Acknowledgements

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ABSTRACT

In 2003, the Ontario Ministry of Transportation commissioned the construction of a pavement trial on Highway 655, some 60 km north of Timmins, in an area that regularly experiences extremely cold temperatures. The seven asphalt cements included in the trial were designed to withstand -34ºC surface temperatures after 8 to 10 years of service according to the Superpave® specification. The trial was hit by record low air temperatures of around -48ºC in early 2004, with surface temperatures reaching -34ºC on two occasions. Early distress consisted of longitudinal cracking in three sections, but today significant longitudinal, transverse, and random cracking has appeared in five sections, while the two remaining sections are largely free of distress.

This paper discusses the physical and chemical differences that are responsible for the extraordinary variation in performance. Double-Edge-Notched Tension (DENT, LS-299) and extended Bending Beam Rheometer (extended BBR, LS-308) results on laboratory residues and recovered asphalt cements are able to explain why two sections have performed according to expectation and the others have failed in an unacceptable manner. Chemical analysis has shown that the likely presence of waste engine oil residues, air-blown residues, and/or acids in some of the asphalt cements may have contributed to excessive cracking.

RÉSUMÉ

En 2003 le Ministère des Transports de l’Ontario a commandé la construction d’un essai sur chaussée sur la route 655, à quelques 60 km au nord de Timmins, en un endroit qui expérimente régulièrement des températures extrêmement froides. Les sept bitumes inclus dans l’essai ont été conçus pour supporter une température de surface de -34ºC après 8 à 10 ans de service selon la spécification Superpave®. L’essai a été frappé d’une basse température record de l’air d’environ -48ºC tôt en 2004, avec des températures de surface atteignant -34ºC en deux occasions. La première détérioration a consisté en fissures longitudinales sur trois sections, mais aujourd’hui des fissures longitudinales, transversales et aléatoires sont apparues sur cinq sections, pendant que les deux sections restantes sont largement libres de détériorations.

Cet exposé discute des différences physiques et chimiques qui sont responsables de l’extraordinaire variation de la performance. Les résultats du tensiomètre à double encoche en paroi (DENT, LS-299) et du rhéomètre à poutre en flexion étendu (extended BBR, LS-308) sur les résidus de laboratoire et les bitumes récupérés sont capables d’expliquer pourquoi deux sections ont eu une performance selon les attentes et les autres ont failli de manière inacceptable. L’analyse chimique montre que la présence probable de résidus de vieille huile à moteur, les résidus du bitume soufflé à l’air ou les acides dans plusieurs bitumes peuvent avoir contribué à la fissuration excessive.
1.0 INTRODUCTION

Asphalt producers are naturally motivated to use low-cost modification technologies for their products. These are often introduced without field trials that assess long-term performance, and in the absence of accurate performance-based specifications they present a potential liability. The Strategic Highway Research Program (SHRP) asphalt binder specification method, commonly known as Superpave™ (Superpave) was introduced in the early 1990s with the objective of allowing user agencies to specify asphalt cement with a 98 percent reliability that in a given year the road surface would not be exposed to damaging low temperatures. Superpave has provided an improvement over historical penetration and viscosity grading methods. However, binders of the same performance grade can still show very different field behaviour, and it is widely recognized that more than a few binders graded under Superpave protocols fail prematurely due to low temperature exposure [1-14].

The need for improvement in Superpave protocols was illustrated early on with performance data from Canadian Strategic Highway Research Program (C-SHRP) pavement trials in Hearst, Ontario, and Lamont, Alberta, as well as a Long-Term Pavement Performance Program Special Pavement Site (LTPP SPS-9A) near Petawawa, Ontario [1, 4-6, 9]. Two C-SHRP test sections on Highway 631 west of Hearst, which were constructed in 1991 with binders of the exact same grade, showed a difference in transverse cracking severity of nearly a factor of 20 [9]. Two C-SHRP sections on Highway 637:02 east of Lamont, constructed with binders of nearly the same grade, showed a difference in cracking severity of a factor of 30 [6, 11]. Finally, a PG 58-28 and both of the PG 58-34 sections constructed in 1996 as part of the SPS-9A experiment on Highway 17 in Petawawa, which were exposed to minimum surface temperatures of approximately -27°C in their first winter and again in 2003, and hence should not have cracked, were damaged by a significant 169, 52, and 65 transverse cracks/km, respectively [9]. Early studies have shown that all these differences can be explained if improved binder specification tests are used [7-14].

In 2003, the Ontario Ministry of Transportation embarked on an effort to develop improved low temperature and fatigue specification tests and concurrent specifications for asphalt cement. The laboratory research phase was supplemented with the construction of a number of full-scale pavement trials on Highway 655 (Phase I in 2003, Phase II in 2007), Highway 417 (2006), and Highway 427 (2008). Figure 1 provides a map with the location of the five current Ontario trials as well as the location for the C-SHRP trial near Hearst, which has since been reconstructed. The research on these 37 test sections is providing a wealth of data for the development of improved low temperature and fatigue cracking specifications for asphalt cement [9, 13]. While the trials in Lamont, Hearst, and Petawawa were constructed with asphalt cements from different sources and of very different low temperature grades, the Highway 655 Phase I trial near Timmins contains asphalt cements that all grade in a narrow range between -35 and -37°C, just above the required 98 percent confidence limit of -38°C [15]. This reduces the number of variables and thus should make the interpretation of performance data easier. The other trials were also designed with fewer variables and are targeted to develop specifications for the control of reflection cracking (Highways 417 and 427) and fatigue cracking (Highway 655 Phase II).

Thermal cracking is a complex process with various contributing and sometimes confounding causes. It has historically been linked to excessive stiffness of the asphalt, although other factors such as traffic loading and asphalt cement content are also considered to be important [16-27]. Our objective has been to develop an asphalt cement grading approach that is blind to source, composition, and modification method. However, this paper discusses both physical and chemical analysis results from recovered asphalt cements in order to confirm the validity of the DENT (LS-299) and extended BBR (LS-308) methods in the most unambiguous manner.
Figure 1. Locations for Ontario Pavement Trials in Support of the Development of Improved 
Asphalt Cement Specifications (37 Test Sections)

Note: A = Highway 631 east of Hearst (C-SHRP 1991), B = Highway 17 in Petawawa (SPS-9A 1996), 
C = Highway 655 north of Timmins (2003), D = Highway 417 between Limoges and Casselman (2006), 

2.0 BACKGROUND

2.1 Causes of Thermal Cracking

In winter when the temperature of a road surface drops, the asphalt mixture tries to contract. 
Longitudinally, this contraction is largely prevented due to the restraint in that direction and the friction 
with the granular base. In the transverse direction, the pavement is able to contract to a limited extent due 
to a lack of restraint at the edge of the pavement. However, it should be noted that thermal stresses exist in 
both the longitudinal and transverse directions. If the total stress from thermal shrinkage and traffic 
loading reaches a critical level, the asphalt suffers damage, which can manifest itself as cracking distress 
in areas of weakness (joints, segregated spots, shoulders, etc.). During very cold weather, the granular 
base is solidly frozen, and hence most of the time insufficient movement occurs to create visible cracks. 
The appearance of large cracks is usually delayed until the first spring thaw, when base movement 
facilitates the opening of the damaged pavement [9, 28-30]. Depending on the thickness of the pavement,
the care taken during construction to prevent segregation, drainage issues, the absolute minimum temperatures reached, and the age of the pavement, these cracks can be longitudinal, transverse, or both.

