalt is one of the critical factors contributing to the performance of asphalt pavements. Pavement distresses associated with oxidized or aged asphalt binders are commonly referred to as non-load related distresses: block cracking, raveling, and longitudinal or transverse cracking.

 ΔT_c is calculated using values from the Bending Beam Rheometer (BBR) test by subtracting the BBR m-critical temperature from the BBR stiffness-critical temperature:

$\Delta T_c = (T_{s-critical} - T_{m-critical})$

The critical temperatures, T_{s-critical} and T_{m-critical}, are the temperatures at which the stiffness and m-value specification requirements are met (i.e., S=300 MPa, m-value=0.300) respectively. They can be determined following ASTM D7643, Standard Practice for Determining the Continuous Grades for PG Graded Asphalt Binders; or AASHTO R29, Standard Practice for Grading or Verifying the Performance Grade (PG) of an Asphalt Binder.

The ΔT_c of an aged binder would be more negative, than that of an unaged binder, and would be more likely to exhibit the non-load related pavement distresses described earlier: block cracking, raveling, and longitudinal or transverse cracking.

 ΔT_c values were calculated for the tank asphalts for each binder from each participating laboratory and is presented in the tables below. Table 4-20 shows the calculated ΔT_c values with the standard 20 hours of PAV aging, and Table 4-21 shows the calculated ΔT_c values after 40 hours of PAV aging, which is considered extended PAV aging. Extended aging is not typically done as part of standard PG grading, however with research suggesting that the 20 hours may not be aggressive enough, (Galal & White, 1997; Huber et al., 2012) 40 hours was included in this research to compare the effect of the additional aging time in the lab.

The data shows ΔTc becoming worse (more negative) with extended aging, which supports the understanding of the impact of oxidation on the asphalt properties. However, the percentage of change in ΔT_c between the 20 and 40 hours is not consistent for all the asphalt binder grades and therefore laboratory aging is a key component of any discussion pertaining to ΔTc . It is also important to note that the COV of ΔTc is high for all the samples included in this research, and especially for the higher-grade asphalt binders that are polymer modified shown in both the 20-hour and 40-hour PAV Aged results. This suggests that more data should be collected to analyze the between laboratory variability of this test method, to determine if this parameter is reliable enough to be used in specification due to the high COV noted from this small sample size. Figure 4-19 shows graphically the differences in ΔTc values with the two aging times.

Sample ID	58-34 8-1031	58-34 4-1003	58-34 3-0915	58-28 6-1006	64-28 7-1010	70-28 1-0708	70-28 2-0809
Average ∆T _c (°C)	0.86	0.85	1.34	1.48	0.87	0.74	0.78
Min	0.00	0.40	0.80	0.25	0.34	0.00	-2.00
Max	1.30	1.42	2.00	2.93	1.30	1.90	5.00
StDev	0.61	0.48	0.61	1.12	0.40	0.73	2.56
COV (%)	71	57	46	76	46	99	329
Sample Size	4	4	3	4	4	5	5

Table 4-20: ΔTc Values for 20-hour PAV Aged Tank Asphalt

Table 4-21: ΔTc Values for 40-hour PAV Aged Tank Asphalt

Sample ID	58-34 8-1031	58-34 4-1003	58-34 3-0915	58-28 6-1006	64-28 7-1010	70-28 1-0708	70-28 2-0809
Average ΔT _c (°C)	-1.31	-2.68	-0.64	-1.08	-1.51	-2.22	-2.28
Min	-2.60	-3.10	-1.00	-1.24	-1.90	-3.00	-3.32
Max	0.78	-2.04	-0.12	-1.00	-0.97	-1.55	-1.12
StDev	1.82	0.57	0.46	0.14	0.48	0.73	1.10
COV (%)	140	21	72	13	32	33	48
Sample Size	3	3	3	3	3	3	3





4.3.2 Evaluation of Flow Number of Plant Produced Asphalt Mix

The flow number, is one of three parameters that were identified in NCHRP Project 9-19 as a parameter related to the rutting resistance of asphalt mixes, the others being dynamic modulus and flow time. (Monismith et al., 2001) The flow number is the number of cycles where shear deformation occurs under constant volume. Each load that's applied to the specimen induces a corresponding permanent axial strain. By graphing the accumulated deformation as a function of load cycles, it is possible to see the development of a permanent strain until failure begins. The calculation of the permanent strain for each load cycle and the flow number for individual specimens is performed automatically by the AMPT. Laboratory conditioning has a major impact on flow number, as these samples were plant produced asphalt mixes, they were short-term conditioned for two hours at the compaction temperature, based on the findings of NCHRP Project 9-43 that showed that this level of conditioning reasonably produced the stiffness of asphalt mixes at the time of construction for both hot mix and warm mix asphalt. (Bonaquist, 2011)

Four flow number test specimens were fabricated in accordance with AASHTO R 83 at a target air void content of 7.0 \pm 0.5 percent; determined after cylindrical coring and sawing of the specimen ends. The flow number tests were conducted in accordance with AASHTO TP79 (AASHTO, 2015) at 58°C, which is the 50 percent reliability performance grade temperature at a depth of 20mm for Southern

Ontario obtained from LTPPBind 3.1 software as per the testing protocol. Table 4-22 shows the results for the mixes included in this research.

Sample ID	58-34 8-1031	58-34 4-1003	58-34 3-0915*	58-28 6-1006	64-28 7-1010	70-28 1-0708	70-28 2-0809*
Flow Number							
(FN)	67	28	109	78	136	2659	3119
Min	32	25	78	60	123	1680	440
Max	112	35	162	96	144	4190	4732
StDev	41	6	46	18	11	1342	2336
COV (%)	60	20	43	23	8	51	75

Table 4-22: AMPT Flow Number of Plant Produced Mixes

*Includes 15% RAP

The results show that generally the Flow Number values trend in the manner expected, i.e., higher FN values correspond to mixtures that are more resistant to rutting. This is illustrated in Table 4-22 with higher PG grades having high FN values, and asphalt mixes with 15% RAP have higher FN values compared to their equivalent virgin mixes. It is also important to note that the standard deviations of the Flow Number test results are high for the asphalt mix with PG 70-28 and even higher when RAP is incorporated in the mix. This suggests that more data should be collected to better understand the repeatability within and between laboratories before considering this test in specifications.

4.3.3 Semicircular Bend Test on Plant Produced Asphalt Mix

The Semi-Circular Bending (SCB) Test was developed as a fracture test to characterize the lowtemperature cracking resistance of asphalt mixtures. The University of Illinois modified the SCB procedure, called the Illinois Flexibility Index Test (I-FIT), based on a thorough investigation of test temperatures, loading rates and sample geometry to quantify the cracking potential of asphalt mixtures at intermediate temperature. (Ling et al., 2017) The Flexibility Index (FI) parameter combines mixture fracture energy and post-peak failure behavior of the mixture to determine cracking resistance of asphalt mixtures. Researchers have found that the I-FIT shows consistent and repeatable trends for changes in asphalt mix design properties, and the FI parameter is shown to provide a greater distinction between mixtures' fracture properties relative to the total fracture energy parameter alone. (Ling et al., 2017)

I-FIT test uses semi-circular bending (SCB) specimen geometry to determine the fracture resistance of an asphalt mixture at an intermediate temperature. The provisional standard test method, AASHTO

TP-124, "Determining the Fracture Potential of Asphalt Mixtures Using the Semi-Circular Geometry at Intermediate Temperature," calls for 50-mm thick, 150-mm diameter semi-circular specimens to be tested using a three-point bending principle, at the constant displacement rate of 50 mm/min. Figure 4-20 presents a photograph of the I-FIT test arrangement. A 15-mm deep, 1.5-mm wide notch is cut along the specimen's axis of symmetry to force the failure location. Prior to testing, the test specimen is conditioned for two hours in an environmental chamber at 25°C, the standard test temperature.



