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# **Innovative Testing of Ontario's Asphalt Materials**

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Ames, Iowa  
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# Presentation Outline

- ❖ Background
- ❖ Ash Content Test
- ❖ Double Edge Notched Tension (DENT) Test
- ❖ Multiple Stress Creep Recovery (MSCR)
- ❖ Extended Bending Beam Rheometer (ExBBR) Test
- ❖ X-Ray Fluorescence (XRF)
- ❖ Fourier Transform Infrared (FTIR) Spectroscopy
- ❖ Highlights of Mixture Testing
- ❖ Future Work, Conclusions

# Background

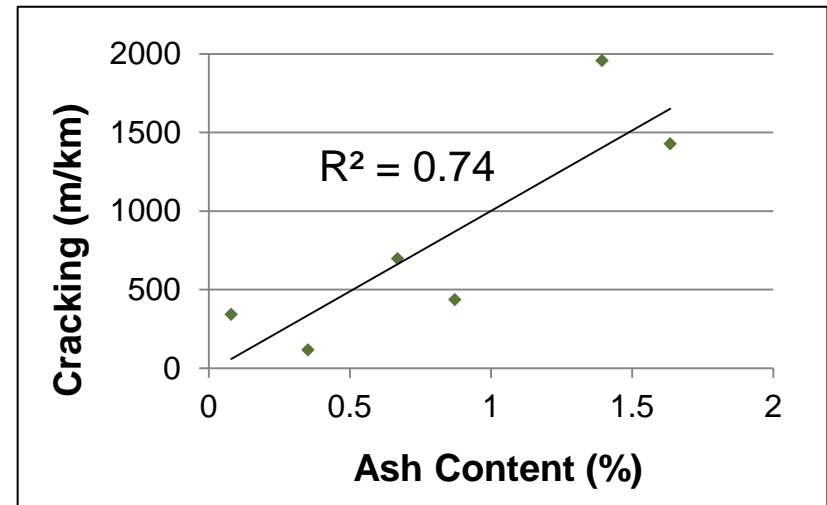
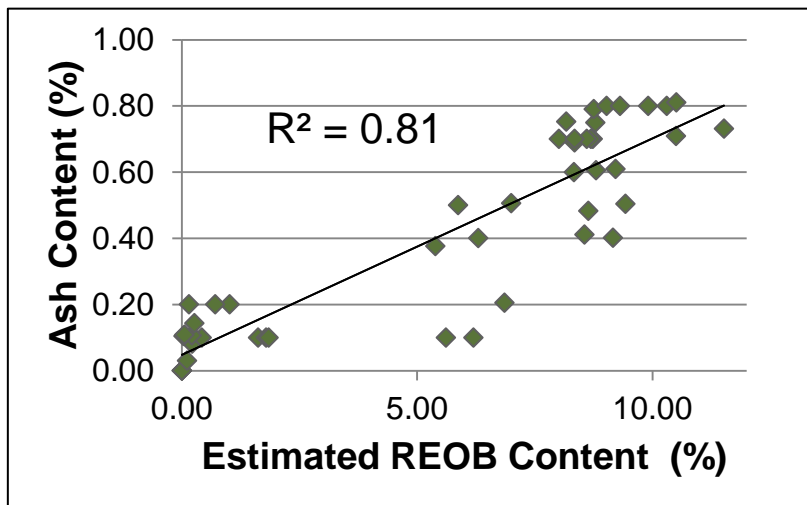
- ❖ MTO implemented performance grading grading in 1997 and was only used Superpave mix designs since 2005
- ❖ Rutting has all but disappeared
- ❖ Cracking is still a concern
- ❖ MTO uses a PG plus specification to mitigate cracking concerns and will be refocusing our attention to the properties of the mix

# Ash Content Test

❖ Ash Content test was implemented to prevent over-modification with Re-Refined Engine Oil Bottoms (REOB)

- Analysis of over 80 samples showed an excellent correlation between ash content and estimated REOB content

- Limited analysis to date shows excellent correlation between 5 year pavement cracking and ash content