Localized weak spots and stress concentrations associated with longitudinal cracks can sprout transverse cracks in subsequent winters. This process progresses until the thermal stresses no longer reach above the tensile strength of the asphalt mixture. Hence, pure thermal cracking is a process that normally reaches a limiting level that depends on the strength of the asphalt mixture/pavement, the friction with the granular base, the traffic levels, and the degree to which the asphalt cement is under-designed for a particular climatic condition. However, long-term chemical and physical aging of the asphalt cement can slowly increase both thermal and fatigue cracking, eventually necessitating reconstruction of the entire pavement.

It has been found that with properly constructed pavements, the overriding factor governing the severity of thermal cracking distress is the quality of the asphalt cement. Since the introduction of the Superpave specification in the 1990s, a number of low-cost modification technologies have appeared on the market with the potential for premature and excessive cracking well before the desired 50 year design life (for instance, [31-35] and others). In response, the Ministry of Transportation of Ontario constructed a pavement trial on Highway 655 north of Timmins in 2003 with six side-by-side test sections of highly modified asphalt cements in order to support the development of improved test methods and specifications to reduce unwanted thermal cracking.

2.2 Historical Development of the AASHTO M320 Specification

The American Association of State and Highway Transportation Officials (AASHTO) M320 specification employs a limit on the creep stiffness, \( S(t) \), and the slope of the creep stiffness master curve, \( m(t) \), for the asphalt binder at low temperatures [27]. A thin asphalt beam is cooled for one hour, after which it is loaded for 240 seconds and unloaded for 10 seconds. The deflection at various times is used to calculate the creep stiffness using regular bending beam theory. A double-logarithmic plot of the creep stiffness versus the loading time is fitted to a polynomial function from which the creep stiffness and the slope of the master curve at 60 seconds are calculated, \( S(60 \, \text{s}) \) and \( m(60 \, \text{s}) \). The current BBR specification sets an upper limit of 300 MPa on the creep stiffness and a lower limit of 0.3 on the m-value. If a binder meets both criteria it passes the specification test and can be used in a particular climatic zone where the pavement surface temperature reaches 10ºC below the testing temperature once every 50 years. (The 10ºC difference between testing and design temperatures comes from a time-temperature shift, since 60 seconds of loading at the test temperature is equivalent to 2 hours of loading at a 10ºC lower temperature.)

The philosophy behind the AASHTO M320 specification dates back to the 1950s and 1960s, when researchers at Shell Laboratories in Amsterdam found a reasonable correlation between the binder stiffness at a fixed loading time and various failure properties in the binder and mixture [16-19, 21, 24]. Van der Poel [16, 17] used penetrations and ring and ball softening points to determine the stiffness of binders as a function of loading time and temperature. One of his early papers demonstrated that the failure conditions in the Fraass test could be related to the binder stiffness reaching a critical value at 11 s of loading [16, 17]. Based on van der Poel [16, 17] and other unpublished work at Shell Laboratories, Krom and Dormon [36] were the first to present a binder specification scheme that limits the binder stiffness at specific loading times and temperatures to control cracking due to traffic (\( t = 10^5 \, \text{s} \) and low temperatures) and thermal stresses (\( t = 10^4 \, \text{s} \) and low temperatures).

Heukelom [18] went further, testing a wide range of binders for which he found a high correlation between binder stiffness and actual failure properties. Hence, it was suggested that the stiffness, which had become relatively easily accessible through van der Poel’s nomograph, was a good surrogate for the
failure properties. Following this early work at Shell Laboratories, many other researchers have focussed their attention on stiffness as a binder specification parameter at low temperatures (e.g., [20, 22-26] and others). It was not widely recognized, however, that the correlation made by Heukelom [18] was only valid for unmodified asphalt binders and that there was a significant degree of scatter. While modifiers were used sparingly in the 1960s, in some areas today nearly all binders are modified (air-blown, polymer modified, gelled, “engineered,” etc.). Different modification techniques result in binders with vast differences in failure properties in both brittle and ductile states [7, 8, 35, 37]. Such differences are now believed to explain in part the vast in-service performance differences found for binders of the same AASHTO M320 grade.

A second and perhaps more important factor on which the early Shell publications are largely silent is that almost all binders suffer some degree of reversible structuring when stored at low temperatures. Although this phenomenon was regularly discussed in publications from the 1930s [38, 39], 1950s [40-42], and 1970s [43], generally the publications by van der Poel [16, 17] and Heukelom [18, 21] and those following them make only indirect mention of it.

2.3 Development of Ontario’s Improved Binder Specification Tests (LS-299 and LS-308)

2.3.1 Double-Edge-Notched Tension Test Method (LS-299)

Ductility and force-ductility failure tests were originally developed to supplement the rheological penetration test in order to provide a measure of strain tolerance in the non-linear regime. The SHRP researchers considered the typical displacement levels in these tests to be unrealistically high for the pavement and therefore decided to focus their efforts on the development of a new failure test in the ductile-to-brittle and brittle regime [45]. The Direct Tension Test (DTT) was designed under SHRP to measure failure strain and stress in a dog-bone sample stretched at a controlled rate to failure at low temperatures. The DTT has now been largely abandoned since the reproducibility of such brittle failure tests is inherently poor.

The development of the DENT test circumvents the technical issues of the DTT by testing samples in their ductile state, generally providing excellent reproducibility [7, 8, 44]. Three DENT specimens with varying notch depths are pulled at a controlled rate in a water bath until complete failure. The energy under the force-displacement curve is used to calculate the specific total work of failure which is plotted against the ligament length (distance between the notches). The specific total work of failure is then extrapolated to a zero ligament length to provide the specific essential work of failure. This essential work is divided by the peak net section stress in the 5 mm ligament specimen to provide an approximate critical Crack Tip Opening Displacement (CTOD). This approximate CTOD provides a measure of strain tolerance in the ductile state under conditions of severe confinement. As such it is expected to provide a better correlation with the events that happen during ductile failure in the asphalt pavement where asphalt binder and mastic films are highly confined between coarse aggregate particles.