Figure 4-20: I-FIT Test using Semi-Circular Bending (SCB) specimen geometry.

One of the primary outputs of I-FIT is the fracture energy, which represents the energy dissipated by the crack propagation. This parameter is calculated as the area under the load-displacement curve divided by the area of the crack that propagates during testing. The fracture energy is a function of both the strength and ductility of the material, which are related to the peak load and maximum displacement, respectively. The I-FIT test quantifies the cracking resistance of asphalt mixtures using the Flexibility Index (FI), which includes the fracture energy and post-peak behavior of a mixture. Generally, the higher the fracture energy, the better the cracking resistance. (Kaseer et al., 2018)

Sample ID	58-34 8-1031	58-34 4-1003	58-34 3-0915*	58-28 6-1006	64-28 7-1010	70-28 1-0708	70-28 2-0809*
Average FI	5.8	9.2	5.8	10.8	6.2	7.0	2.2
Min	4.3	7.6	4.9	9.3	5.3	5.7	1.8
Max	7.3	10.3	8.2	12.9	7.4	8.9	2.5
StDev	1.5	1.3	1.6	1.6	1.0	1.6	0.3
COV (%)	25	14	27	15	16	22	14

Table 4-23: IFIT Flexibility Index Results for Plant Produced Asphalt Mixes



Illinois Flexibility Index Test

Figure 4-21: Graph of IFIT Results for Plant Produced Asphalt Mixes

The I-FIT test quantifies the cracking resistance of asphalt mixtures using the Flexibility Index (FI), and the results of the asphalt mixes in the research suggest that the asphalt mixes with 15% RAP had lower FI values than their equivalent virgin asphalt mixes, meaning these RAP mixes would be less resistant to fatigue cracking at intermediate temperature than the virgin mixes. It was noted that the standard deviation and COV of the small sample size from this research was better than those obtained in the Flow Number. More data should be collected to understand the between-laboratory variability of this test before implementation in specifications.

4.4 Correlation Tables Comparing Tank Asphalt and Recovered Asphalt

To evaluate how the properties of the asphalt binder and plant-produced asphalt mix correlate with each other and with performance, correlation tables were produced to evaluate separately the properties of tank asphalt and recovered asphalt.

The first correlation is shown in Figure 4-22 for PG High Temperature values of tank and recovered asphalt samples. The graph shows $R^2 = 0.57$ which not a strong correlation, and interesting to note how the recovered PG High temperature results are consistently higher than the tank asphalt, and the impact of RAP as indicated with the red data points.



Figure 4-22: Correlation of PG High Temp (Tank vs Recovered). Red data points indicate asphalt mix with RAP

The second correlation is shown in Figure 4-23 for Grade Loss from the Extended BBR test for low temperature tank and recovered asphalt samples. The graph shows $R^2 = 0.0795$ which is a poor correlation, and interesting again to note how the recovered Grade Loss results generally higher than the tank asphalt. Asphalt mixes with RAP are highlighted as red data points.



Figure 4-23: Correlation of Grade Loss from Extended BBR (Tank vs Recovered). Red data points indicate asphalt mix with RAP.

Lastly, to evaluate how all the properties of the tank asphalt and recovered asphalt compare to asphalt mix performance properties, two correlation tables were prepared to include all the asphalt binder properties as follows: Table 4-24 is a correlation table that summarizes the material properties determined for the tank asphalt binder, including Delta Tc, along with rutting and cracking properties determined from the Flow Number and I-FIT respectively for plant produced asphalt mixes. Table 4-25 summarizes all the material properties determined for the recovered asphalt binder from plant produced mix, along with the rutting and cracking mix properties from the plant produced mixes.

For a correlation table:

- -1 indicates a perfect negative linear correlation between two variables.
- 0 indicates no linear correlation between two variables.
- 1 indicates a perfect positive linear correlation between two variables.

Understanding that not all the parameters have linear relationships, highlighted in the correlation tables are parameters that produce "strong" linear correlations (between |0.7 - 1|).

Highlighted in Table 4-24 are the material properties determined for the tank asphalt and the rutting and cracking properties from plant produced asphalt mix that have a strong correlation (between |0.7 - 1|). Highlighted in Table 4-25 are the material properties determined for the recovered asphalt and the rutting and cracking properties of plant produced asphalt mix that have a strong correlation. For both tables, cells highlighted orange are properties with a negative correlation, cells highlighted blue have a positive correlation.

	-0.6	-0.6	-0.3	-0.9	-0.6	0.2	0.2	0.3	0.6	0.6	-0.5	0.4	0.9	-0.3	0.4	AMPT Flow
		0.8	-0.1	0.7	0.3	-0.2	-0.3	0.4	-0.2	-0.4	0.4	-0.3	-0.6	0.4	-0.6	IFIT Slope
			-0.3	0.8	0.2	-0.4	-0.5	0.1	-0.3	-0.6	0.7	-0.2	-0.7	0.4	-0.7	IFIT
				0.2	0.4	0.7	0.7	0.0	-0.6	-0.4	0.2	0.1	-0.3	-0.4	0.2	Delta Tc (40Hr)
					0.5	-0.2	-0.2	-0.1	-0.6	-0.8	0.8	-0.4	-1.0	0.4	-0.4	Delta Tc (20Hr)
						-0.3	-0.4	-0.3	-0.7	-0.2	0.1	-0.8	-0.5	0.7	-0.4	СТОР
							1.0	0.5	0.0	-0.1	0.0	0.5	0.1	-0.8	0.5	LTLG (40Hr)
								0.4	0.0	-0.1	0.1	0.6	0.1	-0.9	0.5	LTLG (20Hr)
									0.4	0.1	0.0	0.2	0.2	-0.2	0.2	Grade Loss (40Hr)
										0.7	-0.6	0.2	0.6	-0.1	0.4	Grade Loss (20Hr)
											-1.0	-0.1	0.8	0.1	0.3	MSCR %Rec 3.2kPa
												0.2	-0.7	-0.1	-0.3	MSCR Jnr
													0.3	-0.9	0.1	PG Low
														-0.3	0.3	PG High
															-0.3	Ash
																RAP
AMPT Flow	IFIT Slope	IFIT	Delta Tc (40Hr)	Delta Tc (20Hr)	СТОР	LTLG (40Hr)	LTLG (20Hr)	Grade Loss	Grade Loss	MSCR %Rec	MSCR Jnr	PG Low	PG High	Ash	RAP	TANK ASPHALT

