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RESEARCH REPORT

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Improving Quality Control of Asphalt Pavement with RAP Using a Portable Infrared Spectroscopy Device

By

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Pavement Preservation Systems, LLC

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16. Abstract
This project has investigated the effectiveness of a Portable Infrared Spectrometer (PIRS) device in estimating percent of Reclaimed Asphalt Pavement (RAP) and its contribution into oxidative aging of a new asphalt mixture immediately after production. Based on the findings from a limited demonstration study, the research team has developed a draft field procedure and implementation plan for Idaho Transportation Department (ITD) to incorporate PIRS testing of RAP into QC/QA of asphalt pavement construction. The team has also provided recommendations for validation and pilot implementation of the PIRS procedure on future paving projects. Lastly, the team has suggested incorporating the PIRS test into the pavement management program to monitor rate of deterioration and determine optimal timing for preservation treatments of asphalt pavements.

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Infrared spectroscopy, reclaimed asphalt pavement, asphalt oxidation, nondestructive testing

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Symbol	When You Know	Multiply By	To Find	Symbol	Symbol	When You Know	Multiply By	To Find	Symbol
		LENGTH					LENGTH		
in	inches	25.4		mm	mm	millimeters	0.039	inches	in
ft	feet	0.3048		m	m	meters	3.28	feet	ft
yd	yards	0.914		m	m	meters	1.09	yards	yd
mi	Miles (statute)	1.61		km	km	kilometers	0.621	Miles (statute)	mi
		AREA					AREA		
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ft^2	square feet	0.0929	meters squared	m^2	m^2	meters squared	10.764	square feet	ft^2
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mi^2	square miles	2.59	kilometers squared	km^2	ha	hectares (10,000 m^2)	2.471	acres	ac
ac	acres	0.4046	hectares	ha					
		MASS (weight)					MASS (weight)		
oz	Ounces (avdp)	28.35	grams	g	g	grams	0.0353	Ounces (avdp)	oz
lb	Pounds (avdp)	0.454	kilograms	kg	kg	kilograms	2.205	Pounds (avdp)	lb
T	Short tons (2000 lb)	0.907	megagrams	mg	mg	megagrams (1000 kg)	1.103	short tons	T
		VOLUME					VOLUME		
fl oz	fluid ounces (US)	29.57	milliliters	mL	mL	milliliters	0.034	fluid ounces (US)	fl oz
gal	Gallons (liq)	3.785	liters	liters	liters	liters	0.264	Gallons (liq)	gal
ft^3	cubic feet	0.0283	meters cubed	m^3	m^3	meters cubed	35.315	cubic feet	ft^3
yd^3	cubic yards	0.765	meters cubed	m^3	m^3	meters cubed	1.308	cubic yards	yd^3
Note: Volumes greater than 1000 L shall be shown in m^3									
		TEMPERATURE (exact)					TEMPERATURE (exact)		
°F	Fahrenheit temperature	5/9 (°F-32)	Celsius temperature	°C	°C	Celsius temperature	9/5 °C+32	Fahrenheit temperature	°F
		ILLUMINATION					ILLUMINATION		
fc	Foot-candles	10.76	lux	lx	lx	lux	0.0929	foot-candles	fc
fl	foot-lamberts	3.426	candela/m^2	cd/cm^2	cd/cm^2	candela/m^2	0.2919	foot-lamberts	fl
		FORCE and PRESSURE or STRESS					FORCE and PRESSURE or STRESS		
lbf	pound-force	4.45	newtons	N	N	newtons	0.225	pound-force	lbf
psi	pound-force per square inch	6.89	kilopascals	kPa	kPa	kilopascals	0.145	pound-force per square inch	psi

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List of Acronyms

AASHTO	American Association of State Highway and Transportation Officials
ASTM	American Society for Testing and Materials
ATR	Attenuated Total Reflectance (or Reflection)
COV	Coefficient of Variation
DR	Diffuse Reflectance (or Reflection)
ESAL	Equivalent Single Axle Load
FTIR	Fourier Transform Infrared
HMA	Hot Mix Asphalt
ITD	Idaho Transportation Department
JMF	Job Mix Formula
MSE	Mean Squared Error
NMAS	Nominal Maximum Aggregate Size
OxS	Oxidation Signal
PG	Performance Grade
PIRS	Portable Infrared Spectrometer
QA	Quality Assurance
QC	Quality Control
RAP	Reclaimed Asphalt Pavement
SB	Styrene-butadiene
SHRP	Strategic Highway Research Program
SNR	Signal-to-Noise Ratio
TCE	Trichloroethylene

Glossary

Term	Definition
Calibration Curve	A calibration curve is a general method for determining the concentration of a substance in an unknown sample by comparing the unknown to a set of standard samples of known concentration.
Coefficient of Determination (R^2)	R^2 is a number that indicates how well data fit a statistical model. It provides a measure of how well observed outcomes are replicated by the model, as the proportion of total variation of outcomes explained by the model.
Correlation coefficient	Correlation coefficient is a measure of dependency between two variables. It is obtained by dividing the covariance of the two variables by the product of their standard deviations.
F-statistics	F-statistics value is defined as a ratio of explained variance over unexplained variance or a ratio of between-group variability over within-group variability.
Repeatability	Repeatability or test-retest reliability is the variation in measurements taken by a single person or instrument on the same item.
Reproducibility	Reproducibility is the ability of an entire experiment or study to be reproduced either by the researcher or by someone else working independently.
Student's t-test	A t-test is any statistical hypothesis test in which the test statistic follows a Student's distribution if the null hypothesis is supported.

Executive Summary

A 2012 survey sponsored by the National Pavement Association (NAPA) in partnership with the Federal Highway Administration (FHWA), showed significant growth in the use of Reclaimed Asphalt Pavement (RAP) between 2009 to 2012 nationwide. Similarly, in an effort to support and promote sustainable practice, the Idaho Transportation Department (ITD) has allowed increasing RAP usage in their asphalt paving projects. Transportation Departments understand that the higher RAP percentage in any given project equates to the lowering of project related costs normally expended in acquisition of virgin aggregates and binder from the projects' contractor. However, there are inherent risks in the use of RAP. The interaction between the oxidized RAP asphalt and new liquid asphalt during production may lead to a shorter service life of the new pavement depending, among the other factors, on the concentration of RAP in a newly mixed asphalt concrete. The volumetric quantities of each constituent in the mix design are verified during field testing except for one, the amount of RAP being introduced at the HMA plant. The field determination of RAP percentages is critical to the verification of the approved mix design. Once the RAP is combined and processed at the HMA plant it becomes almost impossible to determine this all important percentage. Therefore, there is a need for a rapid yet reliable QC/QA field verification test procedure that effectively verifies the amount of RAP in any given HMA mix immediately after its production and in-place pavement verification. A procedure, critical to the verifications of the approved mix design, has been proposed and submitted to AASHTO for balloting.

This project has investigated the effectiveness of a Portable Infrared Spectrometer (PIRS) device in quantifying the percentage of RAP via its contribution to the total oxidative aging of a blended asphalt mixture immediately after production. The two primary goals of this project were:

1. To evaluate the capability of a Portable Infrared Spectrometer (PIRS) to estimate the percentage of RAP in a new asphalt mixture before, during, and after production.
2. To provide information and recommendations necessary for ITD to make decisions regarding the use of PIRS for estimating the percentage of RAP on future ITD paving projects as well as information on the acquisition and operating costs for PIRS and recommended field QC procedures.

The three projects selected for this study differed in RAP percentage (17%, 45%, and 54% total weight of aggregates) as well as in asphalt mixture volumetric properties, such as gradation and binder content. The mixtures also utilized asphalt binders of different performance grade.

Laboratory samples of RAP-HMA mixtures were fabricated for each project with various percentages of RAP in order to develop a project specific calibration curve (oxidation versus percent RAP). For validation purposes, samples were also collected from the HMA production plants and the paving sites. From each calibration RAP-HMA sample, the liquid asphalt component was extracted with TCE solvent and was recovered using Rotovapor apparatus. The recovered binder blends were then tested by PIRS without further alteration.

A portable Agilent ExoScan FTIR spectrometer was used to conduct PIRS scanning of laboratory and plant samples as well as pavement surfaces. The research team used diffused reflectance (DR) scanning mode for mixture samples, whereas the attenuated total reflectance (ATR) mode was used for recovered binder blends. An oxidation signal (OxS) value was computed from each PIRS spectrum as normalized absorbance intensity at 1700 cm^{-1}, which corresponded to the relative amount of oxidation in a tested sample. The OxS values were then superimposed with corresponding RAP percentage within sample to produce calibration curves. The statistical analysis of PIRS data was conducted to establish standard deviation of the test and evaluate the influencing factors of variability. The OxS data from plant and paving samples were used to establish deviation from the lab-determined calibration trends.

The regression analysis of PIRS measurements revealed significant differences in the linear trend intercepts between the three projects with minor difference in trend slopes. The difference in the intercept was attributed to the initial aging properties of virgin binder. The slopes of the calibration curves were controlled by the difference between RAP and virgin binder performance grades. Twenty percent of PIRS measurements on mixtures were recognized as “bad” data. Once bad measurements were removed, the average standard deviation remained 15±9 percent of sample mean. Consequently, the mean OxS values for the fixed RAP content in all three projects followed the linear trend within one standard deviation from the mean function, making it possible to predict amount of RAP from oxidation signals with absolute precision of 10 percentage points. An extensive analysis of variability in PIRS measurements indicated RAP fine aggregate content, RAP binder content, and %RAP within a mixture as primary influencing factors.

The validation phase of mixture sample testing showed good agreement between JMF, plant-produced, and pavement surface samples for all projects. This evaluation showed that the difference in oxidation between laboratory, plant, and pavement samples did not exceed standard deviation from the mean established for a project. Therefore, the research team has concluded that there was no evidence of deviation from the prescribed RAP percentage in post-production mixtures.

Analysis of oxidation measurements in recovered binder blends revealed similarity of calibration curves for Fruitland and Lewiston project with noticeable convex-shaped deviation from linearity. This result indicated a decrease in the effect of RAP on oxidation with an increase in RAP percentage. The two possible reasons for that could be (1) partial (as opposed to full) blending between RAP and virgin binder or (2) inability of extraction procedure to recover all binder from the RAP aggregates.

Overall, the demonstration study indicated high probability of correctly determining RAP content in HMA from the oxidation signal measured in accordance with a proposed AASHTO procedure. Therefore, the research team has recommended as follows:

- The proposed AASHTO procedure for QC of RAP in HMA is recommended for implementation.
- The implementation plan for the PIRS method is proposed to include personnel training and expanded validation study in all ITD districts.
- The validation study should include at least 3 projects with fixed mix design parameters within each ITD district to establish acceptance thresholds of oxidation in RAP-HMA.
- Further evaluation of the instrumentation market should be conducted to determine the best combination of instrument capabilities and operation costs.

Chapter 1

Introduction

Purpose of the Project

This project was sponsored by the Idaho Transportation Department (ITD) with the purpose of demonstrating the recently proposed AASHTO standard method for Evaluation of Oxidation Level of Asphalt Mixtures by a Portable Infrared Spectrometer (PIRS). A limited demonstration study resulted in the development of a field procedure and implementation plan for the ITD to incorporate PIRS testing of RAP-into QC/QA of asphalt pavement construction. The team has also provided recommendations for validation and pilot implementation of the PIRS procedure on future paving projects. Lastly, the team has suggested incorporating the PIRS test into the pavement management program to monitor rate of deterioration and determine optimal timing for preservation treatments of asphalt pavements.

Problem Statement

RAP is currently used in the majority of highway construction projects in Idaho. Although RAP has economic benefits, there are inherent risks in the use of RAP. The asphalt binder found in RAP oxidizes over time, which results in increased brittleness of the binder. When the oxidized RAP asphalt interacts with new liquid asphalt during production, it can reduce the durability of the new pavement depending on concentration of RAP in a newly mixed asphalt concrete as well as on the other factors. Effectively, the service life of pavement built with RAP may be shortened by early fatigue and low-temperature cracking as well as by raveling. Current asphalt mix designs approved by ITD include material adjustments intended to alleviate the stiffening effect. Once a mix design is approved, ITD is confident that the percentage of RAP proposed by a contractor will not adversely affect the pavement, due to corrective measures built into the design process. The quantity of each component of the asphalt pavement in current ITD projects is verified in the field except for the amount of RAP in the mix. Therefore, there is a need for a rapid yet reliable QC/QA procedure to verify the amount of RAP in the mix after production and placement.

Objectives and Research Approach

The two primary goals of this project were:

1. To evaluate the capability of a Portable Infrared Spectrometer (PIRS) to estimate the percentage of RAP in a new asphalt mixture before, during, and after production.
2. To provide information and recommendations regarding the use of PIRS for estimating the percentage of RAP on future ITD paving projects as well as information on the acquisition and operating costs for PIRS and recommended field QC procedures.

The research work plan (see flowchart in Figure 1) included the following five tasks:

- Task 1. Select projects, collect materials, and conduct preliminary PIRS testing.
- Task 2. Prepare calibration mix and binder samples in laboratory.
- Task 3. Conduct PIRS testing on the calibration HMA samples and develop calibration curves.
- Task 4. Conduct PIRS testing at mixing plant and on paving site and validate calibration curves.
- Task 5. Interpret results, develop recommendations, and prepare final report.

