
SHRP 2 Renewal Project R06B

Evaluating Applications of Field Spectroscopy Devices to Fingerprint Commonly Used Construction Materials (Phase IV—Implementation)

SHRP 2

TRANSPORTATION RESEARCH BOARD

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DETAILS

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Evaluating Applications of Field Spectroscopy Devices to Fingerprint Commonly Used Construction Materials (Phase IV—Implementation)

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The research described in this report was performed by the University of Connecticut (UConn) in collaboration with the Connecticut and Maine Departments of Transportation. Maria Chrysochoou, Associate Professor, UConn Department of Civil and Environmental Engineering, was the principal investigator. Iliya Yut and Chad Johnston served as lead researchers, performing laboratory and field experiments with spectroscopic instruments and contributing to this report. Master students Hanyi Yang and Jacqueline Oakes and undergraduate student Lucia Petriccione assisted with laboratory testing.

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CHAPTER 1

Background

Problem Statement

In the earlier phases (1 through 3) of Project R06B, the research team explored the potential for broad use of portable spectroscopic devices to fingerprint construction materials commonly used in transportation infrastructure. The most promising technologies were recommended for implementation by transportation agencies to incorporate them in quality assurance/quality control (QA/QC) programs. The proposed applications included fingerprinting bridge coatings and water-based traffic paints using a portable X-ray fluorescence (XRF) instrument and detecting chemical admixtures in portland cement concrete (PCC) using a portable attenuated total reflection Fourier transform infrared (ATR FT-IR) spectrometer. In addition, a procedure to detect high levels of moisture and oxidation in recycled asphalt pavements by ATR FT-IR was proposed. Further details may be found in the R06B Report.

The preliminary field tests conducted during Phase 3 of the projects indicated that the proposed procedures were viable for field application and draft AASHTO specifications were developed for field application. However, additional experience with implementation was required in order for the proposed methods to mature for adoption by state highway agencies (SHAs). Given the limited experience of most SHA materials testing labs with spectroscopic techniques, additional testing and closer collaboration of the research team with the future end users was deemed necessary for the proposed testing methods to reach the implementation stage.

Research Objectives

The scope of this pre-implementation phase of the project included the following proposed AASHTO methods:

1. Identification of Water Reducing, Accelerating, and Retarding Chemical Admixtures in Fresh Portland Cement Concrete by Attenuated Total Reflection Infrared Spectrometer.
2. Standard Method of Test for Determination of Titanium Content in Traffic Paints by Field-Portable X-Ray Fluorescence Spectroscopy.
3. Evaluation of Oxidation of Rap-Modified Asphalt Mixtures Using Attenuated Total Reflection Infrared Spectrometer.

To achieve the ultimate goal of promoting the integration of these advanced testing methods into routine QA/QC processes, the following objectives were set:

- Develop and refine agency-oriented specifications for the spectroscopy technologies based on the standards and procedures developed during Phase 3.

- Conduct pilot projects with at least two transportation agencies to demonstrate agency-oriented specifications.
- Prepare a template of an implementation plan applicable to a state highway agency (SHA).
- Organize and conduct a webinar targeting future end users of SHAs to disseminate the project results.

CHAPTER 2

Methodology

Tasks

Choosing Champion Agencies

To ensure success of the pre-implementation phase of this project, it was imperative to identify transportation agencies that would be interested in trial testing and future implementation of the three proposed spectroscopic methods. For that purpose, the research team used the results of an electronic survey conducted in a previous phase to identify DOTs (Departments of Transportation) that could champion the implementation activities. Another criterion was the proximity of the DOTs to the University of Connecticut (UConn), in order to facilitate the field-testing trips within the available budget and time constraints. Thus, the outreach effort was mostly focused on DOTs of the Northeast. The materials engineers of those agencies were contacted and supplied with copies of the proposed AASHTO standards.

Concurrently, introductory meetings with demonstration sessions were scheduled. To familiarize the participating agencies with the scope and work plan of the project, the kick-off meetings were arranged at the headquarters of the participating agencies. The discussions included presentation of the project background, introduction into methodology, and in-room demonstration of the proposed testing procedures. In addition, the preliminary scope of materials and schedules for laboratory and field tests was established. Follow-ups with the agencies indicated which ones showed an interest in the methods and commitment to collaborate and supply the necessary personnel for the needs of the pilot tests.

Refinement of AASHTO Specifications

The applicability and attractiveness of the proposed AASHTO testing procedures to the local settings (e.g., materials, level of personnel training, existing QA/QC procedures) were evaluated by seeking an initial input from potential users. Specifically, the research team looked for comments on language and special test parameters (e.g., set-up, need for calibration, sample preparation). Based on that feedback, the procedures were refined and used for demonstration and pilot testing.

Demonstration and Pilot Testing

The next step involved collection of material samples from participants and fingerprinting those samples with the use of portable devices in the laboratory settings. Once signature spectra of the materials were determined, field trips were arranged to verify applicability of the methods in real-world project environments and the ability of the devices to verify the composition of materials delivered on-site.

Development of Agency-Oriented Specifications

Based on the feedback from agencies and the results of pilot testing, the proposed AASHTO standards were customized into state-oriented procedures to reflect the specific range of materials/brands used by the participating states. Those specifications included operations manuals for the equipment and guidelines for interpretation of test results.

Development of a Generic Implementation Plan for the Proposed AASHTO Standards

A successful incorporation of the innovative testing methods into an agency's QC/QA program is not feasible without having a road map for the implementation. The implementation plan includes a step-by-step outline of the activities and processes that should be undertaken to facilitate enhancement of the routinely performed QC test by adapting any of the spectroscopic testing methods proposed here.

Project Deliverables

Agency-Specific Testing Procedures

The proposed earlier draft AASHTO standards had to be modified to address the local specifics of the participating transportation agencies. The modified and agency-oriented testing procedures included, for example, benchmark spectral characteristics for the range of materials whose samples were provided by the agencies (See Appendix A).

Spectral Libraries for Participating Agencies

Libraries of spectra for the agency-approved materials were created for each of the participating state transportation agencies. These libraries comprised signature ATR FT-IR spectra of chemical admixtures for portland cement concrete (PCC) and typical XRF results for traffic paints and bridge coatings. The spectral database is expected to supplement the procedures developed under this project and can be potentially available online for QC/QA specialists.

Generic Implementation Plan

A generic implementation plan outlines the activities and processes that should be undertaken to incorporate the proposed spectroscopic testing procedures into the QC/QA process of highway/bridge construction (See Appendix D). As a rule, the implementation activities should include (1) identification of personnel who would champion the process, (2) purchase and calibration (if needed) of the equipment, and (3) training of the personnel. Additional steps tailored to the status and singularities of each method are included for each method separately.

Webinar

Based on the project results and deliverables of the pre-implementation phase of this project, the research team has prepared materials for a SHRP 2 webinar that outlines the proposed methods and provides guidance on implementation. The materials are derived from the previous presentations at the Transportation Research Board annual meetings and publications in the American Society of Civil Engineers (ASCE) and Transportation Research Board journals. The research team is ready to conduct a national webinar and/or TRB workshop to present and discuss the deliverables of this project.

CHAPTER 3
Findings and Applications

Identification of Participating Agencies

The pre-implementation phase of this project started with identifying a group of participating DOTs that would be willing to provide their input on the following three draft AASHTO standard practices that were proposed in the earlier phase of the project (Zofka et al., 2013):

1. Identification of Water Reducing, Accelerating, and Retarding Chemical Admixtures in Fresh Portland Cement Concrete by Attenuated Total Reflection Infrared Spectrometer
2. Determination of Titanium Content in Bridge and Traffic Paints by Field-Portable X-Ray Fluorescence Spectroscopy
3. Evaluation of Oxidation Level of Asphalt Mixtures by Attenuated Total/Diffused Reflection Infrared Spectrometer

The research team contacted materials specialists from Connecticut, Maine, New Hampshire, New Jersey, Pennsylvania, and Rhode Island Departments of Transportation (DOTs). Each contact was supplied with the objectives of the project and electronic copies of draft AASHTO standards, and asked to schedule a kick-off meeting. Pennsylvania DOT personnel indicated that they were basically interested in the XRF application, but they did not have the time for full-blown participation in the project. The demonstration sessions for all three methods were held in the headquarters of Connecticut, New Hampshire, Maine, and Rhode Island DOTs. The kick-off meetings revealed different degrees of interest from the potential participants, as shown in Table 3.1. Eventually, Connecticut and Maine stayed on board to provide feedback on the draft AASHTO standards and participate in lab and field testing for all three methods.

In general, the method that attracted the most interest from the various agencies was XRF. Portable XRF analyzers may be used for a variety of applications besides paints, including detection of lead (Pb) and arsenic (As) in glass beads, lead in paints and soils, chromium (Cr) in PCC, and other trace metals in a variety of construction materials; thus the acquisition of the equipment can serve multiple purposes simultaneously. In addition, the current QA/QC methods on paint testing do not involve any chemical specifications that can be used to verify that the paint quality is consistent. Thus, interest in this method was expressed by all contacted agencies that responded to the contact attempts.

The agency representatives’ reactions to the PCC admixtures specification were, conversely, consistently reluctant. The agency representatives either rejected outright the need for this test or merely consented to try out the method in the field. Specifically, the materials testing professionals felt that the performance-based QA/QC methods for accelerators or retarders were adequate and that early detection of the presence of the compound in the wet admixture would not serve any additional purpose. Because of this limited interest, the team later had difficulty identifying suitable projects on which to conduct the field testing.

Finally, the recycled asphalt pavement (RAP) analysis method was received with markedly different reactions depending on the agency. Both agencies that ended up participating were enthusiastic that such a method could become available and expressed an urgent need to have a means of evaluating the RAP content and the degree of oxidation in asphalt. Conversely, other DOTs had no interest in trying the method and found no usefulness in the application. The reactions to the method reflected to some degree the extent of RAP application in the particular state and the views of the asphalt-related personnel on the nature and properties of RAP.

On the basis of this feedback, it is considered that the XRF method is likely to be more attractive to state DOTs, contractors, and manufacturers as a quality control tool. The ATR method for PCC does not seem to hold much promise except in states where portland cement concrete is more widely used, which was not the case with the participating DOTs. The RAP method requires additional development and education of professionals to become a viable method. However, the research team concludes that there is substantial need for the method in areas with intensive RAP use. It should be noted that these conclusions hold true for the more common paradigm of design-bid-build, in which the DOTs are responsible to ensure that their design specs were made. However, the team considers that XRF may also be an attractive method for other project management paradigms, in which it used more as an internal standard QA/QC tool, or to assess the current status of materials status, e.g., in bridges. Additionally, changes in design specifications for the service life of bridges, asphalt surfaces, and other materials, may render the method more or less attractive.

Table 3.1. Summary of Agency Participation in Pre-implementation Phase

Agency	**Proposed AASHTO Standard**		
	ATR of Admixtures for PCC	**XRF of Paints**	**ATR of RAP and Oxidized Asphalts**
Connecticut DOT	Actively participated in lab and field testing	Actively participated in lab and field testing	Actively participated in lab and field testing
Maine DOT	Actively participated in lab and field testing	Actively participated in lab and field testing	Actively participated in lab and field testing
Rhode Island DOT	Not interested	Participated in demonstration only	Participated in demonstration only
New Hampshire DOT	Not interested	Limited interest	Not interested
New Jersey DOT	Not discussed	Unresponsive to contact	Not discussed
Pennsylvania DOT	Not discussed	Indicated interest but limited availability	Not discussed

Revision of Proposed AASHTO Standards

The next three subsections provide brief descriptions of the tests, the nature of agencies' comments, and implemented revisions for each draft AASHTO standard separately. A full description of the standards can be found in Appendix A, whereas the background on methods of spectroscopic evaluation of materials can be found elsewhere (Zofka et al. 2013). It was found that the comments obtained by the materials specialists were of a general nature and not geared toward the particular needs of the local DOT. Thus, it is considered that there is no need for agency-oriented specifications; instead a single improved AASHTO draft was produced by incorporating all comments received.

ATR FT-IR of Chemical Admixtures for Concrete

The identification of the type of a chemical admixture for portland cement concrete (PCC) using a compact ATR FT-IR spectrometer is based on the qualitative comparison of the infrared absorbance spectrum of a pure admixture sample with the spectrum of the PCC chemical mixture. A pure admixture sample is obtained from the storage or feeding tank in a concrete plant. A pure admixture sample is scanned by ATR FT-IR to obtain its absorbance spectrum. The absorbance spectrum presents peaks at specific frequencies that are considered characteristic of the particular chemical. A fresh PCC sample is scanned by ATR to obtain the spectrum of the mixture. If the characteristic absorption bands of the chemical are observed, then the presence of the chemical can be established.

The comments on this proposed method provided by the materials specialists were primarily of an editorial nature. One valuable suggestion was to include a sample spectrum for each type of admixture considered in the method. The draft document was edited accordingly (See Appendix A).

XRF of Bridge and Traffic Paints

The draft AASHTO XRF standard was reviewed by materials engineers of the Maine and Connecticut DOTs. Comments generally fell under one of three categories: the need for additional safety instructions and documentation regarding the hazards and prevention of exposure to ionizing radiation; not enough specific detail in the field and laboratory testing procedures; and more specificity in quality control and calibration sections. The first issue was addressed by including two additional sections of references to radiation safety literature and relevant regulatory standards. Additional detail was included in the procedure to ensure that specific steps are taken by the XRF operator to mitigate the potential for exposure to either the operator or nearby persons, and to ensure compliance with state and federal occupational safety legislation.

Substantial detail was added to the procedure to make the steps easier to follow for a field operator. This included additional steps for selecting suitable testing locations, preparation of samples for lab testing, and the optimal technique for data collection, which is utilized by most portable XRF analyzer manufacturers. In addition, a list of potential vendors and manufacturers of XRF analyzers and related materials was included as an appendix. The quality control sections were updated with specific instructions for instrument calibration and periodic standardization. Instructions for how to recognize as well as mitigate commonly encountered interference effects were updated in the procedure. The full text of the revised standard can be found in Appendix A.

ATR of Recycled and Oxidized Asphalts

No comments from either Connecticut or Maine DOTs were received on this method. This is attributed to the limited familiarity of materials testing engineers with spectroscopy and also with RAP testing, which is not a standard practice, given the lack of relevant standards. In general, while there is considerable interest in development of testing methods and improved understanding of RAP properties, the method requires additional maturing to reach the stage of widespread application as a QA/QC procedure.

It should be noted that Connecticut DOT does not own and, therefore, does not control RAP stockpiles, nor does it specify aging properties of RAP materials. Nevertheless, the asphalt materials specialists expressed their interest in the results of this project. Therefore, the research team proceeded with including samples from RAP stockpiles owned by the major CTDOT contractor, Tilcon Connecticut, Inc., in the testing program. Maine DOT engineers, on the other hand, volunteered large amounts of material from 15 different locations and fully participated in the pilot testing for this method.

Determination of Signature Spectra of State-Approved Materials

Field testing of state-approved materials required the development of appropriate standards, against which the field samples could be compared. Thus, samples of several material types were obtained from both states and tested in the lab with the appropriate procedures described in Appendix B.

ATR of Chemical Admixture to PCC

Several samples of PCC chemical admixtures were obtained from CTDOT and Maine DOT. CTDOT shipped samples directly to Connecticut Advanced Pavement Laboratory (CAP Lab) at UConn, where the laboratory testing occurred. In Maine, the admixture samples were first transferred from different locations/plants to the Freeport, Maine, materials laboratory. The research team picked up the samples from Freeport and took them to CAP Lab for testing. Table 3.2 provides details on the manufacturer, type, and sample size of admixtures tested. More information on material composition and properties can be found in the corresponding material safety data sheets and product descriptions available online.

To perform an ATR test according to the proposed AASHTO standard, about 1 ml of each admixture was probed from the container and placed on top of the ATR diamond plate. Each sample was scanned for about 1 min and the average of 24 absorbance spectra was recorded. Due to the liquid nature of all admixtures, a maximum testing of 2 or 3 subsamples was sufficient to confirm very high repeatability of the method, as shown in Figure 3.1. This way a signature spectrum was determined for each sample listed in Table 3.2, shown in Appendix C.

Table 3.2. Summary of Admixture Samples Tested in CAP Lab

Participating Agency	Admix-ture Name	Admixture Type	Admixture Manufacturer	Source/Plant/ Location	Sample Size
Connecticut DOT	Zyla 630	Water Reducer Type ASTM C494 Type A and D	Grace Concrete Products	Rocky Hill Lab	64 oz.
	Zyla R	Water Reducer and Retarder ASTM C494 Type B and D	Grace Concrete Products	Rocky Hill Lab	64 oz.
Maine DOT	ADVA 140	High Range Water Reducer ASTM C494 Type A and F	Grace Concrete Products	Sargent Materials, Hermon, Maine	32 oz.
	Darex II AEA	Air Entrainer ASTM C260	Grace Concrete Products	Sargent Materials, Hermon, Maine	32 oz.
	Delvo stabilizer	Water Reducer and Retarder ASTM C494 Type B and D	BASF	Dragon Products Co., Portland, Maine	32 oz.
	Glenium 7500	High Range Water Reducer ASTM C494 Type A and F	BASF	Dragon Products Co., Portland, Maine	32 oz.
	Micro Air	Air Entrainer ASTM C260	BASF	Dragon Products Co., Portland, Maine	32 oz.
	Polar Set	Accelerator ASTM C494 Type C	W.R. Grace & Co.	Sargent Materials, Hermon, Maine	32 oz.
	Pozzolith 100XR	Water Reducer and Retarder ASTM C494 Type B and D	BASF	Dragon Products Co., Portland, Maine	32 oz.
	Recover	Retarder ASTM C494 Type D	Grace Concrete Products	Sargent Materials, Hermon, Maine	32 oz.
	Retarder	Unknown	Unknown	Sargent Materials, Hermon, Maine	32 oz.
	Rheocrete CNI	Accelerator/Corrosion inhibitor ASTM C494 Type C	BASF	Dragon Products Co., Portland, Maine	32 oz.

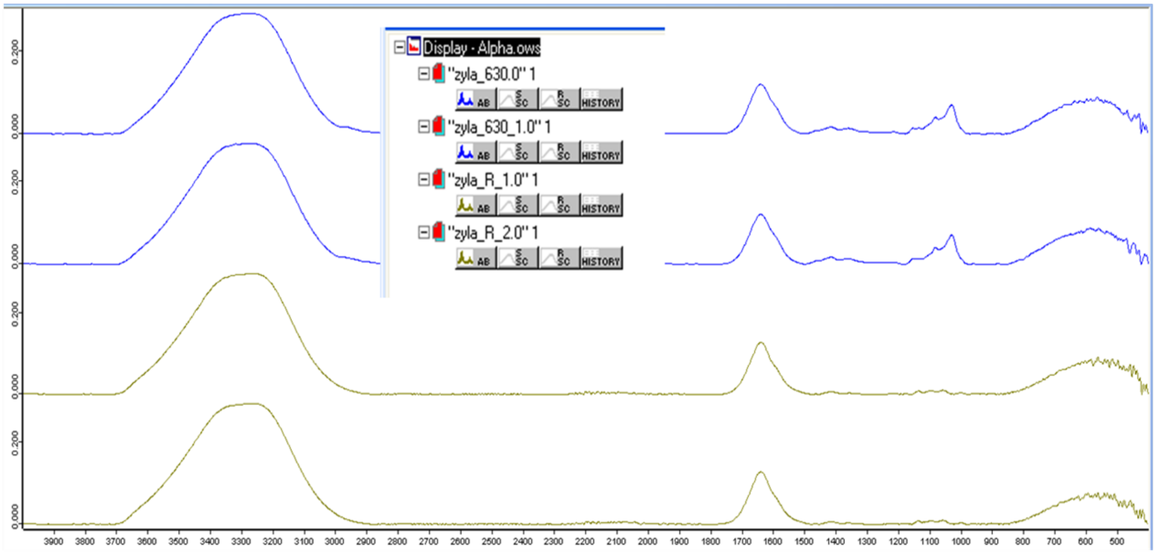

Figure 3.1. ATR absorbance spectra of the CTDOT chemical admixtures.

XRF of Traffic Paints

Initially, Connecticut and Maine DOT provided samples for laboratory testing. Elemental analysis results were obtained for several white and yellow traffic paints, as well as green bridge paints, to establish the chemical composition of the materials used in the field (Table 3.3). The concentrations would serve as a baseline to determine concentrations in the field.

Table 3.3. Titanium Concentrations of Liquid Paint Samples as Determined in the Laboratory by XRF

Paint Name/Type	Color	Average Ti Content (%)	Standard Deviation (%)
ME DOT Corothane	Green	8.82	0.13
ME Spec Waterborne TP	White	6.50	0.11
ME Spec Waterborne TP	Yellow	1.62	0.08
Epoplex LS-50 CTDOT	White	28.78	0.03
Epoplex LS-50 CTDOT	Yellow	5.36	0.08
DM3062AM CTDOT	Yellow	2.05	0.12
Ennis Latex CTY-21-M-2	Yellow	3.65	0.02
Ennis Latex CTW-21-M-1	White	6.77	0.28

The effect of dilution on the XRF signal of latex paints was determined by testing samples corresponding to different paint-to-water ratios (Figure 3.2). Each paint type shows a decay-type curve between approximately 1% to 5% titanium (Ti). A linear relationship is seen between the diluted Ti content and the measure Ti content below 1% Ti. Thus, water-diluted paints may be detected through the development of such calibration curves.

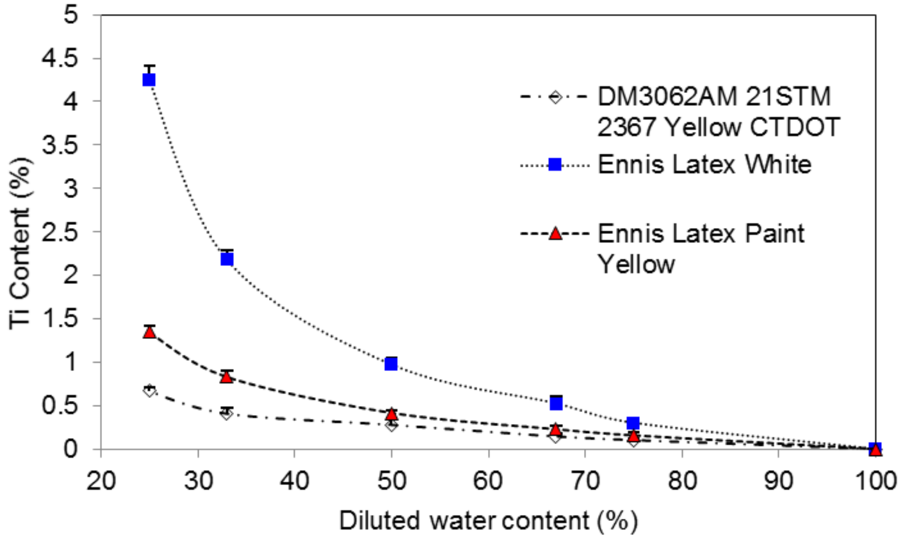

Figure 3.2. Effect of dilution on the Ti XRF signal of three latex-based traffic paints.

ATR of Recycled Asphalt Materials

Sample Preparation and Testing

In Connecticut, RAP samples were collected from three asphalt plants owned by Tilcon Connecticut, Inc., located in Manchester, New Britain, and Groton. Those locations were chosen to represent different climatic subzones, specifically inland hills and shore plains. It was hypothesized that difference in precipitation and location may affect progress of oxidation and moisture content in RAP during the storage. Other factors considered in this study were RAP source (state versus private projects), RAP gradation (milled versus processed to passing #4 sieve), duration of storage (e.g., 2 years versus 6 years), and location within a stockpile (top/ crust versus inside). Table 3.4 summarizes information on Connecticut DOT RAP samples.

In Maine, samples from 12 locations spread over a 250-mi stretch of Interstate 95 between Portsmouth and Houlton were gathered in the Freeport Materials Laboratory and transferred to CAP Lab for testing. The samples differed in gradation (from less than 8% to more than 10% aggregate passing #200 sieve) and variation in asphalt content (from less than 0.3 to greater than 0.5%). Table 3.5 provides a summary of the Maine DOT RAP samples.

All RAP samples were stored in sealed plastic bags or tin containers to preserve the original moisture content. Prior to ATR testing, each sample was sieved through #8, #30, and #50 sieves, and 5 subsamples of the fraction between the #30 and #50 sieves (diameter between

0.59 mm and 0.297 mm) was tested from each sieved sample. This allowed for maximizing the uniformity of particle size and minimizing the variability of sample composition.