Table 4-24: Correlation Table for Tank Asphalt and Plant Produced Mix Properties

RECOVERED ASPHALT	RAP	Ash	PG High	PG Low	MSCR Jnr	MSCR %Rec 3.2kPa	Grade Loss (20Hr)	Grade Loss (40Hr)	LTLG (20Hr)	LTLG (40Hr)	стор	Delta Tc (20Hr)	Delta Tc (40Hr)	IFIT	IFIT Slope	AMPT Flow
RAP																
Ash	0.2															
PG High	0.3	0.5														
PGLow	0.3	0.4	0.5													
MSCR Jnr	-0.3	-0.2	-0.7	0.0												
MSCR %Rec 3.2kPa	0.1	-0.2	0.5	-0.2	-0.8											
Grade Loss (20Hr)	0.1	0.7	0.5	0.3	-0.3	0.0										
Grade Loss (40Hr)	-0.3	0.4	0.1	0.0	-0.1	-0.1	0.3									
LTLG (20Hr)	0.4	0.5	0.5	0.8	-0.1	-0.2	0.4	0.2								
LTLG (40Hr)	0.1	0.7	0.5	0.6	-0.2	-0.1	0.6	0.5	0.8							
СТОД	-0.3	-0.6	-0.5	-0.6	0.1	0.2	-0.8	-0.3	-0.6	-0.6						
Delta Tc (20Hr)	-0.5	-0.5	-0.5	-0.8	0.2	0.1	-0.3	-0.1	-0.7	-0.6	0.5					
Delta Tc (40Hr)	0.1	-0.7	-0.5	-0.2	0.3	0.0	-0.6	-0.5	-0.4	-0.8	0.6	0.3				
IFIT	-0.7	-0.2	-0.6	-0.4	0.6	-0.5	0.0	0.0	-0.4	-0.2	0.1	0.5	0.0			
IFIT Slope	-0.6	-0.2	-0.6	-0.5	0.3	-0.3	0.0	0.1	-0.5	-0.2	0.2	0.6	0.0	0.8		
AMPT Flow	0.4	0.1	0.8	0.4	-0.5	0.6	0.2	-0.1	0.3	0.1	-0.2	-0.3	0.0	-0.6	-0.6	

Table 4-25: Correlation Table for Recovered Asphalt and Plant Produced Mix Properties

Observations made based on from these correlation tables are as follows:

- Ash content (%) determined on tank asphalt (Table 4-24) has a negative correlation with the PG low temperature, i.e., as the ash content increases, the PG low temperature of the binders decreases (becoming less negative). Similar results are noted for the Low temperature limiting grade (LTLG) from the ExBBR test developed by MTO to address the physical hardening phenomenon that changes asphalt binder properties at low temperature.
- PG high temperature of tank asphalt has a positive correlation with MSCR %Recovery and Flow Number, i.e., for the samples in this research, the higher PG high-temperature grades correlate with better resistance to rutting captured by Flow Number.
- As RAP content increased (comparing 0% RAP with 15% RAP mix), the I-FIT Flexibility Index decreased, meaning the mix is more susceptible to cracking compared to the virgin asphalt mix.
- The Delta Tc values of the tank asphalt binders (Table 4-24) showed a positive correlation with the I-FIT Flexibility Index of the resultant mix (i.e., the more positive the Delta Tc value of the tank asphalt, the larger the flexibility index, which means it would be more resistant to non-load related pavement distresses. The Delta Tc showed a negative correlation with Flow Number, supporting our understanding that a less stiff asphalt mix that has a higher I-FIT Flexibility Index, would thus have a lower Flow Number.

4.5 Summary of Research Results

This research compared the physical properties of original asphalt binder to the properties of the same asphalt binder recovered from asphalt mix after plant production. The asphalt mixes included seven surface course mixes, two of which include 15 percent RAP. The asphalt mixes were produced with most common PG grades of asphalt binder used in Ontario.

The results showed that the significant increase in ash content in the recovered asphalt binder coupled with the difference in oxidation between laboratory aging and plant production produced rheological properties that show the recovered asphalt was stiffer and less representative of the tank asphalt. There was a statistically significant difference between the tank asphalt properties and the recovered asphalt properties. Additionally, the physical properties of recovered asphalt showed higher variability than the same physical property tests on tank asphalt.

This research also compared the aging that is simulated by the RTFO and PAV aging in the laboratory, to the short-term aging that occurs during asphalt mix production and placement on a job site. The aging was determined using SAR-AD[®] chemical analysis to determined concentrations of the asphalt fractions (i.e., saturates, aromatics, resins, and asphaltenes).

The concentrations were used to calculate an aging index as an indication of degree of oxidation for laboratory versus field. The outcome of the Bland Altman analysis showed that the level of oxidation offered by RTFO aging of tank asphalt in the laboratory (laboratory short term aging) is less severe and does not simulate the short-term aging obtained in the field through plant production and placement of the hot mix.

Lastly, although the Superpave PG system is an improvement over previous grading systems, the PG system evaluates cracking behavior by only considering properties of asphalt binder and fails to consider the aggregate portion of the asphalt mixes, which makes up about 90 to 95% of the total weight of the asphalt mix. Furthermore, new parameters have been researched since the implementation of Superpave that better characterize the oxidative behavior of asphalt binders. In this research, the Delta Tc (ΔT_c) parameter was evaluated for the asphalt binders, along with Illinois Flexibility Index Test, and Asphalt Mix Performance Tester (AMPT) Flow Number for the asphalt mixes.

Correlation tables indicated that the properties of the tank asphalt binder had better correlation with the IFIT Flexibility Intext and AMPT Flow Number of the asphalt mixes than the recovered asphalt binder properties.

5 CONCLUSIONS AND RECOMMENDATIONS

5.1 General Summary

This research was conducted with the main purpose of developing a framework to better characterize the asphalt binder and asphalt mixes with RAP with field verified test methods and parameters linked to pavement performance. This required the development of specific objectives to 1) Evaluate the inter-laboratory standard deviation of the test methods utilized for acceptance of asphalt binders in Ontario; 2) Compare the oxidative aging that is simulated by the laboratory conditioning, to the shortterm aging that occurs in the field; and 3) Evaluate the asphalt binders and mixes using more recent test methods that have been shown to predict performance.

The analysis of this research showed that the recovered asphalt produced rheological properties that was stiffer and less representative of the tank asphalt. Additionally, the physical properties of recovered binder, were shown to have much higher variability than would be experienced if performing the same physical property tests on tank asphalt. (MTO, 2016; Wakefield et al., 2018)

Furthermore, new parameters and performance tests proposed in this research: the Delta Tc (ΔT_c) parameter for asphalt binder, the Illinois Flexibility Index Test (I-FIT), and Asphalt Mix Performance Tester (AMPT) Flow Number for the asphalt mixes, showed better correlation with other field verified performance criteria than with recovered asphalt binder properties. It is important to note that the sample size in this study was small and therefore the repeatability of these tests should be further evaluated through round robin tests between laboratories before any decision is made about including these in specifications for acceptance.