Studies on materials used in the Pavement Test Facility of the Turner-Fairbank Highway Research Centre of the Federal Highway Administration in McLean, Virginia, have shown a reasonable correlation between fatigue distress and the approximate CTOD of the asphalt cement [46]. The CTOD was also able to explain low temperature performance rankings for test sections in Hearst, Ontario, where the low strain BBR test was unable to correctly rank the various materials [9]. In a more recent study on twenty Eastern Ontario pavement contracts with extraordinary variability in performance, the DENT test was once again able to provide a reasonable correlation with cracking distress. Those asphalt cements that performed well in the DENT test showed little thermal cracking in service [14].
2.3.2 Extended Bending Beam Rheometer Test Method (LS-308)

Asphalt pavements typically cool for periods of up to several weeks and months before they are challenged by cold spells during the mid-winter months of January, February, and March. During such extended periods of conditioning, lower quality asphalt cements are able to consolidate their wax/asphaltene structures [12, 25, 38-43], and to some degree lose their ability to relax thermal stresses (m-values decrease). Hence, the single hour of conditioning, as specified under the Superpave BBR specification test causes most pavements to be under-designed for thermal cracking, with some missing the grade by more than others. The hypothesis underlying the development of LS-308 is that significant reductions in thermal cracking can be realized if binders are conditioned according to a protocol that more realistically reflects service conditions [47].

The slow consolidation of wax/asphaltene structures in asphalt cements has been investigated for a very long time [38-42]. Most of the early work by Traxler and coworkers [38, 39] and Brown and coworkers [40, 41] is particularly informative. In 1959 Blokker and van Hoorn [42] of Shell Laboratories in Amsterdam were the first to coin the term “physical hardening” by which the process is now commonly known in the asphalt literature. They stated that wax precipitation is faster than asphaltene structuring but both are generally slow under high viscosity conditions at low temperatures.

The original Superpave specification included an option to conduct a BBR grading after one hour and 24 hours of conditioning [45, 48]. However, for reasons that are not well documented, the 24-hour option was abandoned soon after the implementation of AASHTO M320. This may be because the recent literature is divided on whether the hardening effect is of any importance for actual pavement performance [25, 49, 50], largely since it has proven difficult to consistently replicate the effect in asphalt mixtures.

Deme and Young [25] stated that measurements on recovered asphalt cements from the Ste. Anne Test Road in Manitoba indicated that low temperature stiffness showed little change over five years of service, which was at odds with the steady increase in low temperature cracking over that period. In contrast, the stiffness of mixture specimens cut from the road showed an increase at all temperature levels, which the authors attributed to the “age hardening” or “structural hardening” effect, distinct from chemical aging.

Romero and coworkers [49] tested AAM-1 and AAM-2 from the SHRP materials reference library but were able to show the hardening effect to be of importance in only AAM-2 using the Thermal Stress Restrained Specimen Test (TSRST). This result may have been due to the TSRST’s inability to replicate conditions that prevail in service and the different wax contents of the two binders (AAM-2 contains 2.2% more wax). It should be noted that the authors provide no physical hardening data for the asphalt cements.

Soenen and coworkers [50] studied the physical hardening ratio in asphalt cement and mixtures as defined by the ratio of stiffness after one hour and 24 hours. This study showed that clearly “there can be an increase in stiffness in mixes, increasing with loading time, similar to what was found in binder tests” [50]. At loading times of 240 s, the binder stiffness increased by as much as 90 percent, and the mix stiffness increased by as much as 60 percent over only 24 hours of conditioning. This shows that for longer periods of conditioning and at longer loading times, which are more typical of those in service, the hardening effect can have far-reaching consequences.

Much low temperature cracking (transverse and longitudinal) occurs at temperatures well above those encountered in a typical TSRST test and in situations that contain a considerable amount of stress concentration in the presence of pre-existing cracks [9, 25, 51, 52]. For these reasons the present study primarily considers field data and reversible aging tendencies of the recovered asphalt cements.
The LS-308 extended BBR method is a simple extension of the regular BBR test [47]. Samples are conditioned at two temperatures above the pavement design temperature, \( T_{\text{design}} + 10 \) and \( T_{\text{design}} + 20 \). The continuous limiting temperatures where creep stiffness, \( S(60 \text{ s}) \), reaches 300 MPa and \( m(60 \text{ s}) \) reaches 0.3 are determined by pass/fail testing after 1, 24, and 72 h of conditioning. The warmest temperature determined in this manner sets the grade for the physically aged asphalt cement. In addition to a requirement to meet the LTPPBind® 98 percent confidence level after three days of conditioning, the maximum grade loss permitted is set at 6ºC, assuring that very poor quality asphalt cements that contain large amounts of wax and/or unstable asphaltene dispersions are excluded from the asphalt supply.

Testing of binders from the C-SHRP trials in Hearst [9] and Lamont [11], the SPS-9A trial in Petawawa [9], and a series of twenty Eastern Ontario contracts [14] has shown that those asphalt cements that perform well in LS-308 perform according to expectation in service while those that perform poorly in LS-308 often fail prematurely and excessively in service. Grade losses of only 6ºC can reduce the confidence that in a given year a pavement is not exposed to damage from the intended 98 percent to less than 50 percent for a typical Canadian climatic condition. A loss of 12ºC can reduce the confidence level to less than 10 percent, suggesting that the road would be exposed to many damage events each winter.

This paper discusses the findings for LS-299 and LS-308 testing of the recovered asphalt cements from the Highway 655 Phase I trial north of Timmins, Ontario. Besides a discussion of the physical properties, a chemical analysis will be provided in order to validate the new methods from a chemical perspective.

### MATERIALS AND EXPERIMENTAL PROCEDURES

#### Trial Design, Superpave, LS-299 and LS-308 Grading Information

The trial sections were designed with asphalt cements of the same low temperature grade but different modification technologies. Table 1 provides a list of the technologies requested from each supplier, other additives found through spectroscopic analysis of the asphalt cements, and grading information (Superpave, LS-299 and LS-308). Table 2 includes additional pertinent information for each material.