# Double Edge Notched Tension (DENT) Test

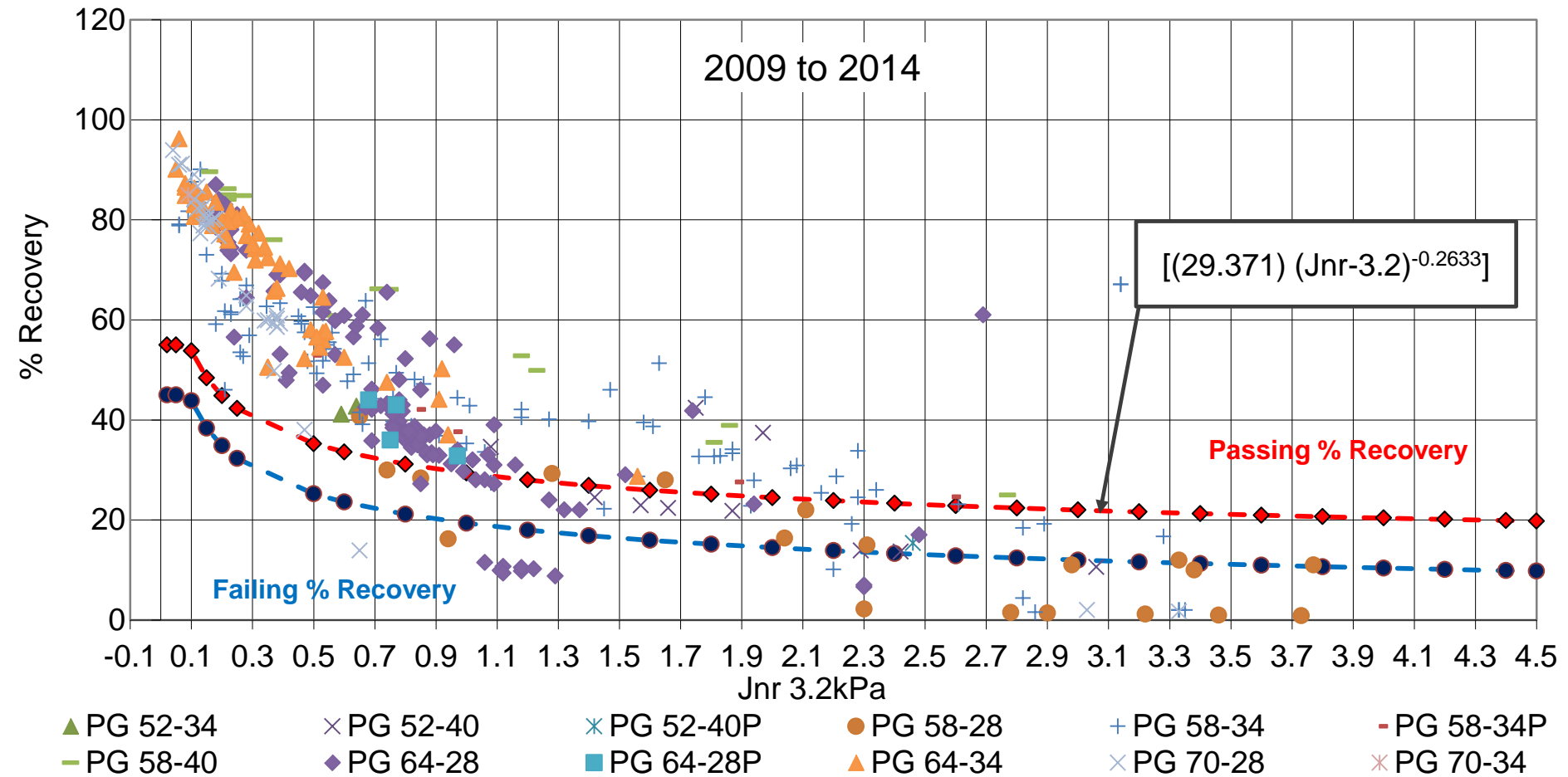
- ❖ Used for acceptance as a measure of asphalt cement's:
  - elasticity
  - ability to stretch and
  - resist cracking
- ❖ AASHTO TP113 specifies silicone molds with aluminum end pieces and testing at 25°C
- ❖ Ontario uses brass molds and tests at:
  - 15°C for PG XX-28 and -34 grades
  - 4°C for PG XX-40 grades



# Multiple Stress Creep Recovery (MSCR)

- ❖ Ontario's pavement performance concerns have focused on premature cracking and not rutting
- ❖ The % recovery portion of the test is used as an indicator of the presence of an elastomeric polymer
- ❖ Ontario's asphalt cement (AC) specifications include  $J_{nr-3.2}$  and % recovery for acceptance on modified grades since 2012
- ❖  $J_{nr\text{diff}}$  (%) is carried out only for information purposes

# MTO Experience with MSCR- Elastic Response



# MSCR Specification for High Temperature

- ❖ Paving first trial on Hwy 11 near Kapuskasing, ON started in 2016. Uses PG 52H-34 in place of PG 58-34
- ❖ Required to meet other MTO AC test requirements