If the testing protocols used for acceptance are not both accurate and precise, owners will have a challenge distinguishing between good and poor performing materials. As such, it is recommended that users should exercise caution when comparing values of recovered asphalt to test criteria and variability derived for unrecovered (original) asphalt.

5.2 Major Findings and Conclusions

The major findings of this research can be summarized as follows:

- There is a statistically significant difference in test results for tank and recovered asphalt when testing for ash content for all PGAC grades.
- There is a statistically significant difference in test results for tank and recovered asphalt when RAP is incorporated in the asphalt mix.
- There is no difference in test results when testing PG low temperature of tank and recovered asphalt, except for PGAC 64-28.
- There is a statistically significant difference in test results for CTOD when RAP is incorporated in the mix and recovered for testing.
- For the samples used in this research, chemical analysis of asphalt fractions through SAR-AD analysis suggest that the level of aging offered by RTFO of tank asphalt in the laboratory (laboratory short term aging) is less severe and does not simulate the short-term aging obtained in the field through plant production and construction of the asphalt mix.
- The Flow Number values from the AMPT showed that higher high temperate PG grades and polymer modified grades had higher FN values, correspond to mixtures that are more resistant to rutting. Asphalt mixes with 15% RAP also had higher FN values compared to their equivalent virgin mixes.
- The I-FIT test for cracking resistance of asphalt mixtures showed that the asphalt mixes with 15% RAP had lower FI values than their equivalent virgin asphalt mixes, meaning these RAP mixes would be less resistant to fatigue cracking at intermediate temperature than the virgin mixes.
- Correlation tables of tank asphalt and recovered asphalt binder properties with asphalt mix properties through field verified performance tests showed that the properties of the tank asphalt binder showed better correlation with the IFIT Flexibility Index and AMPT Flow Number of the asphalt mixes than with the recovered asphalt binder properties.

5.3 Significant Contributions

Based to the laboratory work conducted in this research, major contributions of the study are highlighted in Table 5-1 through the data collected which was not available prior to this study:

Test Description	Purpose of Test
Extraction of Asphalt Cement and Analysis	Remove asphalt binder from asphalt mix
of Extracted Aggregate	
Recovery of Asphalt from Solution by Abson	Remove solvent from asphalt and solvent solution.
or Rotary Evaporator	
Ash Content	Percentage of inorganic materials in asphalt.
	This test was not required for recovered asphalt
	prior to this research.
Grading or Verifying the Performance Grade	Determine PG high and low temperatures.
of an Asphalt Binder	
Multiple Stress Creep Recovery Test of	Tendency to permanent deformation.
Asphalt Binder Using a Dynamic Shear	There was no data in Canada comparing original
Rheometer	(tank) and recovered asphalt for this test, and
	there was no ILS on recovered asphalt testing.
Performance Grade of Physically Aged	Physical hardening of asphalt.
Asphalt Cement using Extended Bending	This test is unique to Ontario and there was no ILS
Beam Rheometer (ExBBR)	was available on recovered asphalt specifically on
	plant-produced asphalt mixes prior to this study.
Asphalt Cement's Resistance to Ductile	Resistance to ductile failure.
Failure Using Double Edge Notched Tension	This test is unique to Ontario and there was no ILS
(DENT) Test	was available on recovered asphalt specifically
	from plant-produced asphalt mixes prior to this
	study.
Accelerated Aging of Asphalt using Pressure	Asphalt binder extended aging.
Aging Vessel Protocols	This test is not used in specification, but a method
	exists in Ontario. There was no data on ΔTc of 40-
	hours PAV aging until this study.
Delta Tc	Asphalt binder relaxation.
	This research generated a database of ΔTc values
	of various binder grades used in Ontario including
	PG 58-34, 58-28, 64-28, and 70-28. This includes
	ΔTc after 20-hours and 40-hours PAV aging.

Table 5-1. Summary of Significant Contributions	Table	5-1:	Summary	of	Significant	Contributions
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*New data contribution

• With respect to other properties measured such as Grade Loss from ExBBR test, Non-Recoverable Creep Compliance from the MSCR test, and the CTOD parameter from the DENT test, at the time of this research there was no ILS conducted for recovered asphalt binder. The results for five labs in this study showed that the higher ash content in the recovered asphalt and/or differences in oxidation in laboratory and plant production produced generally stiffer results than the tank asphalt.

- There is a statistically significant difference in test results for tank and recovered asphalt when testing for ash content for all PGAC grades, as well as when RAP is incorporated in the asphalt mix.
- Chemical analysis of asphalt fractions and subsequent calculation of aging index of laboratory aged and recovered asphalt from plant produced asphalt mix showed that RTFO + 20 hours PAV produced the smallest difference in aging index as obtained from plant production and construction.
- Correlation tables of tank asphalt and recovered asphalt binder properties with asphalt mix properties through field verified performance tests showed that the properties of the tank asphalt binder showed better correlation with the IFIT Flexibility Index and AMPT Flow Number of the asphalt mixes than with the recovered asphalt binder properties.
- Although this research included limited laboratories in the ILS, this is a start for a database of test results that can be referenced and expanded for future use by industry as agencies continue to explore the use of recovered asphalt properties for investigative purposes.

Since the publication of these results, user agencies in Ontario have made the following to their specifications:

- User agencies require Ash content test on asphalt binder and now include a limit <1.0% in specification in Ontario when testing recovered asphalt.
- User agencies are opting to collect more data on recovered asphalt from plant produced mixes before adopting any specifications.
- User agencies are using recovered asphalt testing to set limits of high and low temperature grade only, to ensure responsible use of RAP in asphalt mixes and opting to test other properties of recovered asphalt for information purposes only.

5.4 Recommendations and Future Work

In this research, asphalt binder and plant-produced asphalt mixes were investigated with a purpose of developing a framework to better characterize the asphalt binder and asphalt mixes with RAP with field verified test methods and parameters linked to pavement performance.

The sample size of the ILS is understandably small, due to the complexity of collecting asphalt binder and asphalt mix samples as the various stages of the production process. Nonetheless the findings of the research determined that there is a statistically significant difference in test results for tank and recovered asphalt for several parameters due to the increased fines in the recovered asphalt which produced an overall stiffer binder. During the time of this research, MTO was developing a new test procedure to refine the solved extraction and recovery procedure to reduce the number of residual fines. It is recommended that any changes to test procedure to be followed up with another industry analysis to determine the impact of the procedural changes on the results with similar analysis of statistical significance and should also include asphalt mixes with RAP incorporated at various percentages as this research included 15% only.

The research also showed that there was no difference in test results when testing PG low temperature of tank and recovered asphalt, except for PGAC 64-28. This suggests that there is an opportunity to use the results from testing PG low temperature to calculate Delta Tc values for recovered asphalt from plant produced asphalt mix. It was noted that the standard deviation and coefficient of variance was higher for Delta Tc based on the limited sample size and therefore more work is needed to understand the cause of this high variability and to improve the test method prior to using this parameter in specification.