### Table 1. Asphalt Cement Compositions and Grading Properties [13]

<table>
<thead>
<tr>
<th>Asphalt Cement</th>
<th>Modification Requested</th>
<th>Additional Additives Detected</th>
<th>M320 Grade, ºC</th>
<th>LS-308 Grade, ºC</th>
<th>LS-299 CTOD, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>RET + PPA</td>
<td>-</td>
<td>65-36</td>
<td>-34.5</td>
<td>18</td>
</tr>
<tr>
<td>2</td>
<td>Ox</td>
<td>SB</td>
<td>65-36</td>
<td>-28.4</td>
<td>9</td>
</tr>
<tr>
<td>3</td>
<td>SB</td>
<td>Zn</td>
<td>65-36</td>
<td>-32.5</td>
<td>19</td>
</tr>
<tr>
<td>4</td>
<td>SB</td>
<td>Zn + P&lt;sup&gt;31&lt;/sup&gt;</td>
<td>67-35</td>
<td>-30.1</td>
<td>17</td>
</tr>
<tr>
<td>5</td>
<td>SB</td>
<td>-</td>
<td>66-35</td>
<td>-31.3</td>
<td>41</td>
</tr>
<tr>
<td>6</td>
<td>Ox</td>
<td>Zn + P&lt;sup&gt;31&lt;/sup&gt;</td>
<td>59-35</td>
<td>-28.7</td>
<td>6</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td>Zn + P&lt;sup&gt;31&lt;/sup&gt;</td>
<td>54-35</td>
<td>-28.5</td>
<td>9</td>
</tr>
</tbody>
</table>

Note: RET = Reactive Ethylene Terpolymer; PPA = Polyphosphoric Acid; Ox = Oxidized; SB = Styrene-Butadiene type polymer; Zn = Zinc; and P<sup>31</sup> = additive containing Phosphorous (zinc could have originated from zinc dialkyldithiophosphate, a universal anti-wear/anti-oxidant additive found in engine oil, or from zinc carbonate or zinc sulphate, sometimes used to scavenge hydrogen sulphide). M320 grades are rounded averages from three laboratories, and varied by less than 1.9ºC. LS-308 grades are the warmest of the limiting temperatures after three days of conditioning at -22ºC, slightly different from the most recent standard which requires testing after three days of conditioning at \( T_{\text{design}} + 10 \) and \( T_{\text{design}} + 20 \). LS-299 Crack Tip Opening Displacements (CTOD) were taken at 25ºC at 100 mm/min, slightly different from the most recent standard which requires testing at 15ºC and 50 mm/min.
Table 2. Limiting BBR Grading Temperatures and RTFO Mass Losses [13]

<table>
<thead>
<tr>
<th>Asphalt Cement</th>
<th>Unaged, ºC</th>
<th>RTFO mass change, %</th>
<th>RTFO-aged, ºC</th>
<th>PAV-aged, ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-37.7</td>
<td>-0.57</td>
<td>-36.7</td>
<td>-35.7</td>
</tr>
<tr>
<td>2</td>
<td>-40.9</td>
<td>-0.51</td>
<td>-39.3</td>
<td>-35.7</td>
</tr>
<tr>
<td>3</td>
<td>-42.0</td>
<td>-0.55</td>
<td>-40.9</td>
<td>-37.0</td>
</tr>
<tr>
<td>4</td>
<td>-41.0</td>
<td>-0.28</td>
<td>-40.3</td>
<td>-36.0</td>
</tr>
<tr>
<td>5</td>
<td>-38.9</td>
<td>-0.46</td>
<td>-37.7</td>
<td>-35.4</td>
</tr>
<tr>
<td>6</td>
<td>-40.2</td>
<td>-0.25</td>
<td>-39.3</td>
<td>-34.6</td>
</tr>
<tr>
<td>7</td>
<td>-40.8</td>
<td>-0.42</td>
<td>-39.7</td>
<td>-34.7</td>
</tr>
</tbody>
</table>

Note: All grades were obtained at Queen’s University hence Pressure Aging Vessel (PAV) numbers differ slightly from Table 1. BBR is Bending Beam Rheometer and RTFO is Rolling Thin Film Oven.

The spectroscopic analysis of these asphalt cements is reviewed in the discussion section. However, it should be noted that those materials that tested positive for zinc and phosphorous could have been modified with waste engine oil residues to meet the low temperature performance grade [33, 34, 53]. Besides zinc, a range of other metals that are typically present in waste engine oil streams were detected (copper, iron, chromium, etc.). A preferred method to reach a certain performance grade appears to be through the dilution of hard asphalt with low-cost waste engine oil flux, followed by the adjustment of the high temperature grade with either polymer or gelling agent [33, 34, 53].

3.2 Recovery of Materials

The surface course mix was used for all experiments in this study. It consisted of a Superpave 12.5 mm design with an optimum asphalt binder content of 5.2 percent. The mixture contained coarse aggregate and screenings fractions of hard volcanic rock and a natural sand fraction from a local river source.

Core samples were taken from all seven test sections in the middle of the northbound lane at equal intervals. The 50 m long sampling areas, just south of each monitoring portion of the trial sections, were utilized to obtain representative materials for each asphalt type. Binder lifts were cut off with a diamond saw, and the surface lifts were tested in DENT configuration at 22ºC. After testing, the surface lift fragments were dissolved in solvent to recover the asphalt cement for further testing.

Asphalt cements were recovered through washing with minimal amounts of tetrahydrofuran (THF) until the solvent remained clear. Between 4 and 6 L of solvent was used to recover approximately 200 g of asphalt cement. The solvent was removed through gentle heating in a rotary evaporator. Once no further solvent could visibly be removed, the temperature of the evaporator’s oil bath was increased to 150ºC and the material subjected to an additional 30 minutes of high vacuum.

3.3 Experimental Procedures

3.3.1 Double-Edge-Notched Tension Testing of Core Samples

The core fragments were tested in an MTS 810 test frame equipped with an environmental chamber able to control the temperature to within ± 1ºC of the set point of 22ºC. Notch depths for all samples were kept constant at approximately 30 mm on either side. Samples were tested at a rate of 0.5 mm/min until failure. Between four and six specimens were tested for each section in order to obtain reasonably accurate averages for the peak stress, displacement at peak load, and specific energy to failure. Digital image correlation analysis was done on images captured during the test.
3.3.2 Double-Edge-Notched Tension Testing of Recovered Asphalt Cements

Recovered asphalt cement samples were poured into silicone moulds and tested according to LS-299 procedures [44] in a Petrotest DDA-3 tensile stress ductilometer. In brief, samples were conditioned at 15ºC for 24 h prior to testing. Specimens with ligaments of 5, 10, and 15 mm were tested at a constant rate of 50 mm/min in a temperature-controlled water bath at 15ºC. Duplicate tests were done to provide a total of six force-displacement traces. The energy under each curve was used to calculate the specific total work of failure. The specific essential work of failure was obtained from the intercept of a plot of specific total work of failure versus ligament length. The specific essential work of failure was divided by the peak net section stress in order to calculate an approximate critical CTOD.