Table 3.4. Summary of Connecticut DOT RAP Sample Information

RAP Stockpile Location	RAP Source	Duration of Storage	Milled or Processed	Top or Inside
Groton	State	1–2 years	Processed	Inside
Groton	Private	N/A	Processed	Inside
Groton	State	2 years	Milled	Top
Manchester	Private	N/A	Processed	Top
Manchester	Private	N/A	Processed	Inside
Manchester	State	N/A	Processed	Top
Manchester	State	N/A	Processed	Inside
New Britain	State	1–2 years	Milled	Inside
New Britain	State	1–2 years	Milled	Top
New Britain	State	5–6 years	Milled	Inside
New Britain	State	0–1 year	Processed	Top
New Britain	State	0–1 year	Processed	Inside

Table 3.5 Summary of Maine DOT RAP Sample Information

RAP Stockpile ID	Nearby Location	Latitude, °N	Longitude, °W	RAP Classification	Passing #200	Standard Deviation in Asphalt Content
LAN-HE13-CR-Q	Hampden, Maine	44.785	–68.8319	Pure aggregate	N/A	N/A
LAN-SM13-II	Smyrna, Maine	46.1301	–68.1579	Class II	≤10%	≤0.5
PII-WE13-I	Wells, Maine	Wells, Maine	Wells, Maine	Class I	≤8%	≤0.3
PII-HE13-I	Hampden, Maine	44.7783	–68.8395	Class I	≤8%	≤0.3
PII-FF13-II	Fairfield, Maine	44.5908	–69.6134	Class II	≤10%	≤0.5
LAN-WB13-II	Wetsbrook, Maine	43.7321	–70.3527	Class II	≤10%	≤0.5
PII-PNH13-I	Portsmouth, N.H.	43.0466	–70.7794	Class I	≤8%	≤0.3
CMP-SC13-III	Dayton, Maine	43.5294	–70.5931	Class III	>10%	N/A
PII-WB13-I	Wetsbrook, Maine	43.6742	–70.3320	Class I	≤8%	≤0.3
LAN-PI13-II	Presque Isle, Maine	46.7359	–67.9614	Class II	≤10%	≤0.5
PII-PO3-I	Poland, Maine	44.0165	–70.3491	Class I	≤8%	≤0.3
LAN-WA13-III	Washington, Maine	44.2226	–69.3853	Class III	>10%	N/A

To obtain an infrared (IR) absorbance spectrum, each sample was scanned 24 times on the ATR diamond sampling plate and the average spectrum was recorded. The spectrum was then processed in accordance with the proposed draft AASHTO method and the analysis of oxidation level in terms of carbonyl content and comparison of moisture content was performed. Note that the proposed AASHTO method of measuring oxidation in asphalt mix samples is qualitative by nature and therefore only comparative analysis is provided in this report. Development of absolute standards would require a much larger effort of collecting RAP nationwide, evaluating its properties and developing a database that could provide a basis for comparison.

Analysis of Oxidation and Moisture in RAP Samples

In Connecticut, the results indicated that it was possible to compare both oxidation and moisture in samples from stockpiles with different storage duration. For example, Figure 3.3 compares the carbonyl and aromatic content in different RAP samples, as determined by the absorbance peaks around 1700 cm^{-1} and 1600 cm^{-1}, respectively. An increase is evident in signals from the carbonyl and aromatic groups with storage time. The signal from the water presence at around 3350 cm^{-1} indicates higher moisture content for the 5-year-old stockpile. It is assumed that the seasonal variation of moisture occurs simultaneously in both piles because of their proximity (within one plant).

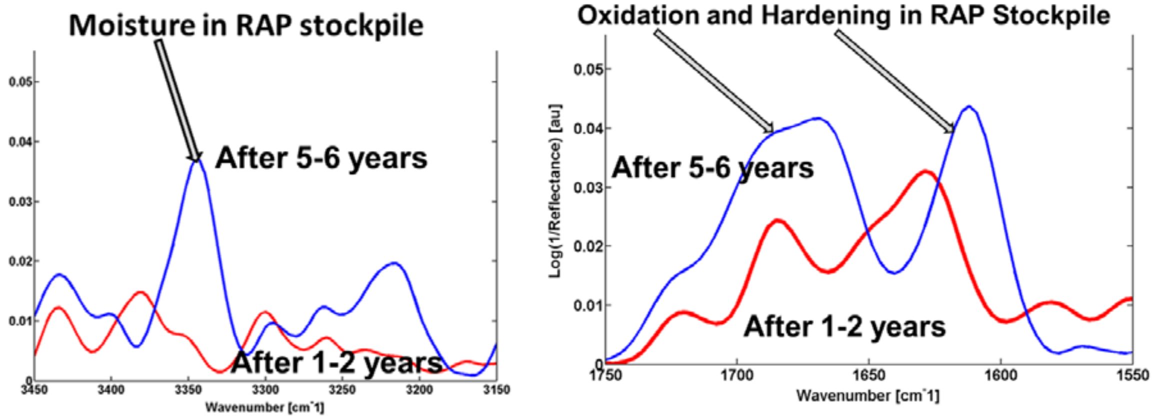

Figure 3.3. Comparison of moisture (left) and oxidation and hardening (right) in RAP stockpiles after 1 to 6 years of storage.

The main challenge in testing RAP or any asphalt mix sample is a relatively high (up to 25% for five subsamples tested in this study) standard deviation from the mean. Ultimately, after gaining experience with the testing method, the analyst may conduct 10 analyses and choose the five most representative of the bulk material to reduce variability to as low as 10% to 15%. Considering the very short duration of a test (less than 1 min), reliable results for a sample prepared as a single batch can be obtained within 10 to 15 min, which complies with the objective of this research. The limiting factor is that an experienced technician is required to judge the quality and representativeness of the spectra.

In Maine, the following two factors of RAP oxidation were investigated: (1) effect of location and (2) effect of RAP class/gradation/asphalt content. A limitation to studying the influence of other factors was that the age of RAP and duration of its storage in a stockpile were unknown. Interestingly, it was found that on average, oxidation in RAP samples was decreasing with latitude, as shown in Figure 3.4. One possible explanation of this trend is the use of binder heavily modified with styrene–butadiene–styrene (SBS) in northern latitudes, such as PG 76-28, which is significantly stiffer than, for example, PG 58-28 and PG 64-22. The stiffer binders are known to oxidize at a slower rate. As far as RAP classification is concerned, the average oxidation was higher in Class I, followed by Classes III and II, in that order (Figure 3.5). A statistical significance of this phenomenon should be further investigated on a larger set of samples. In summary, however, the ATR method revealed considerable variability in oxidation levels of RAP from different projects, which further indicates the need of controlling this parameter, especially when producing mixes with high RAP content.

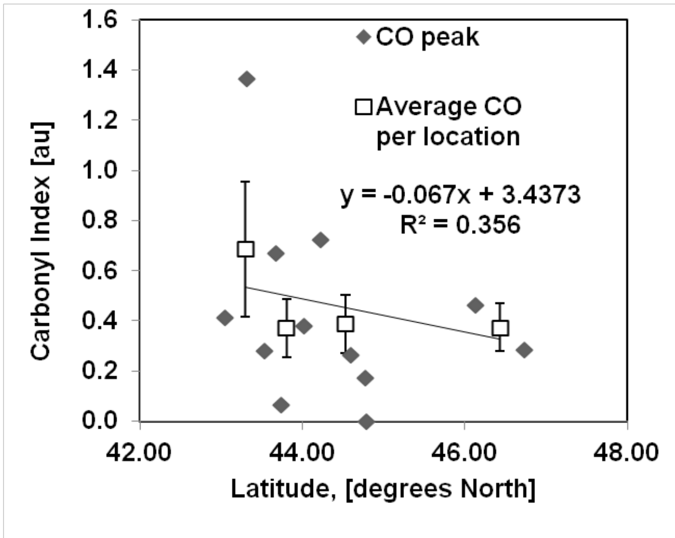

Figure 3.4. Effect of location on RAP oxidation in Maine.

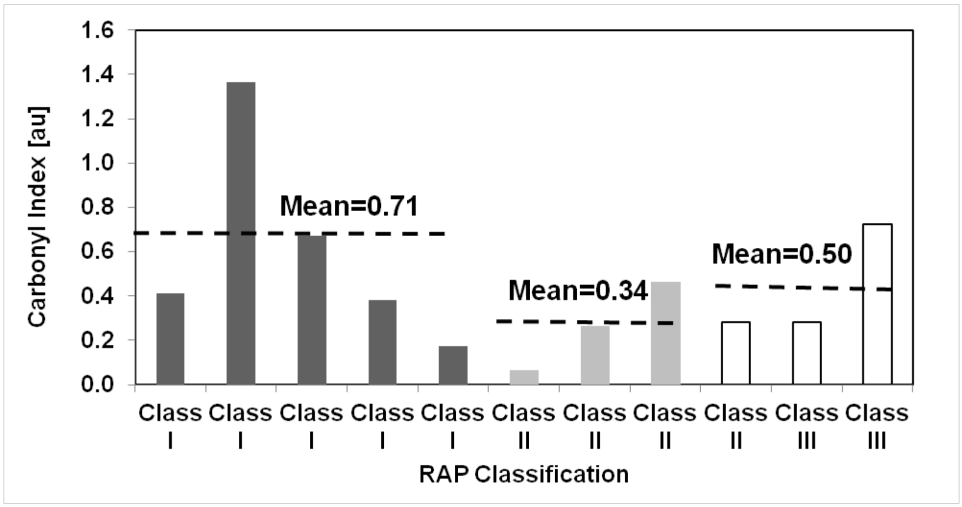

Figure 3.5. Summary of oxidation in different RAP classes.

Pilot Field-Test Results

ATR of Admixtures

No field tests were performed for this method because no suitable project could be identified. This is related to the limited need and interest of the DOTs for the method, given that these admixtures could only be tested for QA/QC in the designated tank at the manufacturer or the concrete ready-mix plant. In general, a stable chemical composition of admixtures and their liquid nature renders them suitable for QA/QC analysis; however, the point of application is the manufacturing location and/or material source, rather than the construction site.

XRF of Bridge and Traffic Paints in Maine

In situ XRF testing was performed in collaboration with the Maine Department of Transportation. The first field test was conducted on a bridge overpassing I-95 one mi north of Exit 174. The site is located on Hinkley Hill Road in Herman, Maine, approximately 8 mi west-southwest of central Bangor. X-ray data were collected by directly applying the XRF analyzer on the green-painted girders on the underside of the bridge. Points of measurement were accessed by means of a boom lift stationed on the right southbound lane of I-95. Pictures of the field-testing processes are shown in Appendix C.

The most abundant detectable elements were Ti, iron (Fe,) and zinc (Zn) (Table 3.6). Smaller concentrations of zirconium (Zr) and Pb were detected at fractional mass-percentages. Comparison to the lab-based results revealed little variation in the Ti content between the sampling techniques. The block samples and liquid samples had slightly lower and higher Ti concentrations, respectively, compared to the bride girder. This similarity suggests that the Ti content of painted girders measured in situ is generally representative of the lab-based characterization. In contrast, substantial variation is seen among the elemental concentrations of Fe, Zn, and Pb. This is suggestive of interference from elements present in the substrate materials which are absent from the pure paint.

Table 3.6. Elemental Composition of Bridge Paint as Detected by XRF for Liquid Samples, Coated Metal Wedge Samples, and the Painted Bridge Girder

Element	**Laboratory Ti Content (%)**				**Field Ti (%)**	
	Liquid Paint	**std**	**Painted Wedges**	**std**	**Bridge**	**std**
Ti	8.82	0.13	2.67	2.67	9.21	0.29
Fe	0.02	0.01	13.30	13.30	17.83	3.68
Zn	0.01	0.01	ND	–	34.20	4.85
Pb	ND	–	ND	–	0.02	0.01

The additional elements detected in the field may be readily explained by the chemical composition of the underlying steel and residual coatings. Lead-based paints were used extensively for corrosion inhibition on steel bridges prior to regulatory changes in the 1970s. Thus, the Pb detected by the XRF likely resides in residual paint layers from the previously applied coatings. Measurements performed on an unpainted section of a girder showed an average of 0.1% Pb, indicating that approximately 20% of the Pb remained after the coating. At about 17% and 37%, Fe and Zn were the most abundant elements detected, respectively. Lab-based tests on liquid paint samples showed that the paint did not contain appreciable amounts of Fe (<0.04%) or Zn (<0.01%). This indicates that the X-rays penetrated depths beyond the paint coating into the girder substrate material, which is most certainly galvanized steel. The galvanization process produces a thick protective layer of Zn on steel, an alloy of primarily Fe and carbon. Thus, the detection of Fe further indicates that the XRF probed the bulk material at depths beyond the exterior paint and Zn coatings.

Collectively, these results show that XRF is effective for field testing the Ti content of bridge paint, in that in situ measurements closely matched lab measurements. The wide variation exhibited by other elements is suggestive of potential for interference effects depending on the substrate. It is critical, therefore, to test an uncoated sample to verify that the measured concentrations are quantitatively representative of the true paint concentration, since it would be difficult to separate the fluorescence contributions from multiple sources. If the paint is titanium-based, which is the case for most brands, then, Ti is a suitable element for QA/QC testing of the paint itself, given that there are no substantial Ti contents in most materials where paint is applied.

Field measurements were also performed on strips of traffic paint dried on Jersey barriers situated behind the Bangor DOT office. The results are quite close to the characteristic concentrations obtained for liquid samples in the laboratory (Figure 3.7). The concentrations were slightly higher in the field, which is likely due to drying.

Table 3.7. Titanium Content in Painted Lines at DOT, Bangor, Maine

	Ti Content (%)	
	White	**Yellow**
Sample 1	7.36	1.86
Sample 2	7.47	1.93
Sample 3	7.89	1.82
Average	7.57	1.87
Std	0.28	0.06

XRF Testing of Traffic Paints in Connecticut

Field XRF measurements were also performed on painted white and yellow lines in conjunction with the ConnecticutDOT at four locations in southeastern Connecticut: the Exit 9 off-ramp on Route 9 in Higganum, Conn., Route 154 in Old Saybrook, Conn., and Exits 64 and 66 on Interstate 95 in Westbrook, Conn. (See Figure 3.8.) The only major difference in locations was that the Route 154 site had been freshly painted one or two days prior to measurement.

The Ti contents of the white paints tested at both I-95 locations are nearly identical, which indicates a similar paint mix, paint type, or application technique. The Ti content in the yellow paint was slightly lower at Exit 64. The Route 9 site had the highest Ti content at 22%, which is comparable to the high-Ti Epoplex paint characterized in the lab. This site also produced a higher standard deviation, which is either a consequence of the higher Ti content or an inconsistency in the paint thickness. Freshly applied paint tested at the Old Saybrook site had the lowest Ti concentration (9%).

Unfortunately, the XRF device could not be used by any DOT workers in either Maine or Connecticut because of the OSHA guidelines regarding the use of X-ray devices. Nevertheless, the measurement process and draft standard were demonstrated and workers were present for all field tests. The response and feedback were positive in both states. Most comments were centered around the simplicity and ease of use of the device, as well as the significant potential for quality control applications. In Connecticut, interest was expressed in linking XRF data to thickness measurements and other techniques for the purposes of enhancing quality control and optimizing the paint application procedure.

Table 3.8. Titanium Concentrations in Pavement Markings as Determined by XRF

Location in Connecticut	**Ti Content (%)**	
	White	**Yellow**
Exit 66, I-95, Westbrook	12.71	3.44
	12.89	4.25
	11.25	3.99
	12.14	3.51
Average	12.25	3.80
Std	0.74	0.39
Exit 64, I-95, Westbrook	11.75	2.08
	12.17	2.90
	11.43	2.32
Average	11.78	2.43
Std	0.37	0.42
Route 154, Old Saybrook	7.80	
	10.79	
	9.04	
Average	9.21	
Std	1.50	
Exit 9, Route 9, Higganum	22.19	2.83
	26.11	2.75
	19.93	2.26
	18.57	3.22
Average	21.70	2.76
Std	3.30	0.40

ATR of RAP and Oxidized Asphalt

Pilot field testing of aged pavement surfaces was performed in Maine. Three pavement locations with a similar pavement structure and mix properties yet of different age (0.5, 5, and 11 years) were selected by Maine DOT from the thin overlay preservation projects. The powdered samples of asphalt were collected by drilling and tested on-site using the portable ATR spectrometer installed on the back of a van. One sample from each pavement section was collected and three probes from each sample bag were tested. Note that the whole operation occurred at an ambient temperature of 35°F, which is much lower than the minimum of 64°F prescribed by the manufacturer. Nevertheless, the instrument did not exhibit any operation problems, nor was the data processing by a laptop computer affected by the low temperature.

The results of testing are presented in terms of the carbonyl index, which is determined as the ratio of the absorbance peak at 1700 cm^{-1} over the peak value at 2920 cm^{-1}. Figure 3.6 plots the resulting carbonyl index as a function of pavement age, showing a fairly significant (R^2 =

0.46) linear trend of increase in oxidation with pavement age. This result is aligned with results on pavement aging reported for Connecticut and Rhode Island pavements in the previous phases of the project. For example, in Connecticut, pavement sections with binder grade of PG 64-XX exhibited intercept of 0.003 and slope of 0.003.

Figure 3.6. Trend of the carbonyl index determined by ATR analysis with the pavement age in Maine DOT field tests.

During the field testing of pavement surfaces, a material engineer from Maine DOT was asked to collect a sample and perform an ATR test. It was confirmed that no special training is required for sample collection, whereas a basic training on the instrument software interface was needed to familiarize an operator with basic spectrum manipulation options.

CHAPTER 4

Conclusions and Recommendations

This chapter summarizes major findings and deliverables of the pre-implementation phase of the project and presents generic implementation plan for incorporating selected AASHTO procedures into state agencies' QA/QC programs.

Summary

This project constitutes the pre-implementation phase that follows the three experimental and pilot testing phases of project R06B. Specifically, three spectroscopic methods for specific materials classes were identified as promising for field implementation during the earlier phases of the project. These included (1) Fingerprinting of chemical admixtures (retarders, accelerators and water reducers in PCC using ATR FT-IR); (2) QA/QC of traffic paints using the Ti content as determined by portable XRF analysis as the quality parameter; and, (3) Evaluation of moisture and oxidation levels in RAP mixes using ATR FT-IR.

The objectives of Phase 4 of the project were to work with two SHAs to further refine the proposed AASHTO methods developed in Phases 1–3, to conduct field tests that would further verify the viability of the methods in real field conditions, and to produce a generic implementation plan for the adoption of the methods by SHAs nationwide.

The initial approach of several DOTs to identify the participating agencies showed that there were varied responses to the three proposed methods. In general, the ATR analysis of PCC admixtures was not met with interest by any DOT, including the ones that eventually participated in the field-testing phase. Existing QA/QC procedures are considered adequate and the proposed method was deemed more suitable for adoption by the chemical manufacturers or the concrete mixing plants, not state DOTs. Conversely, the response to the XRF method for paints was encouraging by practically all responding DOTs. This is because of the applicability of the method for a wide variety of materials, the apparent need for more composition-based QA/QC criteria for paints, and the user-friendly nature of portable XRF analysis. Finally, the ATR RAP testing protocol was either hailed as an essential development in the field or as completely redundant. Ultimately, Maine and Connecticut DOTs agreed to fully participate in the implementation phase of the project.

The collaboration with the two DOTs proceeded in three steps. First, the draft AASHTO methods were sent to the material engineers to obtain feedback. The received comments were incorporated in the methods, and it was observed that there was no need to produce state-specific testing protocols, since the received comments were generic in nature. Thus, a single updated version of each of the draft AASHTO methods was produced.

Then, samples of state-approved materials from all three categories (PCC admixtures, paints, and RAP) were obtained in order to test them in the lab and develop appropriate standards to compare against in the field. For the PCC admixtures, similar results were observed compared to Phase 3, in that highly reproducible ATR spectra could be obtained for the pure compounds. For XRF paints, initial testing showed that the Ti content was specific to the paint type and thus a suitable standard has to be developed in the lab for comparison with field testing. Additionally, detection of diluted paint by water was deemed to be highly successful with this method. Testing of additional samples of glass beads, screws, and other metallic materials supplied by Connecticut DOT showed that XRF has wide applications that meet DOT needs. Finally, lab testing of RAP was performed for a wide range of RAP samples collected from three locations in Connecticut and several locations in Maine. The results from the Connecticut samples indicated that the method yielded higher water contents and increased aging in RAP collected from older stockpiles. The results from the Maine samples, for which no aging information was available, indicated that samples from higher latitudes had a lower degree of oxidation. Overall, the method was deemed successful in producing comparative results of oxidation and moisture in RAP samples.

In the next stage, field tests were conducted for XRF of paints in Maine and Connecticut and for ATR for RAP in Maine. Field tests for ATR of PCC admixtures could not be arranged because QA/QC procedures could only take place in private manufacturer or concrete mixing plants, and this was outside the scope of the project. For RAP in Connecticut, the state DOT is not responsible for RAP management; thus, sampling of the stockpiles managed by the company Tilcon were sampled and tested in the lab only. The field results of the XRF analysis yielded data consistent with the lab observations, and positive feedback was obtained from the participating DOT personnel in both states in terms of the ease of handling and interpretation. An important observation was that the measured concentrations in the field were somewhat lower compared with the lab results, which is attributed to the limited thickness of the paint after it is applied and is potentially due to matrix effects. Thus, development of lab-based standards should be done under similar conditions to the expected field conditions. For RAP testing, the operation of the device was found to be straightforward according to the draft AASHTO method, while data interpretation requires some additional training that has to take place prior to going out in the field.

R06B Deliverables

Revised AASHTO Standards

The feedback obtained from the participating DOTs in the project indicated that revisions were necessary in order to render two of the proposed AASHTOs (XRF and ATR for RAP) more user-friendly. However, no comments were received that warranted the production of state-specific protocols. Accordingly, revisions were made to the draft methods developed in Phase 3 and a single updated version of each method was prepared. These are included in Appendix A.

Spectral Database for Fingerprinting State-Approved Materials

A library of ATR spectra and XRF results for pure materials was created. This library can be used for the identification of those materials in the field. The database will supplement the standards developed under this project and can be potentially available online for QC/QA specialists. The electronic copy of the spectral library will be provided in an electronic version together with a final version of this report.

Recommendations for Implementation

Recommendations for implementation are provided in the Generic Implementation Plans developed for each method separately in Appendix D.

Reference

Zofka, A., M. Chrysochoou, I. Yut, and C. Johnston. 2013. SHRP 2 Report *S2-R06B-RR-1: Evaluating Applications of Field Spectroscopy Devices to Fingerprint Commonly Used Construction Materials*. Transportation Research Board of the National Academies, Washington, D.C.

APPENDIX A
Revised Draft AASHTO Methods

Standard Practice for
Standard Method of Test for Determination of Titanium Content in Traffic Paints by Field-Portable X-Ray Fluorescence Spectroscopy
AASHTO Designation SP XX-14

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1. Scope and Overview

1.1. This guide covers the use of field-portable X-ray fluorescence (XRF) spectroscopy for the determination of titanium content traffic paints.

1.2. XRF spectroscopy is a proven analytical technique for measuring elemental concentrations. Increased sophistication of XRF technology has led to the development of field-portable devices that can be used for rapid, non-destructive analyses for on-site quality control in a variety of industrial settings.

1.3. The XRF analyzer determines the concentrations of metals by measuring the intensity of the fluorescent radiation emitted by atoms at their characteristic energies upon bombardment by high-energy X-rays.

1.4. Because the specific operation varies greatly among the available XRF devices, no specific instructions are provided herein, and the user should refer to the operating instructions provided by the manufacturer.

1.5. The specific elements able to be detected by XRF depend on the type and calibration of the analyzer. In general, organic compounds cannot be detected using XRF, and so this guide is applicable only for inorganic substances.

2. Referenced Documents

2.1. *ASTM Standards:*

- ASTM D3925-02: Standard Practice for Sampling Liquid Paints and Related Pigmented Coatings. In *Annual Book of ASTM Standards*, Vol. 06.01. West Conshohocken, PA, 2010.
- ASTM D4764-01: Standard Test Method for Determination by X-ray Fluorescence Spectroscopy of Titanium Dioxide Content in Paint. In *Annual Book of ASTM Standards*, Vol. 06.01. West Conshohocken, PA, 2012.