The I-FIT test for cracking resistance of asphalt mixtures showed that the asphalt mixes with 15% RAP had lower FI values than their equivalent virgin asphalt mixes, additional data can be collected going forward to determine the appropriate FI threshold to provide desirable premature fatigue cracking resistance. The standard deviation and coefficient of variance should be evaluated between laboratories as this research showed good standard deviation for testing in one lab.

Agencies will need to establish the precision and bias statements for the performance tests they chose to adopt, by setting up experiments to determine the between lab variability of performance tests. Agencies will also need to establish a relationship between the laboratory produced samples for mix design, and plant-produced field samples for quality assurance. Extensive testing will be required to have collect data to prepare standard deviations and coefficient of variations to determine precision and bias. This research is a starting point toward that effort.

Furthermore, pavement distress data in the field can be collected from in-service highways where the asphalt mixes investigated in this research were constructed. The test data obtained for Delta Tc, I-FIT Flexibility Index, and AMPT Flow Number in conjunction with the in-service pavement distress data can be used to fine-tune preliminary acceptance criteria for I-FIT and AMPT FN, to allow asphalt mixes to be eventually accepted based on asphalt mix performance testing. The research included a limited sample size however forms a good starting point for a database of test results of asphalt binder properties and plant produced asphalt mixes that can be referenced as agencies continue to investigate new parameters and specification limits to characterize asphalt mix durability.

REFERENCES

- AASHTO, A. A. of S. H. T. O. (2004a). AASHto T 240: Effects of heatand air on moving film of asphalt (rolling thin film oven test). American Association of State Highway Transportation Officials AASHTO.
- AASHTO, A. A. of S. H. T. O. (2004b). *T240: Effects of Heatand Air on Moving Film of Asphalt (Rolling Thin Film Oven Test)*. American Association of State Highway Transportation Officials AASHTO.
- AASHTO, A. A. of S. H. T. O. (2012a). AASHTO R28: Standard practice for accelerated aging of asphalt binder using a pressure aging vessel (PAV). American Association of State Highway Transportation Officials AASHTO.
- AASHTO, A. A. of S. H. T. O. (2012b). *R 29-08: Grading or Verifying the Performance Grade (PG) of an Asphalt Binder*. American Association of State Highway Transportation Officials AASHTO.
- AASHTO, A. A. of S. H. T. O. (2014a). AASHTO T 350: Standard method of test for multiple stress creep recovery (mscr) test of asphalt binder using a dynamic shear rheometer (dsr). American Association of State Highway Transportation Officials AASHTO.
- AASHTO, A. A. of S. H. T. O. (2014b). *T 350: Standard Method of Test for Multiple Stress Creep Recovery* (*MSCR*) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR). American Association of State Highway Transportation Officials AASHTO.
- AASHTO, A. A. of S. H. T. O. (2015). *TP 79-12: Standard Method of Test for Determining the Dynamic Modulus and Flow Number for Hot Mix Asphalt (HMA) Using the Asphalt Mixture Performance Tester (AMPT)*. American Association of State Highway Transportation Officials AASHTO.
- AASHTO, A. A. of S. H. T. O. (2016). *M320: Standard Specification for Performance-Graded Asphalt Binder*. American Association of State Highway Transportation Officials AASHTO.
- Abson, G. (1933). Method and Aparatus for the Recovery of Asphalt. ASTM, II, 704–714.
- Abson, G., & Burton, C. (1960). The Use of Chlorinated Solvents in the Abson Recovery Method. Association of Ashalt Paving Technologists, 29, 246–252.
- Albritton, G. E., Barstis, W. F., & Crawley, A. B. (1999). *Polymer Modified Hot Mix Asphalt Field Trial* (Issue FHWA/MS-DOT-RD-99-111). Mississippi Department of Transportation.

Anderson, R. M. (Asphalt I. (2007). Asphalt Handbook, Manual Series No. 4 (MS-4) 7th Edition. Asphalt

Institute.

Anderson, R. M. (Asphalt I. (2011). MS-26 Asphalt Binder Handbook. Asphalt Institute.

- Angius, E., Ding, H., & Hesp, S. A. . (2018). Durability assessment of asphalt binder. *Construction and Building Materials*, 165, 264–271.
- Arnold, T. ., & Shastry, A. (2015). Analysis of Asphalt Binders for Recycled Engine Oil Bottoms by X-ray Fluorescence Spectroscopy. *Transportation Research Board*.
- ASTM, A. S. for T. and M. I. (1993). *D5404: Standard Practice for Recovery of Asphalt from Solution Using* the Rotary Evaporator.
- ASTM, A. S. for T. and M. I. (2014). Standard Practice for Recovery of Asphalt from Solution Using Toluene and the Rotary Evaporator.
- ASTM, A. S. for T. and M. I. (2015). *D1856-09: Standard Test Method for Recovery of Asphalt From Solution* by Abson Method.
- ASTM, A. S. for T. and M. I. (2017). D2172/D2172M-17: Standard Test Methods for Quantitative Extraction of Bitumen from Bituminous Paving Mixtures.
- ASTM, A. S. for T. and M. I. (2020). *D7313-20, Standard Test Method for Determining Fracture Energy of Asphalt Mixtures Using the Disk-Shaped Compact Tension Geometry*. ASTM International.
- ASTM C670. (2015). Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials. *ASTM International*, 1–9. www.astm.org,
- ASTM E177. (2014). Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods. ASTM Standards, 177(E177).
- Aurilio, M., Mikhailenko, P., & Baaj, H. (2017). Predicting HMA Fatigue Using the Double Edge Notched Tension Test and Multiple Stress Creep Recovery Test. *Proceedings of the Sixth-Second Annual Conference of the Canadian Technical Asphalt Association (CTAA), November*, 267–290.
- Batioja-Alvarez, D., Lee, J., & Haddock, J. E. (2019). Understanding the Illinois Flexibility Index Test (I-FIT) using Indiana Asphalt Mixtures. *Transportation Research Record*, 2673(6), 337–346. https://doi.org/10.1177/0361198119841282

Blankenship, P., Anderson, R. M., King, G. N., & Hanson, D. I. (2010). a Laboratory and Field Investigation

to Develop Test Procedures for Predicting Non-Load Associated Cracking of Airfield HMA Pavements. Airfield Asphalt Pavement Technology Program, 09.