# Extended Bending Beam Rheometer (ExBBR) Test

- ❖ Determines if AC meets the low temperature performance grade after a physical hardening process that occurs with extended conditioning at cool temperatures
- ❖ Test is published as AASHTO TP122-16
- ❖ Found best able to predict cracking
- ❖ Use for acceptance of all grades
- ❖ ExBBR determines low temperature grade over 72 hours vs. 1 hour for standard grading

# Estimation of 72 Hour Stiffness and Creep

- ❖ MTO developed multivariable regression formulae to predict the 72 hour ExBBR test based on 1 and 24 hour properties:

$$m\text{-value at 72 hrs } (T_{ht}) = 0.03239*(m\text{-value @ 1 hr}) + 0.88952*(m\text{-value @ 24 hr}) + 0.01129$$

$$m\text{-value at 72 hrs } (T_{lt}) = 0.17770*(m\text{-value @ 1 hr}) + 0.795125*(m\text{-value @ 24 hr}) - 0.00869$$

$$S \text{ at 72 hrs } (T_{ht}) = 0.13495*(S @ 1 \text{ hr}) + 0.94721*(S @ 24 \text{ hr}) + 3.34123$$

$$S \text{ at 72 hrs } (T_{lt}) = 0.16874*(S @ 1 \text{ hr}) + 0.93364*(S @ 24 \text{ hr}) + 0.14202$$

Where:

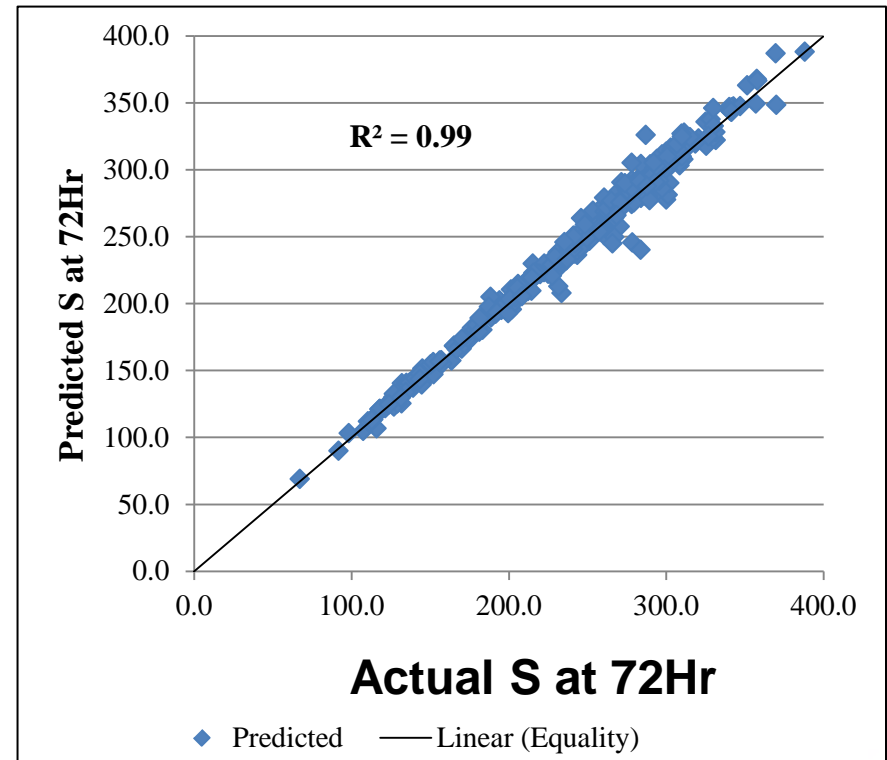
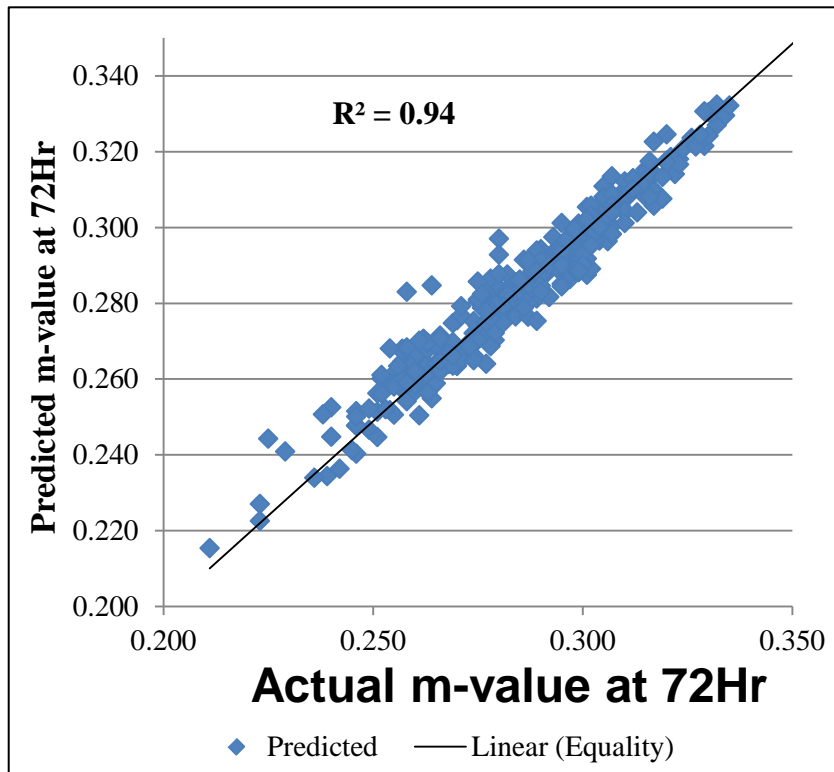
$T_{ht}$  = high test temperature