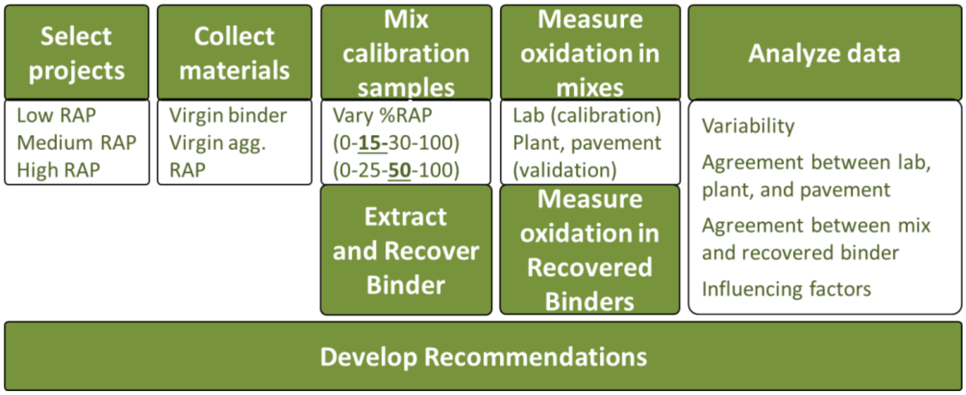


Figure 1. Flow Chart of Research Approach

Organization of the Report

This report summarizes the research methodology, interprets the results, and provides recommendations for the implementation of the proposed method of RAP testing by PIRS. The report starts with introduction to the problem statement, objectives, and description of research work plan (Chapter 1). Chapter 2 provides historical background and review of previous research on measuring oxidation in asphalt by infrared spectroscopy methods. Chapter 3 expands on task activities relevant to this project, while the major research findings are discussed in Chapter 4. The final chapter of the report (Chapter 5) includes the most important conclusions, recommendations on implementation, and suggestions of the future research.

Chapter 2

Literature Review

Usage and Standard Testing of RAP

Recent trends in improving sustainability of asphalt pavement have led to an increased use of Reclaimed Asphalt Pavements (RAP) in paving operations. The majority of transportation agencies in the U.S. continue to limit RAP content from 15 to 20 percent by weight of asphalt mix aggregates (*1*). However, quite a few agencies, including the Idaho Transportation Department (ITD), are allowing RAP content in their new pavements of 50% or more. The main challenge in using an increased amount of RAP is an increase in initial stiffness of newly constructed asphalt layer due to contribution of long-term oxidized RAP component. The increase in initial stiffness leads to an overall higher cracking (fatigue and thermal) and moisture susceptibility of RAP-containing pavement. The common practice of controlling the asphalt mix stiffness and permeability is to use a softer binder or rejuvenating agents where RAP content exceeds about 16% (*2, 3*).

With the ultimate goal of an adequate asphalt mix design being to ensure durability and serviceability of asphalt pavement, it is an accepted practice to measure the initial mechanical properties of RAP-containing asphalt mixtures. The standardized RAP tests involve measuring mixture modulus and flow number as well as volumetric properties, such as binder content and air voids (*4, 5, 6*). However, it is equally important to monitor changes in stiffness of RAP-containing pavements to evaluate their aging rate and to determine optimal timing for pavement preservation treatments. For the moment, field performance of RAP is evaluated by testing asphalt cores retrieved from pavement surface as well as testing asphalt binders extracted and recovered from those cores (*7, 8*). Those procedures are time consuming as they also involve using hazardous solvents.

Infrared Spectroscopy

The infrared (IR) spectroscopy measures intensity of infrared light, which is transmitted, reflected, or absorbed by a material sample. The intensity of a signal produced by a specific chemical bond or component is expected to be proportional to the amount of this component within a mixture (*9*). Thus, a chemical composition of a material can be elucidated from the analysis of an infrared spectrum of light intensities at specified frequencies. Transmission and reflection scanning modes are the most common sampling methods of infrared spectroscopy (Figure 2).

Transmission mode employs dissolution of a solid sample in an aliphatic solvent and mixing it with a neutral K-BR paste to produce sample pellets of known thickness. This mode allows for determining a concentration of a particular component in a mixture. The main drawback of the transmission IR mode is that it is not suitable for testing solids. As such, the transmission mode is obviously a destructive testing method (*10*).

The Attenuated Total Reflection (ATR) method measures the amount of reflectance remaining after the IR light has traveled within a few micron of the sample surface. This mode is suitable for liquids, thin films, and fine powders. The main drawback of the ATR method is that it only investigates a few micron of the sample surface, which may not always be representative of the bulk sample. Although the ATR technique does not alter the chemistry of the sample, it requires obtaining a sample of a material and bringing it to the instrument, which makes it a mildly invasive method (*11*).

The Diffuse Reflection (DR) method measures the amount of infrared light reflected by a bulk sample after being bounced between sample particles. Currently available instruments are capable of testing 3 to 5mm thick bulk samples with a rough top surface. The DR method then is most suitable for bulk granular materials and rough surfaces. The best results can be obtained for powders with 150-micron particle size (*12*). The currently available portable DR devices allow for testing rough surfaces directly without any sample preparation (*13*)

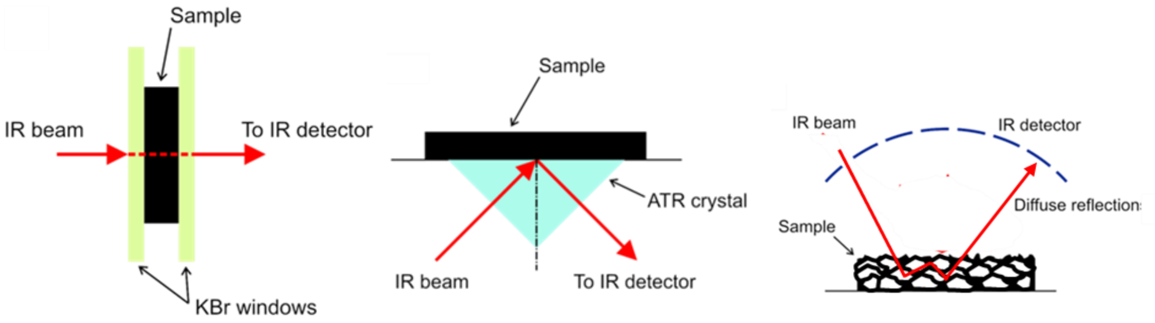


Figure 2. Sampling Modes of Infrared Spectroscopy: Transmission (left), ATR (center), and DR (right)

FT-IR Spectroscopy of Oxidized Asphalts and RAP

Since as early as the late 1960s, the asphalt researchers have been using Fourier Transform Infrared (FTIR) spectroscopy to evaluate chemical properties of asphalt. Change in amount of oxidized chemical components in asphalt is a major indicator of asphalt aging. Asphalt oxidative aging (or oxidation) is a result of a reaction between polar functional groups and atmospheric oxygen, which leads to increase in molecular weight and increase in asphalt viscosity, or hardening. The hardening of asphalt occurs rapidly during production and placement and continues at a slower rate during the service (*14*).

The first Strategic Highway Research Program (SHRP) project established a strong relationship between stiffness of asphalt binder and its level of oxidation. The binder shear modulus was related to amount of oxidized functional groups within binder chemical structure. Those findings were supported by correlating the binder shear modulus measured in a Dynamic Shear Rheometer (DSR) with the carbonyl moiety signal calculated from the binder infrared absorption spectrum under laboratory conditions (*15, 16, 17*). It has been also found that the binder is the main contributor to the oxidation of asphalt mixes, which primarily occurs at intermediate and high temperatures (*15, 17, 18*). It should be noted that the

SHRP study employed research grade FTIR equipment in transmission mode and, therefore, no direct FTIR tests on asphalt mixtures were conducted.

During the most recent Second Strategic Highway Research Program (SHRP 2) project R06B, a survey of DOT materials engineers (Figure 3) revealed an interest in using spectroscopic methods for measuring oxidation in RAP-containing asphalt mixtures and, in particular, determining the amount of RAP in a mixture (*19*). Therefore, the project researchers used a compact bench-top infrared spectrometer to measure oxidation directly on solid asphalt mix samples in the field (*19*). With respect to RAP, the study found a strong correlation between the amount of RAP in the mix and its oxidation levels as measured by infrared spectroscopy. The successful results of pilot implementation of portable IR test method in Connecticut and Maine led to the development of a draft AASHTO procedure for measuring oxidation in asphalt mixes by IR reflection spectrometry (*20)*. Building upon SHRP2 R06B results, a modified draft AASHTO standard for testing aged asphalts and RAP by means of portable infrared spectrometer has been recently submitted by PPS, LLC to AASHTO's Subcommittee on Materials (SOM) for balloting as a Provisional Test Method (*21*).

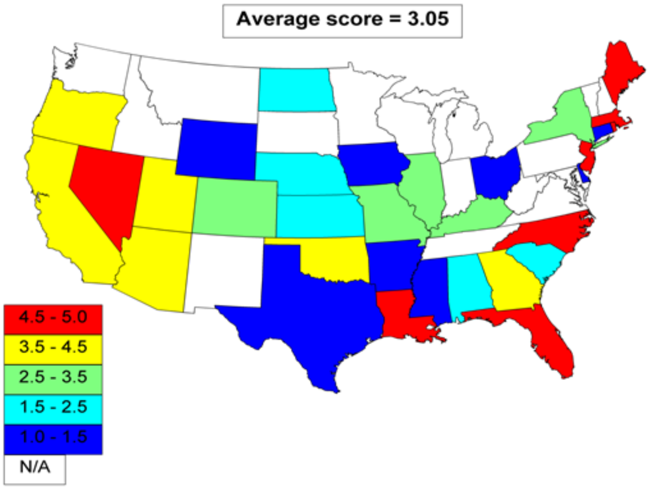


Figure 3. Results of SHRP2 R06B Survey of Material Engineers With Respect to Spectroscopic Testing of RAP

In summary, the literature review, including previous work done by the research team working on this project, led to a firm belief that implementing of an FT-IR technology will prove successful for field verification testing of asphalt RAP mixes when specified on ITD's asphalt paving projects. Therefore, PPS, LLC proposed a research project to demonstrate the ability of Portable Infrared Spectrometer (PIRS) to measure oxidation and determine the amount of RAP in the real asphalt mixture samples before, during, and after production. A portable Agilent ExoScan FTIR spectrometer was selected as the PIRS for this study.

Chapter 3
Methodology

Project Selection

This study targeted the demonstration of PIRS capability of measuring oxidation levels in asphalt mixes containing RAP ranging between 15% and 60% by total weight of aggregate. The following three projects were included with the approval from the ITD:

- Project #1 (Key No. 12046) at the intersection of Karcher and Middleton Roads in Nampa, ID (17% RAP)
- Project #2 (Key No. 13462) on the US-95 between intersection with I-84 and Fruitland Road in Fruitland, ID (54% RAP)
- Project #3 (Key No. 13435) on the US-95 at Lewiston Hill in Lewiston, ID (45% RAP)

Table 1 compares major asphalt mix characteristics for those projects. All three mixes in discussion had nominal maximum aggregate size (NMAS) of 12.5 mm (1/2- in). Mixes for projects #1 and #2 were designed for intermediate traffic volume of 1 to 10 million ESALs, whereas the mix for Project #3 was designed for high traffic volume of 10 to 30 million ESALs. It is also worth noting that although softer asphalt binders were used for both mixes with high RAP content, a two-grade dump was used for Project #2 with 54% RAP whereas only one-grade dump was advised for Project #3 with 45% RAP. Project #3 also stood apart from the other two projects by the fact that its RAP component originated from two sources. Forty percent of RAP came from the processed millings of the replaced surface layer and the other 5% were taken from a pre-existing RAP stockpile in the vicinity of the project.

Table 1. Summary of Mix Design Parameters for Selected Projects in Idaho

Project Key No.	12046	13462	13435
Project ID	“Karcher”	“Fruitland”	“Lewiston”
Location	Nampa, ID	Fruitland, ID	Lewiston, ID
Traffic Level [ESAL]	1 - 10 mln	1 – 10 mln	10 – 30 mln
NMAS	12.5 mm	12.5 mm	12.5 mm
Target PG	70-28	70-28	76-28
RAP [by agg.]	**17%**	54%	45% **(40% millings + 5% pit)**
Adjusted virgin PG	70-28	52-34	70-34
Target P_b	5.3	5.3	5.6
RAP P_b	5.3	5.2	**6.0**
RAP PG (est.)	Unknown	PG 88-XX	PG 76(82)-XX (millings)
Liquid Antistrip	0.5%	0.5%	0.75%

Although all mix designs in this study had the same NMAS, the target gradation as well as RAP gradations varied significantly. As shown in Figure 4, Lewiston RAP had significantly lower amount of fine sand (0.6 to 2mm size) as compared with the other two projects. On the other hand, the target Fruitland mixture was designed with more fine sand than Karcher and Lewiston. Those differences in gradation turned out to be significant factors of the oxidation in RAP-HMA mixtures as discussed later in this report.