- Bonaquist, R. (2011). Mix Design Practices for Warm-Mix Asphalt. In *Mix Design Practices for Warm-Mix Asphalt*. https://doi.org/10.17226/14488
- Bonaquist, R., Adams, J. J., & Anderson, D. A. (2021). Asphalt Binder Aging Methods to Accurately Reflect Mixture Aging. In *Asphalt Binder Aging Methods to Accurately Reflect Mixture Aging*. https://doi.org/10.17226/26089
- Boysen, R., & Schabron, J. (2015). Automated HPLC SAR-AD Separation: Fundamental Properties of Asphalts and Modified Asphalts III Product: FP 01. Federal Highway Administration.
- Burke, K., Wright, L. ., Hesp, S. A. ., Marks, P., & Raymond, C. (2011). Effects of engine oil residues on asphalt cement quality (January 20). ResearchGate. https://www.researchgate.net/publication/303162535
- Burr, B. ., Glover, C. ., Davison, R. ., & Bullin, J. . (1993). New Apparatus and Procedure for the Extraction and Recovery of Asphalt Binder from Pavement Mixtures. *Transportation Research Board*, 1391, 20–29.
- Christensen, D. W., & Tran, N. (2021). Relationships Between the Fatigue Properties of Asphalt Binders and the Fatigue Performance of Asphalt Mixtures. In *Relationships Between the Fatigue Properties* of Asphalt Binders and the Fatigue Performance of Asphalt Mixtures. https://doi.org/10.17226/26302
- Davis, H. L. (1983). Progress Report: AR Grading System Subcommittee to Investigate ASTM D 1856-75, Abson Recovery Test Method. In *Pacific Coast User-Producer Conference*.
- Delfosse, F., Plance, J. ., Largeaud, S., Drouadaine, I., & Turner, F. (2017). Base Asphalt Variability: An Opportunity for Development of Innovative Characterization Approaches. *Canadian Technical Asphalt Association*, *65*, 23–50.
- Ding, Y., Huang, B., & Shu, X. J. (2018). Blending efficiency evaluation of plant asphalt mixtures using fluorescence microscopy. *Construction and Building Materials*, *161*, 461–467.
- Dubois, E., Mehta, D. Y., & Nolan, A. (2014). Correlation between multiple stress creep recovery (MSCR) results and polymer modification of binder. *Construction and Building Materials*, *65*, 184–190.

https://doi.org/10.1016/J.CONBUILDMAT.2014.04.111

- Farcaș, F. A. (2012). Evaluation of Asphalt Field Cores with Simple Performance Tester and X-ray Computed Tomography.
- Feng, C., Wang, Z., Ma, Y., Zhang, Y., Wang, L., & Bi, Y. (2017). Ultrathin graphitic C3N4 nanosheets as highly efficient metal-free cocatalyst for water oxidation. *Applied Catalysis B: Environmental*, 205, 19–23. https://doi.org/10.1016/j.apcatb.2016.12.014
- Galal, K. A., & White, T. D. (1997). SHRP PG classification and evaluation of in-service asphalts after eight years. In ASTM Special Technical Publication (Vol. 1322). https://doi.org/10.1520/stp12085s
- Hesp, S. A. ., Genin, S. ., Scafe, D., Shurvell, H. ., & Subramani, S. (2009). Five Year Performance Refiew of a Northern Ontario Pavement Trial: Validation of Ontario's Double-Edge-Notched Tension (DENT) and Extended Bending Beam Rheometer (BBR) Test Methods. *Canadian Technical Asphalt Association*, 54, 99–126.
- Hesp, S. A. ., Johnson, K.-A. N., & Andriescu, A. (2014). Double-Edge-Notched Tension Testing for the Fatigue Grading of Straight and Modified Asphalt Binders. *International Journal of Pavements*, *13*(January), 1–12.
- Heyes, G. B. (1993). ASTM E691–87 Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method. *Journal of Quality Technology*, 25(4), 313–314. https://doi.org/10.1080/00224065.1993.11979478
- Hofko, B., & Hospodka, M. (2016). Rolling Thin Film Oven Test and Pressure Aging Vessel Conditioning Parameters. *Transportation Research Record*, 111–116.
- Hou, X., Xiao, F., Wang, J., & Amirkhanian, S. (2018). Identification of asphalt aging characterization by spectrophotometry technique. *Fuel*, 226, 230–239. https://doi.org/10.1016/J.FUEL.2018.04.030
- Huber, G., Marks, P., Brown, A., & Raymond, C. (2012). Relationship of Asphalt Binder Properties to Cracking in Five Year Old Test Sections. *Canadian Technical Asphalt Association*, 321–348.
- Hugener, M., & Pittet, M. (2016). Extraction and Recovery of Polymer Modified Bitumen. In *Eurosphalt & Eurobitume Congress*.
- Illinois-DOT. (2016). Determining the Fracture Potential of Asphalt Mixtures Using the Illinois Flexibility Index Test (I-FIT).

- Kaseer, F., Yin, F., Arámbula-Mercado, E., Epps Martin, A., Daniel, J. S., & Salari, S. (2018). Development of an index to evaluate the cracking potential of asphalt mixtures using the semi-circular bending test.
 Construction and Building Materials, 167(February), 286–298.
 https://doi.org/10.1016/j.conbuildmat.2018.02.014
- Lane, B. (2015). Addressing Asphalt Cement Quality Concerns (M. T. O. Ministry of Transportation (ed.)).
- Ling, C., Swiertz, D., Mandal, T., Teymourpour, P., & Bahia, H. (2017). Sensitivity of the Illinois Flexibility Index Test to Mixture Design Factors. *Transportation Research Board*, *2017*.
- Loeber, L., Sutton, O., Morel, J., Valleton, M., & Muller, G. (1996). New direct observations of asphalts and asphalt binders by scanning electron microscopy and atomic force microscopy. *Journal of Microscopy*, *182*(1), 32–29. https://doi.org/10.1046/j.1365-2818.1996.134416.x
- Lv, Q., Huang, W., Sadek, H., Xiao, F., & Yan, C. (2019). Investigation of the rutting performance of various modified asphalt mixtures using the Hamburg Wheel-Tracking Device test and Multiple Stress Creep Recovery test. *Construction and Building Materials*, 206, 62–70. https://doi.org/10.1016/J.CONBUILDMAT.2019.02.015

Lysyk, B. (2016). 2016 Annual Report. Office of the Auditor General of Ontario.

- McDaniel, R. ., & Anderson, R. . (2001). *Recommended Use of Reclaimed Asphalt Pavement in the Superpave Mix Design Method: Technician's Manual* (Issue Report 452). National Cooperative Highway Research Program (NCHRP).
- Menapace, E., Masad, E., & Bhasin, A. (2016). Evolution of the microstructure of unmodified and polymer modified asphalt binders with aging in an accelerated weathering tester. *Journal of Microscopy*, 263(3), 341–356. https://doi.org/10.1111/jmi.12405
- Meng, F., Shuyi, M., M, Y., & Jung, L. (2020). Analysis of virgin asphalt brands via the integrated application of ftir and gel permeation chromatography. *Arabian Journal of Science and Engineering*, 45. https://doi.org/10.1007/s13369-020-04539-x
- Mikhailenko, P., Ataeian, P., & Baaj, H. (2019). Extraction and recovery of asphalt binder: a literature review. International Journal of Pavement Research and Technology, 13. https://doi.org/10.1007/s42947-019-0081-5

Mikhailenko, P., & Baaj, H. (2017). Survey of Current Asphalt Binder Extraction and Recovery Practices. In

Transportation Association of Canada.