$T_{lt}$  = low test temperature

- ❖ Regression analysis was conducted on over 330 ExBBR tests

# Estimation of 72 Hour Stiffness and Creep

- ❖ The predicted m-value and S can be used to estimate ExBBR Low Temperature Limiting Grade that could be useful for quality control purposes



## $\Delta T_c$ From BBR/ExBBR Test

- ❖ Another useful outcome from the BBR test is:

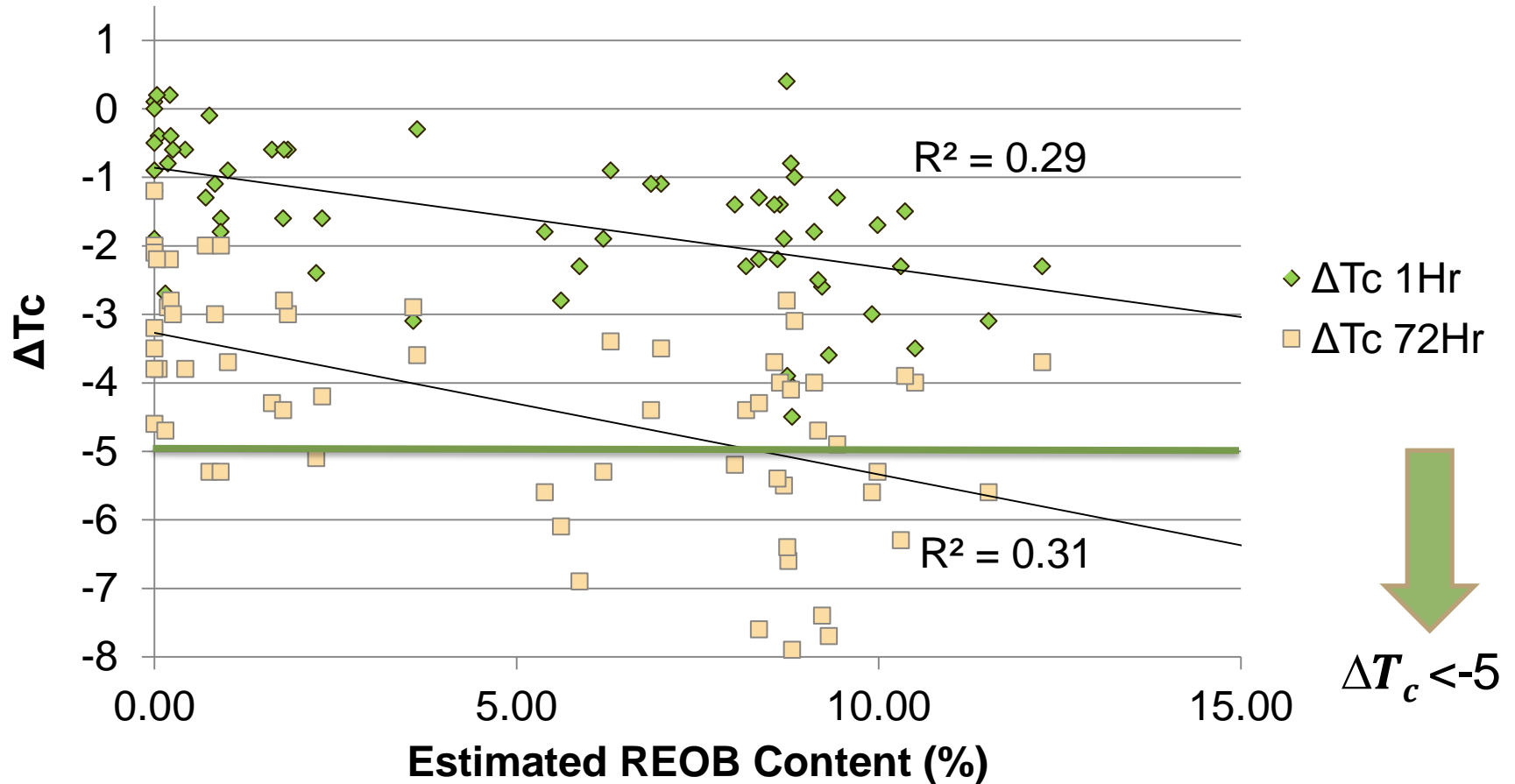
$$\Delta T_c = T_{stiffness} - T_{creep}$$