3.3.3 Extended Bending Beam Rheometer Testing of Recovered Asphalt Cements

Recovered asphalt cement samples were tested according to LS-308 procedures in a Cannon Instruments Thermoelectric BBR to assess their tendency to physically harden during isothermal conditioning [47]. In brief, specimens were tested after storage for 1, 24, and 72 h at -12 and -24ºC. The absolute grades and grade losses for each of the conditioning times and temperatures were recorded for comparison with original grades and pavement cracking distress.

3.3.4 Nuclear Magnetic Resonance Spectroscopy Testing of Original Asphalt Cements

Nuclear Magnetic Resonance (NMR) spectroscopy was used to detect the presence of phosphorous (P-31-NMR) and to confirm the presence of Styrene-Butadiene (SB) type polymers (H-1-NMR). Samples were dissolved in THF (50/50 w/w) and scanned in a 500 MHz Avance spectrometer for a constant number of scans to qualitatively detect phosphorous, from zinc dialkyldithiophosphate or phosphoric acid, and vinyl protons, from SB-type polymers.

3.3.5 X-Ray Fluorescence Testing of Original Asphalt Cements

X-ray Fluorescence (XRF) spectra for all materials were collected using a hand-held Innovative X-Ray Technologies model XT-440L analyser. In brief, the XRF equipment irradiates the surface of the material with high energy X-rays, causing the ejection of inner shell electrons from heavy elements. The vacancies produced are reoccupied by electrons from outer shells. The descent of electrons from outer shells to inner shells is accompanied by the emission of a lower energy X-ray with a characteristic energy for the element being irradiated. The XRF analyser detects the emitted radiation, and a plot of intensity versus the X-ray energy provides qualitative as well as quantitative information on the presence of a range of heavy elements.

4.0 RESULTS

4.1 Construction Details and End Result Specification Findings

The construction of the test sections was uneventful except for one scheduling problem. The paving crew left for another job just prior to the arrival of the asphalt cement for the surface course of Section 5. Consequently, the asphalt cement for this section ended up in storage for six weeks and the binder course was left open to traffic for over 90 days, nearly twice as long as what the other test sections endured. As a result, the surface course for Section 5 was placed rather late in the season on October 7, 2003, more than six weeks after the previous surface course was placed on August 22, 2003.
During July 2005 a detailed Falling Weight Deflectometer (FWD) and soils investigation was conducted [54]. Measurements were taken in the northbound lane, in the wheel path and mid-lane, every 20 metres throughout the monitoring portions of each section. In addition to the FWD testing, two boreholes per test section were advanced through the shoulder to a depth of three metres. The depths of the granular base, subbase, and subgrade as well as the water table were measured. Samples were taken for subsequent moisture determination. The borehole analysis showed that the site had a consistent structure, with only Section 6 having a lower base thickness and overall stiffness [54].

The detailed FWD data showed that the site is fairly homogeneous, and there appears to be no relationship between pavement stiffness and the observed distress. However, it should be recognized that the FWD testing was done in July 2005, while most of the early distress occurred from March to April of 2004. Hence, the subgrade consistency may have been somewhat different at the time of testing.

End Result Specification (ERS) testing was conducted on all materials of each test section. The asphalt cements all graded according to AASHTO M320 between -35ºC and -36ºC (table 1). The asphalt cement contents, air voids contents, and pavement thicknesses were all kept within narrow tolerances. Table 3 provides a summary of the most pertinent ERS findings.

<table>
<thead>
<tr>
<th>Section</th>
<th>AC, %</th>
<th>Voids, % Plate</th>
<th>Voids, % In Situ</th>
<th>Pavement Thickness, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.81</td>
<td>5.6*</td>
<td>7.7</td>
<td>92.0</td>
</tr>
<tr>
<td>2</td>
<td>4.95</td>
<td>4.6</td>
<td>7.6</td>
<td>88.7</td>
</tr>
<tr>
<td>3</td>
<td>4.85</td>
<td>4.3</td>
<td>7.1</td>
<td>86.3</td>
</tr>
<tr>
<td>4</td>
<td>4.80</td>
<td>4.7</td>
<td>6.3</td>
<td>86.3</td>
</tr>
<tr>
<td>5</td>
<td>4.92</td>
<td>4.3</td>
<td>7.6</td>
<td>91.0</td>
</tr>
<tr>
<td>6</td>
<td>4.85</td>
<td>4.0</td>
<td>6.2</td>
<td>85.4</td>
</tr>
<tr>
<td>7</td>
<td>4.82</td>
<td>4.3</td>
<td>5.5</td>
<td>98.7</td>
</tr>
<tr>
<td>AVERAGE ± SD</td>
<td>4.9 ± 0.1</td>
<td>4.5 ± 0.5</td>
<td>6.9 ± 0.9</td>
<td>89.8 ± 4.7</td>
</tr>
</tbody>
</table>

Note: All values were obtained as averages from three loose mix samples (plate) or cores (in situ) taken during and after construction of the trial. * This lot was rejectable based on the Standard Deviation (SD), even though all three plate samples were acceptable within the limits. AC is asphalt cement content.

4.2 Weather Monitoring

The temperature on the site is monitored with two thermocouples placed alongside the road and eight thermocouples glued into the pavement surface. The loggers are battery operated and record a temperature reading every 30 minutes for an entire year. Batteries are replaced and data downloaded during spring.

The air temperature reached a record low of approximately -48ºC on January 9, 2004. The pavement temperature reached the design value of -34ºC on two occasions on January 9 and 15, and below -30ºC on eight separate occasions during the first winter. In early 2005, the air temperatures reached around -40ºC on six occasions, while the pavement at 5 mm below the surface reached -30ºC or slightly lower on five occasions. In early 2006, the two lowest air temperatures recorded were around -39ºC with what would have been corresponding surface temperatures of around -30ºC or slightly lower. Air temperatures for 2007 reached between -35 and -40ºC on a total of 16 occasions, with corresponding pavement temperatures in the -25 to -30ºC range. For 2008, the lowest air temperature of approximately -35ºC was reached on January 20, with corresponding pavement surface temperatures of approximately -24ºC. It is
clear that this pavement trial is located in an ideal area where pavement surface temperatures reach close to the design value on regular occasions.

4.3 Distress Monitoring

The test sections were visited twice during the first year. In late January 2004, just weeks after the severe cold spell, the pavement was inspected and very few visible cracks were noted. In late April 2004, the site was visited again, at which point a significant amount of wheel path distress was observed on the southbound lane in Sections 2, 3, and 4 [9].

Detailed cracking distress surveys were done during each of the last five springs, followed by a comprehensive pavement condition survey during the summer of 2008. The results of the spring 2008 survey are provided in Figure 2, while the more detailed results of the summer 2008 survey are presented in Figures 3 and 4. Figure 5 provides representative photographs of Sections 1 through 5. (No photographs were taken for Sections 6 and 7 in 2008.)