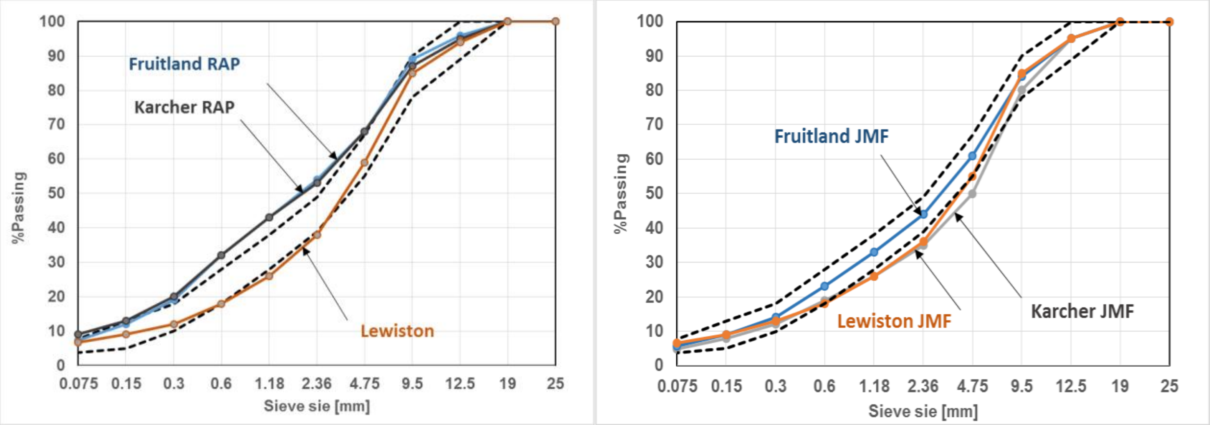


Figure 4. RAP Gradation (left) and Target Gradation (right) for the Selected Mix Designs

Sample Fabrication and Processing

Material Collection and Laboratory Mix Designs

Once a Job Mix Formula (JMF) was approved for each project, samples of the original components of the mix, namely virgin binder, antistripping agent, aggregate blends, and RAP from the pile, were collected in the amount sufficient to prepare a set of laboratory samples. The laboratory samples of RAP-HMA mixtures were fabricated for each project with various RAP percentages in order to develop a project-specific calibration curve. Such a curve should superimpose a specified RAP content with the intensity of an "oxidation" signal as computed from an infrared absorbance spectrum of laboratory samples with fixed RAP content. The designed proportions for the calibration mix samples are summarized in Table 2. The range of RAP contents was chosen uniquely for each project in order to capture a possible deviation from the target percentage of RAP. At least one sample for each project included JMF-prescribed RAP content, as highlighted in Table 2. Note that the added virgin binder content in calibration samples was computed based on the assumption of full blending between virgin and RAP binder. Based on this assumption, a linear trend in oxidation versus RAP percentage was anticipated.

Table 2. Summary of Proportions for Calibration Mixture Ingredients

Project	JMF RAP [by total weight of aggregates]	Virgin Binder [by total weight of aggregates]	Virgin Agg. [by total weight of aggregates]	RAP [by total weight of aggregates]
Karcher	17%	5.6%	100%	0%
		4.8%	85%	15%
		3.9%	70%	30%
		3.1%	55%	45%
Fruitland	54%	5.6%	100%	0%
		4.1%	75%	25%
		2.7%	50%	50%
		1.3%	25%	75%
Lewiston	45%	5.8%	100%	0%
		3.7%	69%	31%
		2.8%	55%	45%
		2.0%	42%	58%

Laboratory Mixing Procedure

The mixing procedure followed the AASHTO M 323 standard. The virgin aggregates submitted by the contractor were oven dried then screened on the 3/4", 1/2", 3/8” and #4 sieves in a Gilson sieve separator. The individual screen size increments were collected in buckets and identified with labels. The passing #4-sieve portion was tested for gradation to ensure that it reasonably matched a contractor’s JMF. The RAP was used as received, processed by a contractor to 100% passing the 5/8” screen. Once the aggregate was ready, the JMF was translated into a batch formula using the proportions from Table 2 to the amount of 10 kg of resultant mix.

The asphalt binder specified in the ITD construction contract was heated to a mixing temperature determined by the supplier. Heated virgin and RAP aggregates were placed in a pre-heated mixing container and the asphalt binder was added in the portion specified on the batch sheet. All components were then mixed in a mechanical device used for the Superpave Mix Design process. The aggregate and asphalt binder combination was mixed for 45 – 60 seconds to ensure adequate coating of the aggregate.

Mixture Sample Processing

The laboratory samples of RAP-HMA mixtures were cooled to the room temperature and stored in standard boxes. The pure RAP from each project was stored in separate pans. In order to prepare a PIRS testing probe, a material from each box was screened through sieves No. 8 and No. 30 to isolate particles of 0.6-2mm in diameter. This fine fraction of a mixture was then compacted in a sampling cup to prepare a sample for PIRS testing. Five samples were tested from each box. Figure 5 illustrates the process of PIRS probe preparation in the laboratory.

Figure 5. Process of Mixture Sample Processing for the Purpose of PIRS Testing: Storage in Boxes (top left), Sieving (top right), Sampling Cup Filling (bottom left), and Compaction (bottom right)

For validation purposes, samples were also collected from the HMA production plants. The plant samples were compacted into sampling cup without prior sieving because of higher temperature at the time of collection (Figure 6). The newly paved asphalt surfaces were tested with no prior sample preparation.

Figure 6. Process of Plant Mixture Sample Processing for PIRS Testing: Sampling from a Box (left), Filling (center), and Compaction (right)

From each calibration RAP-HMA sample, the liquid asphalt component was extracted with TCE solvent and recovered with use of Rotovapor apparatus. The recovered binder blends were then tested by PIRS without further alteration.

PIRS Testing Method

PIRS Testing Protocol

A portable Agilent ExoScan FTIR spectrometer was used to conduct PIRS scanning of laboratory and plant samples as well as of pavement surfaces. The research team used diffused reflectance (DR) scanning mode for mixture samples, whereas the attenuated total reflectance (ATR) mode was used for recovered binder blends. The 24 scans within a frequency interval between 4000 and 650 cm^{-1} wavenumbers with 8-cm^{-1} resolution for mixtures and 4-cm^{-1} resolution for binders were averaged to generate one infrared spectrum per test. A PIRS test was repeated three times on each mixture or binder sample.

To perform measurement on a loose granular material, such as sand and RAP, the PIRS instrument with DR sampling accessory was pressed down against the sampling cylinder (Figure 7 [left]). It was found, however, that a cylinder with slightly compacted material such as fresh HMA could be put on the top of PIRS sampling accessory as shown in Figure 7 (center). In this manner, a dependency on the ability of the operator to hold the instrument still with constant pressure was minimized.

In order to validate the calibration curves developed in the laboratory, asphalt mix samples from the HMA plants and paving sites were collected and scanned by the PIRS instrument. The research team had no access to production line in Fruitland plant and, therefore, Fruitland samples were collected in cardboard boxes by contractor personnel and handed to the PPS team shortly after collection. Karcher plant samples were tested near the plant. In Lewiston, plant mixture was not sampled because the HMA plant was closed at the time of testing. The samples were prepared using sampling cylinders similar to those in the laboratory while their PIRS testing was conducted after the temperature of asphalt mix dropped to about 50^oC.

On paving sites, sampling was performed directly on paved surface (Figure 7 [right]) once the temperature of asphalt dropped to about 50^oC. This temperature of testing was chosen due to the limitations of the instrument operation manual. For each project, five locations spaced 30ft apart were tested with three repetitions each.

Figure 7. PIRS Testing with DR Sampling Accessory in Downward (left) and Upward (center) Position in Laboratory and in the Field (right)

The recovered binder blends were tested by sampling about 0.1g of material from 2oz cans and smearing it over the sampling plate of a PIRS's ATR module (Figure 8). Three samples were tested from each binder can.

Figure 8. PIRS Testing of Binders with ATR Sampling Accessory

Computation of Oxidation Signals

An Oxidation Signal (OxS) value was computed from each PIRS spectrum as normalized absorbance intensity at 1700 cm^{-1}. This corresponded to the relative amount of oxidation in the tested sample. The oxidation signal values were then superimposed with corresponding RAP percentage within the sample to produce calibration curves. Figure 9 illustrates the modified method for calculating OxS values. This method employs computing the baselined absorbance peaks at 2920 cm^{-1} and 1700 cm^{-1} as shown in Figures 9 and 10, respectively, followed by normalizing the latter with respect to the former to arrive at OxS value (Figure 11). Figure 12 illustrates those calculations. Such an approach allows for elimination of the effect of the differences in IR sampling methods (DR for HMA and ATR for extracted binders) on OxS values. This also eliminates need for elaborated manipulation of the whole IR spectrum, i.e. baseline correction followed by atmospheric correction and normalization. Note that the basic software provided by Agilent did not include any of the manipulation options.

$$P2920 = A2920\text{-}A2750$$

where *A2920* and *A2750* are measured IR absorbance at 2920 and 2750 cm-1, respectively.

Figure 9. Equation for Computing IR Absorbance Peak at 2920 cm^{-1}

$$P1700 = A1700\text{-}A1820$$

where A1700 and A1820 are measured IR absorbance at 1700 and 1820 cm-1, respectively.

Figure 10. Equation for Computing IR Absorbance Peak at 1700 cm^{-1}

$$OxS = P1700/P2920$$

Figure 11. Equation for Computing Oxidation Signal Value

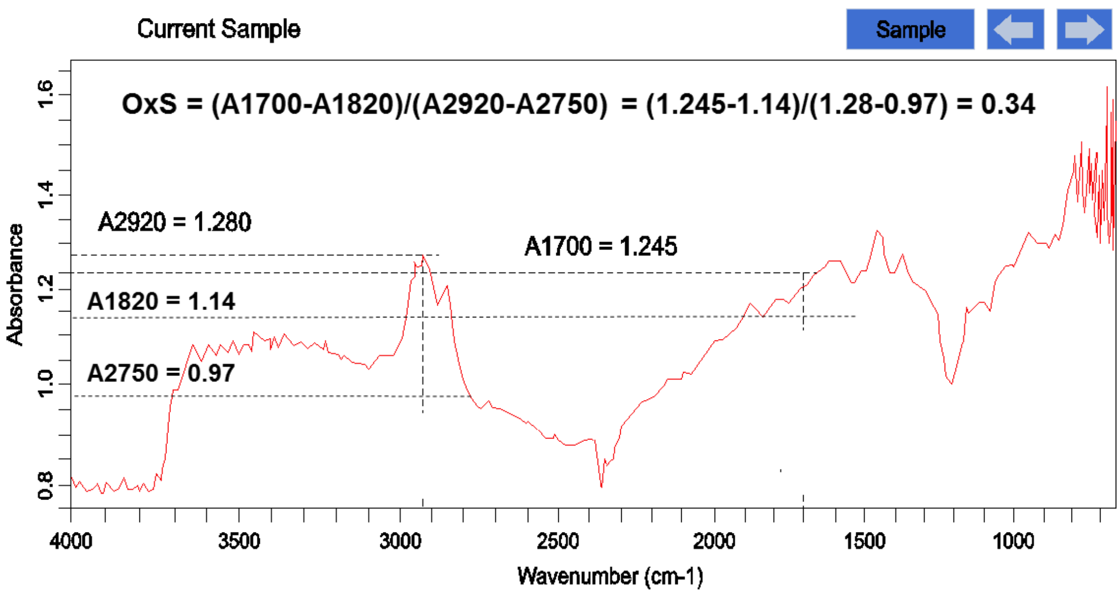


Figure 12. Example of an Absorbance Spectrum of Asphalt Mix Sample

Statistical Analysis Approach

The statistical analysis of PIRS data was conducted to establish standard deviation of the test and evaluate the influencing factors of variability. The analysis began with determining variability in measurements on the same sample in terms of coefficient of variation (COV) and signal-to-noise ratio (SNR) as shown in Figures 13 and 14, respectively.

$$C.O.V. = std/mean$$

where

std = standard deviation from sample mean
mean = sample mean

Figure 13. Equation for Computing the Coefficient of Variation

$$SNR=1/C.O.V.$$

Figure 14. Equation for Computing the Signal-to-Noise Ratio

The second phase of the analysis involved evaluating the linear regression models with OxS as a response and RAP percentage as a predictor. The goodness of fit of a calibration curve for each project was represented by R-squared value. The second phase of the analysis included enhancing the regression models by including various test parameters, such as binder PG, RAP gradation, and RAP binder content as factors. For each factor, its individual contribution into variability of the regression model was evaluated by F-statistics computed as shown in Figure 15. The F-statistics was then used as a measure of importance of a particular factor for a model. The larger the factor's F-value was the higher rank of importance was assigned to this factor.

$$F = \frac{MSE_{predictor}}{MSE_{\mathrm{mod}el}}$$

where:

F = F-value

$MSE_{predictor}$ = Mean squared error of the mean predicted OxS response associated with an individual predictor under question

MSE_{model} = Mean squared error of the mean predicted distress output when all other predictors are in the model

Figure 15. Equation for Computing the F-statistics

In the validation phase of the analysis, the significance of differences between the JMF, plant, and pavement measurements were evaluated with Student's t-tests for difference in means. In the final stage of the analysis, the JMF-specific predictive equations were developed to estimate RAP percentage within an asphalt mixture from the OxS measurements.