Where:  $T_{stiffness}$  = critical temperature for stiffness (S)

$T_{creep}$  = critical temperature for creep (m-value)

- ❖ For the over 60 samples tested, MTO compared single PAV aged BBR and ExBBR results
- ❖ REOB estimates ranged from 0 to 12%

# Estimated REOB Content vs. $\Delta T_c$



# $\Delta T_c$ From BBR/ExBBR Test

Recovered AC $\Delta T_c$ ( $^{\circ}\text{C}$ )		
% Recycled	BBR	ExBBR
0% RAP + RST	-6.2	-8.3
10% RAP + 1%RST	-6.0	-10.6
	-7.6	-13.3
10% RAP + 0% RST	-8.2	-10.8
0% RAP + 2% RST	-8.3	-8.7
	-4.2	-7.2

AC $\Delta T_c$ ( $^{\circ}\text{C}$ )	
BBR	ExBBR
-4.8	-8.0
-0.5	-6.1
-7.9	-9.0
-1.0	-3.1

# X-Ray Fluorescence (XRF)

- ❖ XRF detects the elemental content of a sample
- ❖ Transportation agencies, including MTO, are looking at XRF to identify over-modification of REOB in asphalt cement
- ❖ Elemental intensity peaks obtained are all relative to other elements found, so calibration curves are required for each element in a material to be quantified (in ppm)
- ❖ The four key elements and the levels detected in a REOB sample are:

Calcium	10,000 ppm
Zinc	3,000 ppm
Molybdenum	300 ppm
Copper	100 ppm

# X-Ray Fluorescence (XRF)

- ❖ MTO created calibration curves from base asphalt cement samples with varying percentages of REOB
- ❖ A linear regression curve was created for each element
- ❖ Equations currently used by MTO for estimating REOB content based on each element follow:

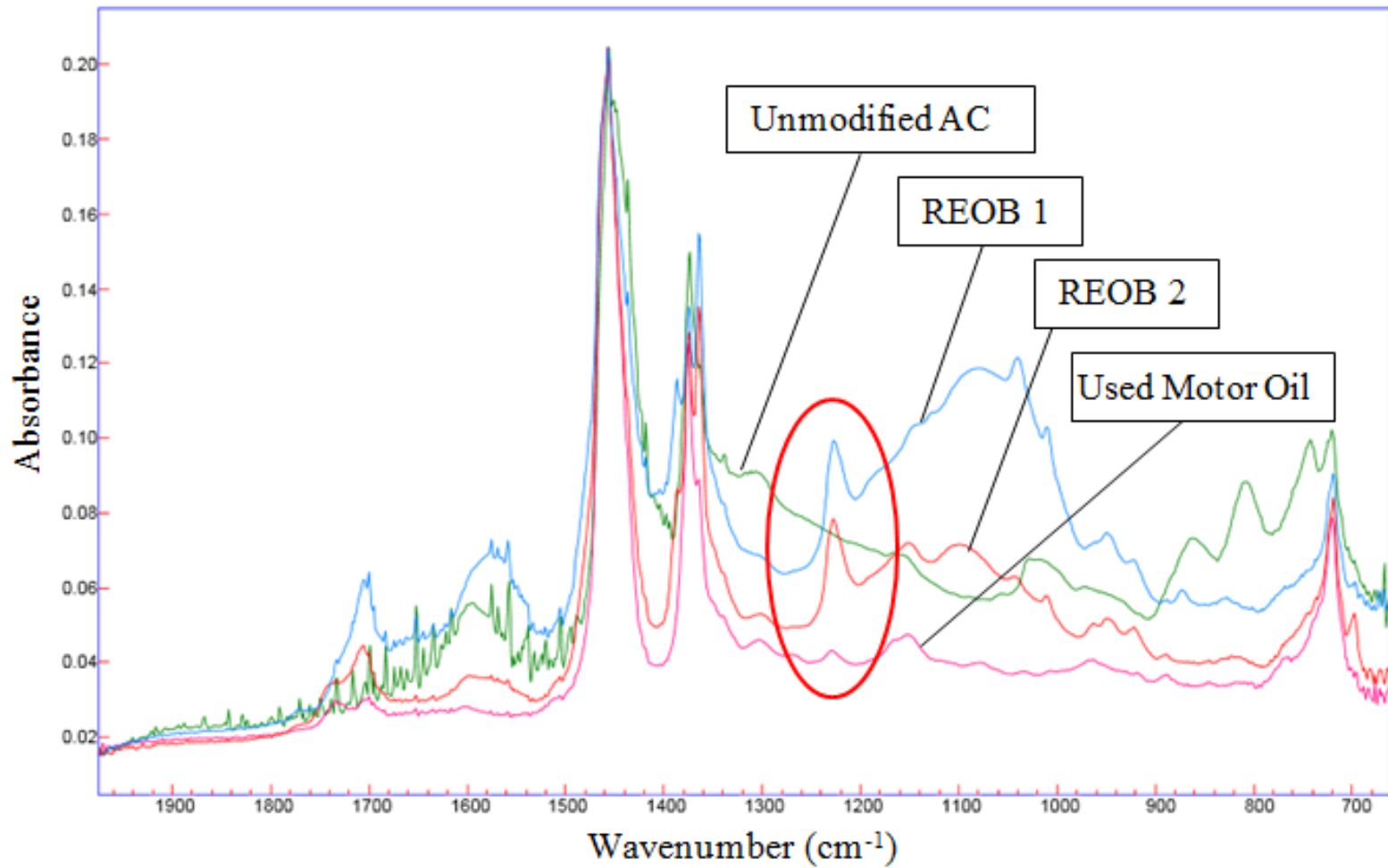
Element	Equation for Estimating REOB Content
Calcium	$REOB\%(Ca) = \frac{XRF(Ca) - 16}{109}$
Zinc	$REOB\%(Zn) = \frac{XRF(Zn) - 14}{48}$
Molybdenum	$REOB\%(Mo) = \frac{XRF(Mo) - 18}{4}$
Copper	$REOB\%(Cu) = \frac{XRF(Cu)}{1.5}$