![Figure 2. Transverse and Longitudinal Cracking Distress in Seven Test Sections (Spring 2008)](image)

Note: Centerline longitudinal, shoulder longitudinal, and borehole associated cracking is not included in the above summary. Counts refer to transverse cracks only and include those sprouting into the driving lane from centerline joints and shoulders. Cracks less than a quarter of a lane long were not included in the above summary. First and second columns are for transverse and longitudinal, respectively, while third column is for crack counts.

The findings of both 2008 distress surveys show that test Sections 1 and 5 remain largely free of serious cracking distress and continue to hold a relatively good pavement condition rating. Sections 2, 3, 6, and 7 are damaged at moderate to severe degrees, while Section 4 is damaged throughout and excessively for a five year old pavement.

The detailed survey results collected during the summer agree reasonably well with those from the spring, except for some minor differences related to the fact that the somewhat more cursory spring survey excluded very short cracks of less than a quarter lane in length.
Figure 3. Transverse and Longitudinal Cracking Distress in Seven Test Sections (Summer 2008)

Note: Centerline longitudinal, shoulder longitudinal and borehole associated cracking is not included in the above summary. Counts refer to transverse cracks only and include those sprouting into the driving lane from centerline joints and shoulders. All cracks over 10 cm long were included in the above summary. First and second columns are for transverse and longitudinal, respectively, while third column is for crack counts.

Figure 4. Pavement Condition and Ride Comfort Ratings (Summer 2008)
Figure 5. Representative Photographs of Test Sections (Summer 2008)

Note: (a) & (b) = 655-1; (c) = 655-2; (d) 655-3; (e) = 655-4; and (f) & (g) = 655-5.
No photographs of Sections 655-6 and 655-7 were taken during 2008.
4.4 Core Testing

Asphalt core samples were tested in double-edge-notched tension configuration at 22 ± 1°C. The peak load, displacement at peak load, and specific energy to failure were recorded. The findings of this series of tests are provided in Figure 6. For six of the seven mixtures, the displacements at peak load are very close to those measured on laboratory-prepared samples [13]. However, it should be noted that the earlier experiments were done on samples with different dimensions and at higher displacement rates.

![Figure 6. Double-Edge-Notched Tension Results for Asphalt Core Samples](image)

Note: Error bars provide two standard deviations (n = 4-6), Temperature = 22°C and Rate = 0.5 mm/min.

4.5 Recovered Asphalt Cement Testing

4.5.1 Double-Edge-Notched Tension Testing According to LS-299

The DENT test results on the recovered asphalt cements are provided in Figure 7. No error bars are provided in this figure, since the experiments were done in duplicate only and generally provided excellent reproducibility. The testing rate was kept constant at 50 mm/min, and the temperature was controlled at 15°C to an accuracy of ± 1°C.

4.5.2 Extended Bending Beam Rheometer Testing According to LS-308

The extended BBR test results are provided in Figures 8-10. The limiting grade temperature after one hour of conditioning at -24°C provides a good estimate for the AASHTO M320 grade. The LS-308 grade is determined from the warmest of the limiting temperatures determined after 1, 24, and 72 h of conditioning at both -12 and -24°C. (Note: The actual conditioning temperatures are specified as T_{design} + 10 and T_{design} + 20, but for convenience in this study these were kept at -12 and -24°C.)

The worst grade loss over the three day conditioning period at -12°C was recorded, since it was found during an earlier study of twenty Eastern Ontario contracts to show a strong correlation with cracking severity [14]. Those asphalt cements that lose less generally perform better than those that lose more due to isothermal conditioning.
Figure 7. Double-Edge-Notched Tension Results on the Recovered Asphalt Cements

Note: $T = 15^\circ$C and Rate = 50 mm/min.

Figure 8. Limiting Grades after One Hour of Conditioning at -24$^\circ$C

Note: This temperature is very close to the grade determined under AASHTO M320.

Figure 9. Limiting Grades after Three Days of Conditioning at -12 and -24$^\circ$C (LS-308)

Note: The warmest limiting temperature where $S = 300$ MPa or $m = 0.3$, after 1 h, 24 h, and 72 h of conditioning at both temperatures, determines the LS-308 grade.
4.6 Chemical Analysis

4.6.1 Phosphorous and Proton Nuclear Magnetic Resonance

Phosphorous and proton nuclear magnetic resonance ($^{31}$P-NMR and $^1$H-NMR) spectra are provided in Figures 11(a) and 11(b).

The $^{31}$P-NMR samples were scanned 10,240 times, with the exception of 655-1 which was scanned only 1,853 times. The $^1$H-NMR samples were scanned only 16 times, given the higher abundance of protons in these samples.

4.6.2 X-Ray Fluorescence Analysis

X-ray fluorescence findings are provided in Figure 12, which shows the X-ray emission intensity as a function of X-ray energy. The spectrum provides a qualitative analysis of the presence of heavy elements from sulphur (S), calcium (Ca), vanadium (V), iron (Fe), nickel (Ni), zinc (Zn), and a host of other metals.

Zinc is a fingerprint element for waste engine oil residues, since it is typically present in large quantities after the refining of waste engine oil and never occurs in either straight asphalt cement or the aggregate.

Figures 11 and 12 show that materials used in Sections 4 and 7 contain significant quantities of phosphorous and zinc and, hence, were likely modified with significant quantities of waste engine oil residues to reach the low temperature target grade. Section 6 likely contains much less waste engine oil residue, yet it still has a detectable XRF peak at 8.6 keV. This could have originated from contamination in the asphalt cement storage tank during construction.
While the primary objective of this research has been the development of an improved specification grading method that is blind to asphalt production method and modification type, it was decided to conduct NMR and XRF analysis in order to further explain the findings from LS-299 and LS-308. As it has proven challenging to convince others of the merit of both methods, a chemical explanation of the findings was sought to provide an added validation of this new asphalt cement grading approach.

The detection of phosphorous and zinc, as well as different types of polymers and oxidation catalysts indicates that waste engine oil residues, air-blown asphalt cements, and polymer modified asphalt cements were all present in the trial. These elements are known to either promote or prevent the effects of isothermal conditioning, as will be discussed in the following section.

However, the findings of this study should not be considered in isolation, since our research on low temperature cracking has also investigated C-SHRP pavement trials in Hearst, Ontario [9], and Lamont, Alberta [11], an SPS-9A trial in Petawawa, Ontario [9], and over twenty Eastern Ontario contract sites with either superior or objectionable performance [14]. The conclusions we have reached are consistent in that physical hardening tendencies and strain tolerances or intolerances can explain observed performance differences where current Superpave specification properties failed.
Figure 12. X-Ray Fluorescence Spectra for Asphalt Cements

Note: The broad background peak between 6 and 15 keV is associated with inelastic scattering of incident X-rays.
S = Sulphur, Ca = Calcium, V = Vanadium, Fe = Iron, Ni = Nickel, and Zn = Zinc.