Chapter 4
Major Findings

Initial Evaluation of Oxidation Trends and Removal of Outliers

The analysis of PIRS measurements started with initial evaluation of oxidation trends with respect to RAP percentage where all the lab samples were considered. At this point, some unusual data points that did not follow expected monotonic trends were noticed. Those potential outlier values are circled in Figure 16 as they lead to overall low correlation (R-squared of 0.25 to 0.35) between OxS values and RAP percentage, especially for Karcher and Lewiston projects.

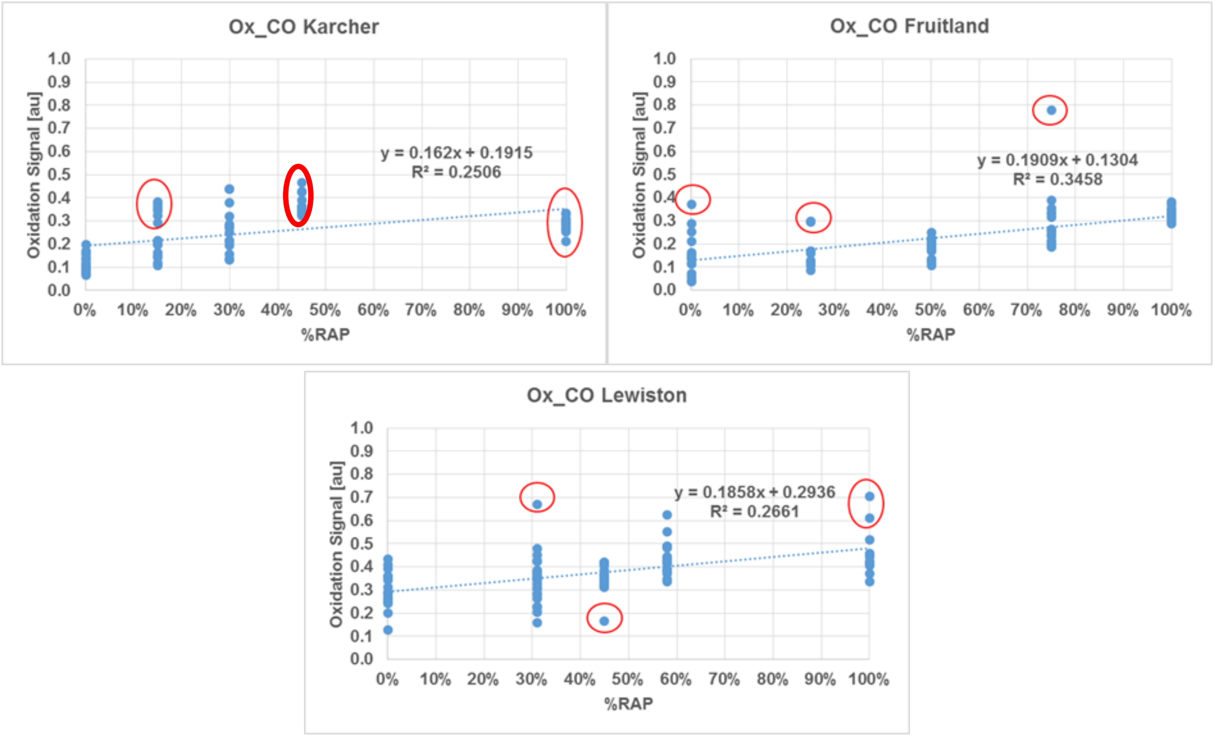


Figure 16. Initial Linear Trends for Selected Idaho Projects: Karcher (top left), Fruitland (top right), and Lewiston (bottom)

The initial regression analysis of PIRS measurements revealed significant differences in the linear trend intercepts with minor difference in trend slopes between the three projects (Table 3). The difference in the intercept was attributed to the initial oxidation in the corresponding virgin binders.

Table 3. Summary of Preliminary Regression Analysis

Project	Slope	Intercept	R-squared
Karcher	0.162	0.192	0.25
Fruitland	0.191	0.130	0.35
Lewiston	0.186	0.294	0.27

The correlation values, however low, were found highly significant (p-value < 0.0001) for the relatively large dataset of about 230 points collected in this study. Therefore, the analysis of normality was performed next to identify the outlier values. The normality of distribution of measurements was evaluated through the visual analysis of histograms and cumulative probability plots for all data and each of the three paving projects separately.

Figure 17 (left) shows histograms of OxS values for all data and each project separately. It can be noticed that the total distribution, while having an approximately "bell" shape, has a distinctive shoulder toward OxS=0.2, and a distinctive albeit low peak centered on OxS=0.7. A visual evaluation of individual project histogram reveals that the shoulder originates from Fruitland and Karcher projects, whereas the Lewiston data contributes to the "outlier" peak on the right. Another important observation is that Lewiston data is more centered around its mean value as compared with widely spread Fruitland and Karcher values. Lastly, the cumulative probability plot in Figure 17 (right) confirms that on average (at 50% probability) Lewiston measurements are higher than the Fruitland and Karcher numbers.

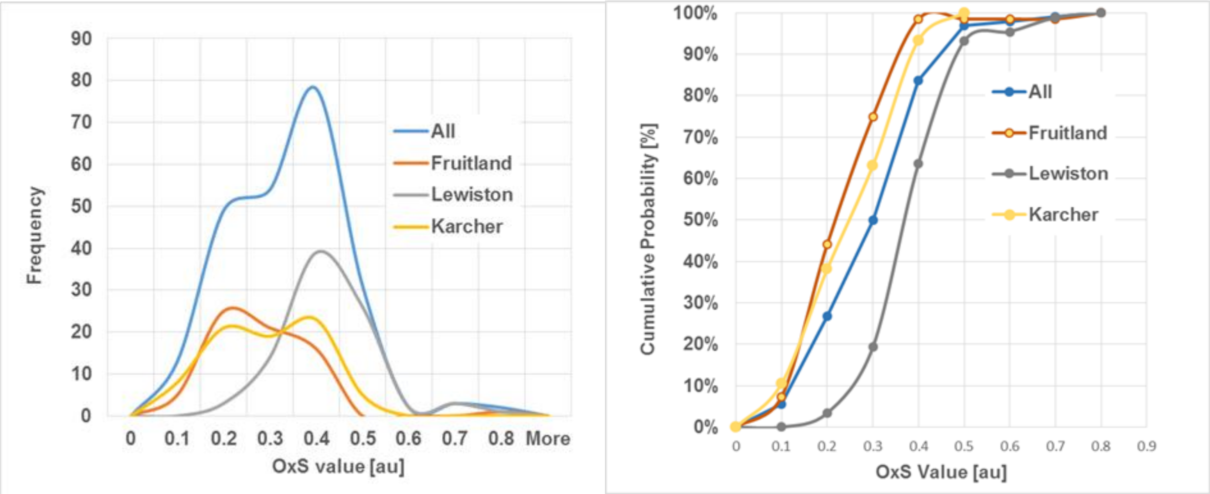


Figure 17. Histogram (left) and Cumulative Probability Plot (right) of OxS Values

The aforementioned observations called for identifying and removing outlier values with cumulative probability lower than 2.5% and higher than 97.5%. This approach resulted in removal of approximately 22% of data (52 out of 232 points), while preserving enough measurements for a fixed RAP percentage within each project (Table 4). It should be also noticed that removal of outliers had resulted in reduction of average coefficient of variation from 28% to 15%.

Table 4. Summary of Outlier Analysis and Variability in OxS Measurements

Project	**Karcher**					**Fruitland**					**Lewiston**				
RAP [%]	**0**	**15**	**30**	**45**	**100**	**0**	**25**	**50**	**75**	**100**	**0**	**31**	**45**	**58**	**100**
C.O.V. before [%]	32	39	36	11	11	60	46	21	47	8	20	30	16	16	23
Total cases	18	15	15	15	13	15	8	17	16	12	16	23	19	20	10
Cases retained	12	9	11	11	10	11	6	13	15	12	9	21	18	18	5
Percent data retained	67	60	73	73	77	73	75	76	94	100	56	91	95	90	50
C.O.V. after [%]	18	22	14	4	6	41	23	12	24	6	14	12	11	11	8

Oxidation Trends in Laboratory RAP-HMA Samples

Once the outlier measurements were removed, the oxidation trends were determined for each project by means of linear regression modeling of OxS as a response from RAP percentage as a predictor. The mean trend functions were then analyzed for deviation from linearity. An ideal linear trend would be expected at full blending between virgin and RAP binder. Therefore, an ideal linear trend could be computed as shown in Figure 18. A deviation of a mean function from an ideal linearity would occur due to lack of blending (negative deviation), over-heating of a sample (positive deviation), or some artifacts of IR light scattering during reflectance measurement (either negative or positive deviation). The latter is an expected deviation from Beer-Lambert Law of Proportionality for an infrared signal from a mixture (*11, 12*).

$$OxS = (1 - C_{RAP})*OxS_0+C_{RAP}*OxS_{RAP} \qquad \text{or} \qquad OxS = OxS_0 +C_{RAP}*(OxS_{RAP} - OxS_0)$$

where

C_{RAP} = RAP percentage in a mixture
OxS_0 = oxidation signal from a zero-RAP mix
OxS_{RAP} = oxidation signal from 100% RAP

Figure 18. Equations for Computing a Linear Oxidation Trend

Figure 19 depicts measured (real) and expected (ideal) oxidation trends for Karcher, Fruitland, and Lewiston projects, while Table 5 compares real and ideal slopes and intercepts. The error bars in Figure 13 depict standard deviation values from the mean OxS value for each fixed RAP percentage shown as hollow and filled markers for real and ideal values, respectively. It is easily observed that in Fruitland and Lewiston projects, most of the real measurements fell within one standard deviation from the ideal linear trends. A clear exception from this observation is a 58% RAP sample from Lewiston with higher than expected OxS values. A lesser yet noticeable degree of deviation is shown for a 25% RAP sample from Fruitland with lower than expected OxS values. Evidently, the Karcher project stands apart with an obviously non-linear trend (R-Squared = 0.54), despite a similarity between ideal and real slopes. For the moment, the reason for such a poor agreement with the Beer-Lambert law for a Karcher project is unknown. Apparently, the magnitude of intercepts for all three projects is controlled by the initial oxidation in the corresponding virgin binders, whereas the slopes depend on difference between oxidation values in virgin and RAP binder. Interestingly, Table 5 shows steeper oxidation slopes for larger differences in high-temperature PG between virgin and RAP binder.

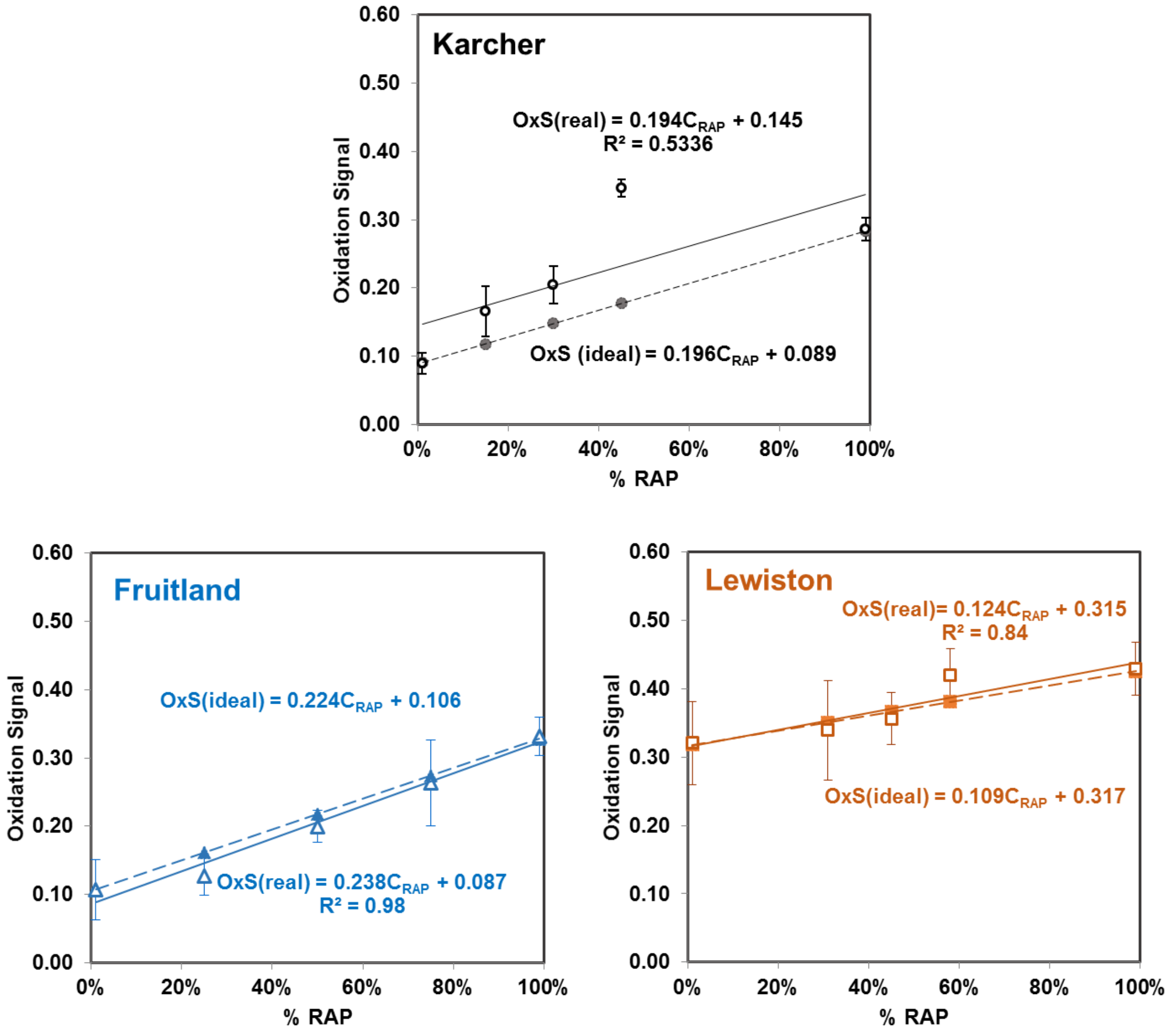


Figure 19. Real and Ideal Oxidation Trends in RAP-HMA: Karcher (top), Fruitland (bottom left), and Lewiston (bottom right)