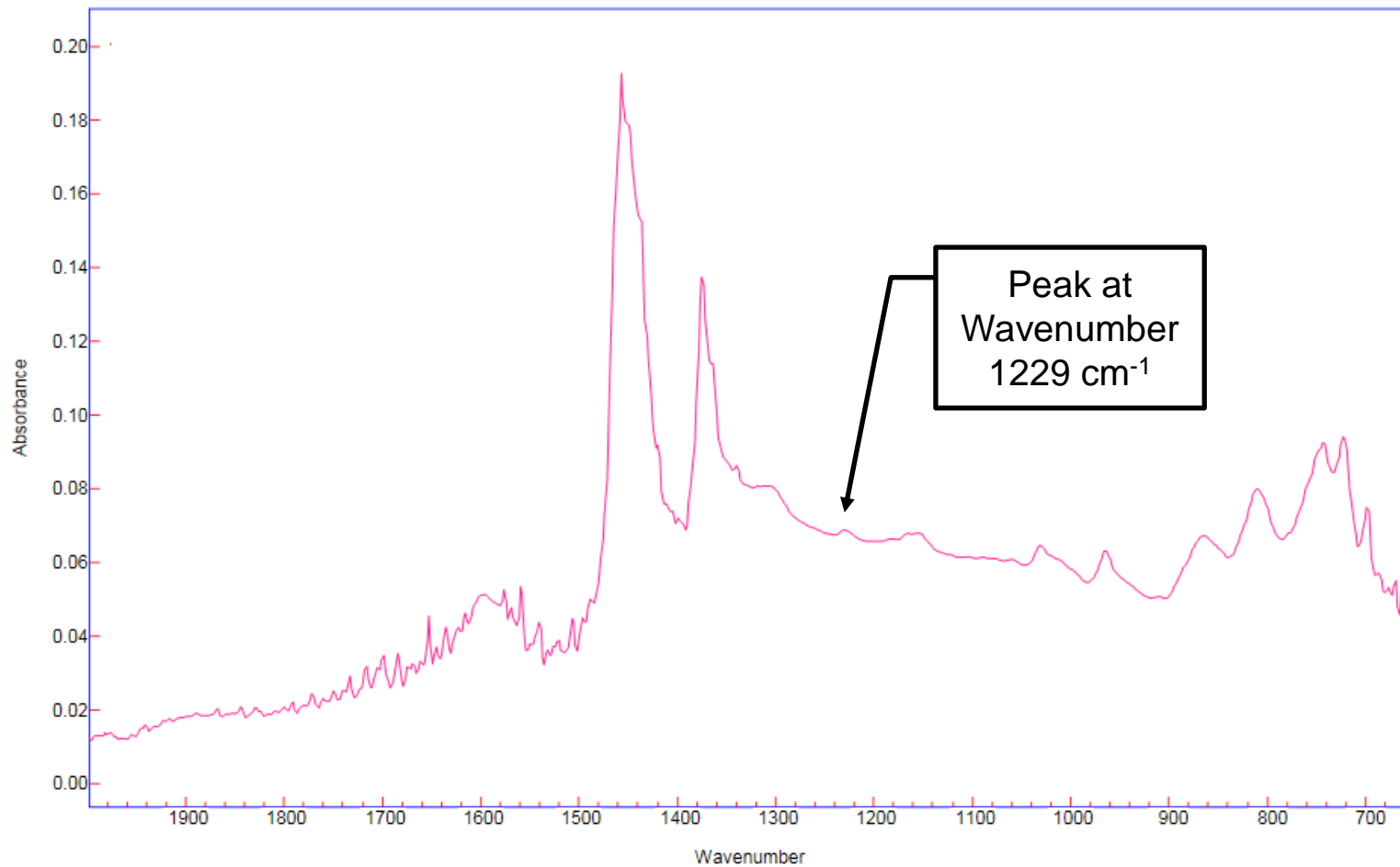
# Fourier Transform Infrared (FTIR) Spectroscopy