- ASTM D5381-93: Standard Guide for X-Ray Fluorescence (XRF) Spectroscopy of Pigments and Extenders. In *Annual Book of ASTM Standards*, Vol. 06.01. West Conshohocken, PA, 2009.

2.2. *Radiation Safety Literature*

- EPA-402-K-07-006, *Radiation: Risks and Realities*, U.S. Environmental Protection Agency, 2007.
- Shapiro, J. *Radiation Protection. 4th ed.*, Harvard University Press, 2002.

2.3. *Regulatory Standards:*

- OSHA 29 CFR 1910.1096, Ionizing Radiation: General Industry
- OSHA 29 CFR 1926.53, Ionizing Radiation: Construction Industry

3. Significance and Use

3.1. The method described herein is effective for the rapid, non-destructive, on-site determination of titanium in traffic paints and related coatings by XRF for quality control purposes.

3.2. The method is suitable for measurements of liquid paint samples and in situ measurements of pavement markings.

4. Safety

4.1. XRF analyzers produce ionizing radiation, which damages biological tissue. Significant advancements in engineered safety features have reduced the risk of occupational exposure to ionizing radiation when using XRF devices. However, necessary precautions must be followed to further ensure safety and minimize radiation exposure (Shaprio, 2002; US EPA, 2007).

4.2. XRF analyzers should be used only by operators who have documented proficiency in radiation safety and have been trained by a representative of the instrument manufacturer. The XRF device must be used only as described in the instruction manual provided by the manufacturer. The instruction manual should be present anytime the instrument is in use. The operator should attend radiation safety training in accordance with the instructions issued by the manufacturer and applicable federal and state occupational safety regulations (e.g., OSHA 29 CFR 1910.1096 and 1926.53).

4.3. Safety should the primary objective when using portable XRF analyzers. The operator should avoid direct contact from the X-ray beam. Exposure from scattered X-rays should be minimized by measuring high-density samples, never performing measurements on handheld samples, and maximizing the distance between the operator and the sample.

4.4. The XRF operator is responsible for maintaining a safe environment during use of the XRF instrumentation. The XRF analyzer should never be pointed directly at another person. Nearby persons should be alerted of the radiation exposure hazards and kept at a safe distance from the XRF analyzer.

4.5. The XRF operator should wear a radiation dosimeter badge to monitor radiation exposure, in accordance with relevant state regulations.

4.6. Engineered safety features, such as trigger locking mechanisms and sample proximity sensors, are designed to minimize radiation exposure and should never be tampered with or altered in any way.

5. Apparatus

5.1. *Portable XRF Analyzer*—This is typically designed as a handheld device that is easily transported to and from field sites and measurement locations therein. The detectable elements depend on the instrument and manufacturer. The XRF analyzer should have proof of calibration for absolute measurements. This method is only applicable to XRF instruments capable of detecting titanium. Typical accessories include batteries, charging adapters, and a personal digital assistant installed with the necessary measurement software.

5.2. *Sampling containers*—Sampling containers are typically cylindrical plastic cups and should be selected based on manufacturer recommendations. These are required when the material of interest must be sampled prior to analysis, i.e., for ex situ XRF measurements.

5.3. *X-ray transparent tape or film*—The sample containers should be covered by an X-ray transparent tape or film, such as Mylar or Kapton, such that X-rays can penetrate the sample in close proximity without damaging the instrument.

6. Sample

6.1. Sampling of liquid paints should be conducted in accordance with ASTM Method D3925.

7. Potential Interferences

7.1. *Moisture Effects*—Caution must be exercised when analyzing and comparing XRF results obtained for paints that have variable moisture content. Titanium in the paint is necessarily concentrated as paint the dries. In situ measurements should be performed on dried paint coatings for consistency and to avoid damaging the XRF instrument.

7.2. *Sample Preparation*—Samples should be uniform, homogenized, and randomly sampled, such that the results obtained are representative of the bulk of the material.

7.3. *Spectral Overlap*—When interpreting XRF results, one must be aware of potential overlap of signals from different elements with similar characteristic energies. This type of interference is usually resolved by the manufacturer in operating software of the XRF instrument.

7.4. *Penetration Depth*—X-rays' penetration depth is usually on the order of micrometers to millimeters and is a complicated function of X-ray energy and the properties of the material. If the sample is thinner than the depth of X-ray penetration, the results will include contribution from the substrate. Measurements performed on exceedingly thin coatings should be compared to measurements performed on an adjacent uncoated surface to evaluate the possibility of this type of interference.

8. Standardization

8.1. Follow the instructions for device startup and standardization as described by the manufacturer. This typically involves a warm-up period of 15 to 30 minutes prior to performing any analysis.

8.2. Typically, this is performed using a standardization material (e.g., Alloy 316) after a specified instrument warm-up period. Most devices have a digital screen or PDA that will prompt the user to perform the specific method for internal standardization.

9. Procedure

9.1. For *in situ* measurements of pavement coatings:

9.1.1. The measurement should be performed on an area of the coated pavement surface with thickness that is representative of the entire coating. The coating surface should be clean and smooth as to minimize potential interference from dirt or other debris and to ensure a close contact with the XRF instrument. The coating should be sufficiently dry as to not damage the XRF analyzer.

9.1.2. Quality control measurements should be performed on paint samples prior to the application of glass beads, which may potentially interfere with the measurements.

9.1.3. Follow the instructions for instrument warm-up and standardization.

9.1.4. Gently apply the XRF analyzer directly to the clean coating, such that it is flush against the device and away from the adjacent exposed pavement. Portable XRF analyzers are usually operated by depressing a trigger button. Once the trigger is released, the instrument stops emitting radiation.

9.1.5. Fluorescent X-ray counts are usually collected for a period of 1 to 2 minutes. The actually time required will depend on the instrument and desired level of accuracy. In general, the standard deviation of the measurement decreases with measurement time.

9.1.6. Measurements should be performed in multiple locations of larger coatings to ensure that results are representative of the entire desired coating surface.

9.1.7. The results will be displayed in the form of concentration units such as % total concentration or parts per million by mass.

9.2. For *ex situ* measurements:

9.2.1. The paint should be thoroughly mixed such that the sample is representative of the bulk material.

9.2.2. Place the uniform, homogenized, and representative amount of the liquid paint into a sample container such that it is at least halfway filled.

9.2.3. Cover the opening of the container with piece of X-ray transparent film. Sample containers generally come with a plastic fastener to hold the film in place and seal the container. Once sealed, check that the container is sealed and that the film window is as flat and straightened out as possible to ensure close contact with the X-ray source.

9.2.4. Perform the measurement in accordance with the operating instructions for the device. Some manufacturers provide an apparatus to house the XRF analyzer for bench-top measurements that do not require the instrument to be handheld and trigger-operated.

9.2.5. Fluorescent X-ray counts are usually collected for a period of 1 to 2 minutes. The actually time required will depend on the instrument and desired level of accuracy. In general, the standard deviation of the measurement decreases with measurement time.

9.2.6. The results will be displayed in the form of concentration units such as % total concentration or parts per million by mass.

9.3. Quality assurance can be addressed by restandardizing the instrument, as recommended by the manufacturer. This is typically performed before the first sample, after every 10 to 20 samples, and after the very last sample. The standardization can be verified by measuring a known standard and performing a blank measurement. If necessary and possible, collect a sample for laboratory analysis to verify the accuracy of in situ measurements and to identify possible matrix effects.

10. Report

The report shall include the following:

10.1. The titanium content in units of percent concentration or parts per million by mass.

10.2. The mean and standard deviation associated with each sample.

10.3. Proof of verified instrument calibration (e.g. by ASTM Method D4764) if absolute concentration is reported.

10.4. Limits of detection.

10.5. Specify measurement increments and sample size. It is recommended that 5 readings per increment and 8 to 10 increments to represent a sample size of 1,000 linear ft.

11. Precision and Bias

11.1. Ti concentration may vary within ± 0.3% total concentration by mass between duplicate samples.

Appendix A to Revised Draft ASHTO Methods: Portable XRF Manufacturers

The following is a partial list of manufacturers of field portable XRF analyzers.

- Olympus NDT, Inc.
  48 Woerd Avenue
  Waltham, MA 02453
  (781) 419-3900

- Bruker Optics
  19 Fortune Drive
  Billerica, MA 01821
  (978) 439-9899

- Thermo Scientific Portable Analytical Instruments
  2 Radcliff Road
  Tewsbury, MA 01876
  (978) 670-7460

- SPECTRO Analytical Instruments
  160 Authority Drive
  Fitchburg, MA 01420
  (978) 342-3400

Standard Practice for
Identification of Water Reducing, Accelerating, and Retarding Chemical Admixtures in Fresh Portland Cement Concrete by Attenuated Total Reflection Infrared Spectrometer
AASHTO Designation SP XX-14

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1. SCOPE

1.1 This method covers the qualitative identification of the type of a chemical admixture for portland cement concrete (PCC). The method is based on the qualitative analysis of the infrared absorbance spectra of a pure admixture sample. A pure admixture sample is obtained from the storage or feeding tank in a concrete plant. A pure admixture sample is scanned by attenuated total reflection (ATR) infrared spectrometer to obtain its absorbance spectrum. Next, the type of admixture is determined, based on characteristic absorption bands associated with the particular admixture type. Lastly, a fresh PCC sample is scanned by the ATR spectrometer, and the absorption bands associated with presence of the admixture are identified.

1.2 It is desired to perform ATR testing of an admixture sample immediately after sampling. Long exposure of an admixture to air can result in the evaporation of water or oxidation of an organic content of the admixture, which would alter its chemical composition.

1.3 It is required that PCC sample be tested by the ATR spectrometer within 5 minutes after its removal from framework. Furthermore, the ATR scanning should occur within 2 minutes after its placement on the ATR sampling plate. This is to avoid the damage to the testing apparatus due to fast drying of a thin PCC paste sample.

1.4 This procedure may involve hazardous materials, operation, and equipment. This procedure does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this procedure to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitation prior to use.

2. REFERENCED DOCUMENTS

2.1 *ASTM Standards:*

- C494, Standard Specification for Chemical Admixtures for Concrete
- C1017, Standard Specification for Chemical Admixtures for Use in Producing Flowing Concrete
- C260, Standard Specification for Air-Entraining Admixtures for Concrete

3. APPARATUS

3.1 Sampling equipment

3.2 Pipette for sampling liquid admixture

3.3 Spoon for sampling PCC

3.4 Spectroscopic equipment

3.5 Infrared spectrometer equipped with diamond single reflection ATR accessory and load applicator

3.6 Cleaning tools

3.7 99% Acetone for cleaning ATR sampling plate after sample is removed.

3.8 Soft cloth or tissue for sample removal.

4. SAMPLE PREPARATION

4.1 **Pure chemical admixtures.** Normally, no sample preparation is required for liquefied chemicals when the horizontal ATR sampling plate is used.

4.2 **Fresh PCC.** A PCC sample with maximum particle size of 1 mm is collected from a mixer or from the formwork with using a sampling spoon. Special care should be taken to avoid particles bigger than 2 mm and to preserve representative moisture content in a PCC sample.

5. SPECTROSCOPIC EQUIPMENT SETUP

5.1 The ATR spectrometer should be placed on a firm horizontal surface to avoid any vibrational interference with the instrument signal.

5.2 A reliable source of electric power (AC or DC) should be provided to ensure no interference with the spectrometer signal.

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

5.4 The ATR spectrometer should be connected to a data acquisition system (normally, a computer with an accompanying software) all the time during a test.

6. PROCEDURE

6.1 ATR testing of pure admixture sample

6.1.1 Clean up the surface of the ATR sampling plate by applying soft tissue wetted in 99% Acetone.

6.1.2 Collect and store the background spectrum in accordance with the ATR spectrometer manual.

6.1.3 Collect admixture sample using a pipette and place 3 to 5 drops of a sample on the ATR sampling plate.

6.1.4 Operate ATR spectrometer in accordance to the instrument manual to obtain infrared absorbance spectrum of a sample. Use accompanied data acquisition software to subtract background spectrum, correct baseline, and remove atmosphere- and water-vapor-related absorption bands from the sample spectrum. Store the ATR absorbance spectrum in numerical format for further processing as needed.

6.1.5 Repeat steps described in 6.1.1 through 6.1.4 two more times to establish standard deviation of the test.

6.1.6 Interpret the absorbance spectrum of the admixture sample and determine the type of admixture as explained in Sections 7.XX and 7.XXX of this Standard.

6.2 ATR testing of Fresh PCC Sample

6.2.1 Clean up the surface of the ATR sampling plate by applying soft tissue wetted in 99% acetone.

6.2.2 Collect and store the background spectrum in accordance with the ATR spectrometer manual.

6.2.3 Collect PCC sample using a sampling spoon and place enough of a sample t o entirely cover ATR sampling plate. Ensure sample to be as explained in section 4.2. Apply pressure to a sample using load applicator supplied with an instrument.

6.3 **Note:** If no load applicator supplied with the ATR instrument, it is recommended to: (a) ensure sample particle size be not larger than 0.3 mm to avoid increased variability in results, and (b) apply pressure to a sample through the flat surface of a sampling spoon.

6.3.1 Operate ATR spectrometer in accordance to the instrument manual to obtain infrared absorbance spectrum of a sample. Use accompanied data acquisition software to subtract background spectrum, correct baseline, and remove atmosphere- and water-vapor-related absorption bands from the sample spectrum. Store the ATR absorbance spectrum in numerical format for further processing as needed.

6.3.2 Repeat steps described in 6.2.1 through 6.2.4 four more times to establish standard deviation of the test.

6.3.3 Interpret the absorbance spectrum of the admixture sample and verify presence of the admixture, as explained in Section 8 of this standard.

7. SPECTRAL DATA PROCESSING

7.1 Need in data processing for identification of characteristic absorption bands

7.2 Identification of absorption bands in simple compounds such as chemical admixtures to PCC is normally done using software supplied with an infrared spectrometer by a manufacturer. The software generates an output in both tabular and graphic formats. In both formats, the reciprocal wavelength, or wavenumber, at the center of an identified band is reported along with the corresponding intensity of infrared absorption at that wavenumber. Alternatively, the user can identify the absorption peaks from visual analysis of a spectrum.

7.3 The absorbance is directly proportional to the concentration of particular component of a compound or a mixture. The concentration of the admixtures in a PCC sample is expected to range between as low as 0.05% to 1%, which may make visual analysis of its spectrum difficult. In addition, the default sensitivity of the instrument software may not be sufficient to identify weak but narrow absorption bands associated with the admixture. However, a relatively simple mathematical manipulation of a spectrum using a second-derivative method for extraction of absorption peaks of any intensity can be used. This method is documented elsewhere and it is beyond the scope of this standard practice.

8. INTERPRETATION OF RESULTS

This section provides guidelines for the interpretation of the absorbance spectra of a pure admixture and a PCC sample. The class of admixture is determined based on the characteristic infrared absorption bands on a spectrum. The unique absorption bands are attributed to specific chemical components (functional groups) within an admixture. Those characteristic spectral features are used to verify the presence of the admixture in a resultant PCC mix sample. Appendix A provides example chemical composition and list of characteristic spectral features for the admixtures, along with spectra graphs.

8.1 Identification of the Type of Admixture by Infrared Absorption Bands

8.1.1 Non-chloride Accelerators (ASTM C494, Type C).

8.1.1.1 **Sodium thiocyanate** is identified by the weak to medium band centered on 2070±5 cm^{-1} wavenumbers. When in aqueous solution, it can additionally give rise to a strong and wide band centered around 1330±5 cm^{-1} with distinctive shoulder at 1410±5 cm^{-1}.

8.1.1.2 **Calcium nitrate** is identified by a strong and wide band at 1330±5 cm^{-1} with distinctive shoulder at 1410±5 cm^{-1}due to NO_2 and two medium and sharp peaks at 1047±5 and 826±5 cm-1 associated with nitrate anion NO_3.

8.1.2 Water Reducers (ASTM C494, Types A and D).

8.1.2.1 **Polycarboxylate ether** is identified by a very strong absorption band centered around 1086±5 cm^{-1} with distinctive shoulder at about 1140 cm^{-1} associated with polyether backbone.

8.1.2.2 **Carbohydrates** are typically identified by the coupled C-O-C vibrations yielding medium peaks at 1300 and 1250 cm^{-1} as well as by C-OH vibrations with a strong corresponding peak at 950 cm^{-1}.

8.1.3 Retarders (ASTM C494, Type B).

8.1.3.1 **Sodium gluconate** is identified by a prominent terminal carboxylate in its structure, which yields characteristic split of the water band (1649 and 1592 cm^{-1}) and a strong band splitted at 1084 and 1038 cm^{-1} due to vibrations of the multiple OH groups.

8.1.3.2 **Carbohydrates –** See 8.1.2.2.

8.2 Verification of Presence and Type of Admixture in Fresh PCC Sample

8.2.1 On the spectrum of a fresh PCC sample identify characteristic peaks described in 8.1

8.2.2 Assign characteristic peaks in accordance with 8.1

8.3 **Note:** Positive verification of the presence of a particular admixture is limited to those added in minimum 0.5% of the total PCC batch weight or 2% of the cement weight.

9. PRECISION

9.1 This method based on the qualitative evaluation of the ATR spectra in regards to location of characteristic infrared absorption bands. It cannot be used for quantitative assessment of the admixture content in PCC.

9.2 Location of the characteristic peaks on an ATR spectrum can vary within ± 10 cm^{-1} from the values given in this method.

Appendix A. Example of chemical composition and spectral features for PCC admixtures

Table A-1 summarizes classification and chemical composition of the admixtures described in this standard. Figures A-1 through A-4 superimpose absorbance spectra of pure Portland cement concrete, an admixture, and their mix for each of the admixtures described in Table 1.

Reference

Yut I. and Zofka A., “Fingerprinting of Chemical Admixtures in Fresh Portland Cement Concrete by Portable Infrared Spectrometer,” Transportation Research Record: Journal of Transportation Research Board, No. 2290, pp.1-9, 2012

TABLE A-1. Chemical Composition of Admixtures (after Yut and Zofka, 2012)

Admixture Class	ASTM Type	Chemical class	Ingredients (Weight %)	Chemical Functionalities
High-Range Water Reducer (Superplasticizer)	C494 Type A and F, ASTM C1017 Type I	Acrylic co-polymer	Water (90–99) Carboxylated polyether (1–10)	H_2O; -COO-; $-CH_2-O-CH_2-$;
Air Entrainer	AEA C260	Sodium abietate	Water (>60) Tall oil, sodium salt (10–30) 4-Chloro-3-methylphenol (<1)	H_2O; $C_{19}H_{29}$-,COOH, Na^+; $-C_6H_3OH$; $-CH_3$; -Cl
Water Reducer/ Accelerator	C494 Type C and E	Calcium nitrate	Water (40–70) Calcium nitrate (40–70)	H_2O; $Ca(NO_3)_2$
Water Reducer/ Retarder	C494 Type D	Sodium gluconate	Water (>60) Sodium gluconate (30–60) 4-Chloro-3-methylphenol (<1)	H_2O; -CHOH-; CH_2OH; $CONaO-C_6H_3OH$; $-CH_3$; -Cl

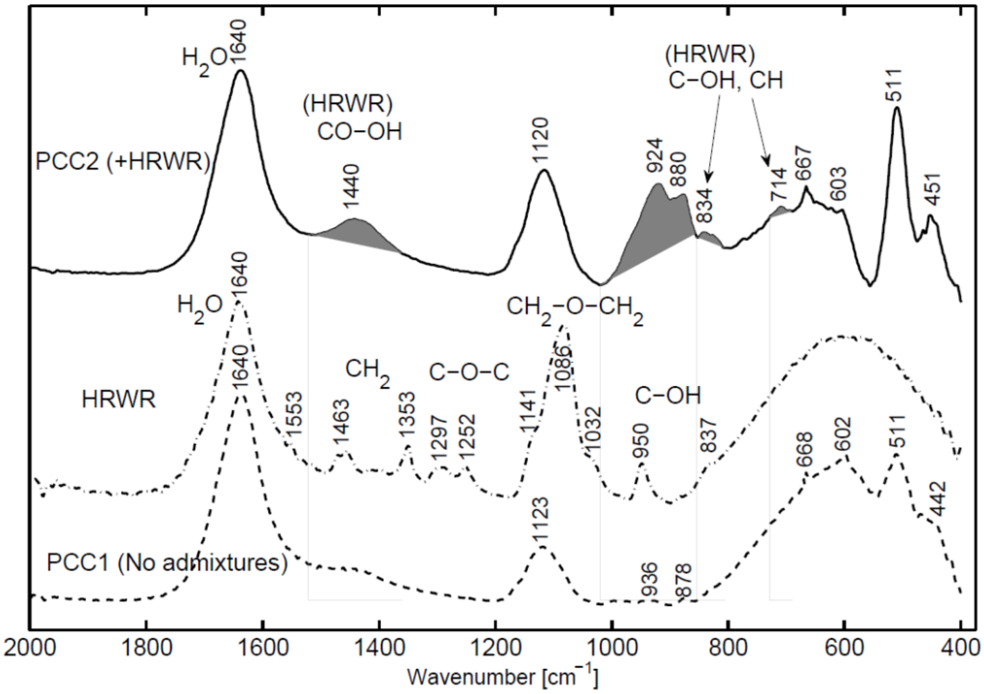

FIGURE A-1. Composition of the ATR spectrum of PCC sample modified with High Range Water Reducer (HRWR) (after Yut and Zofka, 2012).

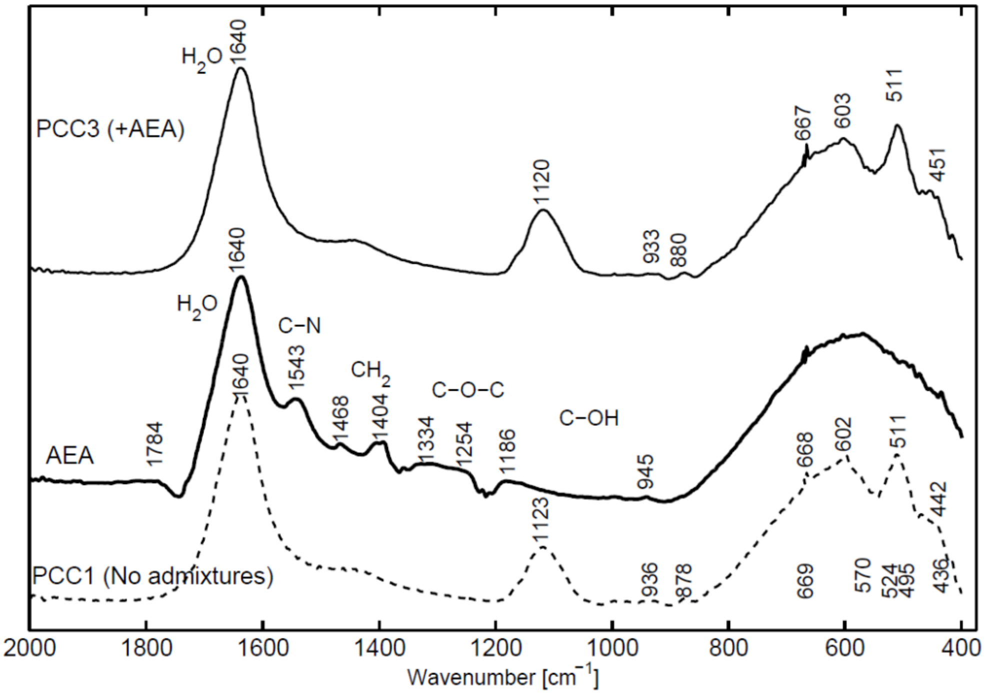

FIGURE A-2. Composition of the ATR spectrum of PCC sample modified with Air Entraining Admixture (AEA).