5.0 DISCUSSION

5.1 Construction and Design Issues

The construction details are unremarkable, and those factors that are frequently associated with poor fracture performance were generally well controlled in this trial. Air voids, voids in the mineral aggregate, asphalt cement contents, pavement thickness, and pavement stiffness varied within expected limits from the beginning to the end of this 3.5 km stretch of Highway 655 [13].

The fact that the binder course for Section 5 was left open for nearly twice as long as the other six sections shows that damage of this layer could not have been a significant factor in the explanation of the early and/or excessive distress in any of these sections, given that Section 5 remains in the best overall condition (see Figure 4). This agrees with the findings for the SPS-9A trial in Petawawa where the sole
section that remains largely free of distress today was paved in late November 1996 when snow was falling.

5.2 Distress Survey Results

A somewhat cursory distress survey was done in the spring of 2008. The cracking distress data for this survey are provided in Figure 2. More detailed cracking surveys were done in three randomly selected 50 m stretches for each section during the summer of 2008. The distress data in Figure 3 and the pavement condition indices in Figure 4 show the sums/averages of the three surveys for each section. However, before LS-299 and LS-308 findings are compared with the distress rankings and pavement condition indices, it is important to take note of the difficulties inherent in crack surveying.

The data in Figures 2 and 3 exclude the centerline longitudinal, borehole, and shoulder longitudinal distress. Had this been included in Figure 3, the picture would have been slightly different. Section 2 has about 17 m of borehole-related transverse cracking. Section 4 has about 50 m of longitudinal alligator cracking associated with boreholes in a northbound wheel path. Section 7 has nearly 150 m of a continuous longitudinal crack in the inside shoulder of a curve, likely associated with the cold joint to the fully paved shoulder.

Hence, depending on how the cracking survey results are presented, the performance of the sections is ranked in slightly different order. Transverse crack lengths, longitudinal crack lengths, and transverse crack counts all rank the sections differently. Spring and summer surveys rank the sections in different order due to the different survey distances and the different criteria that were used to record cracks. Hence, in our opinion, it is best to separate the performance into the following three categories:

(1) Sections 1 and 5 are performing as expected/desired;
(2) Sections 2, 3, 6, and 7 are cracked significantly more than desired; and
(3) Section 4 is cracked excessively for a five year old trial.

A more detailed analysis of the cracking data is likely not justified due to the inherent difficulties associated with cracking distress surveys. In years to come, the picture will become clearer with Sections 1 and perhaps 5 surviving largely free of distress and the others deteriorating to states requiring reconstruction at an early date.

5.3 Double-Edge-Notched Tension Testing of Core Samples

The core samples were tested in DENT configuration, and the results in Figure 6 show that the general ranking agrees reasonably well with the performance: Sections 1 and 5 are largely free of distress, and this is reflected by their high strain tolerance (displacement at peak load). Section 4 performed worst in both the core DENT test and the field. Sections 2, 3, 6, and 7 performed somewhat in between, with intermediate strain tolerance and significant cracking distress.

However, the ambient temperature test results are close together, and the errors in the individual measurements are significant. Further, the ambient tests fail to recognize that different materials were exposed to varying levels of low temperature stress due to differences in low temperature properties. Hence, recovered asphalt cements were also tested according to both LS-299 and LS-308. A review of all test results is needed to provide a complete explanation of the performance differences in the trial.
5.4 Asphalt Cement Testing

5.4.1 RTFO/PAV Materials

The data in Table 1 show that the current AASHTO M320 specification, as applied to laboratory-aged materials, passes all the seven asphalt cements. The lowest measured pavement temperature for the site was -34°C suggesting these asphalt cements should not have failed. In contrast, the LS-308 grades show that all but the material for Section 1 were expected to fail during a -34°C cold spell. Section 5 performed satisfactorily, which is likely due to the high strain tolerance in LS-299. Hence, a specification based on LS-299 and LS-308 testing provides a significant improvement over the current M320 approach.

What the data in Table 1 fail to explain is the significant differences between some of the poor performing sections. In order to better understand this variability, it is useful to consider the grading results on recovered materials.

5.4.2 Recovered Materials

The asphalt cements were recovered from the 50 mm surface layer of the pavement. It is well known that aging of asphalt cement in a pavement varies with the depth from the surface and that most hardening occurs in the top 5 to 10 mm [55, 56]. Hence, it is likely that somewhat different (and perhaps better) correlations would have been obtained if only the asphalt cement from a thinner surface layer had been extracted. Further, the cracks measured were those visible at the surface, and no investigation was made into how deeply these cracks penetrated into the surface and binder courses. Hence, the correlations between recovered asphalt cement properties and pavement performance need to be considered in the proper context of these particular study limitations.

Double-Edge-Notched Tension Testing According to LS-299

The recovered asphalt cement samples were poured into silicone moulds and cooled overnight at 15°C before the essential work of fracture and the approximate critical CTOD were determined for each material. The findings are presented in Figure 7 and show one sample with superior performance (Section 5) and three with inferior performance (Sections 2-4), while the remaining samples fall somewhat in between (Sections 1, 6, and 7).

Taken in isolation, these results do not directly correlate with the crack survey results. This is due to the fact that the low temperature asphalt cement properties vary a great deal. Hence, as for the laboratory-aged materials, LS-299 results should only be discussed in combination with LS-308 findings.

Extended Bending Beam Rheometer Testing According to LS-308

The extended BBR results are provided in Figures 8-10. The limiting temperatures determined after one hour of conditioning at -24°C are provided in Figure 8. The results show a reasonable correlation with the field performance in that asphalts recovered from superior Sections 1 and 5 graded at -39 and -36°C, lower than the others. Section 4 material graded at -29°C, higher than the others and in agreement with its poor field performance.

However, the materials recovered from Sections 2, 3, 5, and 7 all grade in the same range (-34 to -36°C). Yet three of these are cracked significantly more than what was anticipated, while only Section 5 is in a desired condition. Obviously, the one hour conditioning time overrates the performance of materials recovered from Sections 2, 3, and 7.
LS-308 data presented in Figures 9 and 10 show that the grade after three days of conditioning provides once again a significantly improved measure of performance. The materials recovered from Sections 1 and 5 grade at respectable temperatures of -37 and -30°C and lost relatively little when stored at -12°C. The fact that Section 5 has survived largely free of distress is likely due to combined LS-308 and LS-299 performance. It has a high strain tolerance, which may offset to some extent the disadvantage that it does not make the grade for the trial area (Figure 9). Materials recovered from Sections 4 and 6 graded at -19 and -21°C and lost 6.4 and 10°C after three days of conditioning at -12°C, respectively, indicating that these sections will likely need early reconstruction. Materials recovered from Sections 2, 3, and 7 also graded well above the required -38°C limit, suggesting that these will also suffer from excessive thermal distress in years to come. These findings are largely in agreement with earlier findings from the C-SHRP trials in Hearst, Ontario [9], and Lamont, Alberta [11], the SPS-9A trial in Petawawa, Ontario [9], and a large number of premature low temperature failures in regular Eastern Ontario contracts [14].