- ❖ FTIR detects the infrared energy absorbed in a sample
- ❖ Comparing FTIR spectra of an unknown sample to a “standard” sample can be used to spot modifications made to an “unknown” sample
- ❖ FTIR also provides information on the molecular bond and functional groups of modifications that are made to a material
- ❖ We found a unique FTIR absorbance peak corresponding to REOB
- ❖ A peak was observed near wavenumber  $1229\text{ cm}^{-1}$  - believed to correspond to polyisobutylene, an additive used in engine oil

# FTIR Spectra



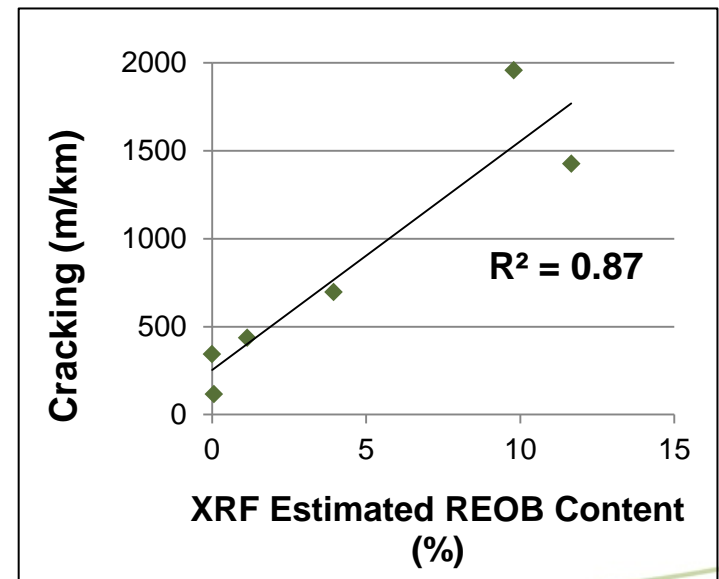
# FTIR Spectra for a Contract Sample



# REOB Estimation using FTIR and XRF

- ❖ FTIR can identify whether REOB is present in the AC
- ❖ MTO is currently estimating % REOB in AC with XRF for information purposes
- ❖ Results are provided below for:
  - comparison between FTIR peak and XRF estimated REOB content; and
  - five year pavement cracking performance

Sample	FTIR Absorption		XRF Count (ppm)				Average REOB Estimate (%)
	at 1229 cm <sup>-1</sup>	Peak Present?	Ca	Cu	Zn	Mo	
1	172	<b>Yes</b>	937	24	668	79	<b>13</b>
2	181	<b>Yes</b>	1378	9	331	36	<b>10</b>
3	135	<b>No</b>	23	0	27	10	<b>0.1</b>
4	46	<b>No</b>	0	0	11	0	<b>0</b>
5	282	<b>Yes</b>	945	0	509	29	<b>5.5</b>



# Highlights of Mixture Testing

- ❖ Moisture Sensitivity Tests
- ❖ Performance Tests using AMPT
- ❖ Performance Tests using DTS-30
- ❖ Bitumen Bond Strength Test (BBS)

# Stripping by Static Immersion Test

- ❖ Determines the stripping susceptibility of the different components of an asphalt mix (MTO LS-285)
- ❖ Aggregates are blended with asphalt cement and the blended material is submerged in distilled water at 49°C for 24 hours
- ❖ Stripping susceptibility of the asphalt mix is assessed visually based on the percentage of the retained coating on the aggregate