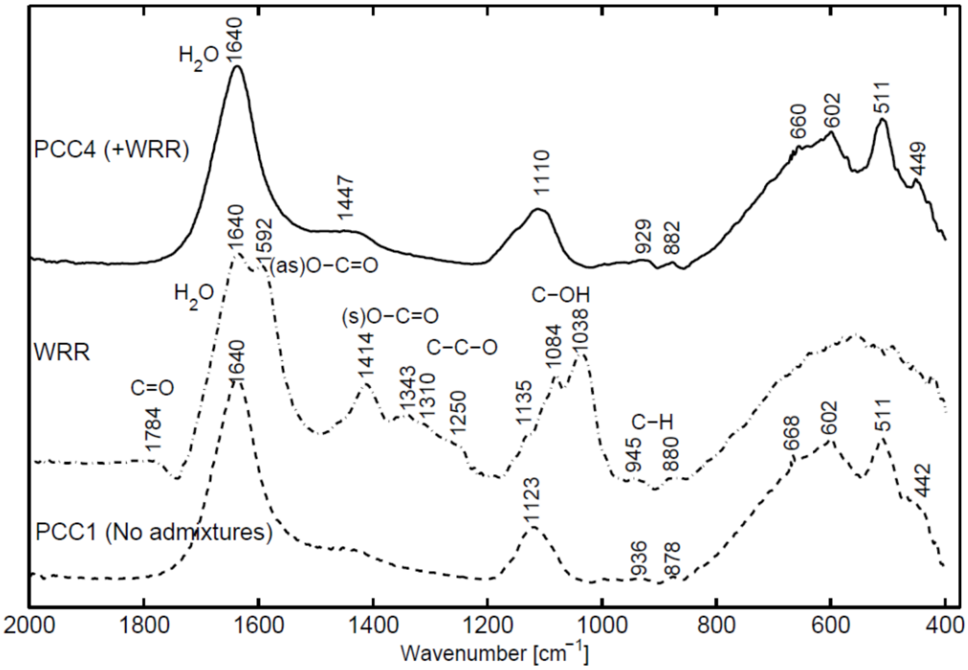

FIGURE A-3. Composition of the ATR spectrum of PCC sample modified with Water Reducer/Retarder (after Yut and Zofka, 2012).

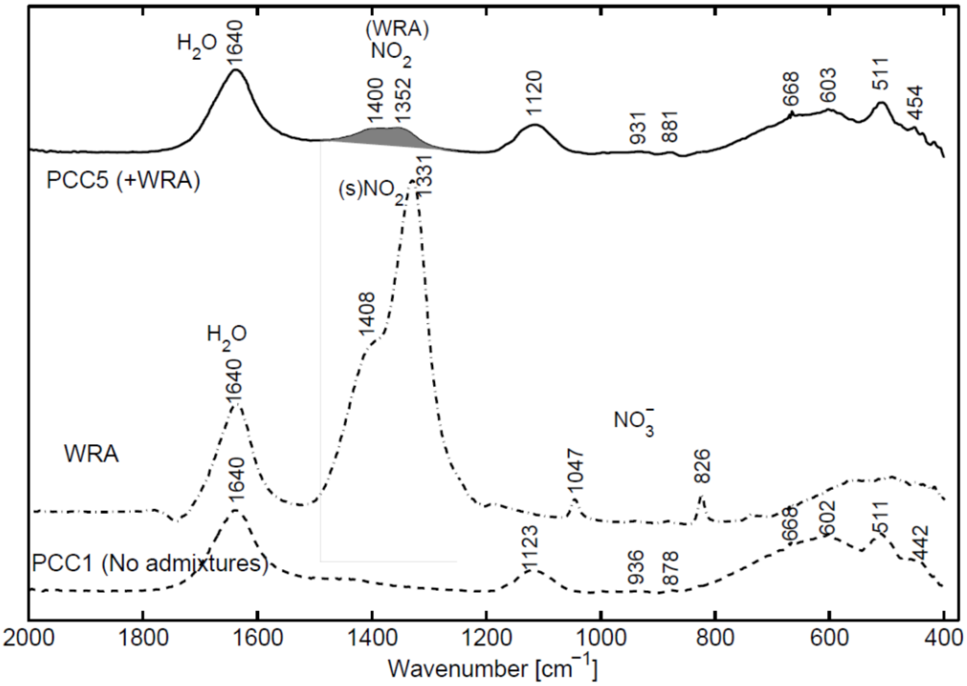

FIGURE A-4. Composition of the ATR spectrum of PCC sample modified with Water Reducer/Accelerator (after Yut and Zofka, 2012).

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Standard Practice for

Evaluation of Oxidation Level of Asphalt Mixtures by Attenuated Total/Diffused Reflection Infrared Spectrometer

AASHTO Designation SP XX-14

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1. SCOPE

1.1 This method covers the semi-quantitative evaluation of the oxidation level in an asphalt mixture by measuring its carbonyl content. The method is based on the quantitative analysis of the infrared absorbance/ log (Reflectance) spectrum of an asphalt mixture sample. This sample can be obtained from the pavement surface and may be modified by adding recycled asphalt pavement or shingles (RAP and RAS, respectively). The carbonyl content of a sample in question is compared with predetermined carbonyl content of a non-oxidized (freshly paved) and fully oxidized (milled/removed) samples of asphalt mix. Those samples are obtained from an asphalt plant and a recycled asphalt stockpile, respectively. The asphalt mix samples are scanned by attenuated total reflection (ATR)/ diffused reflection (DR) infrared spectrometer to obtain their corresponding absorbance/ log(Reflectance) spectra. Next, the oxidation level in the samples is calculated based on the absorbance/ log(Reflectance) intensity in the infrared region of frequencies associated with carbonyl content.

1.2 The asphalt mix samples must be in a loose state (not compacted), while the maximum size of aggregate in a sample should pass U.S. sieve #30 (600 μm).

1.3 It is desired to perform ATR/DR testing of an asphalt mix sample at a temperature not exceeding 40°C.

1.4 This procedure may involve hazardous materials, operation, and equipment. This procedure does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this procedure to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitation prior to use.

2. REFERENCED DOCUMENTS

2.1. *ASTM Standards:*

- To be updated
- Yut, I., and Zofka, A., Spectroscopic Evaluation of Recycled Asphalt Materials, Paper 12-1259, In: CD-ROM, *91st Annual TRB Meeting Compendium*, January 2012
- Yut I., Bernier A., and Zofka A., Development of a Compact Laboratory Aging Procedure for Asphalt Binders, *Journal of Association of Asphalt Pavement Technologists*, Vol. 81, 2012

3. APPARATUS

3.1 Sampling equipment

3.2 Hammer drill, scoop, and brush for sampling from pavement surface

3.3 Standard AASHTO sieves #8, #16, and #30 for sifting a sample of the loose mix

3.4 Spectroscopic equipment

3.5 Infrared spectrometer equipped with diamond single reflection ATR accessory and load applicator/diffused reflection infrared fourier transform (DRIFT) spectrometer

3.6 Cleaning tools

3.7 99% acetone for cleaning ATR sampling plate after sample is removed.

3.8 Soft cloth or tissue for sample removal.

4. SAMPLE PREPARATION

4.1 **Loose Mix Sample.** Sieve about 1 kilogram of loose mix through standard US #8, #16, and #30 sieves. Use passing #30 fraction for spectroscopic testing.

4.2 **Field Pavement Sample.** A sample of powdered asphalt mix from the in-situ pavement surface can be obtained by following procedures:

4.2.1 Drill a ½-in deep and ½-in diameter hole in pavement surface. Use brush and scoop to collect the asphalt powder residue.

4.2.2 Use sampling spoon to scrub binder mastic sample from the in-situ pavement surface.

5. SPECTROSCOPIC EQUIPMENT SETUP

5.1 The ATR/DRIFT spectrometer should be placed on a firm horizontal surface to avoid any vibrational interference with the instrument signal.

5.2 A reliable source of electric power (AC or DC) should be provided to ensure no interference with the spectrometer signal.

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

5.4 The ATR spectrometer should be connected to a data acquisition system (normally, a computer with an accompanying software) all the time during a test.

6. PROCEDURE

6.1 ATR testing of asphalt mix sample

6.1.1 Clean up the surface of the ATR sampling plate by applying soft tissue wetted in 99% acetone.

6.1.2 Collect and store the background spectrum in accordance with the ATR spectrometer manual.

6.1.3 Collect asphalt mix sample using a sampling spoon and place the sample on the ATR sampling plate in amount sufficient for covering the entirety of diamond reflection window.

6.1.4 Apply pressure to the sample by using load applicator attached to the instrument.

6.2 **Note:** If no load applicator is supplied with the ATR instrument, it is recommended to: (a) ensure sample particle size be not larger than 0.15 mm to avoid increased variability in results, and (b) apply pressure to a sample through the flat surface of a sampling spoon.

6.2.1 Operate ATR spectrometer in accordance to the instrument manual to obtain infrared absorbance/log(Reflectance) spectrum of a sample. Use accompanied data acquisition software to subtract background spectrum, correct baseline, and remove atmosphere- and water-vapor-related absorption bands from the sample spectrum. Store the ATR absorbance spectrum in numerical format for further processing as needed.

6.2.2 Repeat steps described in 6.1.1 through 6.1.4 four (4) more times to establish standard deviation, as explained in Section 9 of this standard.

6.2.3 Interpret the absorbance spectrum of the asphalt mix sample and determine the level of oxidation, as explained in Section 8 of this Standard.

7. SPECTRAL DATA PROCESSING

7.1 Normalization and smoothing of the absorbance spectrum

7.1.1 The absorbance spectrum of a sample should be normalized to the absorbance intensity A at 2920 cm^{-1} wavenumbers (A2920) by subtracting minimum intensity value for the given spectrum (Amin) from each reported intensity and dividing the result by difference between A2920 and Amin, as in Equation [1].

7.1.2 $$Anorm = \frac{A - A\min}{A2920 - A\min} \qquad [1]$$

7.1.3 The normalized absorbance spectra can be smoothed to reduce noise along the spectral line and thus facilitate determination of relevant absorbance peaks. This can be done using Savitzki-Golay algorithm.

7.2 **Note:** Both normalization and smoothing of a spectrum can be performed with an accompanied to the ATR/**DRIFT** instrument software or by an external computational software.

8. DETERMINATION OF OXIDATION LEVEL

8.1 The extent of oxidation is defined as a relative concentration of carbonyl(C=O) and sulfoxide(S=O) chemical functional groups within the sample. The relative concentration, or index of C=O and S=O, is defined as a value of absorbance/log(Reflectance) intensity at 1700 cm^{-1} and 990 cm^{-1} wavenumbers, correspondingly.

8.2 To evaluate level of oxidation, the relative concentrations of C=O and S=O in the sample in question should be compared with those in the fully oxidized and non-oxidized samples as shown in Figure A.8.1.

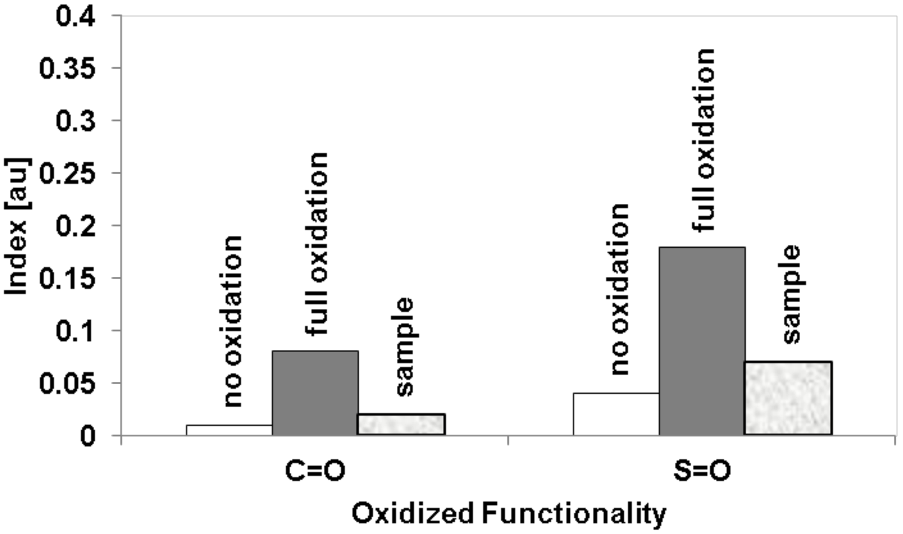

Figure A.8.1 Comparison of oxidation levels between oxidized and non-oxidized asphalt samples.

9. PRECISION

9.1 The coefficient of variation (C.O.V.) in measured oxidation indices for one sample is established as a ratio of standard deviation (std) over the mean index value for at least 5 probes from the sample. If the C.O.V. exceeds 25 percent, additional probes should be taken to achieve this threshold.

9.2 Location of the characteristic peaks on an ATR/DRIFT spectrum can vary within ± 10 cm^{-1} from the values given in this method.

APPENDIX B

Equipment and Testing Protocols

This appendix describes the spectroscopic equipment and testing protocols used in the pre-implementation Phase 4 of this project. The testing protocols include procedures for the preparation of samples, acquisition of data, and interpretation of results for the following two methods:

- Fourier-Transform Infrared Spectroscopy
- X-ray Fluorescence Spectroscopy

Below is a more detailed discussion of these methods and their corresponding experimental protocols.

Fourier-Transform Infrared Spectroscopy

Based on the successful evaluation in the previous stages of the project, a portable Bruker ALPHA ATR FT-IR spectrometer owned by the research team was employed to test chemical admixtures for Portland cement concrete, hot-mix asphalt (HMA) samples, and recycled asphalt pavement (RAP) samples.

Equipment Setup. The ALPHA FT-IR device by Bruker Optics (Figure B.1) has dimensions of 22 × 30 × 25 cm and weighs about 7 kg. It is able to analyze a spectral range from 7500 cm^{-1} to 375 cm^{-1} with a spectral resolution of at least 2 cm^{-1} (0.9 cm^{-1} is optional) and an accuracy of 0.01 cm^{-1}. The device is designed to operate at 18 to 35°C and is powered by 100-240 VAC or by a high-capacity battery. Software, including a comprehensive library of chemical components, is provided with this device. The exchangeable measurement modules include transmission, ATR, and reflection, making it possible to analyze a wide range of materials (liquid, solid, or gas). The Team used the following settings in their laboratory testing:

- A laptop PC with OPUS 6.5 IR spectroscopy software (provided by Bruker Optics).
- The Attenuated Total Reflectance (ATR) mode to acquire the infrared spectra for all materials and their components.
- The spectra for each material/component were obtained in the range from 4,000 to 400 cm^{-1} where most of the chemical components yield detectable absorbance.
- The spectral resolution was set to 1.5 cm^{-1}.
- 24 scans were run for each sample.
- Ambient temperatures during testing ranged between 18 °C in laboratory and 0 °C in the field.

Figure B.1. ALPHA ATR FT-IR portable spectrometer set-up in the field.

Test Sample Preparation. The research team tested the materials/components included in Phase 4 in their original physical state (as provided by manufacturer), i.e., liquid and solid state. Liquid materials, such as PCC chemical admixtures, were sampled using pipettes or syringes and placed on an ATR sampling plate for obtaining their absorbance spectra (Figure B2). Plant–produced RAP samples were collected from stockpiles and transferred for testing (Figure B.3). Before testing, RAP samples were first sieved through a sequence of the U.S. #8, #16, and #30 sieves, after which the passing #30 sieve fraction was tested by ATR spectrometer (Figure B.4). A sample of each material was placed on the ATR diamond surface in an amount sufficient to cover the surface (several drops of liquid or approximately 1 g of solid). For HMA and RAP samples, pressure was applied to ensure full contact between the sample and the ATR diamond surface. The field HMA samples were recovered from the pavement surface by drilling with the use of a hammer drill. The asphalt powdered residue from drilling was placed in a plastic bag and transferred for testing either in a laboratory or in the back of a van directly on the project site. Figure B.6 documents the process of HMA testing in the field.

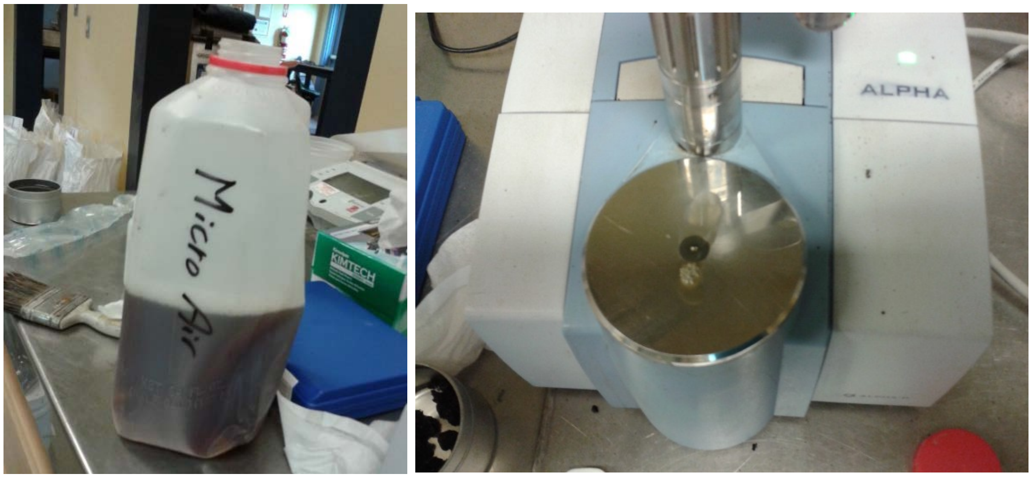

Figure B.2. Air-entraining admixture in a container (left) and on the ATR sampling plate (left).

Figure B.3. RAP stockpile in Manchester, Connecticut (left), and collected RAP samples (right).

Figure B.4. Maine RAP sample in a bag (left) and sieving a RAP sample in the DOT Material Laboratory in Freeport, Maine (right).

Figure B.5. Passing #30 RAP fraction (left) and ATR setup with RAP sample (right).

Figure B.6. Recovering an HMA sample from a pavement surface (left) and placing a pavement surface sample in a bag (right).

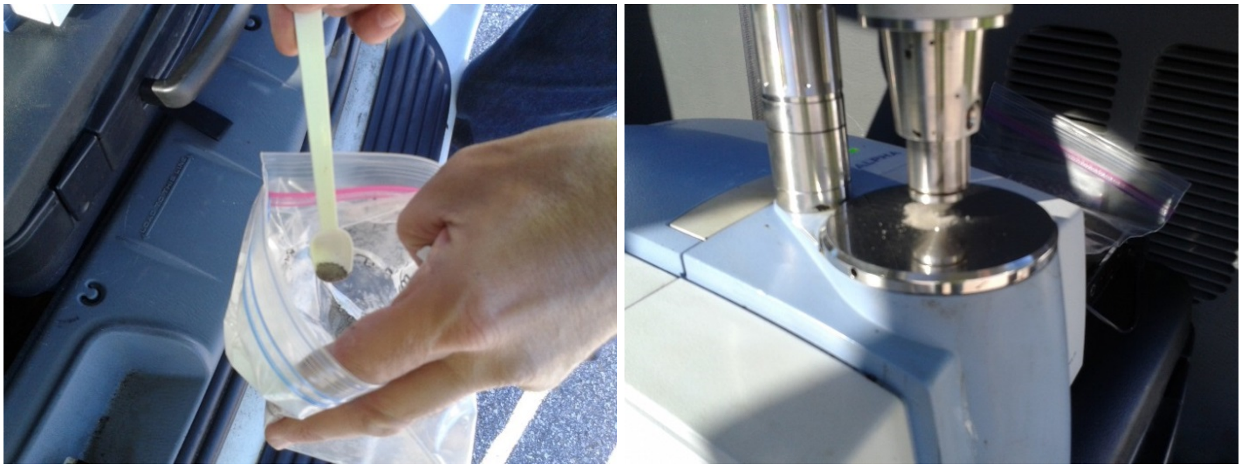

Figure B.7. Placing an in situ HMA sample on an ATR sampling plate.

Testing Procedure. A typical FT-IR testing procedure for any material included the following routines:

- Prepare control sample, if required.
- Obtain infrared spectrum of the control sample.
- Prepare test sample as explained above.
- Obtain infrared spectrum of the test sample.
- Compare the test sample spectrum with that of the control sample by using an appropriate method of analysis, as specified in a corresponding draft AASHTO Standard in Appendix A.

X-ray Fluorescence Spectroscopy

In consideration of promising results in testing traffic paints and bridge coatings, a portable XRF device was included in the pre-implementation phase of this project. The equipment setup and testing procedures are described in detail below.

Innov-X Alpha XRF analyzer

Equipment Setup. The portable Innov-X Alpha XRF analyzer (Figure B.8) owned by the team measures the concentration of the following elements: P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Zr, Mo, Ag, Cd, Sn, I, Ba, W, Hg, and Pb. There are two separate calibration modes for this particular XRF: one for trace elements in soils (concentrations up to 8% to 10% by weight) and one for alloys and ores that have concentrations >1% by weight. However, the alloy mode can only measure elements heavier than Ti, so Ca, K, and other lighter elements can only be measured in relatively low concentrations using this equipment.

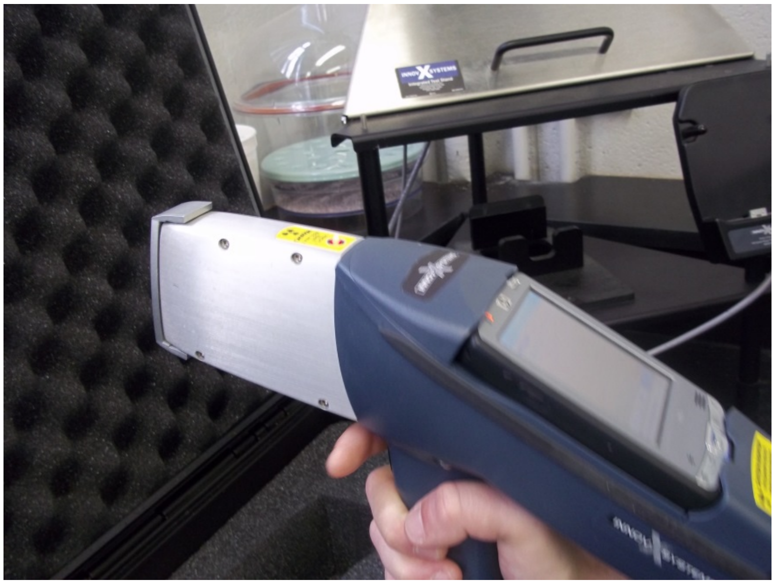

Figure B.8. Innov-X ALPHA XRF analyzer in a calibration mode with stationary setup on the background.

Test Sample Preparation. In general, liquid paint samples were tested in their as-received state by placing 15 g to 30 g of the sample in an XRF sample holder. The field testing of the in situ dried paint samples was performed by applying the sampling window of the XRF instrument to a designated surface and collecting data for 1 to 2 minutes. Figures B.9 and B.10 document the process of testing traffic paints in Connecticut and bridge coatings in Maine.

Figure B.9. XRF testing of a traffic paint control sample (left) and in situ paint strip on Exit 64, I-95, in Connecticut (right).

Figure B.10. Control bridge coating samples in the lab (left) and in situ testing on Exit 174, I-95, in Maine (right).

APPENDIX C
Lab and Field-Test Results

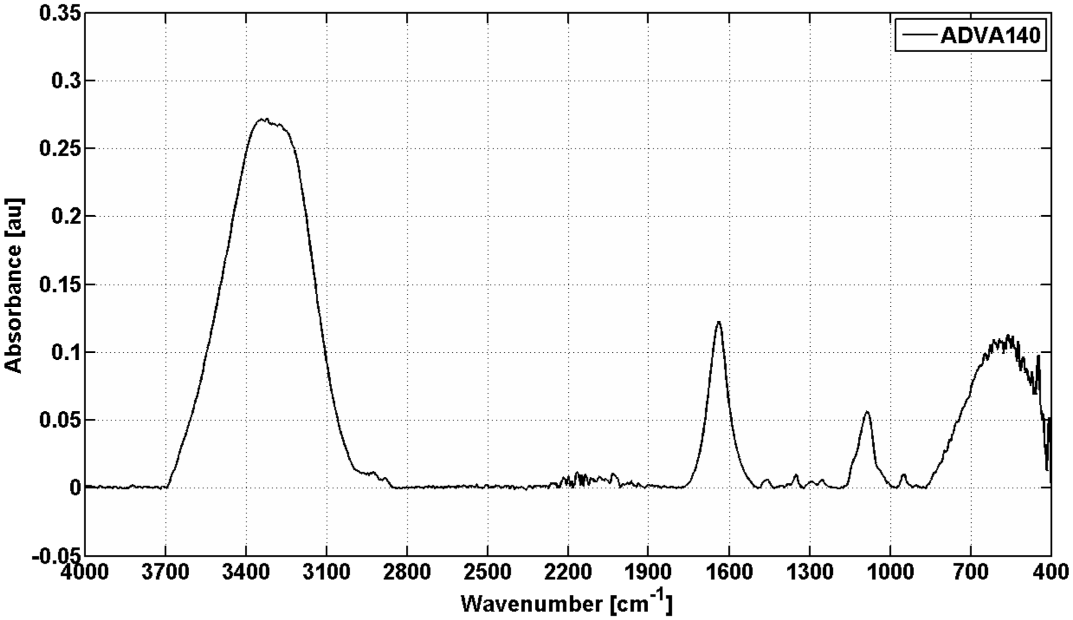

Figure C.1. IR Absorbance spectrum of ADVA-140.