Chemical Analysis

NMR and XRF investigations provided additional insights into the field performance and the correlation with the asphalt cement properties.

P\textsuperscript{31}-NMR data as presented in Figure 11(a) show that four of the recovered asphalt cements tested positive for phosphorous.

In Section 1, the reactive ethylene terpolymer reacted with the asphalt cement using PPA as a catalyst. This was disclosed by the supplier and obviously has not had any significant detrimental effects.

In Section 6, the asphalt cement likely contained phosphorous from PPA or regular phosphoric acid (H\textsubscript{3}PO\textsubscript{4}) as an air blowing catalyst. This was not disclosed by the supplier, but it is a well known practice in the industry to use such additives for air blowing [57]. The fact that air-blown asphalts suffer a great deal from the physical hardening effect (Figure 10) has long been known and is due to the colloidal instability imparted by the oxidation of the aromatics and polar aromatics to produce more asphaltenes and coarser structures [38-43, 57, 58]. The asphaltenes produced are largely insoluble in the remaining saturates and, hence, physical hardening effects worsen in air-blown asphalt cements.

The broad peaks in the P\textsuperscript{31}-NMR spectra for materials from Sections 4 and 7 likely originate from zinc phosphate, which is probably a remnant of zinc dialkyldithiophosphate (ZDDTP), the anti-wear/anti-oxidant additive found in waste engine oils. The blending of waste engine oil residues with asphalt cement is not widely reported in the industry, with only a few publications on this issue; several United States Departments of Transportation ban it for environmental and possibly performance reasons (lead is found in waste engine oils and it can cause skin cancer in mice) [33, 34, 53, 59, 60]. Another possible source of the phosphorous is from phosphoric acid (H\textsubscript{3}PO\textsubscript{4}) or polyphosphoric acid (PPA) which may have been used to reach the high temperature Superpave grade in a low cost manner [34, 35].

H\textsuperscript{1}-NMR spectra presented in Figure 11(b) show that four asphalts were modified with polymers of the styrene-butadiene type. Three vinyl protons show up as peaks at chemical shifts between 4 and 6 ppm.

In Section 5, a high percentage of 1,2-vinyl addition versus 1,4-diene addition in the polybutadiene is revealed by three relatively strong proton signals. The supplier declared that the polymer loading for Section 5 was 4 percent SBS by weight of the asphalt cement. What advantage if any the high 1,2-vinyl content might have had on the performance of Section 5 to date remains to be investigated with
performance data from other trials. However, the low grade loss at -12°C and the high strain tolerance indicate that the amount of polymer could have been a factor in the superior performance.

Judging from the $\text{H}^1$-NMR spectra, the other three materials that showed peaks in the 4-6 ppm range were modified with lower amounts of SB-type polymer.

The broad peaks in the $\text{H}^1$-NMR spectra between 6 and 9 ppm originate from the aromatic protons of the asphalt cements. The figure indicates that all samples had similar aromatic contents, with 655-3 perhaps being slightly higher, explaining its somewhat better performance in Figure 10 (LS-308 grade loss after three days of conditioning at -12°C).

NMR data in Figure 11 and XRF data in Figure 12 reveal that the materials used in Sections 4, 6, and 7 tested positive for zinc and phosphorous, confirming the likely presence of waste engine oil residue. An $\text{H}^1$-NMR spectrum for waste engine oil residue is provided in Figure 13, indicating that this is largely an aliphatic (saturated) material. This may explain why the asphalt cements from these sections lose a significant amount in LS-308 (Figure 10). (Remember that n-heptane is used for the quantitative determination of asphaltenes through precipitation [61].)

Although the asphalt cement from Section 6 was likely contaminated with less waste oil residue, this cement was reportedly oxidized, adding to the colloidal instability imparted by the aliphatic oils [58]. Similar analysis of a large number of premature failures in Eastern Ontario has recently shown that the use of waste engine oil residues and/or air blowing is likely widespread and can produce objectionable performance [62].

These potential problems with asphalt cement modification were discussed more than 70 years ago in the papers of Traxler and coworkers [38, 39] and more than 50 years ago by Brown and coworkers [40, 41]. Hence, the authors hope to move forward with the implementation of both LS-299 and LS-308 to obtain a better handle on low temperature cracking and to prevent further costly failures.

![Figure 13. Proton Nuclear Magnetic Resonance Spectrum of Waste Engine Oil Residue](image-url)
6.0 SUMMARY AND CONCLUSIONS

Given the review of the literature and the findings presented in this paper, the following summary and conclusions are provided:

- The current AASHTO M320 test method and specification criteria failed to explain significant thermal cracking distress in three of five damaged sections of the Highway 655 Phase I trial.
- The LS-308 test method and specification criteria correctly explained thermal cracking distress in all the damaged sections and predict some future distress in Section 5, which has so far remained largely free of cracks. The two best performing asphalt cements lost only 0.4 and 3°C, respectively, from their low temperature grades when stored for three days at -12°C. In contrast, the five poor performing asphalt cements lost between 3.6 and 10°C under the same conditions.
- The LS-299 strain tolerance as reflected by the approximate critical CTOD provided additional insights. Recovered material from Section 5 failed to make the grade in LS-308 but remains largely free of cracks, which is likely due in part to the high strain tolerance as measured in LS-299.
- The problematic use of certain modification technologies (e.g., waste engine oil residues, air-blown residues, gelled asphalt cement) can for the most part explain the observed differences in LS-299 and LS-308 properties and concurrent field performance.

These findings are in agreement with those of earlier studies on premature and excessive thermal cracking in trials and regular contracts.

7.0 FURTHER WORK

The Ontario Ministry of Transportation is working with the asphalt industry to implement both LS-299 and LS-308 for acceptance purposes on all future contracts. This year both methods have been implemented for acceptance purposes on a trial basis in three major paving contracts in Eastern and Northeastern Ontario. The City of Kingston has also implemented both methods on a single reconstruction project.

REFERENCES


LTPPBind®, Version 2.1, Superpave® Binder Selection Program, Developed by Pavement Systems LLC, Bethesda, Maryland, July 1, 1999.