~15%  
retained  
coating



~85%  
retained  
coating

# Stripping by Static Immersion Test

- ❖ Use percent coating to determine what aggregate, AC, and anti-stripping treatment combination, provides better moisture resistance
- ❖ Minimum satisfactory value for this test is 65% retained coating

Aggregate Type	No Treatment	Hydrated Lime	Alternative AST-AGG
Granite	15%	85%	90%

# Tensile Strength Ratio (TSR)

- ❖ AASHTO T283 is used during mix design to determine susceptibility of an asphalt mix to moisture damage
- ❖ Not used to accept production mix
- ❖ In some cases we find this to be insufficient and specify anti-strip to minimize risk of stripping
- ❖ Tests uses a 40 hour freeze-thaw cycle





# Moisture Induced Stress Tester (MIST)

- ❖ An alternative moisture conditioning process to the TSR's freeze/thaw conditioning
- ❖ In addition to a conditioning process, MIST can be used to evaluate specimens based on sample swelling
- ❖ Air voids are measured and the percent swelling is calculated using

$$\textit{Swelling} = \frac{(\textit{BRD}_{\textit{before}} - \textit{BRD}_{\textit{after}})}{\textit{BRD}_{\textit{before}}}$$

Where:

$\textit{BRD}_{\textit{before}}$  = Bulk Relative Density prior to MIST conditioning

$\textit{BRD}_{\textit{after}}$  = Bulk Relative Density after MIST conditioning

# Moisture Sensitivity Test Results

- ❖ The results of liquid anti-stripping treatments (AST-AC) for the moisture sensitivity are:

Aggregate Type	Static Immersion		TSR		MIST -TSR		MIST-Swelling	
	No AST	AST-AC	No AST	AST-AC	No AST	AST-AC	No AST	AST-AC
Granite	15%	90%	67%	98%	62.0%	74.0%	4.2%	3.1%
Diabase	98%	*	84%	98%	69.0%	85.0%	2.0%	1.1%

\* Not tested

- ❖ The sample with the lowest retained coating, also has the lowest TSR, MIST-TSR and highest swelling value
- ❖ Alternately, the diabase had greatest retained coating without AST, the highest TSR, MIST-TSR and lowest swelling
- ❖ More testing required

# Hamburg Wheel Tracking Test (HWT)

- ❖ MTO uses Hamburg Wheel Tracking Machine to:
  - Evaluate antistripping additives for approved product list
  - Evaluate specialty mixes
  - Investigate premature pavement failure
- ❖ Not used to evaluate mixes before or during production



# AMPT

- ❖ MTO's AMPT (IPC Global) can run the following tests:
  - Dynamic Modulus
  - Flow Number (WMA)
  - Cyclic Fatigue (SMA)
  - Texas Overlay(Fiber)



# Performance Testing using DTS-30

- ❖ MTO is purchasing a Dynamic Testing System (Pavement Test) that will allow us to run the following:
  - Dynamic Modulus
  - Flow Number
  - Cyclic Fatigue
  - Texas Overlay
  - Four Point Bending
  - **Semicircular Bend (SCB)**
  - **Disk-Shaped Compact Tension (DCT)**
  - **Indirect Tensile (IDT) Creep Compliance and Strength**
  - Resilient Modulus
  - TSRST (Thermal Stress Restrained Specimen Test)



# Bitumen Bond Strength Test (BBS)

- ❖ The BBS test is a simple procedure to measure moisture resistance of the asphalt-aggregate interface for different combinations of materials
- ❖ *“Pull-Off Strength of Coatings Using Portable Adhesion Testers”*. (ASTM D4541)
- ❖ Just acquired the device



# Future Work

- ❖ FRAASS breaking point: measures the brittleness of binders at low temperature
- ❖ Refining recovery protocol for characterizing recovered binders from loose mix
- ❖ Determine if there is a relationship between double PAV BBR  $\Delta T_c$  and Ontario's pavement performance
- ❖ Establish a mix testing program to evaluate best options for predicting cracking of mixes placed

# Conclusions

- ❖ Strong correlation was found between pavement cracking, estimated REOB and ash content
- ❖ Relationship between  $\Delta T_c$  and estimated REOB was found to be poor
- ❖ Equations developed to predict ExBBR results after 24 hours, correlated well with actual test data - can be used for QC purposes based on grades current used in Ontario
- ❖ Found that FTIR spectroscopy can detect REOB
- ❖ Can estimate REOB content in asphalt cement using XRF spectroscopy



# Questions?



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