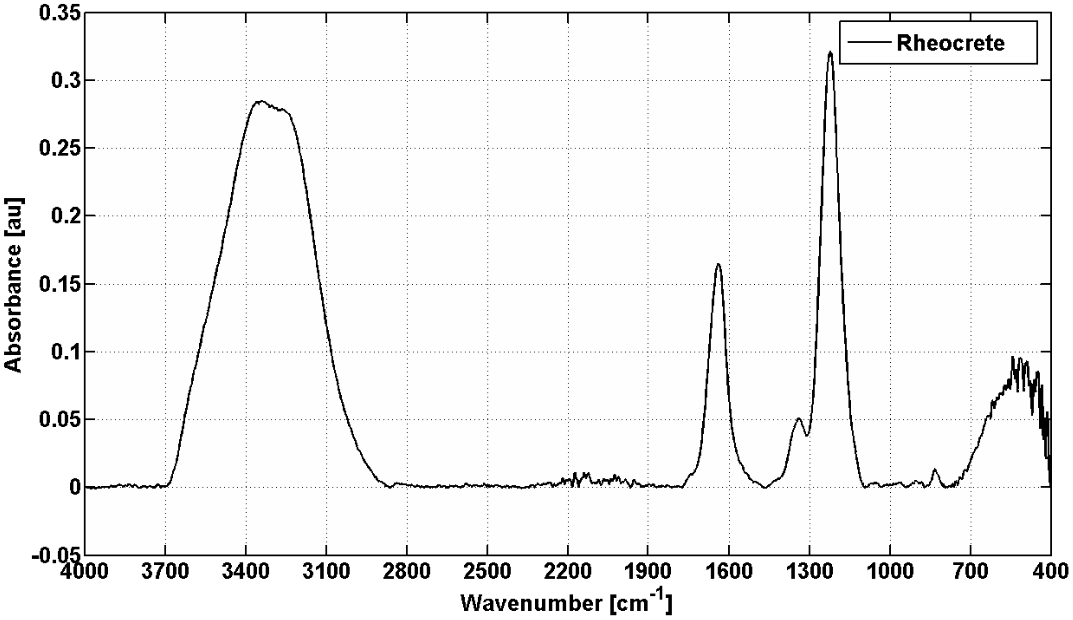

Figure C.2. IR Absorbance spectrum of Rheocrete.

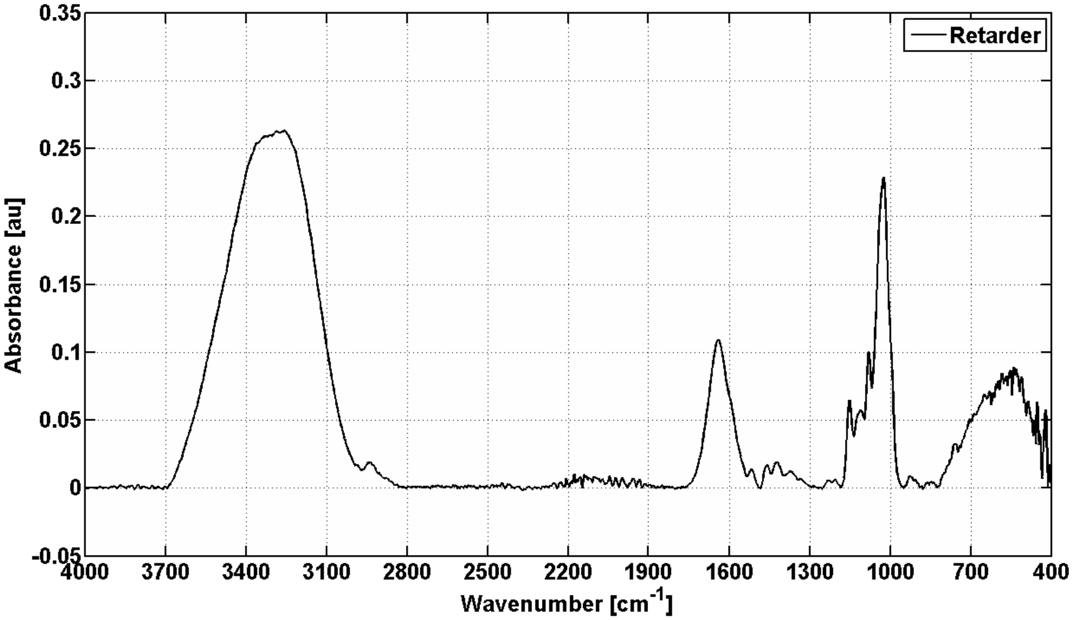

Figure C.3. IR Absorbance spectrum of Retarder.

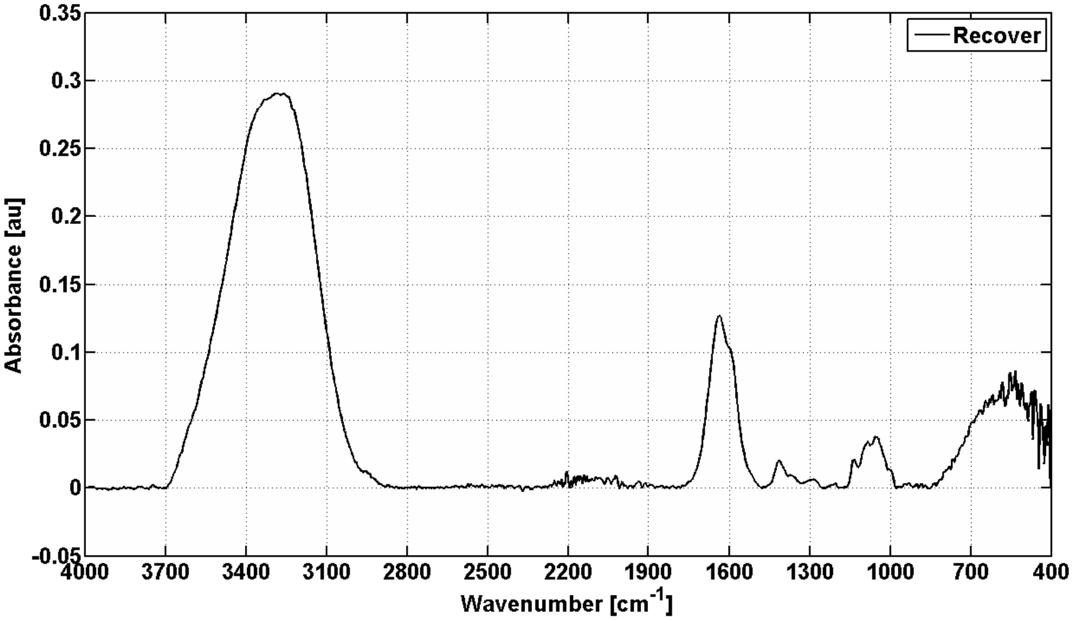

Figure C.4. IR Absorbance spectrum of Recover.

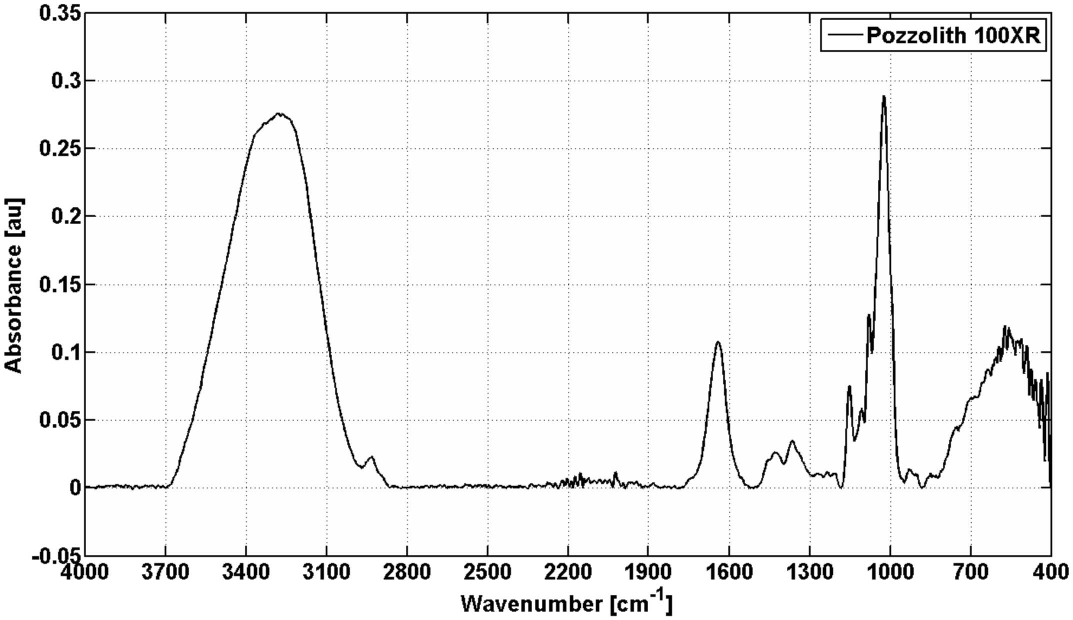

Figure C.5. IR Absorbance spectrum of Pozzolith 100 XR.

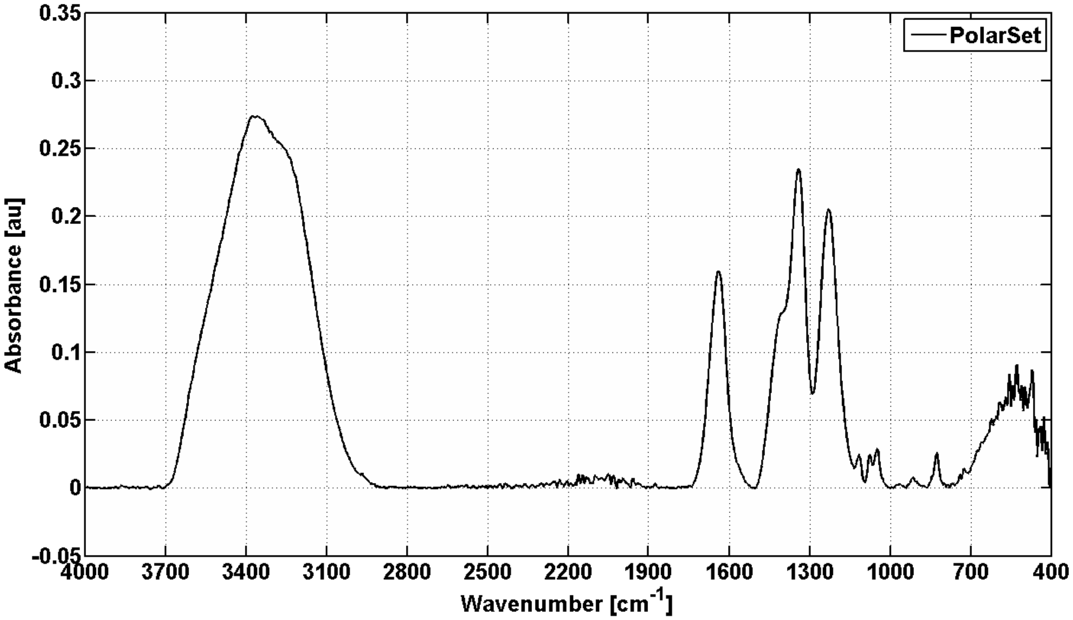

Figure C.6. IR Absorbance spectrum of PolarSet.

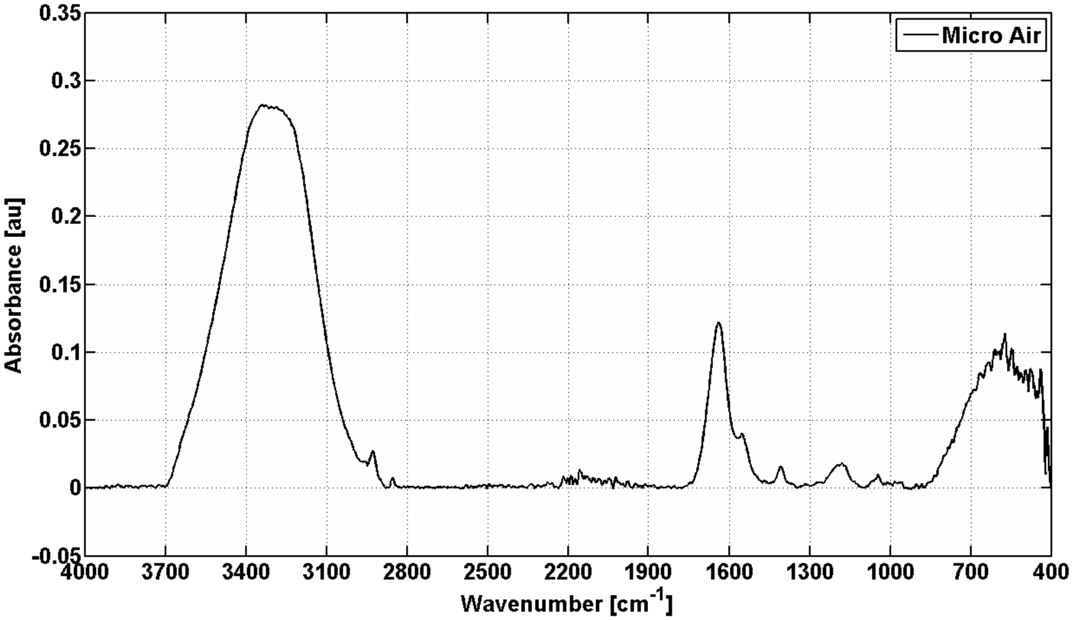

Figure C.7. IR Absorbance spectrum of Micro Air.

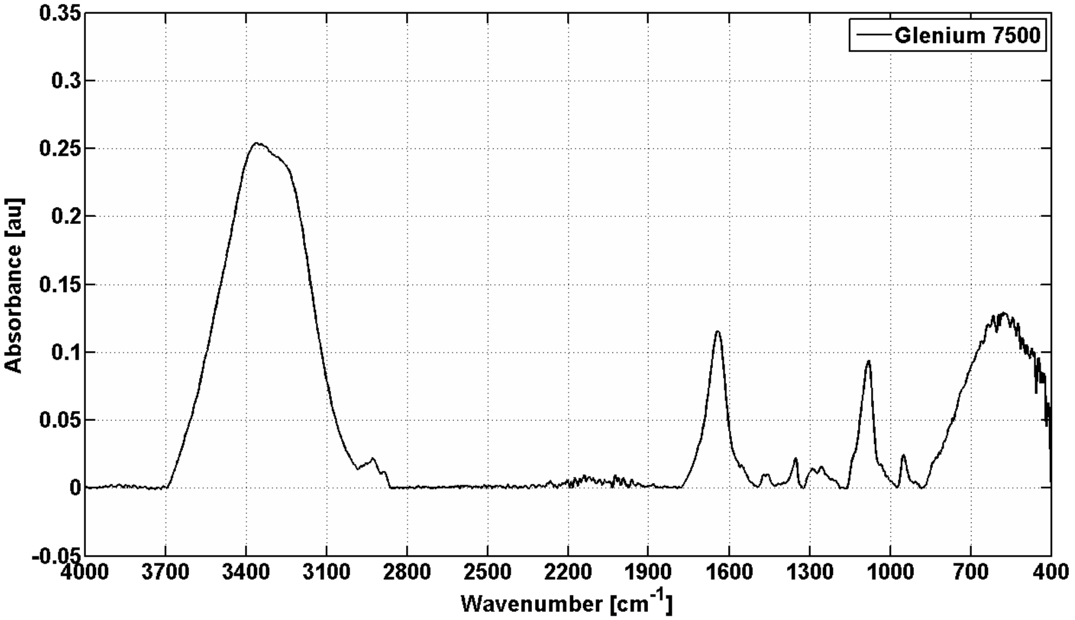

Figure C.8. IR Absorbance spectrum of Glenium 7500.

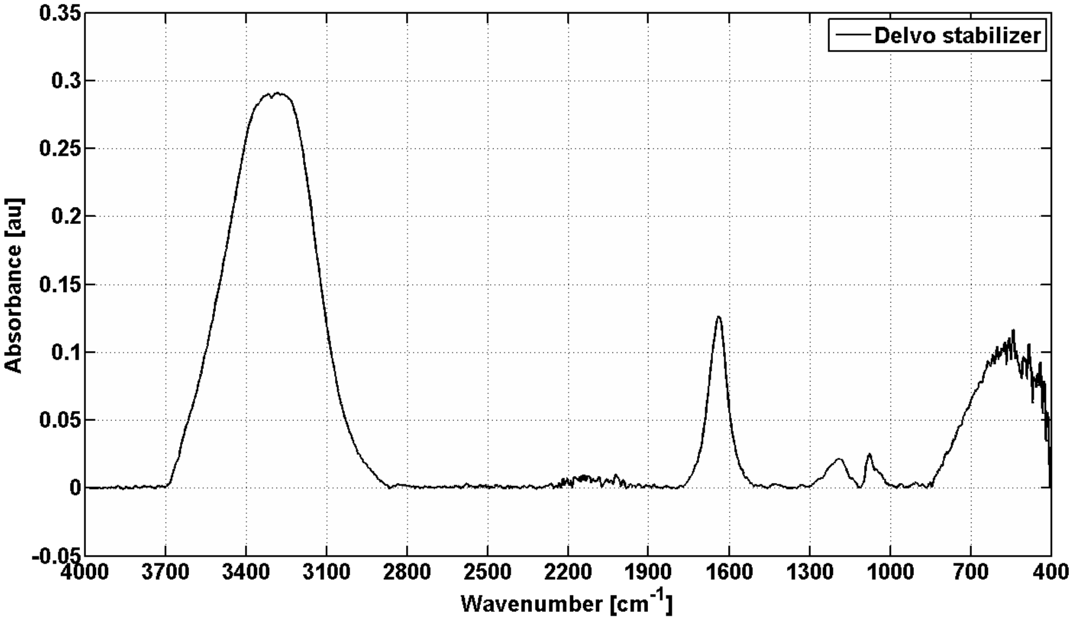

Figure C.9. IR Absorbance spectrum of Delvo Stabilizer.

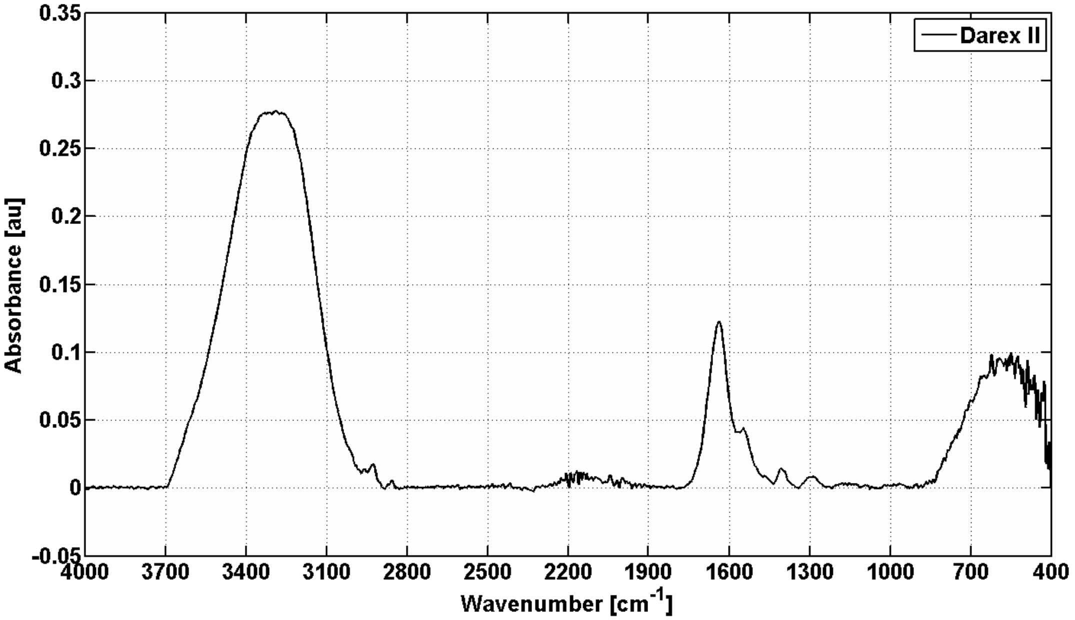

Figure C.10. IR Absorbance spectrum of Darex II.

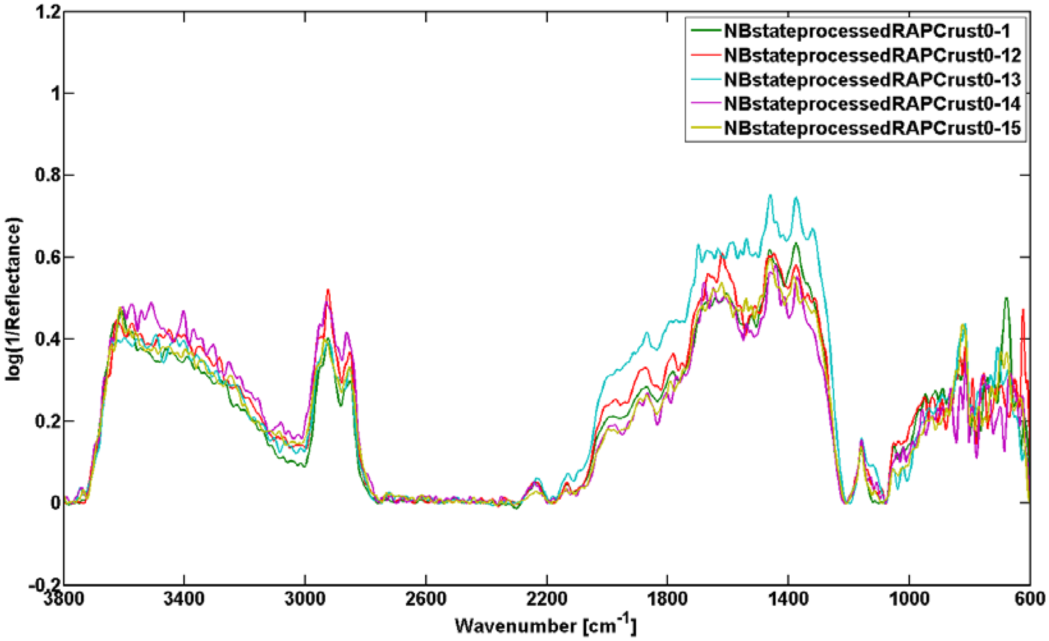

Figure C.11. DRIFTS Reflectance spectra of state-processed RAP from the top of a stockpile in New Britain, Connecticut.

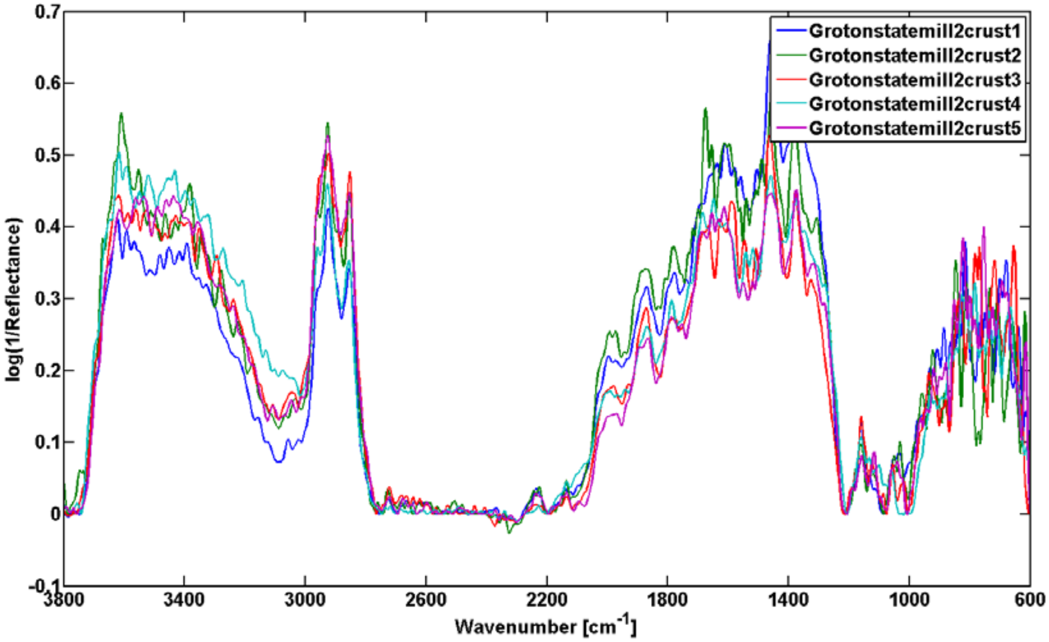

Figure C.12. DRIFTS Reflectance spectra of state-milled RAP from the top of a stockpile in Groton, Connecticut.