Table 5. Summary of Oxidation Trend Parameters for RAP-HMA Samples

Project	Ideal Slope	Ideal Intercept	Real Slope	Real Intercept	R-squared	Difference in PG between RAP and virgin binder
Karcher	0.196	0.089	0.194	0.145	0.54	X-70=??
Fruitland	0.224	0.106	0.238	0.087	0.98	88-52=36
Lewiston	0.109	0.317	0.124	0.315	0.87	82-70=12

Field Validation of Laboratory Oxidation Trends

The ultimate goal of this project is to develop a practical procedure for monitoring oxidation in RAP by PIRS before, during, and after asphalt mixture production. Therefore, the validation phase involved analysis of significant differences in oxidation signal between lab-prepared samples, plant samples, and paved surfaces. The project means and standard deviations are shown in Figure 20. The results of pairwise Student t-tests performed to compare sample means are summarized in Table 6. Both graphic comparison and formal t-test results indicate that Karcher plant and pavement samples were oxidized as much as or less than lab samples. Fruitland tests resulted in plant samples oxidized marginally higher than lab samples, while pavement samples were significantly less oxidized overall. The oxidation in Lewiston pavement samples matched that in the lab samples.

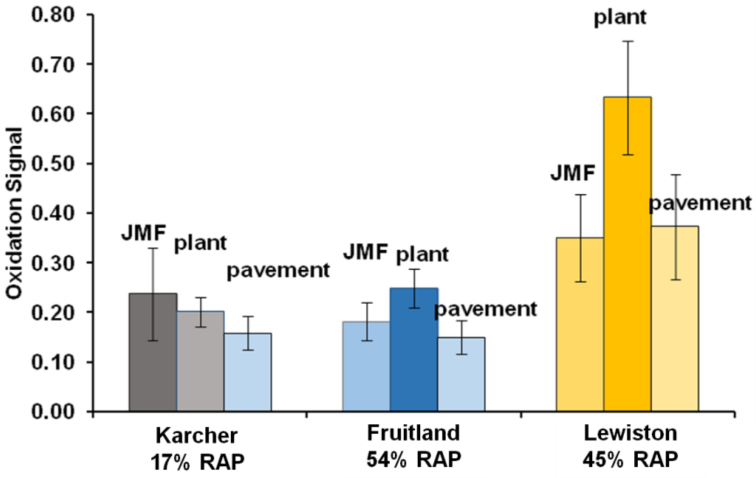


Figure20. Comparison of Mean OxS Values in JMF, Plant, and Pavement Mixture Samples

Table 6. Summary of Significance Tests for Lab, Plant, and Pavement HMA Samples

Project	Parameter	JMF	plant	pavement	
Karcher, 17% RAP	**Mean**	**0.237**	**0.201**	**0.158**	
	Variance	0.009	0.001	0.002	
	Observations	15	10	4	
	Comparison		**plant <= JMF**	**pavement < JMF**	**pavement <= plant**
	P(T<=t) two-tail		**0.194**	**0.026**	**0.102**
	Difference		**Insignificant**	**Significant**	**Insignificant**
Fruitland, 54% RAP		JMF	plant	pavement	
	Mean	**0.182**	**0.248**	**0.150**	
	Variance	0.002	0.002	0.001	
	Observations	17	12	10	
	Comparison		**plant > JMF**	**pavement < JMF**	**pavement < plant**
	P(T<=t) two-tail		**0.000**	**0.047**	**0.000**
	Difference		**Significant**	**Marginal**	**Significant**
Lewiston, 45% RAP		JMF	**plant**	pavement	
	Mean	**0.344**	**Not available**	**0.327**	
	Variance	0.002		0.017	
	Observations	13		12	
	Comparison		**plant vs. JMF** **Not available**	**pavement <= JMF**	**pavement vs. plant** **Not available**
	P(T<=t) two-tail			**0.660**	
	Difference			**Insignificant**	

In summary, the field validation analysis showed that the difference in oxidation between laboratory, plant, and pavement samples did not exceed standard deviation from the mean established for a project. Therefore, the research team has concluded that PIRS testing of post-production mixture samples showed no evidence of deviation from the oxidation level in lab-prepared JMF samples at confidence level α=0.05. Consequently, the proposed procedure for monitoring the quality of RAP would concentrate on using PIRS comparing of oxidation levels in lab-prepared JMF, plant, and pavement samples rather than estimating RAP percentage from oxidation measurements.

Analysis of Influencing Factors of Oxidation in RAP-HMA Samples

To determine what factors influenced OxS measurements on RAP-HMA samples, a multiple linear regression analysis was conducted on PIRS data. As shown in Table 7, the regression analysis dataset included OxS values as a numerical response and RAP percentage (C_{RAP}) as a categorical variable. Treating C_{RAP} as a factor was warranted because (1) each RAP-containing sample was manufactured independently, (2) multiple measurements were done for each RAP increment, and (3) there was an overlap in RAP percentages in the three selected projects. For example, each project had 0 and 100% RAP, both Karcher and Lewiston had 30(31) and 45% RAP samples. The other factors, besides location (PROJECT) included parameters associated with differences in project JMFs, such as RAP binder content (RAP_Pb), and amount of fines in RAP (RAP_#16). Effectively, the following regression models were considered (Figure 21):

$$OxS = \beta_0 + \beta_i F[C_{RAP}]_j + se$$

$$OxS = \beta_0 + \beta_i F[C_{RAP}]_j + \beta_k F[PROJECT]_k + se$$

$$OxS = \beta_0 + \beta_i F[C_{RAP}]_j + \beta_m F[RAP\_Pb]_m + se$$

$$OxS = \beta_0 + \beta_i F[C_{RAP}]_j + \beta_n F[RAP\_\#16]_n + se$$

where

β_0 = intercept value
β_j β_k β_m, and β_n= coefficient estimates
se = standard error of the model

Figure 21. Regression Model Equations for Analysis of Influencing Factors

Table 7. Summary of Statistical Data Set for Influencing Factors Analysis

Variable Description	Variable ID	Response/ Predictor	Numerical/ Categorical (factor)	Values
Oxidation Signal	OxS	Response	Numerical	Min=0.04, Max=0.52, Mean=0.27
RAP percentage	C_{RAP}	Identifier		0, 0.15, 0.3, 0.45, 1 for Karcher 0, 0.25,0.5,0.75, 1 for Fruitland 0, 0.31, 0.45, 0.58, 1 for Lewiston
Factor of C_{RAP}	$F[C_{RAP}]$	Predictor	Categorical	1 if present in the model; 0 if absent from the model
Project JMF	PROJECT	Identifier		Karcher, Fruitland, Lewiston
Factor of PROJECT	F[PROJECT]	Predictor	Categorical	1 if present in the model; 0 if absent from the model
RAP Binder Content	RAP_Pb	Identifier		5.2 (Fruitland) 5.3 (Karcher) 6 (Lewiston)
Factor of RAP_Pb	F[RAP_Pb]	Predictor	Categorical	1 if present in the model; 0 if absent from the model
RAP passing #16 sieve	RAP_#16	Identifier		0.26 (Lewiston), 0.43 (Karcher and Fruitland)
Factor of RAP_#16	F[RAP_#16]	Predictor	Categorical	1 if present in the model; 0 if absent from the model

The multiple regression analysis of each model from Figure 21 was performed with Arc 1.06 open-source statistical software (*22*). Table 8 summarizes regression parameters for those models along with individual F-values of the factors, which indicate their importance for a model. The goodness of fit of the models in Table 8 does not vary significantly. A minimum of 82% of data fits the model with RAP percentage as a sole predictor (R-sq. =0.823). When other factors are considered, any of them improve the goodness of fit to 87%. When individual influence of the factors is considered, amount of RAP passing sieve #16 (RAP_#16) appears to be an even slightly more influencing factor (F=45.8) than RAP percentage (F=41.3). On the other hand, factor of project JMF (PROJECT) yields identical effect to that of RAP binder content (RAP_Pb) with F-value =26.7.

Table 8. Summary of Importance Ranking for Influencing Factors of Oxidation in HMA

Model Regression Parameters					**Factors Influence**	
Model Equation	**R-sq.**	**Standard error**	**F (model)**	**p-value (model**	**F(C_{RAP})**	**F(other factors)**
$OxS = \beta_0 + \beta_i F[C_{RAP}]_j + se$	0.823	0.049	71.8	0.0000	71.8	None
$OxS = \beta_0 + \beta_i F[C_{RAP}]_j + \beta_k F[PROJECT]_k + se$	0.869	0.042	84.2	0.0000	42.1	26.7 (PROJECT)
$OxS = \beta_0 + \beta_i F[C_{RAP}]_j + \beta_m F[RAP\_Pb]_m + se$	0.869	0.042	84.2	0.0000	42.1	26.7 (RAP_Pb)
$OxS = \beta_0 + \beta_i F[C_{RAP}]_j + \beta_n F[RAP\_\#16]_n + se$	0.864	0.043	88.4	0.0000	41.3	45.8 (RAP_#16)

In conclusion, the results of the regression analysis indicated RAP fine content, RAP binder content, and RAP percentage within a mixture as primary influencing factors. That being said, only about 87% of data selected for the analysis could be explained by the controlled factors. The other 13% of variation were attributed to random factors (non-uniformity of samples) and instrumentation artifacts (light scattering).

Predicting RAP Percentage in HMA Mixtures from Oxidation Signals Measured by PIRS

The reliability of predicting RAP percentage within a RAP-HMA sample was evaluated by the regression analysis of a model where C_{RAP} was a response to OxS with an addition of PROJECT as a factor (Equation in Figure 22).

$$C_{RAP} = \beta_0 + \beta_1 OxS + \beta_2 F[PROJECT]$$

where

β_0 = intercept value
β_j β_k β_m, and β_n= coefficient estimates
se = standard error of the model

Figure 22. Regression Model Equations for Predicting the RAP Percentage

The regression equations and goodness-of-fit parameters are shown for each project, separately, in Table 9. One can notice common slope for all three projects with distinct intercepts where the lower the intercept the worse goodness-of-fit is. Accordingly, the best-fit model has been achieved for Fruitland (R-sq. =0.77), followed by Karcher (45% RAP excluded) and Lewiston, in that order (R-sq. of 0.67 and 0.51, correspondingly). Note that R-squared values indicate how much of the data included in a model can be explained by a predicted mean function.

Table 9. Summary of Regression Parameters for Predicting RAP Percentage from PIRS Measurements

Project	Model Regression Parameters				
	Model Equation	**R-sq.**	**Standard error (model)**	**F (model)**	**p-value (model**
Karcher	C_{RAP}=3.36-0.28*OxS	0.67	0.16	80	0.0000
Fruitland	C_{RAP}=3.36-0.19*OxS	0.77	0.15	184	0.0000
Lewiston	C_{RAP}=3.36-0.80*OxS	0.51	0.15	66	0.0000

To illustrate the confidence of predicting the C_{RAP} from OxS measurements, mean functions and their 95% confidence intervals are plotted in Figure 23. From the Figure, it can be surmised that RAP percentage can be predicted within 8 to 10 percentage points for all projects. This means that, for example, an oxidation signal value of 0.25 may indicate 60% to 70% RAP in Fruitland, 50% to 65% RAP in Karcher, or 0% to 15% in a Lewiston mixture sample.