- Modi, K., Tabib, S., Bashir, I., & Marks, P. (2016). Innovative testing of ontario's asphalt materials. *Canadian Technical Asphalt Association*, 195–214.
- Monismith, C. L., Musselman, J. A., Dot, F., Samuels, J. M., Hoel, L. A., Horsley, J. C., Peters, M. E., Highway Administration ROBERT SKINNER, F. E., Jencks, C. F., David Beal, N. B., Barry, T. F., Buffington, J. E., Frosch, R. A., Gilbert, G., Lacy Distinguished Professor, L. A., Mallory, B. L., Meyer, M. D., Morales, J. P., Moreland, J. R., ... Oge, M. T. (2001). *Deputy Secretary of Transportation*. http://www.trb.org/trb/bookstore
- MTO. (2016). Recovered AC Testing Correlation v2_2016-05-10 From MTO.
- MTO, M. of T. (2017). LS-228: Method of Test for Accelerated Aging of Aspalt Cement Using Modified Pressure Aging Vessel Protocols.
- MTO, M. of T. O. (2007). LS-299: Method of Test for Asphalt Cement's Resistance to Fatigue Fracture Using Double-Edge-Notched Tension Test (DENT). In *MTO Laboratory Testing Manual*. Ministry of Transportation of Ontario.
- MTO, M. of T. O. (2011). LS-308: Method of Test for Determining of Performance Grade of Physically Aged Asphalt Cement Using Extended Bending Beam Rheometer (BBR) Method Rev No 26. Ministry of Transportation of Ontario Laboratory Testing Manual.
- MTO, M. of T. O. (2014). Amendment to OPSS 1101, November 2014 Additional Test Results and Samples for Performance Graded Asphalt Cement (PGAC). In *Ontario Provincial Standard Specification* (Issue 111, pp. 1–7).
- MTO, M. T. O. (2015). LS-227: Method of Test for Determination of Ash Content, Rev No 31. In *Ministry of Transportation of Ontario Laboratory Testing Manual*.
- Nosler, I., Tanghe, T., & Soenen, H. (2008). Evaluation of Binder Recovery Methods and the Influence on the Properties of Polumer Modified Bitumen. In *Eurasphalt & Eurobitume Congress*.
- Paliukaite, M., Verigin, M., & Hesp, S. A. M. (2015). Double-edge-notched tension testing of asphalt cement for the control of cracking in flexible asphalt pavements. *Bituminous Mixtures and Pavements VI - Proceedings of the 6th International Conference on Bituminous Mixtures and Pavements, ICONFBMP 2015*, 13–20. https://doi.org/10.1201/B18538-4
- Petersen, J. C. (2009). A Review of the Fundamentals of Asphalt Oxidation. Transportation Research Board of The National Academies.
- Pierard, N., Vansteenkiste, S., & Vanelstraete, A. (2010). Effect of Extraction and Recovery Procedure on the Determination of PmB Content and on the Properties of the Recovered Binder. *Road Materials and Pavement Design*, *11*, 251–279.
- Planche, J. P., Elwardany, M. D., Rodezno, C., & D. Christensen, D. (2018). NCHRP 09-60: Addressing Impacts of Changes in Asphalt Binder Formulation and Manufacture on Pavement Performance through Changes in Asphalt Binder Specifications, Interim Report. https://apps.trb.org/cmsfeed/TRBNetProjectDisplay.asp?ProjectID=4036
- Rahbar-Rastgar Daniel, J.S., Reinke, G., R. (2017). Comparison of Asphalt Binder and Mixture Cracking
 Parameters. *Road Materials and Pavement Design*, 18, 211–233.
 https://doi.org/10.1080/14680629.2017.1389071
- Rasool, R., Hongru, Y., Hassan, A., Wang, S., & Zhang, H. (2018). In-field aging process of high content SBS modified asphalt in porous pavement. *Polymer Degradation and Stability*, *155*, 220–229.
- Shi, L., Sun, M., Li, N., & Zhang, B. (2015). Sinteza i online FTIR spektrofotometrijsko ispitivanje novog emulgatora betainskog tipa za asfalt. *Chemical Industry and Chemical Engineering Quarterly*, 21(1), 113–121. https://doi.org/10.2298/CICEQ140223014S
- Speight, J. G. (2016). Nomenclature and Terminology. *Asphalt Materials Science and Technology*, 3–43. https://doi.org/10.1016/B978-0-12-800273-5.00001-5
- Stroup-Gardiner, M., & Nelson, J. W. (2000). Use of Normal Propyl Bromide Solvents for Extraction and Recovery of Asphalt Cements. National Centre for Asphalt Technology NCAT.
- Terrenzio, L. ., Harrison, J. ., Nester, D. ., & Shiao, M. . (1997). Natural v. Artificial aging: use of diffusion theory to model asphalt and fiberglass-reinforced shingle performance. *International Symposium on Roofing Technology*, 4th.
- West, R., Rodezno, C., Leiva, F., & Yin, F. (2018). Development of a framework for balanced mix design. *Project NCHRP 20-07/Task 406*, 7–20.
- Williams, R. C., Kvasnak, A., & Aurilio, V. (2002). Properties of Recovered Binder Samples and their Relationships to Tank Binder. *Canadian Technical Asphalt Association CTAA*, *47*, 349–363.

- Witczak, M. W., Kaloush, K., Pellinen, T., El-Basyouny, M., & Quintus, H. Von. (2002). Simple Performance
 Test for Superpave Mix Design. In Design.
 http://onlinepubs.trb.org/onlinepubs/nchrp/nchrp_rpt_465.pdf
- Wright, L. ., Moult, E., & Hesp, S. A. . (2011). Oxidative Aging of Asphalt Cements from an Ontario Pavement Trial. *International Journal of Pavement Research and Technology*, 4(5), 259–267.
- Yan, C., Huang, W., & Tang, N. (2017). Evaluation of the temperature effect of Rolling Thin Film Oven aging for polymer modified asphalt. *Construction and Building Materials*, 137, 485–493.
- Youtcheff, J. (2016). Automated High-Performance Liquid Chromatography Saturate, Aromatic, Resin, and Asphaltene Separation. *TechBrief,* 8p. http://www.fhwa.dot.gov/publications/research/infrastructure/pavements/15055/15055.pdf%0A https://trid.trb.org/view/1427383
- Yu, J., Dai, Z., Shen, J., Zhu, H., & Shi, P. (2018). Aging of asphalt binders from weathered asphalt mixtures compared with a shrp process. *Construction and Building Materials*, 160, 475–486. https://doi.org/10.1016/j.conbuildmat.2017.11.060