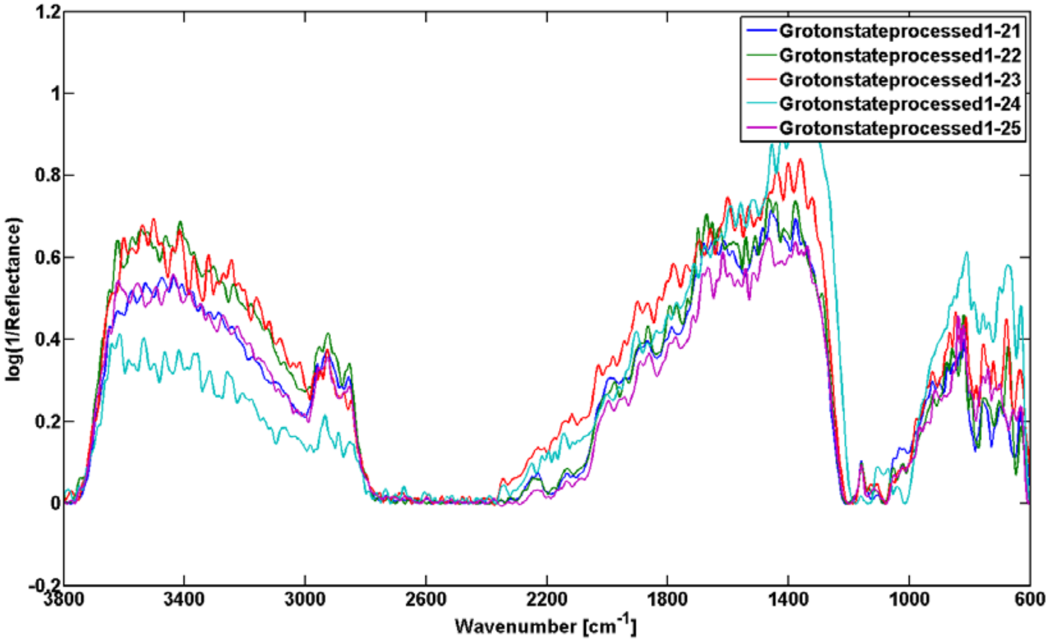

Figure C.13. DRIFTS Reflectance spectra of state-processed RAP in Groton, Connecticut.

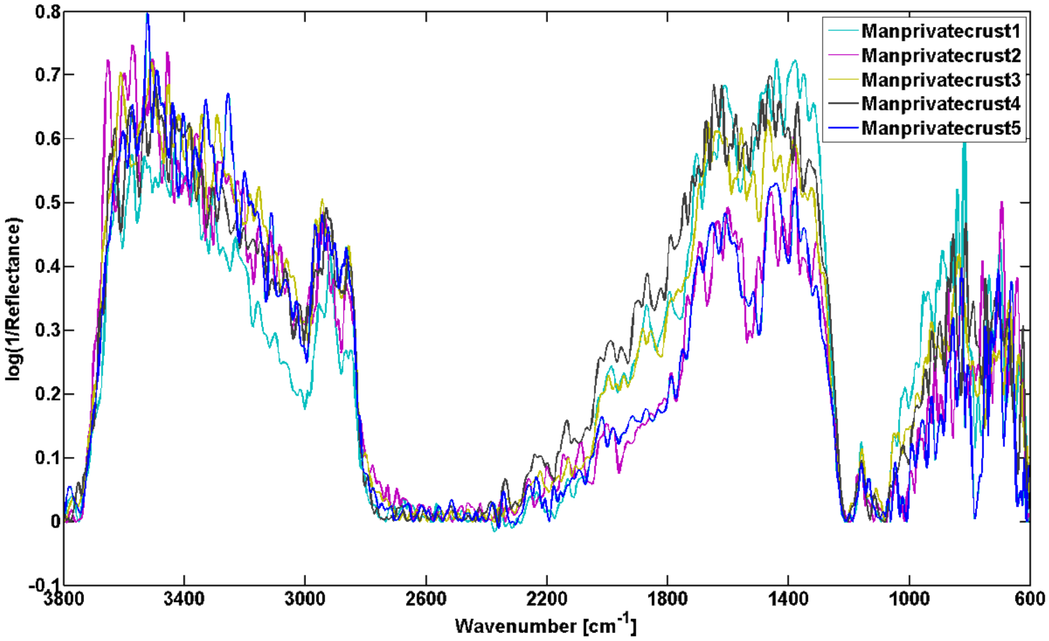

Figure C.14. DRIFTS Reflectance spectra of private RAP from the top of a stockpile in Manchester, Connecticut.

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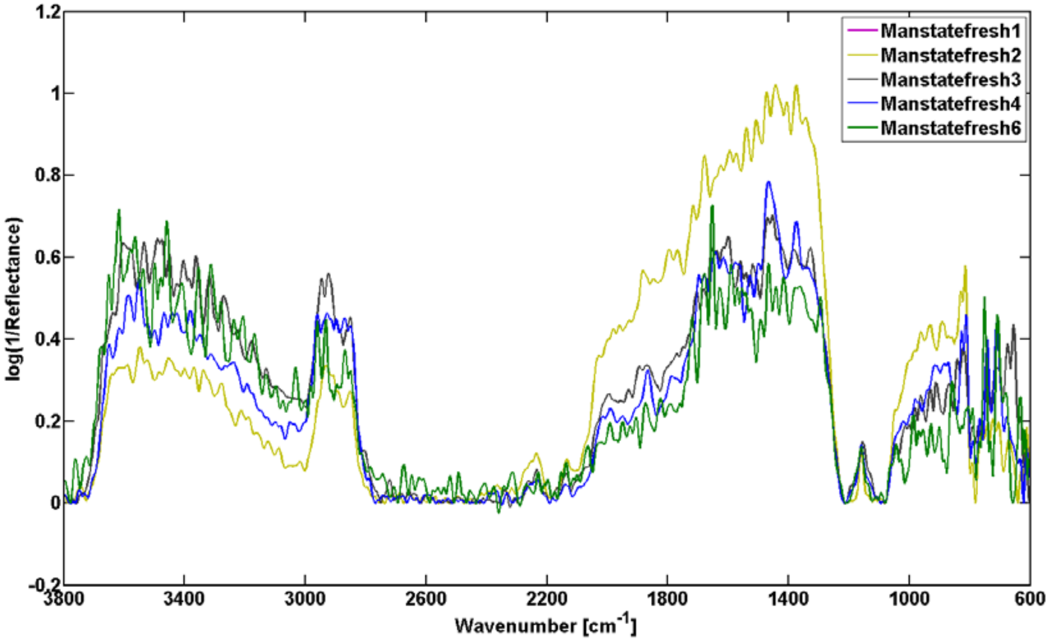

Figure C.15. DRIFTS Reflectance spectra of state-processed RAP from the inside of a stockpile in Manchester, Connecticut.

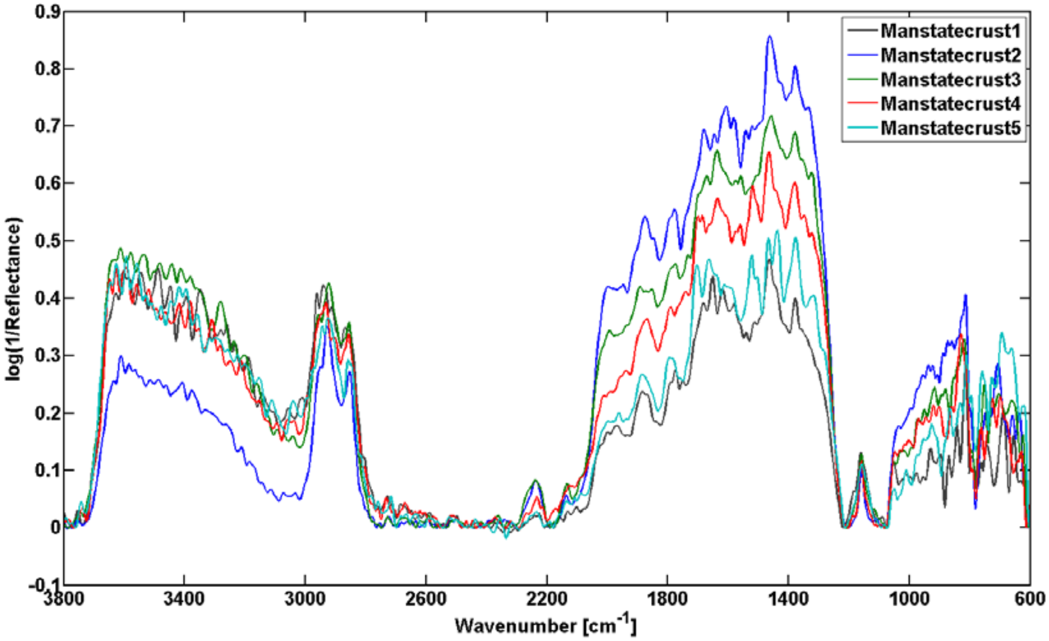

Figure C.16. DRIFTS Reflectance spectra of state-processed RAP from the top of a stockpile in Manchester, Connecticut.

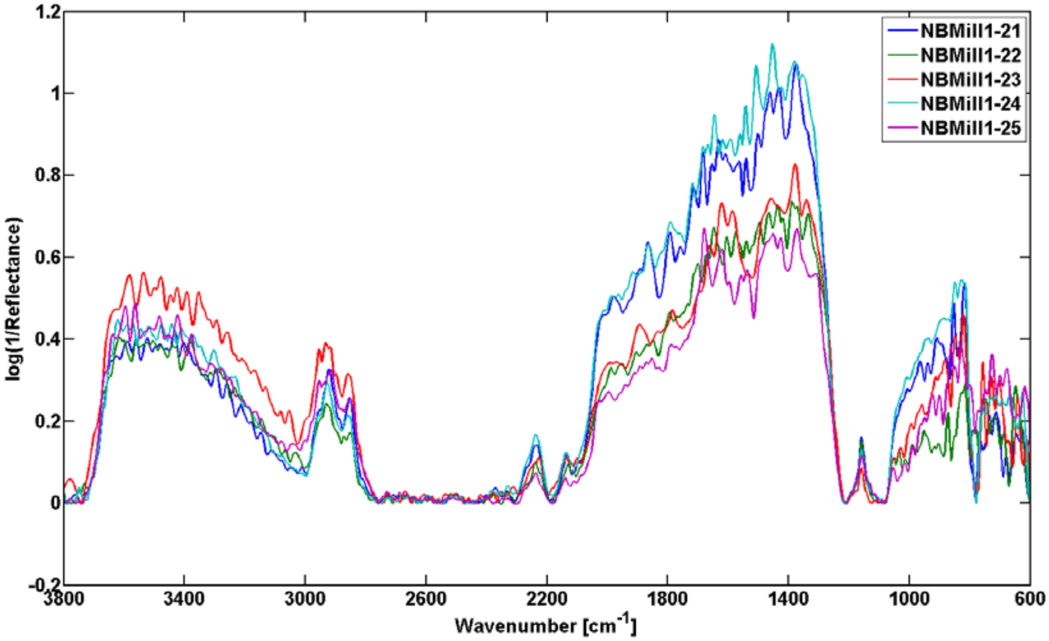

Figure C.17. DRIFTS Reflectance spectra of state-milled RAP (after 1-2 years of storage) from the inside of a stockpile in New Britain, Connecticut.

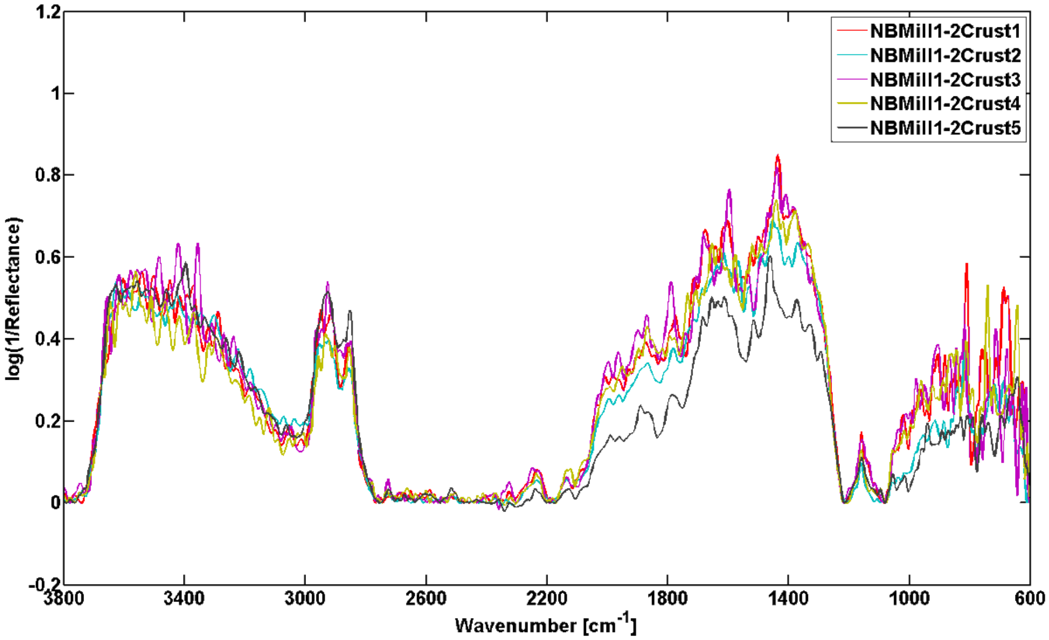

Figure C.18. DRIFTS Reflectance spectra of state-milled RAP (after 1-2 years of storage) from the top of a stockpile in New Britain, Connecticut.

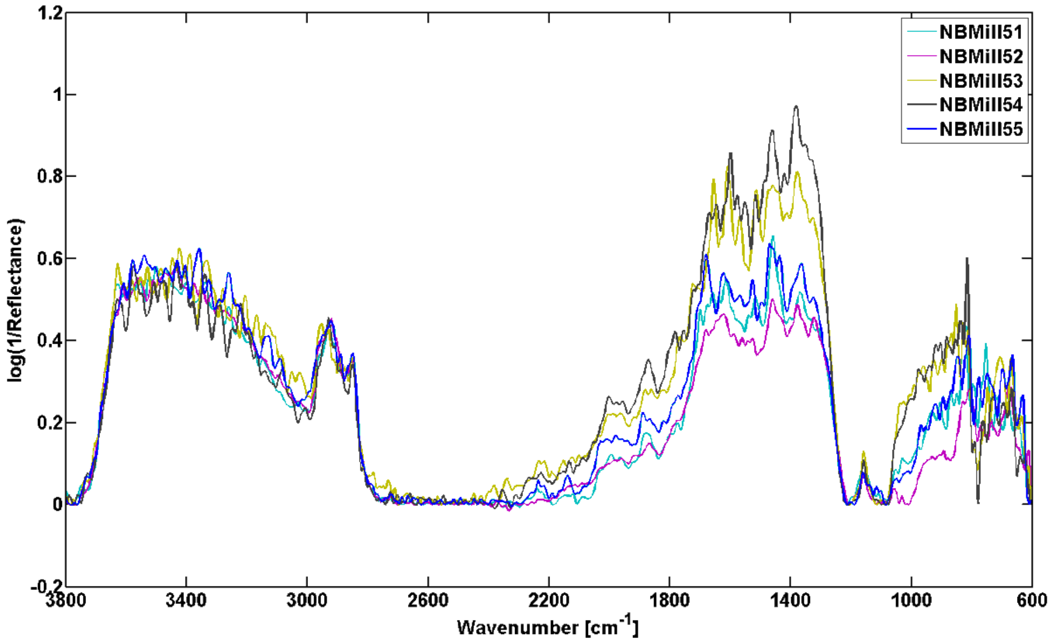

Figure C.19. DRIFTS Reflectance spectra of state-milled RAP (after 5 years of storage) from the inside of a stockpile in New Britain, Connecticut.

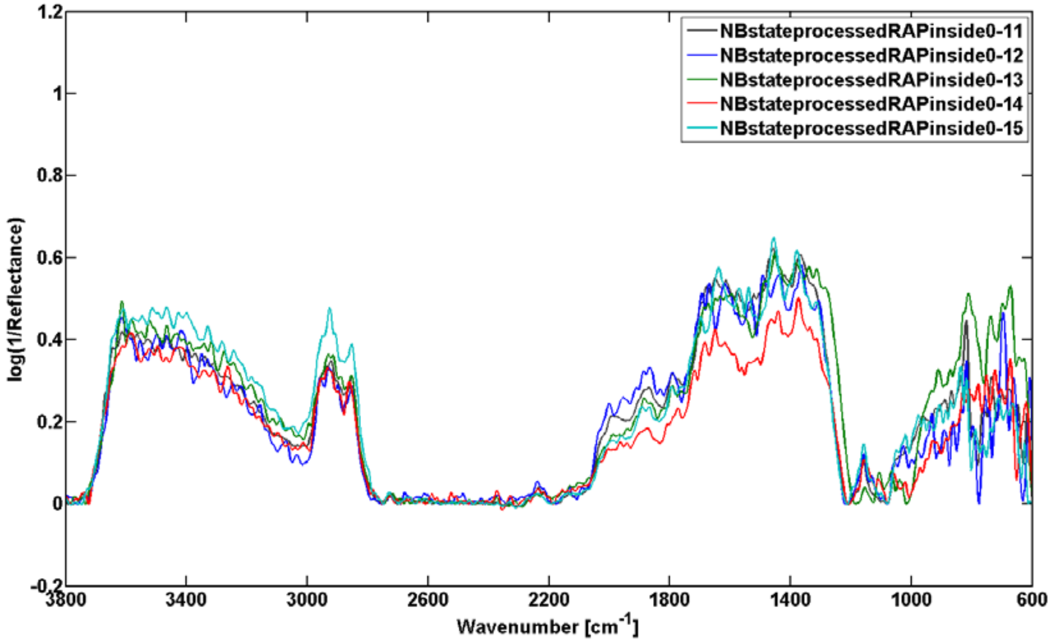

Figure C.20. DRIFTS Reflectance spectra of state-processed RAP from the inside of a stockpile in New Britain, Connecticut.

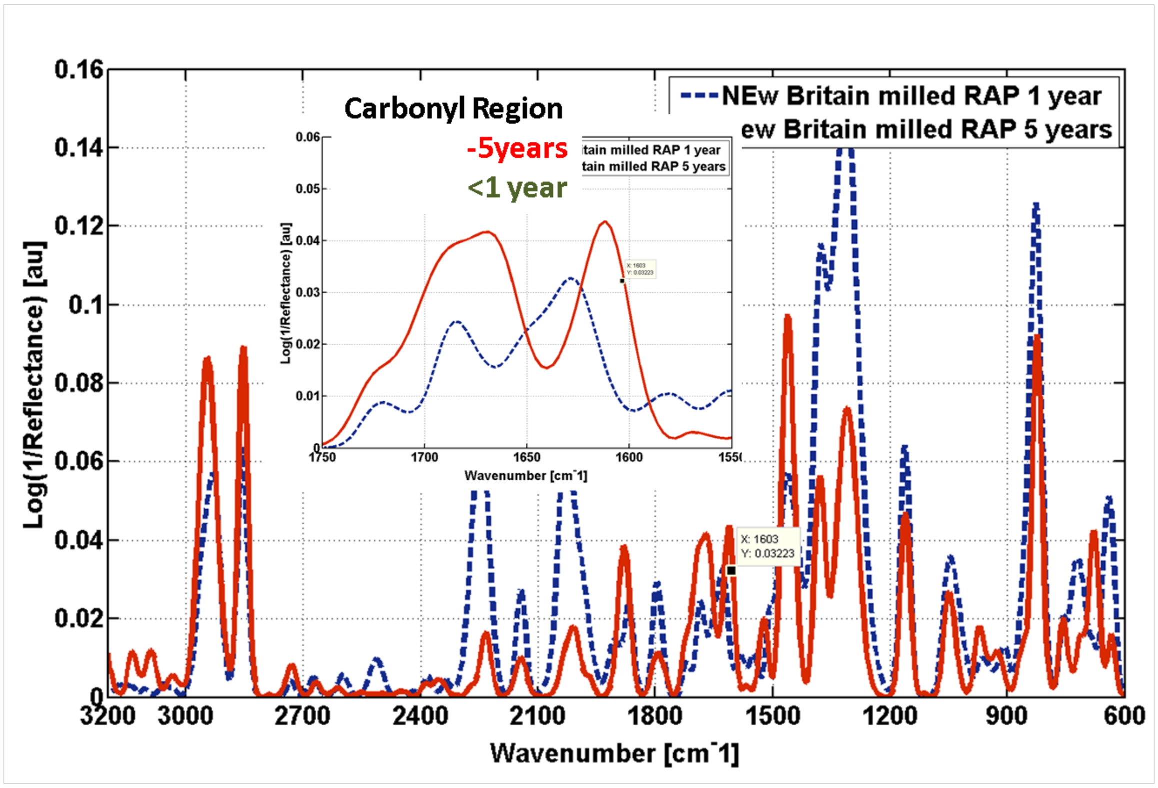

Figure C.21. Log(1/Reflectance) spectra of milled RAP after 1 and 5 years of storage in New Britain, Connecticut.

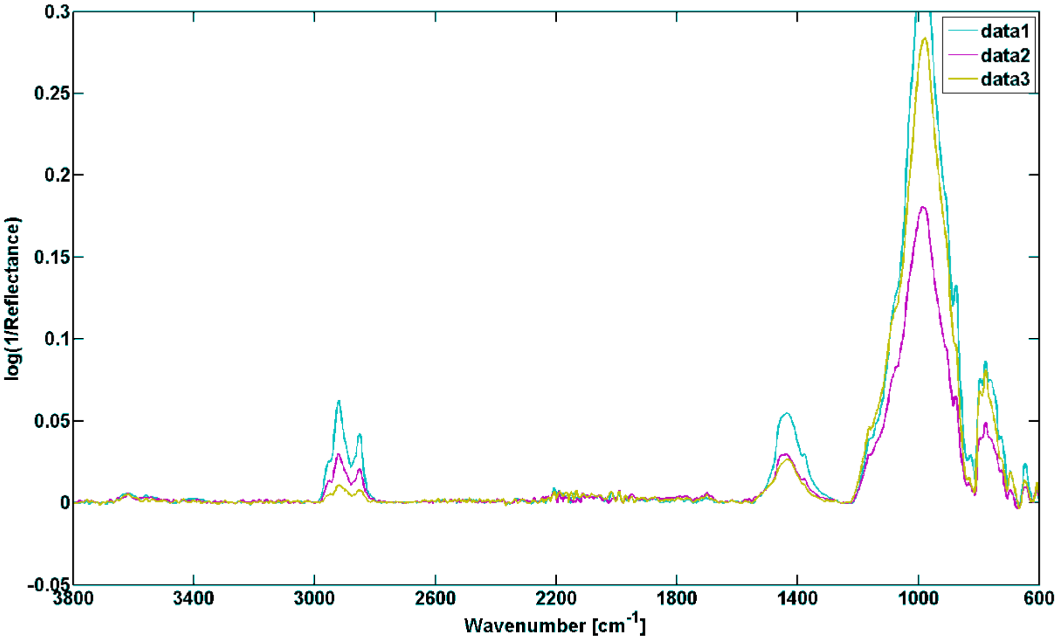

Figure C.22. ATR spectrum of Class II RAP from Smirna, Maine.