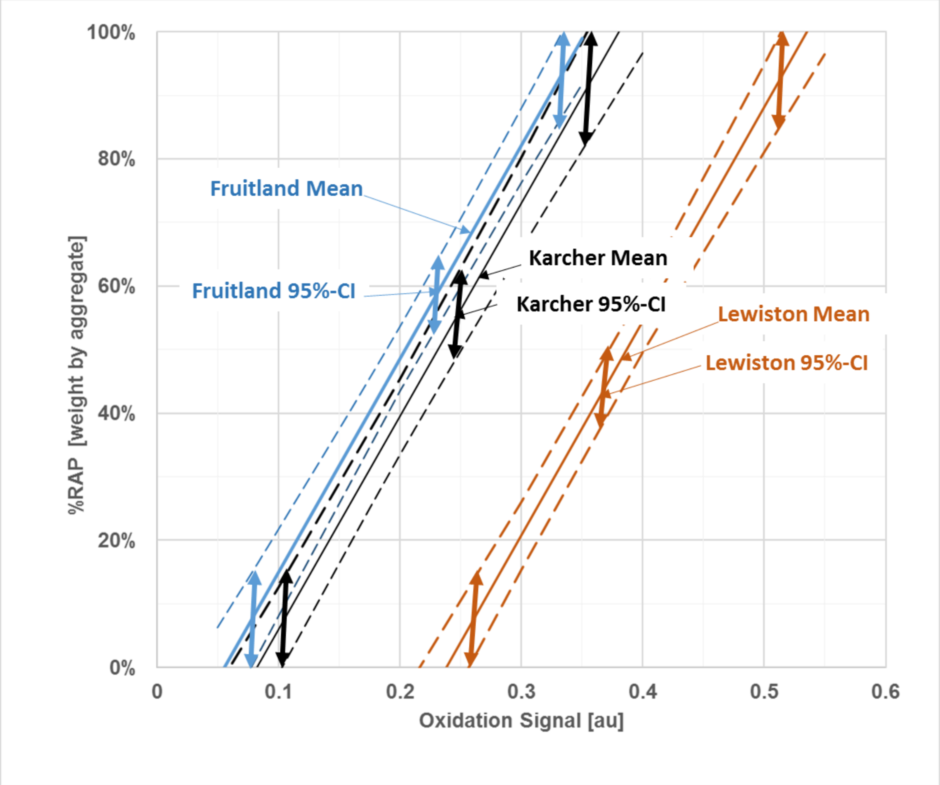


Figure 23. Confidence Intervals of Predicted RAP Percentage

The results depicted in Figure 23, while optimistic, should be considered with caution, because only 72% of "good" data, which was 165 out of a total 230 measurements, are associated with relatively narrow confidence interval of 15 percentage points. Therefore, at this point, the team is going to recommend evaluating differences between JMF, plant, and pavement samples rather than estimating RAP percentage in an asphalt mixture.

Variability in Oxidation of Recovered Binder Blends

Binder blends were recovered from the laboratory RAP-HMA samples in order to validate oxidation trends determined from the PIRS measurements on mixture samples. Similar to the approach to mixture analysis, the validation targeted comparison of OxS trends with respect to increase in RAP percentage accompanied by the analysis of variability. Accordingly, ideal and real trend lines were fitted to the corresponding mean OxS values for fixed RAP percentages and their slopes and intercepts were evaluated.

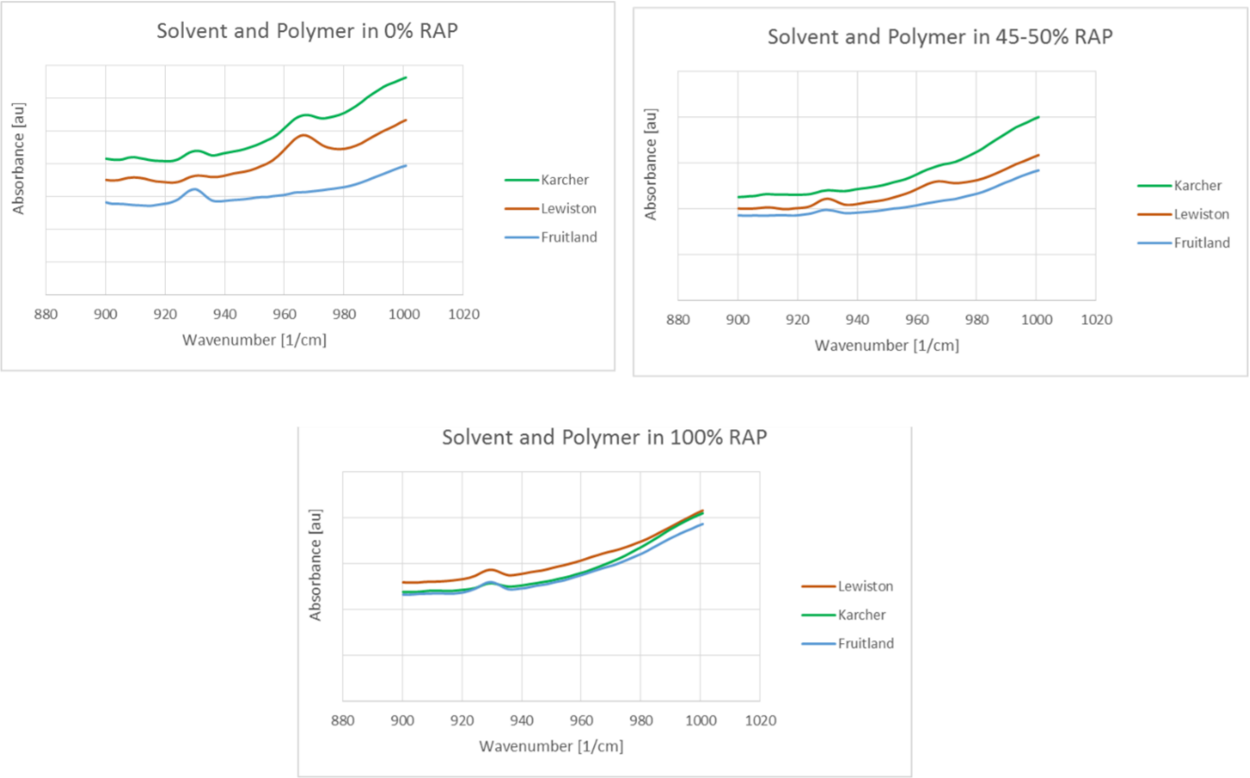
Table 10 reports the variability in PIRS measurements on recovered binders in terms of COV for 3 repetitions of each measurement. The Karcher measurements indicate very low variability (COV=3%) both within and between samples with different RAP percentage. On the other hand, both Fruitland and Lewiston shows much higher variability both within and between samples. Overall variability within Fruitland and Lewiston projects (COV of 10% and 13%, respectively) is much higher than that for Karcher project (COV = 3%). In addition, the COV differs by 5% to 9% in Fruitland and Lewiston data, with only

1% of deviation in Karcher data. Although, the overall variation in binder measurements (~8%) was found lower than that in mixture measurements (~15%), additional investigation was conducted on the effect of solvent and polymer within binder samples, as discussed next.

Table 10. Summary of Variation in PIRS Measurements on Recovered Binders

Project ID					
Karcher		**Fruitland**		**Lewiston**	
RAP Content	**Average C.O.V.**	**RAP Content**	**Average C.O.V.**	**RAP Content**	Average C.O.V.
No RAP	2%	No RAP	20%	No RAP	31%
15% RAP	3%	25% RAP	7%	31% RAP	11%
30% RAP	2%	50% RAP	9%	45% RAP	13%
45% RAP	3%	75% RAP	5%	58% RAP	5%
100% RAP	3%	100% RAP	9%	100% RAP	5%
Average Project C.O.V.	3% ± 1%	Average Project C.O.V.	10% ± 5%	Average Project C.O.V.	13% ± 9%

Presence of solvent was examined by using characteristic peak at 930 cm^{-1}, which is commonly present in a trichloroethylene solvent IR spectrum. The presence of Styrene-butadiene polymer was indicated by the peak at 965 cm^{-1}, which is associated with butadiene HC=CH bond bending vibration. Figure 24 shows example PIRS spectra of binder blends extracted from HMA samples with 0%, 50%, and 100% RAP. On average, TCE peak values for Karcher, Fruitland, and Lewiston projects were 0.018±0.007, 0.026±0.01, and 0.030±0.012, correspondingly. With respect to polymer, no SB polymer was detected in any of the Fruitland samples (base binder PG 52-34) whereas the traces of SB polymer could be detected in both Karcher and Lewiston samples (base PG 70-28 and 70-34, correspondingly) with as much as 50%RAP (Figure 24).



Solvent and Polymer in 0% RAP

Solvent and Polymer in 45-50% RAP

Solvent and Polymer in 100% RAP

Figure 24. Examples of Presence of TCE Solvent and SB Polymer in HMA Samples

The aforementioned results indicated an association between higher variability in binder oxidation measurements and presence of solvent and polymer in mixture samples. This effect could mostly lead to hardening of binder that is exposed to solvent for a prolonged time during extraction (*23, 24*). On the other hand, if not all the binder extracted from a mix sample, the measured oxygen content is expected to be lower than that in the mix (*25*).

Oxidation Trends in Recovered Binders

The ideal and real oxidation trends in recovered binders are depicted in Figure 25, with trend parameters reported in Table 11. One can see that all three projects exhibit good agreement with the expected (ideal) linear trend, while Fruitland and Lewiston data shows a very similar, although relatively small, upward bias in the intercepts. The lack of evidence of such a bias in Karcher measurements might indicate that Fruitland and Lewiston binders underwent a somewhat greater extent of hardening during extraction. Such an indication also agrees with higher concentration of solvent in Fruitland and Lewiston samples and/or longer exposure to solvent at higher temperatures during extraction. Overall, the good agreement with the ideal linear trends is expected because of the uniformity of chemical composition within an extracted asphalt binder.

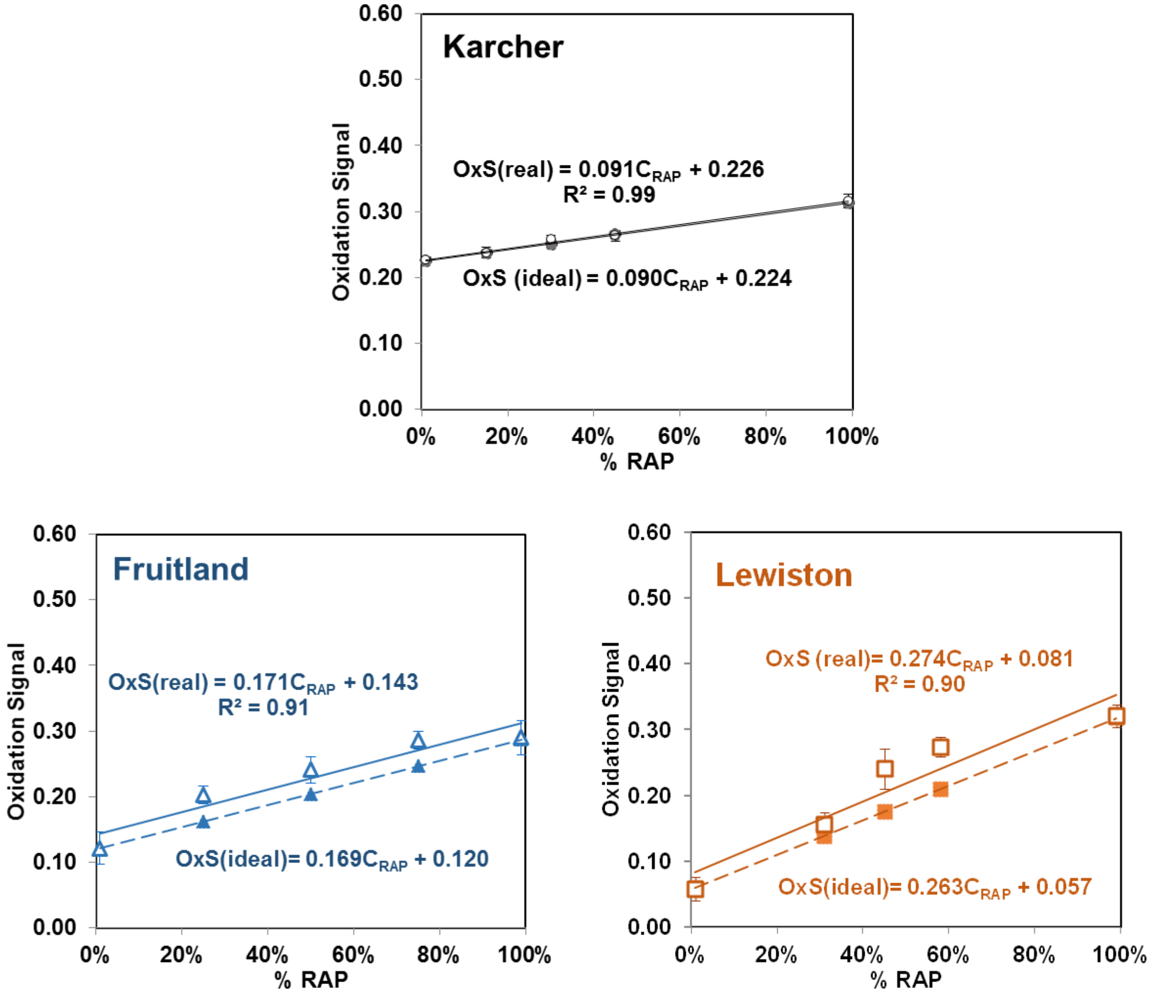


Figure 25. Real and Ideal Oxidation Charts for Recovered Binders: Karcher (top), Fruitland (bottom left), and Lewiston (bottom right)

Table 11. Summary of Oxidation Trend Parameters for Recovered Binder Samples

Project	Ideal Slope	Ideal Intercept	Real Slope	Real Intercept	R-squared
Karcher	0.090	0.224	0.091	0.226	0.99
Fruitland	0.169	0.120	0.171	0.143	0.91
Lewiston	0.263	0.057	0.274	0.081	0.90

At this point of the investigation, significant differences were noticed between OxS values for mixture samples and their corresponding extracted blends (Table 12). Such a difference was previously reported for 25% RAP mixtures, although it did not exceed 0.05 (*15*). However, the variation in differences between mixture and corresponding binder measurements in this study was substantially larger. For instance, all Karcher binders yielded higher OxS value than their corresponding mixtures did. On the other hand, much lower OxS values were reported for Lewiston binders as compared with their corresponding mixtures. The Fruitland data yielded the best agreement between OxS measurements in mixtures and recovered blends.