APPENDIX A

APPENDIX A

PG Grade	RAP	Mix ID	Lab ID	Tank/Rec	Ash	PG	G High PG	Low M	SCR Jnr	Grade Loss	LTLG	СТОР	IFIT FI	IFIT Slope	AMPT Fn
58-28		0 6-1006	Y1	Tank		0.1	60.6	-33.0	2 14	4		97	,		
58-28		0.6-1006	V1	Rec		4.1	51.5	-37.8	7 13	87)				
58-28		0 6-1006	V2	Tank		0.1	51.5	57.0	2.02	2 3	-	12 6			
58-28		0 6-1006	V2	Rec		2.4		-34.7	1 00	2.5	-200	14.7			
50-20 E0 20		0 6 1006	V2	Tank		0.1	E 9 0	-34.7	2.27	3.5	-23.5 1 20 E	14.7	10.9	1	0 70
50-20 E0 20		0 6 1006	V2	Roc		1.2	64.9	-34.5	1.26	2.4 E /	+ -30.5 1 37 E	14.3	10.0	-1	o 70
50=20 E0 20		0 6 1006	13	Tank		0.1	04.0	-34.1	2.22	3.4	+ -27.3	1.34	10.8	-1	.0 70
50-20		0 6 1006	14	Dee		2.4		-55.4	2.22	2.2		17.5			
50-20		0 8 1021	14 V1	Tank		2.4	65.0	25.4	0.63	5.3		3.7			
50-54		0 8-1031	11	Tank		0.2	65.0	-55.4	0.03			23.4			
58-34		0 4-1003	Y1	Tank		0.5	63.5	-35.5	0.44	4.7	-33.4	24.5			
58-34		15 3-0915	¥1 	Tank		0.1	62.0	-37.2	0.67	4.4	+ -32.4	15.8			
50-54		0 8-1031	11	Rec		0.7	67.4	-30.9	0.79	4.0	-52.7	9.0			
58-34		0 4-1003	Y1	Rec		2.7	68.5	-36.0	0.28	6.4	+ -28.1	1			
58-34		15 3-0915	¥1	кес		2.7	75.5	-29.7	0.19	6.9	-22.1	3.2			
58-34		0 8-1031	Y2	Tank		0.1			0.8	3.2	-33./	29.7			
58-34		0 4-1003	Y2	Tank		0.3			0.41	2.3	3 -34.1	28			
58-34		15 3-0915	Y2	lank		0.1	63.5	-36.4	0.53	4.6	-31.8	19.8			
58-34		0 8-1031	Y2	Rec		1.5		-37.9		2.7	7 -34.6	20.8			
58-34		0 4-1003	Y2	Rec		2.1		-39.1	0.3	4.5	5 -32.8	12.3			
58-34		15 3-0915	Y2	Rec		1.7	68.0	-36.4	0.43	6.7	7 -18	8.2			
58-34		0 8-1031	Y3	Tank		0.2	62.7	-36.9	0.91	1.5	5 -29.3	34.9	5.8	-3	.0 68
58-34		0 4-1003	Y3	Tank		0.6	62.5	-39.5	0.38	2.9	9 -36.8	30.4	9.2	-1	.4 28
58-34		15 3-0915	Y3	Tank		0.1	61.9	-36.7	0.52	3.3	3 -27.8	18.1	5.8	-2	.3 109
58-34		0 8-1031	Y3	Rec		2.5	66.9	-36.6	0.63	4.7	7 -30.8	8.98	5.8	-3	.0 68
58-34		0 4-1003	Y3	Rec		1.1	66.9	-38.9	0.23	4.3	3 -30.4	11.88	9.2	-1	4 28
58-34		15 3-0915	Y3	Rec		2.4	68.5	-34.5	0.57	4.6	5 -28.1	-0.14	5.8	-2	.3 109
58-34		0 8-1031	Y4	Tank		0.1		-37.9	0.95	2	-35.2	37.4			
58-34		0 4-1003	Y4	Tank		0.7		-39.9	0.53	4.3	3 -35.6	22.4			
58-34		15 3-0915	Y4	Tank		0.1	61.6	-38.9	0.71	3.7	7 -33.7	21			
58-34		0 8-1031	Y4	Rec		3.9			1.9	6.2	-33.4	15			
58-34		0 4-1003	Y4	Rec		3.2			0.76	5.7	7 -34.9	11.2			
58-34		15 3-0915	Y4	Rec		3.0	71.6		1.32	8	3 -25.5	6.6			
64-28		0 7-1010	Y1	Tank		0.1	65.9	-33.3	0.28	3.7	7 -29.2	6.4			
64-28		0 7-1010	Y1	Rec		7.8	82.6	-31.2	0.05	9.7	7 -20.5	5.4			
64-28		0 7-1010	Y2	Tank		0.1			0.33	3.9	-30.7	12.9			
64-28		0 7-1010	Y2	Rec		3.9		-31.5	0.3	4.4	-28.7	8.9			
64-28		0 7-1010	Y3	Tank		0.1	64.9	-35.5	0.19	3.1	L -31.4	15.6	6.2	-3	.2 130
64-28		0 7-1010	Y3	Rec		1.7	69.9	-32.2	0.38	3.9	-25	4.9	6.2	-3	.2 130
64-28		0 7-1010	Y4	Tank		0.0		-37.1	0.36	3.2	-30.3	21.2			
64-28		0 7-1010	Y4	Rec		6.5			0.79	4.1	L -26.1	7.6			
70-28		0 1-0708	Y1	Tank		0.1	72.8	-36.7	0.1	4.3	3 -28.8	4.7			
70-28		15 2-0809	Y1	Tank		0.0	75.7	-35.6	0.09	6.1	L -28.7	6.6			
70-28		0 1-0708	Y1	Rec		2.8	77.8	-32.8	0.09	3.6	5 -27.1	7.1			
70-28		15 2-0809	Y1	Rec		4.1	93.0	-11.6							
70-28		0 1-0708	Y2	Tank		0.1	71.4	-35.4	0.11	2.4	-32.8	23.1			
70-28		15 2-0809	Y2	Tank		0.1	73.0	-35.2	0.12	3.5	5 -30	12.2			
70-28		0 1-0708	Y2	Rec		2.3	76.4	-35.8	0.05	3.5	-29.6	9.5			
70-28		15 2-0809	Y2	Rec		2.3	77.4	-31.2	0.11	2.3	3 -25.2	6.1			
70-28		0 1-0708	Y3	Tank		0.1	72.4	-34.8	0.05	3.7	7 -30.5	13.2	7.0	-2	.0 2659
70-28		15 2-0809	Y3	Tank		0.1	72.7	-35.3	0.05	3.4	-29.8	12.5	2.2	-7	4 3119
70-28		0 1-0708	Y3	Rec		2.1	76.3	-35.2	0.04	4.8	3 -28.5	11.02	7.0	-2	0 265
70-28		15 2-0809	Y3	Rec		2.8	78.5	-30.2	0.08	4.6	5 -23.2	3.5	2.2	-7	4 3119
70-28		0 1-0708	Y4	Tank		0.1	70.3	-35.8	0,13	5.6	-27.9	20.7			511.
70-28		15 2-0809	Y4	Tank		0.1	71 3	-36.0	0.13	2.7	7 _79	7 1			
70-28		0 1-0708	Y4	Rec		3.6	72.1	55.5	0.12	4 7	2.28.8	9.5			
70-28		15 2-0809	Y4	Rec		4.2	81.2		0.12	4.2	23.0 716) s			
70-28		0 1-0708	Y5	Tank			71 7	-34 8	0.00	5.7	10	2.5			
70-28		15 2-0800	V5	Tank			72.5	-35.4	0.00						
70-28		0 1-0709	V5	Rec			72.6	_2/ 2	0.04						
70-28		15 2-0200	V5	Rec			7 J.U 80 1	-34.0	0.00						
,0-20		10 2-0003	10	NCC			00.1	-33.4	0.08						