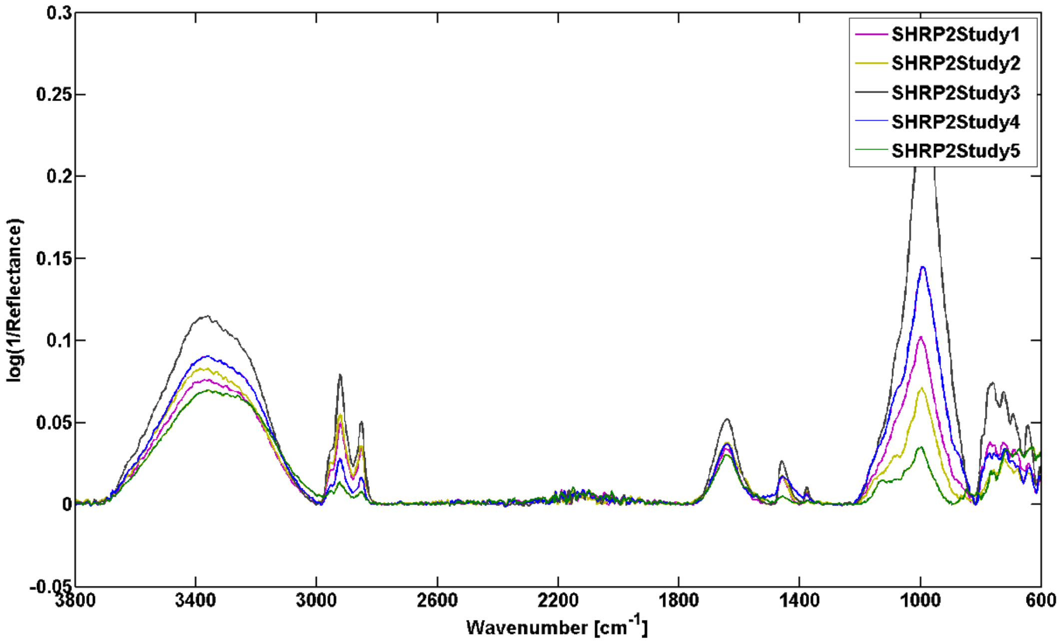

Figure C.23. ATR spectrum of Class I RAP from Poland, Maine.

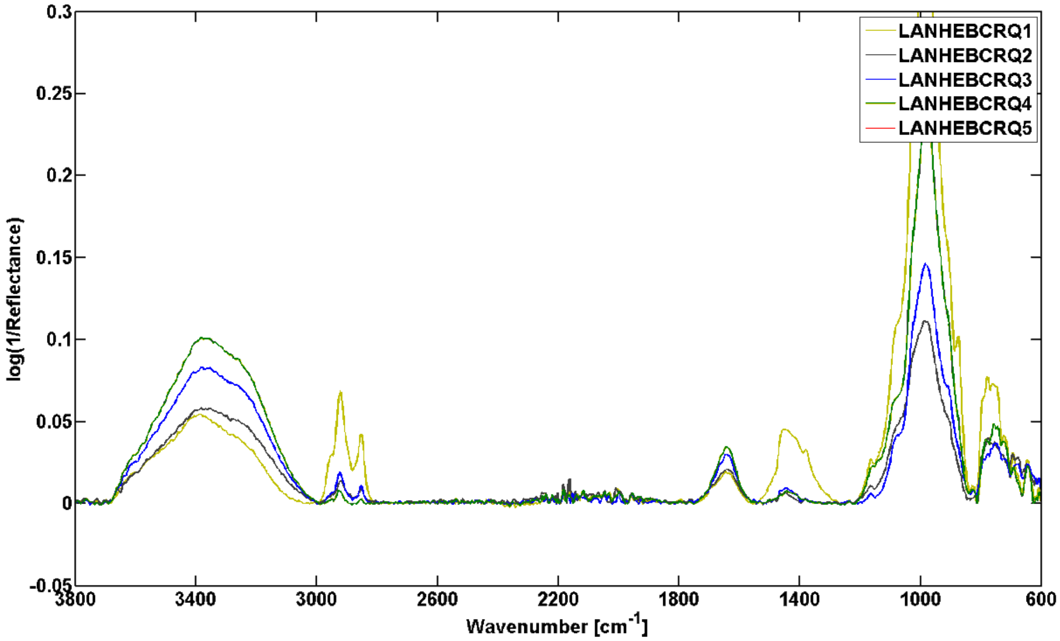

Figure C.24. ATR spectrum of Class III RAP from Washington, Maine.

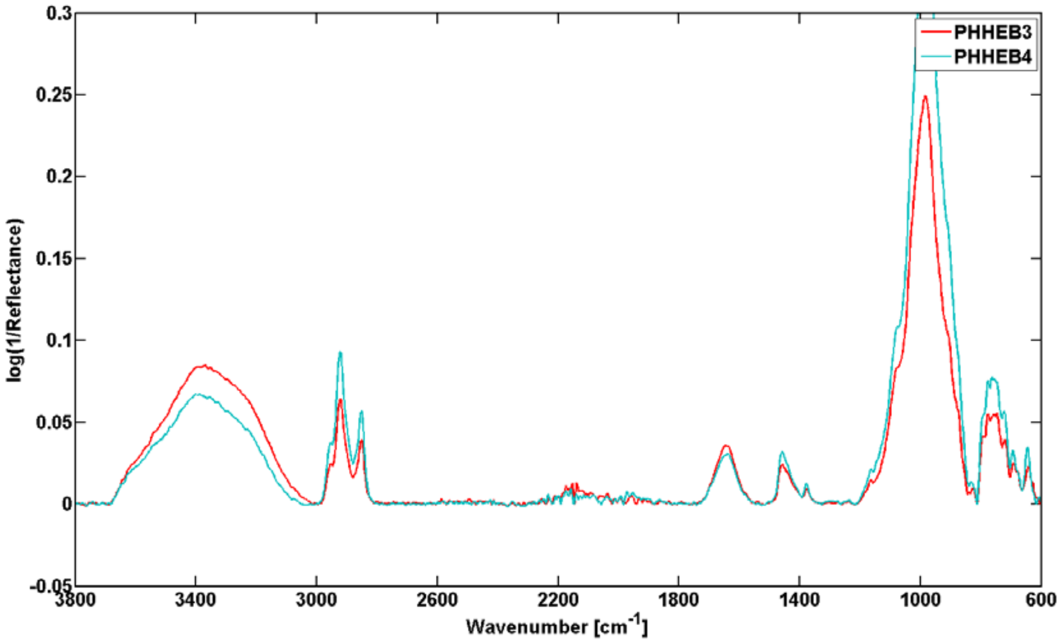

Figure C.25. ATR spectrum of Class I RAP from Hampden, Maine.

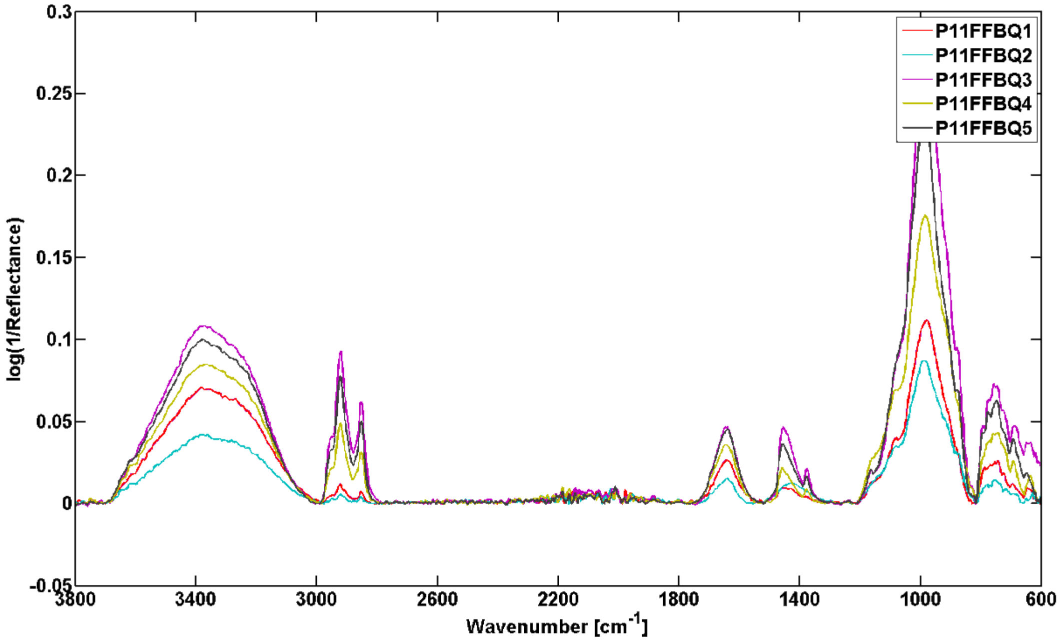

Figure C.26. ATR spectrum of Class II RAP from Fairfield, Maine.

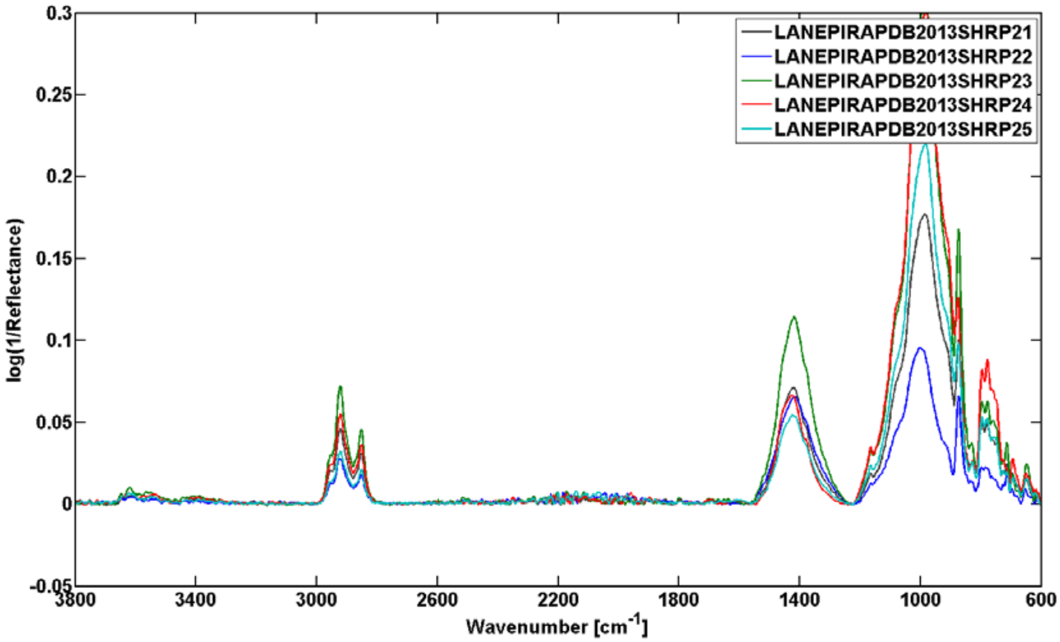

Figure C.27. ATR spectrum of Class II RAP from Westbrook, Maine.

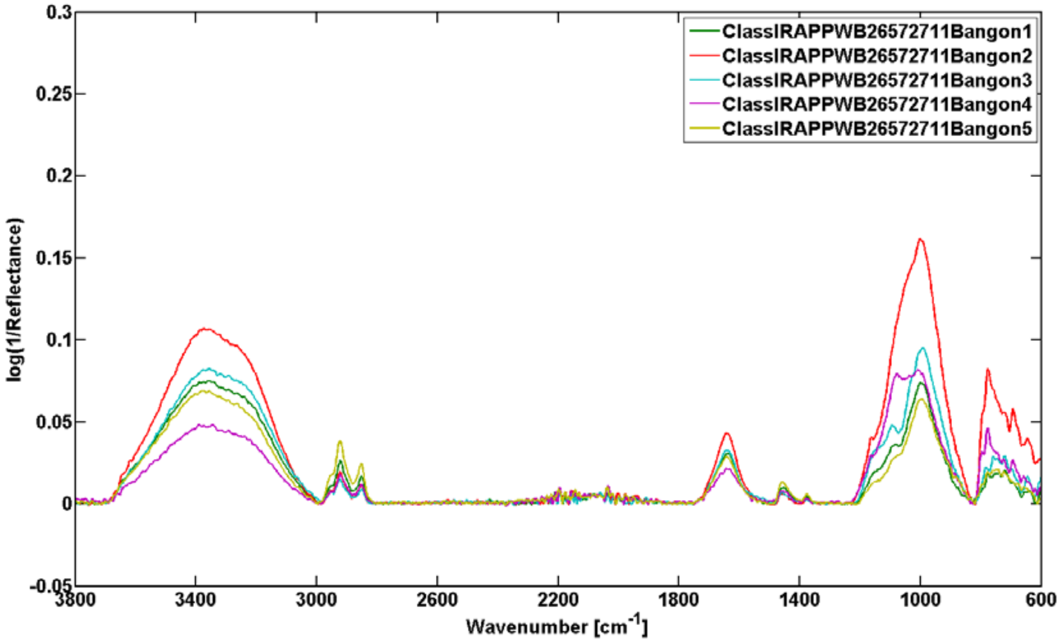

Figure C.28. ATR spectrum of Class I RAP from Westbrook, Maine.

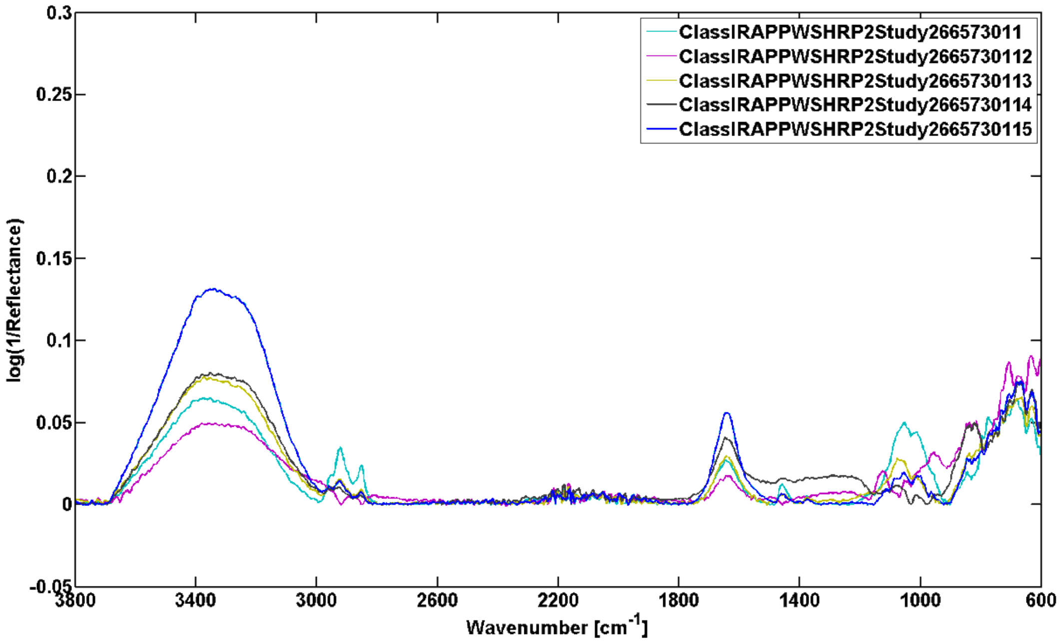

Figure C.29. ATR spectrum of Class I RAP from Wells, Maine.

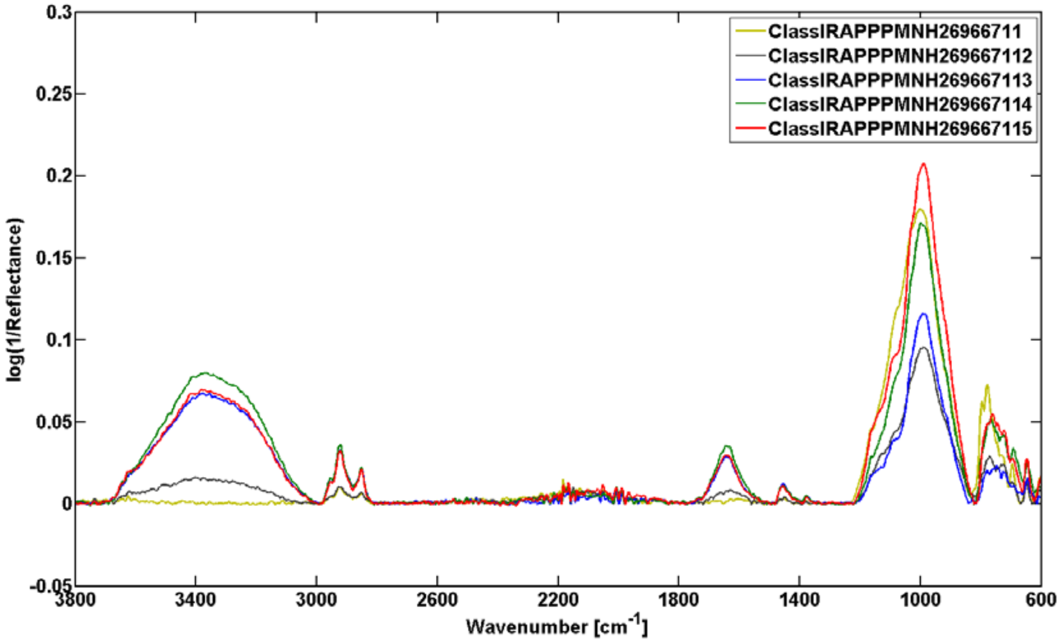

Figure C.30. ATR spectrum of Class I RAP from Portsmouth, New Hampshire.

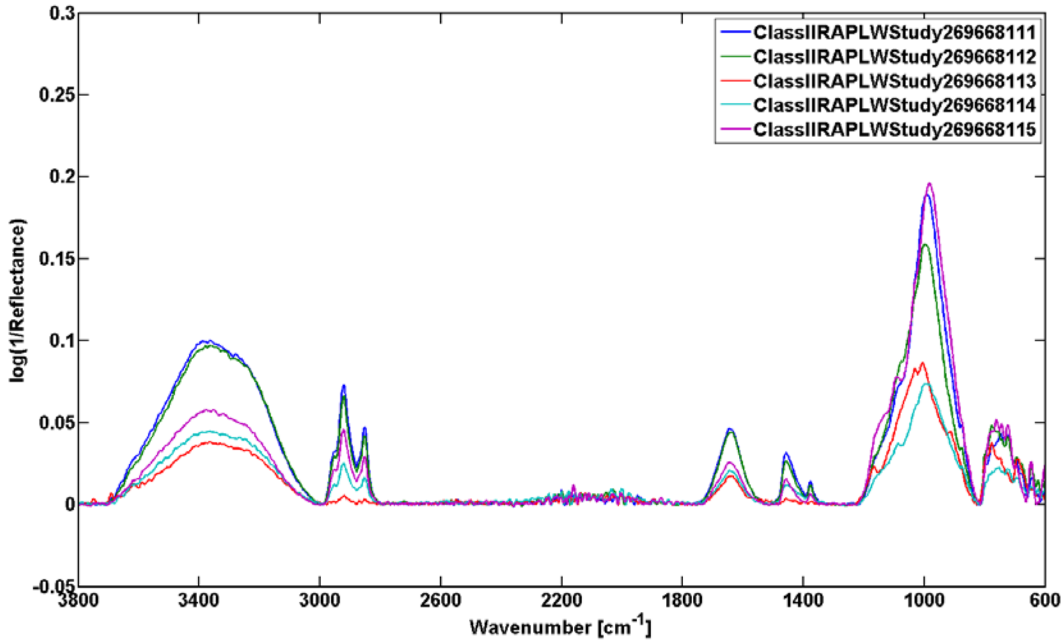

Figure C.31. ATR spectrum of Class II commercial RAP from Presque Isle, Maine.

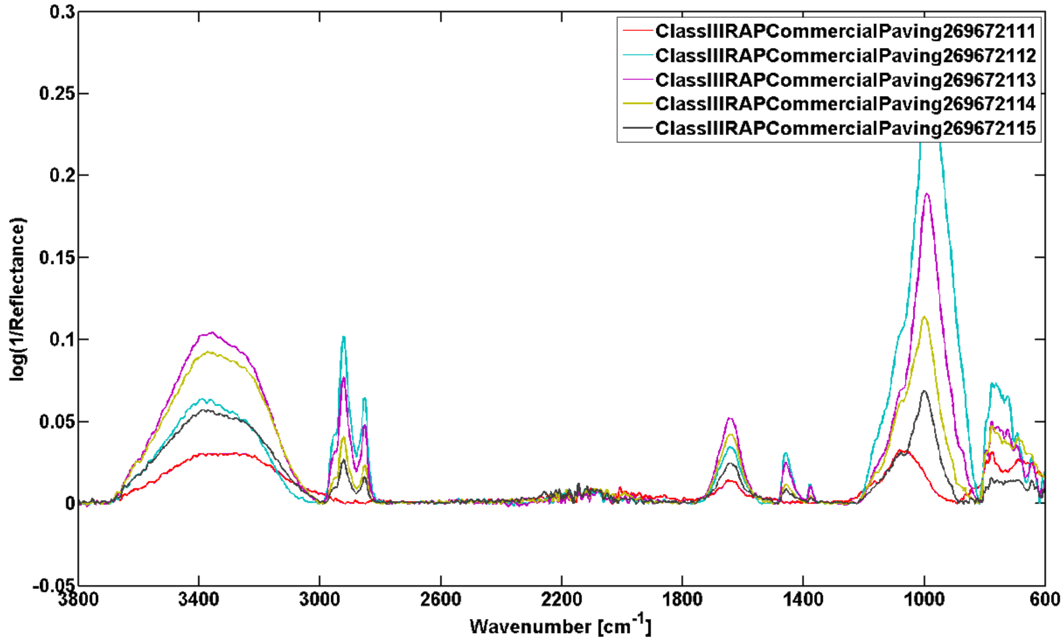

Figure C.32. ATR spectrum of Class II RAP from Dayton, Maine.

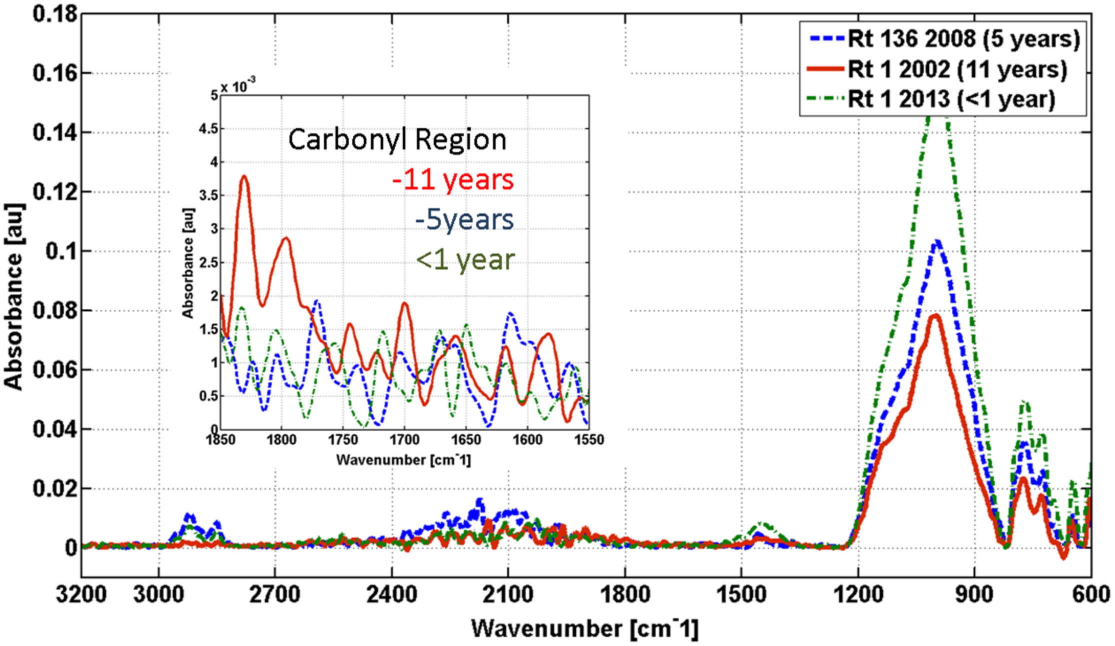

Figure C.33. IR Absorbance spectra of in situ HMA from pavement surfaces of different ages.