Table 12. Comparison Between Real Oxidation Trend Parameters for Mixtures and Recovered Binders

Project	Difference in Intercepts ($OxS_{0\text{-}BINDER}$ - $OxS_{0\text{-}MIX}$)	Difference in Slopes (S_{BINDER} - S_{MIX})
Karcher	0.091	-0.103
Fruitland	0.056	-0.067
Lewiston	-0.234	0.150

In summary, the analysis of oxidation measurements in recovered binder blends revealed similarity of oxidation trends for Fruitland and Lewiston project with noticeable convex-shaped deviation from linearity. This result indicated a decrease in the effect of RAP on oxidation with an increase in RAP percentage. The two possible reasons for that could be (1) partial (as opposed to full) blending between RAP and virgin binder, or (2) inability of extraction procedure to recover all binder from the RAP aggregates. The effect of solvent and polymer on the oxidation of binders during the extraction led us to believe that direct PIRS measurements on solid asphalt mixture samples was more suitable for quality control of RAP in the field.

Chapter 5

Conclusions and Recommendations

Summary and Major Findings

- The reported research project RP 249 included demonstration of the PIRS technology for in-situ measuring of oxidation in asphalt mixtures and validation of the proposed AASHTO procedure for quality control of RAP-containing asphalt mixtures in the field. Three projects in Karcher, Fruitland, and Lewiston, ID, with various JMFs were chosen to conduct PIRS measurements on RAP-HMA samples concluding laboratory-manufactured samples with various RAP content as well as samples from the plant and pavement surface.
- The RAP-HMA samples and their corresponding recovered binder blends were tested with a PIRS instrument with DR and ATR accessories to determine relative amount of oxidation defined as a normalized carbonyl peak at 1700 cm^{-1} wavenumbers. The analysis of measured oxidation signals involved analysis of variability, correlation with RAP percentage within a mixture sample, and direct comparison of JMF, plant, and pavement samples.
- The average variation in DR measurements on solid RAP-HMA samples remained within 15% of sample mean as approximately 80% of measurements ("good" data) were considered for the analysis. Half as much variation was found in all the recovered binder blends. The differences in JMFs, such as RAP fines percentage and RAP binder content, were the major factors of variability in 86% of good measurements. The rest of uncertainty in oxidation measurements was attributed to non-uniformity of sample composition and PIRS method artifacts, such as light scattering.
- With one out of fifteen samples removed, all three projects demonstrated the oxidation measurements in RAP-HMA followed linear Beer-Lambert law within one standard deviation from a mean sample value. A similarly high degree of linearity was also observed in the recovered binder blends, although with a noticeable bias from the expected trends in two out of three projects. The reason for such a bias could be extended exposure to solvent and heating during the extraction and recovery.
- The extensive statistical analysis indicated that RAP percentage within a solid asphalt sample could be determined from PIRS-measured oxidation values with reliability of about 70 percent with expected error of 8 to 10 percentage points.
- Direct comparison of oxidation in JMF, plant, and pavement samples revealed a similar level of oxidation with pavement being similarly or less oxidized than JMF and plant samples for all paving projects.

In summary, the demonstration of PIRS technology on the three ongoing paving projects in Idaho was successful. The analysis of PIRS measurements on HMA mixtures and the corresponding recovered binder blends indicated fairly high reliability of predicting RAP percentage within a mixture. However, a direct comparison of JMF, plant, and pavement samples appears to be a faster and more reliable solution for quality control of RAP in the field.

Project Deliverables

Draft Standard Test Procedure for Quality of RAP with PIRS

The demonstration of PIRS technology on the selected paving projects in Idaho was accompanied by following an earlier proposed draft AASHTO test method for measuring oxidation in aged asphalts. In the process, a sample preparation and data processing techniques were adjusted to facilitate the use of PIRS technology in the field. The draft standard test procedure is a stand-alone document that can be found in Appendix A. It is a research team belief, however, that additional steps to adjust the procedure for specific materials and operation needs in Idaho are warranted. A final version of a QC procedure for monitoring oxidation in RAP is proposed to be developed during a validation study as a part of implementation process.

Implementation Plan for Incorporation of PIRS Technology into ITD QC Program

In order to successfully implement the PIRS technology on Idaho paving projects, a plan of action is proposed as one of this project deliverables. An implementation plan includes the following major steps:

1. Assemble an Implementation team to develop and follow through the implementation plan.
2. Assemble data with regard to current practices of RAP production, maintenance, and utilization in the state.
3. Develop preliminary strategic plan with regard to RAP use and objectives of PIRS testing.
4. Conduct market research to identify the vendor and purchase PIRS equipment.
5. Conduct staff training on using the proposed method on selected equipment.
6. Develop a sampling and testing plan for expanded pilot testing of the PIRS technology statewide.
7. Complete pilot testing of PIRS technology statewide to develop standardized threshold of acceptance
8. Review and finalize the state standard method for PIRS testing of RAP.

Recommendations

- The demonstration of PIRS technology on the three paving projects in Idaho was successful. Therefore, the research team recommends on taking steps to implement this technology as a complimentary QC procedure for controlling and monitoring oxidation in high-RAP asphalt mixtures before, during, and after production.
- The implementation process should include as a minimum (1) an expanded pilot validation study statewide and (2) a "rodeo" of PIRS equipment from multiple vendors to ensure that the best available equipment is chosen.

- Should the ITD decide to implement PIRS technology, the PIRS equipment can be potentially used for other pavement applications as described in the next section.

Other Prospective Applications of PIRS Technology

The previous work by SHRP2 R06B and by the research team for this project identified a wide range of potential applications of PIRS technology to pavement materials. Besides the RAP monitoring technology described in this report, PIRS can be successfully used for the following applications:

- Identification and quantification of polymer additives in asphalt binders and mixtures.
- Fingerprinting of other additives and contaminants to asphalt including, but not limited to, solvents and recycled engine oils.
- Fingerprinting of chemical admixtures in Portland cement concrete.
- Short-term oxidation of asphalt binders to determine their high-temperature performance grade.
- Determination of aging rate of asphalt pavement surfaces to determine optimal timing of preservation treatment.

Should the ITD become interested in the above applications, the research team is ready to assist ITD with implementing the aforementioned technologies.

Instrument Costs

The cost of a hand-held portable FTIR such as the Agilent Technologies used in this project is in rounded figures close to $40,000.00, with no more than a $1,200.00 for yearly operations. This will include the diffuse reflectance accessory which was the principle application for this demonstration project. Adding the attenuated total reflectance (ATR) accessory to measure the asphalt binder would an additional cost of $5,000.00 to the total purchase price of the portable unit. These accessories are easily interchangeable in the field and can also be used in the laboratory to get a quick analysis of the asphalt binder. In the original SHRP2 research a Bruker Optics portable Fourier infrared spectrometer was used and its cost was close to $25,000. However, as in the original research, one would need to drill a sample from the pavement or obtain a sample of the asphalt mixture and bring to the instrument to then perform similar measurements. The Agilent Technologies device is truly a field instrument and all analyses are totally nondestructive, and with the added versatility to measure the oxidation level of the binder by quickly interchanging the diffuse reflectance for the ATR accessory.

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Appendix A
Draft Standard Test Procedure for Quality of RAP with PIRS

Standard Method of Test for

Evaluation of Oxidation Level of Asphalt Mixtures by a Portable Infrared Spectrometer

AASHTO Designation: TP XXX-YY

American Association of State Highway and Transportation Officials
444 North Capitol Street N.W., Suite 249
Washington, D.C. 20001

Standard Method of Test for

Evaluation of Oxidation Level of Asphalt Mixtures by a Portable Infrared Spectrometer

AASHTO Designation: TP XXX-YY

1. SCOPE

1.1. This method covers the measurement of the oxidation level in an asphalt mixture by a Portable Infrared Spectrometer (PIRS). The level of oxidation in an asphalt mixture sample is inferred from a specified peak value on the absorbance spectrum of a sample further called “oxidation signal.” The PIRS measurements of oxidation can be incorporated into pavement management data to assess pavement aging rate. This is obtained by using as a baseline the initial PIRS values and then at some defined frequency obtained other PIRS values that can be used to obtain a pavement aging rate.

1.2. The oxidation signal from an asphalt mixture sample produced from an asphalt plant serves as a reference for measurements on pre-fabricated in laboratory prepared asphalt mixture samples, on the surface of compacted asphalt mixture in the field, and/or on samples of reclaimed asphalt pavement (RAP) material collected from RAP stockpile.

1.3. Samples of loose uncompacted asphalt mixture as well as in-place compacted asphalt mixtures in the field can be scanned by a PIRS operating in a diffuse reflectance mode to obtain their corresponding spectra. When loose uncompacted asphalt mixture samples are scanned by PIRS the nominal maximum aggregate size (NMAS) of the asphalt mixture passing the US sieve should be No. 8 (2.36 mm). When in-place compacted asphalt mixtures in the field are being scanned by PIRS the nominal maximum aggregate size of the asphalt mixture should be #4 (4.76 mm) or larger.

1.4. Do not perform PIRS scanning of asphalt mixture samples at a temperature exceeding 60°C (140°F).

1.5. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See Section 11.

2. TERMINOLOGY

2.1. *Definitions*:

2.1.1. Infrared spectroscopy is the study of the interaction of infrared light with matter. Infrared spectroscopy is the study of the interaction of infrared light with matter.

2.1.2. An infrared spectrum is a plot of the measured infrared intensity versus wavelength (or wavenumbers) of light.

2.1.3. An infrared spectrometer is an instrument used to obtain infrared spectra.

2.1.4. A Fourier Transform Infrared Spectrometer (FTIR) is the most common type of spectrometer.

2.1.5. Diffuse reflectance Fourier transform infrared spectroscopy (DRIFTS) is an infrared technique used on rough surfaces where the infrared light is reflected and transmitted at different amounts depending on the bulk properties of the material.

2.1.6. Oxidation signal is defined as the ratio of two peaks from the infrared spectrum

3. SIGNIFICANCE AND USE

3.1. *Procedure:*

3.1.1. The procedure can be used to monitor the oxidation levels of an asphalt mixture before, during its production and the newly constructed pavement. The procedure can detect differences between the oxidation levels of asphalt mixtures from the initial JMF, plant production and the newly placed pavement.

3.2. *Oxidation Value Uses:*

3.2.1. The reported oxidation values from this procedure can be used to monitor the aging rate in asphalt pavements and determination of optimal timing for pavement preservation treatments. Overtime the oxidation values will increase and an aging rate can be obtained.

3.2.2. The reported oxidation values from the procedure can be used for monitoring consistency of asphalt mixtures containing RAP.

4. APPARATUS

4.1. *Sampling Equipment:*

4.1.1. Metal Scoop

4.1.2. A stainless steel pipe for filling and compacting the asphalt mixture samples. Suitable dimensions are diameter: a minimum of one inch; height: two inches

4.1.3. Hammer to compact the sample in the stainless pipe.

4.2. Standard AASHTO sieves No. 4 (4.75 mm), No. 8 (2.36 mm) and No. 30 (0.6 mm) for sifting a sample of the loose mix.

4.3. Portable Fourier Transform Infrared spectrometer equipped with diffused reflection accessory.

4.4. Soft cloth or tissue for cleaning.

5. SAMPLING PROCEDURE

5.1. *Preparation of a Plant Produced Asphalt Mixture Samples*:

5.1.1. Collect a representative sample of plant produced loose asphalt mixture according to AASHTO T 168 or AASHTO T 2 if sampling for RAP.

5.1.2. Let the asphalt mixture sample cool to a temperature of 60 °C (104 °F) or less before measurement.

5.1.3. Fill the stainless steels pipe with asphalt mixture as shown and compact it with the hammer. A compacted plant sample is shown in Figure 2.

5.1.4. Use a hammer and pivot tube to compact the sample to the top level of the sampling cylinder, to ensure a smooth surface of the sample (Figure 2).

5.1.5. Repeat steps 5.1.1 through 5.1.4 to create five sampling cylinders filled and compacted with asphalt mixture for PIRS scanning.

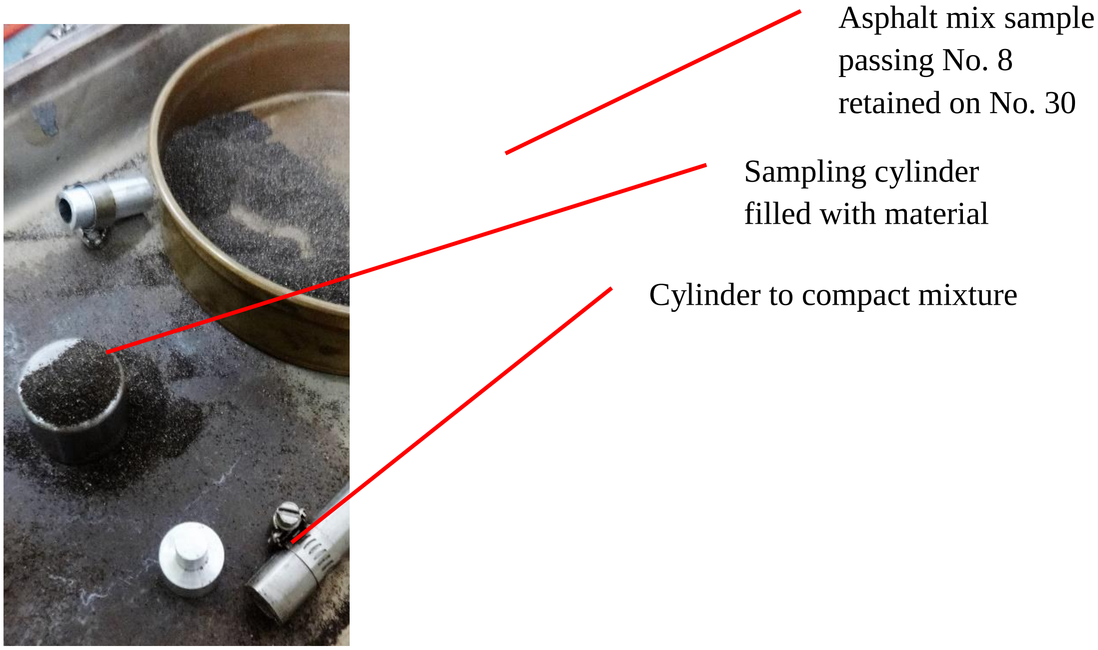

Figure 1—Tools for Preparing Sample from an Uncompacted laboratory Material

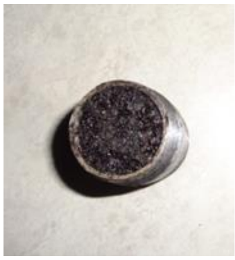

Figure 2—Steel Sampling pipe with compacted mixture: side and top view

5.2. *Preparation of RAP Material Sample or Uncompacted laboratory samples*

Note 1—This step is skipped if the asphalt mixture does not contain RAP.

5.2.1. Obtain approximately 10 lbs of RAP material sample, per AASHTO T 2, from the designated RAP stockpile.

5.2.2. Sieve the RAP sample through No. 4, No. 8, and No. 30 sieves.

5.2.3. Use the material retained on the No. 30 sieve to fill the sampling cylinder (Figure 1).

5.2.4. Use a hammer to compact the sample to the top level of the sampling cylinder (Figure 2).

5.2.5. Repeat steps 5.2.1 through 5.2.4 to create five sampling cylinders for further PIRS scanning.

5.3. *In-Place Pavement Surface Measurement:*

Note 2—No sample preparation is required for PIRS testing of asphalt pavement surfaces. Use a brush to remove any surface dust on the pavement prior to contact of the pavement surface with the PIRS device.

5.3.1. Select and mark designated sampling locations on the pavement surface. Use the built in GPS in the PIRS device associated with the particular scan.

5.3.2. Perform PIRS scanning as described in Section 6 and Section 7 and away from the wheel path.

5.3.3. A minimum of five sampling cylinders for each designated job mix formula (JMF) or pavement location should be scanned by PIRS to establish mean and standard deviation for that JMF for the pavement location.

Note 3—A sample plan may follow the process used to generate random numbers that determine which five sections of the pavement are to be measured similar to that used to determine where density is measured on a compacted pavement. Alternatively, a sampling plan may be defined by the agency.

5.3.4. If an agency sampling plan requires more samples, use agency sampling plan.

6. SPECTROSCOPIC EQUIPMENT SET-UP

6.1. The PIRS instrument should have a sampling accessory in full contact with the asphalt mixture sample in the sampling cylinder or in full contact with the surface of the in-place compacted asphalt mixture in the field. See Figures 3 and 4 respectively.

6.2. The spectrometer should be equipped with a portable battery to ensure reliable power supply during testing.

6.3. It is recommended to follow the instrument manual in regards to the ambient temperature and moisture.

6.4. The diffused reflectance mode should be used for this procedure.

6.5. The number of scans and scanning region of frequencies should be pre-set as prescribed by the PIRS manufacturer. A minimum 24 scans in the region between 3800 and 1000 cm–1 wavenumbers is recommended. Such a setup ensures testing time of maximum one minute per sample.

Figure 3—PIRS Testing Module Attached to Sampling Cylinder

Figure 4—PIRS Testing of the in-place Asphalt Pavement Surface

7. PROCEDURE

7.1. Collect asphalt mix sample as described in Section 5.

7.2. Set up the PIRS instrument as described in Section 6 and in accordance with PIRS operating manual.

7.3. Place the PIRS instrument on the surface of the asphalt sample, ensuring full contact between them.

7.4. Complete PIRS testing of the sample in accordance with PIRS operating manual.

7.5. Store the testing result in a designated library or folder for further processing.

8. SPECTRAL DATA PROCESSING AND INTERPRETATION

8.1. *Determination of Oxidation Signal Value:*

8.1.1. For each sample, a set of spectral data is represented by a scan by the portable infrared spectrometer where the Y-axis is the infrared absorbance values and the X-axis the corresponding wavenumbers (see Figure 5).

8.1.2. The absorbance values plotted against the wavenumber values create an absorbance spectrum for the sample.

8.1.3. Oxidation Signal (OxS) value is calculated as shown in Equation 1.

$$OxS = \frac{A1700 - A1820}{A2920 - A2750} \qquad (1)$$

where:

*A*1700 = peak value (maximum absorbance value) in the region between 1750 and 1650 cm^{-1}, which is associated with the benzylic ketone functional group most commonly present in oxidized asphalt

*A*2920 = peak value (maximum absorbance value) in the region between 2950 and 2850 cm^{-1}, which is associated with the aliphatic hydrocarbon chains present in all asphalts. This peak is also expected to be the highest of all the peaks associated with asphalt binder.(Figure 5)

A1820 and A2750 = baseline absorbance values at 1820 and 2750 cm^{-1}, respectively.

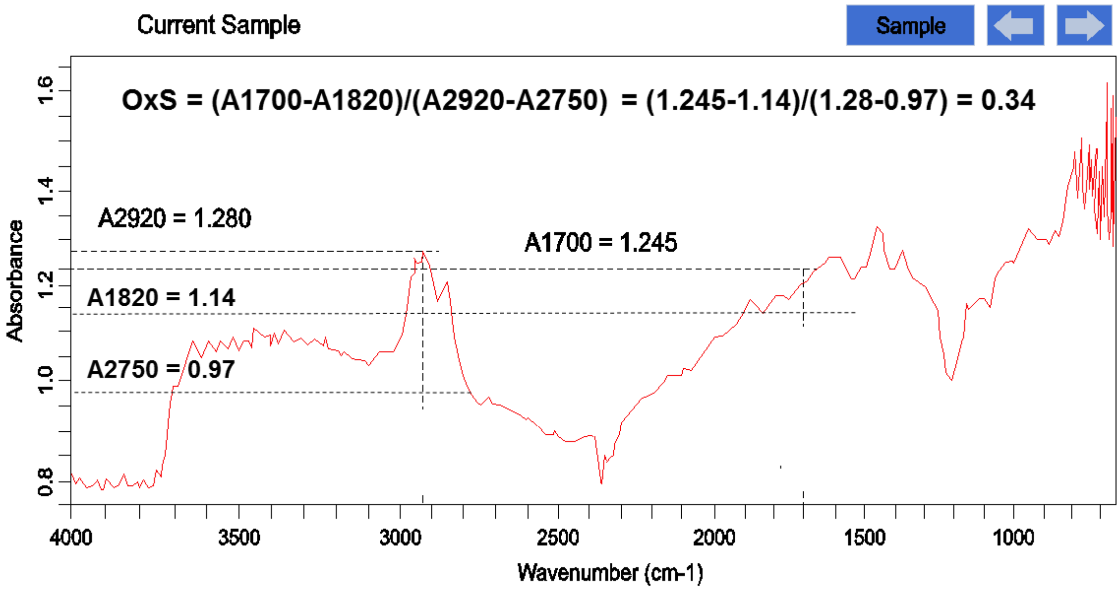

Figure 5—Example of Calculation of an *OxS* Value

Note 2— Note: Location of the characteristic peaks on an absorbance spectrum can vary within ±10 cm^{-1} from the values given in this method.

8.2. Interpretation of Oxidation Value Data:

8.2.1. The OxS data should be used for the quality control purposes to compare the extent of oxidation in plant-produced and compacted samples against a control sample produced in accordance with a JMF. For example, Figure 6 superimposes the mean and standard deviations for the OxS values obtained from the JMF, plant, and the in-place pavement samples. The error bars shown are one standard deviation from the mean of five samples. The apparent overlap between the error bars in Figure 6 indicates that both plant and the in-place pavement measurements have OxS values well within a standard deviation from the mean JMF values. The control sample is the approved JMF and obtained from the mix design laboratory

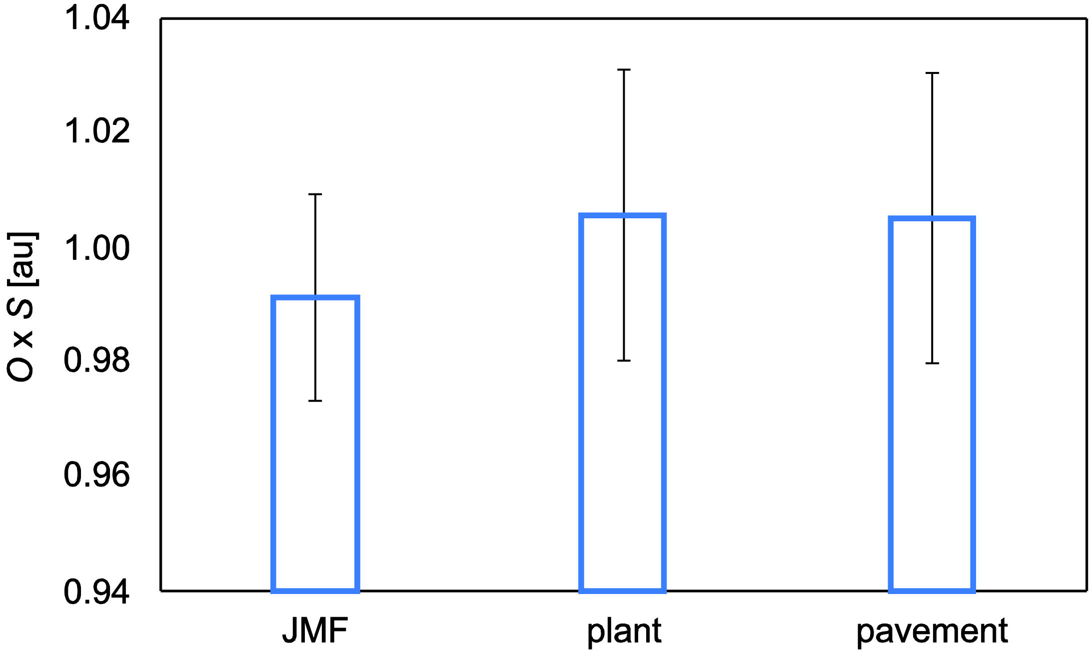

Figure 6—Example of Comparison of Oxidation in JMF, Plant, and Pavement Samples

9. PRECISION AND BIAS

9.1. The coefficient of variation (C.O.V.) in measured oxidation indices for one sample is established as a ratio of the standard deviation (std) over the mean index value for at least five samples from the sample. If the C.O.V. exceeds 6 percent, additional samples should be taken to achieve this threshold.

10. REPORT

10.1. *The report for this procedure should include the following items*:

10.1.1. General project information, such as location, and type of material tested.

10.1.2. Testing parameters, such as instrument type/ID, ambient temperature, and temperature of sample before testing.

10.1.3. A1700, A2920, and OxS values for each sample tested.

10.1.4. Mean and standard deviation for all samples of the same material (such as RAP pile, pavement surface, plant-produced, or JMF).

11. HAZARDS

11.1. This standard practice may employ toxic solvents for cleaning purposes. The user of the practice is referred to OSHA standard 29 CFR 1910 Subpart Z, Toxic and hazardous substances, for safety guidelines.

11.2. The user of the equipment is referred to OSHA standard 1926 Subpart D, Occupational health and environmental controls, for safety guidelines.

12. REFERENCED DOCUMENTS

12.1. *AASHTO Standards:*

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- T 168, Sampling Bituminous Paving Mixtures
- T 2, Standard Practice for Sampling Aggregates

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