Table C.1. Lab XRF Results of Liquid Paint Samples Received by CTDOT (All Data in wt.%)

Liquid Paint Sample	Light Elements	LE +/-	Ti	Ti +/-	Cr	Fe	Fe +/-	Zn	As	Zr	Pb
Epoplex LS-50 white CTDOT	71.21	0.25	28.75	0.23	ND	ND		ND	ND	0.024	ND
Epoplex LS-50 white CTDOT	71.17	0.25	28.80	0.23	ND	ND		ND	ND	0.023	ND
Epoplex LS-50 yellow CTDOT	94.68	0.04	5.31	0.11	ND	ND		ND	ND	0.002	ND
Epoplex LS-50 yellow CTDOT	94.57	0.04	5.41	0.11	ND	ND		ND	ND	0.002	ND
DM3062AM 21STM2367 yellow CTDOT	97.39	0.03	2.13	0.11	ND	0.46	0.01	ND	ND	0.010	ND
DM3062AM 21STM2367 yellow CTDOT	97.61	0.03	1.96	0.10	ND	0.41	0.01	ND	ND	0.010	ND
Ennis latex paint CTY-21-M-2	95.98	0.05	3.66	0.12	ND	0.33	0.01	ND	ND	0.012	0.0074
Ennis latex paint CTY-21-M-2	95.99	0.05	3.63	0.13	ND	0.34	0.01	ND	ND	0.012	0.0061
Ennis latex white CTW-21-M-1	93.33	0.08	6.57	0.17	ND	0.06	0.09	ND	ND	0.021	ND
Ennis latex white CTW-21-M-1	92.92	0.09	6.96	0.18	ND	0.08	0.01	ND	ND	0.02	ND

Table C.2. Lab XRF Results of Dried Paint on Metal Plate Samples by CTDOT (All Data in wt.%)

Paint Dried on Metal Plate Sample	Light Elements	LE +/-	Ti	Ti +/-	Cr	Cr +/-	Fe	Fe +/-	Zn	Zn +/-	As	As +/-	Zr	Zr +/-	Pb	Pb +/-
White painted metal plate with beads	79.97	0.20	19.92	0.24	ND		0.07	0.008	ND		ND		0.03	0.001	ND	
White painted metal plate with beads	79.76	0.20	20.12	0.23	ND		0.08	0.09	ND		ND		0.03	0.001	ND	
Back side of metal plate	94.68	0.07	0.40	0.04	2.21	0.05	0.11	0.01	0.03	0.002	0.31	0.013	ND		2.08	0.01
Back side of metal plate	94.64	0.0	0.35	0.04	2.25	0.05	0.11	0.01	0.03	0.002	0.34	0.014	ND		2.10	0.01

Table C.3. Lab XRF Results of Dried Paint on Glass Bead Samples by CTDOT (All Data in wt.%)

Bead Sample	K	K +/-	Ca	Ca +/-	Ti	Ti +/-	Mn	Mn +/-	Fe	Fe +/-	Zn	Zn +/-	As	As +/-	Ba	Ba +/-	Pb	Pb +/-
TTB 1325D CTDOT	614	156	48820	858	ND	126	22	6	787	20	ND	10	ND	7	ND	30	ND	8
Type1 181B54 CTDOT	910	138	49098	709	ND	112	33	6	915	18	188	7	ND	6	ND	10	ND	6
M247-09 Type 3 large	1061	183	97893	1459	ND	140	40	6	579	16	14	3	ND	6	ND	30	ND	7
TT-B-1325 Type 3 airport beads	ND	1336	36615	1006	210356	4680	ND	108	ND	43	ND	29	ND	17	ND	1990	ND	20
TTB-1325D Type3 airport CTDOT	ND	1245	39638	1039	199242	4382	ND	103	ND	43	ND	25	ND	18	ND	1898	ND	20
Type1 181 B5 3 CTDOT	879	133	47872	679	ND	98	29	5	846	17	22	3	ND	5	ND	24	ND	6
Type3 large 247-09 CTDOT	768	181	99503	1500	ND	144	45	7	587	16	14	3	ND	6	ND	31	ND	7
Type1 TTB 1325D CTDOT	711	134	49248	707	ND	101	37	6	747	16	ND	8	ND	5	ND	24	ND	6

Table C.4. Lab XRF Results of Dried Paint on Various Metallic Samples by CTDOT (1) (All Data in wt.%)

Metallic Sample	LE	LE +/-	Ti	Ti +/-	Cr	Cr +/-	Mn	Mn +/-	Fe	Fe +/-	Co	Co +/-
Large screw	8.74	0.50	ND		ND		ND		0.12	0.01	ND	
Large screw	4.93	0.29	ND		ND		ND		0.17	0.01	ND	
X19MM rebar (#6) - rusted	15.92	0.65	ND		0.06	0.01	0.62	0.03	83.01	0.21	ND	
X19MM rebar (#6) - rusted	12.59	0.54	ND		0.04	0.01	0.61	0.03	86.39	0.22	ND	
X16MM rebar (#5) - green coating	50.25	1.46	5.26	0.12	0.07	0.02	0.23	0.02	43.51	0.16	0.42	0.04
X16MM rebar (#5) - green coating	51.09	1.47	4.93	0.12	0.07	0.02	0.28	0.02	43.00	0.16	0.39	0.04

Table C.5. Lab XRF results of dried paint on various metallic samples by CTDOT (2) (all data in wt.%)

Metallic Sample	Ni	Ni +/-	Cu	Cu +/-	Zn	Zn +/-	As	As +/-	Mo	Mo +/-	Pb
Large screw	ND		ND		90.95	0.18	ND		ND		ND
Large screw	ND		ND		94.68	0.19	ND		ND		ND
X19MM rebar (#6) - rusted	0.10	0.02	0.26	0.02	ND		ND		0.014	0.002	ND
X19MM rebar (#6) - rusted	ND		0.34	0.02	ND		ND		0.015	0.002	ND
X16MM rebar (#5) - green coating	ND		0.22	0.01	ND		ND		0.014	0.001	ND
X16MM rebar (#5) - green coating	ND		0.20	0.01	ND		ND		0.015	0.002	ND

Table C.6. Lab XRF Results for Paint Dilution Tests with Water (All Data in wt.%)

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
DM3062AM 21STM 2367 yellow CTDOT	1:1	99.60	0.01	0.25	0.04			0.14	0.01	0.0067	0.0005		
	1:1	99.60	0.01	0.22	0.04			0.17	0.01	0.0087	0.0006		
	1:1	99.63	0.00	0.22	0.04			0.14	0.01	0.0078	0.0006		
	1:1	99.63	0.01	0.18	0.04			0.19	0.01	0.0096	0.0006		
	1:1	99.54	0.01	0.25	0.04	0.22	0.00	0.19	0.01	0.0115	0.0007		
	2:1	99.34	0.01	0.45	0.05			0.20	0.01	0.0088	0.0006		
	2:1	99.38	0.01	0.42	0.05			0.20	0.01	0.0107	0.0007		
	2:1	99.32	0.01	0.45	0.06			0.22	0.01	0.012	0.0007		
	2:1	99.25	0.01	0.53	0.06			0.21	0.01	0.0109	0.0007		
	2:1	99.29	0.01	0.48	0.05	0.47	0.00	0.22	0.01	0.0113	0.0006		
	1:2	99.71	0.00	0.18	0.03			0.10	0.01	0.0036	0.0004		
	1:2	99.73	0.00	0.16	0.03			0.11	0.01	0.0039	0.0004		
	1:2	99.86	0.00	0.08	0.02			0.05	0.01	0.0031	0.0004		
	1:2	99.86	0.00	0.08	0.02			0.06	0.01	0.0037	0.0004		
	1:2	99.82	0.00	0.09	0.02	0.12	0.00	0.08	0.01	0.0031	0.0004		
	3:1	99.01	0.01	0.79	0.06			0.19	0.01	0.0089	0.0006		
	3:1	99.04	0.01	0.70	0.06			0.25	0.01	0.0096	0.0006		
	3:1	98.92	0.01	0.78	0.06			0.30	0.01	0.0086	0.0006		
	3:1	99.03	0.01	0.68	0.06			0.28	0.01	0.0094	0.0006		
	3:1	99.07	0.01	0.66	0.06	0.72	0.00	0.26	0.01	0.0091	0.0006		

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
	1:3	99.78	0.00	0.13	0.03			0.08	0.01	0.0045	0.0004		
	1:3	99.79	0.00	0.12	0.03			0.08	0.01	0.0047	0.0004		
	1:3	99.73	0.00	0.18	0.03			0.09	0.01	0.0041	0.0004		
	1:3	99.79	0.00	0.12	0.03			0.09	0.01	0.0045	0.0004		
	1:3	99.69	0.00	0.21	0.03	0.15	0.00	0.10	0.01	0.0044	0.0004		
Ennis latex paint, CTY 21M2	1:1	99.32	0.01	0.45	0.05			0.22	0.01	0.0048	0.0005		
	1:1	99.23	0.01	0.50	0.05			0.27	0.01	0.0051	0.0005		
	1:1	99.01	0.01	0.63	0.06			0.36	0.01	0.0051	0.0005		
	1:1	99.40	0.01	0.37	0.04			0.23	0.01	0.0059	0.0005		
	1:1	99.19	0.01	0.54	0.06	0.50	0.01	0.27	0.01	0.0054	0.0006		
	2:1	99.05	0.01	0.70	0.06			0.24	0.01	0.0071	0.0006	0.0031	0.0008
	2:1	98.92	0.02	0.79	0.07			0.28	0.01	0.0069	0.0006	0.0026	0.0008
	2:1	98.63	0.02	1.02	0.07			0.34	0.02	0.0075	0.0006	0.0027	0.0008
	2:1	98.52	0.02	1.13	0.08			0.34	0.02	0.006	0.0006		
	2:1	98.79	0.02	0.94	0.07	0.92	0.01	0.26	0.01	0.0081	0.0006	0.0032	0.0008
	1:2	99.34	0.01	0.57	0.04			0.09	0.01	0.0055	0.0004	0.0022	0.0005
	1:2	99.34	0.01	0.55	0.04			0.10	0.01	0.005	0.0004	0.002	0.0005
	1:2	99.54	0.00	0.37	0.03			0.09	0.01	0.0045	0.0004	0.0021	0.0005
	1:2	99.31	0.01	0.58	0.04			0.10	0.01	0.0066	0.0004	0.0028	0.0006
	1:2	99.33	0.01	0.57	0.04	0.53	0.00	0.09	0.01	0.0056	0.0004	0.0028	0.0006
	3:1	99.74	0.00	0.15	0.03			0.11	0.01	0.0035	0.0004		
	3:1	99.78	0.00	0.08	0.02			0.14	0.01	0.0035	0.0004		
	3:1	99.70	0.00	0.17	0.03			0.12	0.01	0.0034	0.0004		
	3:1	99.72	0.00	0.15	0.03			0.12	0.01	0.0038	0.0004		
	3:1	99.76	0.00	0.11	0.03	0.13	0.00	0.12	0.01	0.0031	0.0004		
	1:3	98.53	0.02	1.23	0.08			0.23	0.01	0.0084	0.0006	0.0025	0.0008
	1:3	98.51	0.02	1.20	0.07			0.28	0.01	0.0075	0.0006	0.0045	0.0008
	1:3	98.39	0.02	1.33	0.08			0.26	0.01	0.0077	0.0006	0.003	0.0008
	1:3	98.43	0.02	1.29	0.08			0.27	0.01	0.0088	0.0006	0.0045	0.0008
	1:3	98.41	0.02	1.32	0.08	1.27	0.00	0.26	0.01	0.01	0.0006	0.0026	0.0007
Ennis latex white CTW-	1:1	99.00	0.01	0.84	0.06			0.15	0.01	0.0081	0.0006		
	1:1	98.76	0.02	1.04	0.07			0.20	0.01	0.0071	0.0006		

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
21-M-1	1:1	98.59	0.02	1.20	0.07			0.20	0.01	0.009	0.0006		
	1:1	98.92	0.01	0.93	0.06			0.14	0.01	0.0091	0.0006		
	1:1	98.68	0.02	1.15	0.07	1.03	0.01	0.16	0.01	0.0099	0.0006		
	2:1	98.07	0.03	1.79	0.09			0.13	0.01	0.0121	0.0007		
	2:1	97.36	0.03	2.48	0.11			0.15	0.01	0.0116	0.0007		
	2:1	97.85	0.03	1.98	0.09			0.16	0.01	0.0119	0.0006		
	2:1	97.70	0.03	2.17	0.10			0.13	0.01	0.0118	0.0006		
	2:1	97.65	0.03	2.20	0.10	2.12	0.01	0.14	0.01	0.0133	0.0007		
	3:1	95.92	0.05	3.96	0.13			0.10	0.01	0.0142	0.0007		
	3:1	95.68	0.05	4.19	0.13			0.12	0.01	0.014	0.0007		
	3:1	95.54	0.05	4.35	0.13			0.09	0.01	0.0155	0.0007		
	3:1	95.60	0.05	4.29	0.13			0.09	0.01	0.0152	0.0007		
	3:1	95.79	0.05	4.11	0.13	4.18	0.00	0.09	0.01	0.0152	0.0007		
	2:1	97.58	0.03	2.25	0.10			0.15	0.01	0.0127	0.0006		
	2:1	97.69	0.03	2.15	0.09			0.15	0.01	0.0124	0.0006		
	2:1	97.63	0.03	2.20	0.10			0.16	0.01	0.0133	0.0006		
	2:1	97.68	0.03	2.16	0.09			0.15	0.01	0.0133	0.0006		
	2:1	97.57	0.03	2.26	0.10	2.20	0.00	0.15	0.01	0.0126	0.0006		
	1:2	99.48	0.01	0.41	0.04			0.11	0.01	0.0063	0.0005		
	1:2	99.48	0.01	0.41	0.04			0.11	0.01	0.0071	0.0005		
	1:2	99.42	0.01	0.47	0.05			0.10	0.01	0.0068	0.0005		
	1:2	99.46	0.01	0.43	0.04			0.11	0.01	0.0067	0.0005		
	1:2	99.48	0.01	0.39	0.04	0.42	0.00	0.12	0.01	0.0066	0.0005		
	1:3	99.70	0.00	0.20	0.03			0.09	0.01	0.0039	0.0004		
	1:3	99.62	0.00	0.26	0.04			0.11	0.01	0.0043	0.0004		
	1:3	99.64	0.00	0.26	0.03			0.10	0.01	0.0035	0.0004		
	1:3	99.64	0.00	0.27	0.04			0.09	0.01	0.0033	0.0004		
	1:3	99.68	0.00	0.22	0.03	0.24	0.00	0.10	0.01	0.0043	0.0004		
Ennis latex paint CTY 21M2 (yellow)	1:1	99.30	0.01	0.48	0.05			0.21	0.01	0.0047	0.0005		
	1:1	99.45	0.01	0.34	0.04			0.20	0.01	0.0056	0.0005		
	1:1	99.44	0.01	0.35	0.04			0.20	0.01	0.0058	0.0005		
	1:1	99.37	0.01	0.41	0.04			0.21	0.01	0.0049	0.0005		

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
	1:1	99.32	0.01	0.42	0.04	0.40	0.00	0.25	0.01	0.0058	0.0005		
	2:1	98.80	0.02	0.90	0.07			0.28	0.01	0.0081	0.0006	0.0027	0.0008
	2:1	98.96	0.01	0.74	0.06			0.28	0.01	0.0072	0.0006	0.0045	0.0008
	2:1	98.96	0.01	0.75	0.06			0.28	0.01	0.008	0.0006		
	2:1	98.92	0.01	0.83	0.06			0.24	0.01	0.0067	0.0006	0.0038	0.0008
	2:1	98.80	0.02	0.94	0.07	0.83	0.00	0.26	0.01	0.0076	0.0006		
	3:1	98.23	0.02	1.42	0.08			0.34	0.01	0.0101	0.0006	0.0038	0.0008
	3:1	98.39	0.02	1.31	0.08			0.29	0.01	0.0081	0.0006	0.0031	0.0008
	3:1	98.45	0.02	1.26	0.08			0.28	0.01	0.0085	0.0006	0.0031	0.0008
	3:1	98.25	0.02	1.33	0.08			0.41	0.02	0.0092	0.0006	0.0051	0.0009
	3:1	98.25	0.02	1.45	0.08	1.36	0.00	0.28	0.01	0.0106	0.0006	0.0039	0.0008
	1:2	99.61	0.00	0.22	0.03			0.17	0.01	0.0046	0.0005	0.0019	0.0006
	1:2	99.63	0.00	0.24	0.04			0.13	0.01	0.0042	0.0005		
	1:2	99.60	0.00	0.25	0.03			0.15	0.01	0.0041	0.0005		
	1:2	99.58	0.00	0.27	0.04			0.14	0.01	0.004	0.0005		
	1:2	99.62	0.00	0.22	0.03	0.24	0.00	0.15	0.01	0.004	0.0005		
	1:3	99.72	0.00	0.17	0.03			0.10	0.01	0.0052	0.0005	0.0019	0.0006
	1:3	99.71	0.00	0.16	0.03			0.13	0.01	0.0051	0.0005		
	1:3	99.68	0.00	0.19	0.03			0.13	0.01	0.0054	0.0005	0.0028	0.0006
	1:3	99.71	0.00	0.16	0.03			0.12	0.01	0.0044	0.0004		
	1:3	99.69	0.00	0.16	0.03	0.17	0.00	0.14	0.01	0.0049	0.0005	0.0023	0.0006
	1:3	99.71	0.00	0.15	0.03			0.13	0.01	0.0045	0.0004		
	1:3	99.64	0.00	0.23	0.03			0.13	0.01	0.0034	0.0004		
	1:3	99.67	0.00	0.20	0.03			0.13	0.01	0.0045	0.0004		
	1:3	99.73	0.00	0.15	0.03			0.12	0.01	0.0044	0.0004		
	1:3	99.77	0.00	0.12	0.03	0.17	0.00	0.11	0.01	0.0049	0.0005		
DM3062AM 21STM 2367 yellow CTDOT	1:1	99.48	0.01	0.35	0.04			0.16	0.01	0.0076	0.0006		
	1:1	99.59	0.01	0.26	0.04			0.15	0.01	0.0068	0.0005		
	1:1	99.59	0.01	0.26	0.04			0.14	0.01	0.0081	0.0006		
	1:1	99.48	0.01	0.39	0.04			0.12	0.01	0.0079	0.0005		
	1:1	99.53	0.01	0.31	0.04	0.31	0.00	0.15	0.01	0.0082	0.0006		
	2:1	99.42	0.01	0.37	0.05			0.20	0.01	0.0084	0.0006		

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
	2:1	99.45	0.01	0.34	0.05			0.20	0.01	0.0088	0.0006		
	2:1	99.35	0.01	0.44	0.05			0.20	0.01	0.0088	0.0006		
	2:1	99.43	0.01	0.38	0.05			0.18	0.01	0.0079	0.0006		
	2:1	99.42	0.01	0.38	0.05	0.38	0.00	0.19	0.01	0.0076	0.0006		
	3:1	99.10	0.01	0.64	0.06			0.25	0.01	0.0092	0.0006		
	3:1	99.04	0.01	0.70	0.06			0.25	0.01	0.0102	0.0006		
	3:1	98.99	0.01	0.72	0.06			0.28	0.01	0.0103	0.0006		
	3:1	99.03	0.01	0.68	0.06			0.28	0.01	0.0095	0.0006		
	3:1	98.98	0.01	0.75	0.06	0.70	0.00	0.27	0.01	0.0099	0.0006		
	1:1	99.59	0.01	0.25	0.04			0.15	0.01	0.0069	0.0005		
	1:1	99.60	0.01	0.26	0.04			0.14	0.01	0.009	0.0006		
	1:1	99.67	0.00	0.19	0.04			0.12	0.01	0.0081	0.0006		
	1:1	99.49	0.01	0.31	0.04			0.19	0.01	0.0088	0.0006		
	1:1	99.48	0.01	0.37	0.05	0.28	0.00	0.14	0.01	0.0084	0.0006		
	1:2	99.76	0.00	0.12	0.03			0.11	0.01	0.0049	0.0005		
	1:2	99.73	0.00	0.15	0.03			0.11	0.01	0.0051	0.0005		
	1:2	99.72	0.00	0.16	0.03			0.11	0.01	0.0052	0.0005		
	1:2	99.74	0.00	0.14	0.03			0.11	0.01	0.0053	0.0005		
	1:2	99.77	0.00	0.11	0.03	0.14	0.00	0.11	0.01	0.0054	0.0005		
	1:3	99.80	0.00	0.10	0.03			0.09	0.01	0.0059	0.0005		
	1:3	99.76	0.00	0.14	0.03			0.10	0.01	0.0053	0.0005		
	1:3	99.75	0.00	0.14	0.03			0.10	0.01	0.0051	0.0005		
	1:3	99.84	0.00	0.08	0.03			0.08	0.01	0.0057	0.0005		
	1:3	99.78	0.00	0.12	0.03	0.12	0.00	0.09	0.01	0.0065	0.0005		
	1:3	99.85	0.00	0.08	0.03			0.07	0.01	0.0043	0.0004		
	1:3	99.91	0.00					0.09	0.01	0.0041	0.0004		
	1:3	99.91	0.00					0.09	0.01	0.0041	0.0004		
	1:3	99.78	0.00	0.13	0.03			0.09	0.01	0.0035	0.0004		
	1:3	99.82	0.00	0.11	0.03	0.10	0.00	0.07	0.01	0.0041	0.0004		
Ennis latex white CTW-21-M-1	1:1	99.01	0.01	0.84	0.06			0.14	0.01	0.0088	0.0006		
	1:1	98.94	0.01	0.90	0.06			0.16	0.01	0.01	0.0006		
	1:1	98.86	0.01	0.97	0.07			0.16	0.01	0.0091	0.0006		

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
	1:1	98.94	0.01	0.91	0.06			0.14	0.01	0.0087	0.0006		
	1:1	99.08	0.01	0.76	0.06	0.88	0.00	0.15	0.01	0.0093	0.0006		
	1:1	98.91	0.01	0.95	0.06			0.14	0.01	0.0082	0.0006		
	1:1	98.95	0.01	0.88	0.06			0.17	0.01	0.0082	0.0006		
	1:1	98.72	0.02	1.13	0.07			0.15	0.01	0.0083	0.0006		
	1:1	98.78	0.02	1.08	0.07			0.13	0.01	0.0079	0.0006		
	1:1	98.94	0.01	0.90	0.06	0.99	0.00	0.16	0.01	0.0086	0.0006		
	2:1	97.76	0.03	2.06	0.09			0.16	0.01	0.0127	0.0007		
	2:1	97.61	0.03	2.23	0.10			0.15	0.01	0.012	0.0006		
	2:1	97.58	0.03	2.24	0.10			0.16	0.01	0.0123	0.0006		
	2:1	97.64	0.03	2.19	0.10			0.15	0.01	0.0133	0.0007		
	2:1	97.68	0.03	2.16	0.10	2.18	0.00	0.15	0.01	0.0138	0.0007		
	3:1	95.85	0.05	4.05	0.13			0.09	0.01	0.0159	0.0007		
	3:1	95.90	0.05	4.02	0.13			0.07	0.01	0.0135	0.0007		
	3:1	95.46	0.06	4.42	0.13			0.11	0.01	0.0148	0.0007		
	3:1	95.71	0.05	4.16	0.13			0.11	0.01	0.0157	0.0007		
	3:1	95.74	0.05	4.14	0.13	4.16	0.00	0.10	0.01	0.0144	0.0007		
	1:2	99.55	0.01	0.37	0.04			0.08	0.01	0.0065	0.0005		
	1:2	99.42	0.01	0.47	0.04			0.11	0.01	0.0061	0.0005		
	1:2	99.47	0.01	0.41	0.04			0.11	0.01	0.0064	0.0005		
	1:2	99.49	0.01	0.38	0.04			0.12	0.01	0.0055	0.0005		
	1:2	99.43	0.01	0.45	0.04	0.42	0.00	0.12	0.01	0.0064	0.0005		
	1:3	99.66	0.00	0.25	0.03			0.08	0.01	0.0068	0.0005		
	1:3	99.59	0.00	0.32	0.04			0.09	0.01	0.0057	0.0005		
	1:3	99.61	0.00	0.28	0.04			0.10	0.01	0.006	0.0005		
	1:3	99.62	0.00	0.28	0.04			0.10	0.01	0.0056	0.0005		
	1:3	99.57	0.00	0.32	0.04	0.29	0.00	0.10	0.01	0.0063	0.0005		
DM3062AM 21STM 2367 yellow CTDOT	1:1	99.35	0.01	0.42	0.05			0.21	0.01	0.0074	0.0006		
	1:1	99.52	0.01	0.28	0.04			0.20	0.01	0.0083	0.0006		
	1:1	99.60	0.01	0.23	0.04			0.16	0.01	0.0083	0.0006		
	1:1	99.58	0.01	0.27	0.04			0.14	0.01	0.0072	0.0006		
	1:1	99.60	0.01	0.20	0.04	0.28	0.00	0.18	0.01	0.0075	0.0006		

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
	2:1	99.31	0.01	0.46	0.05			0.21	0.01	0.009	0.0006		
	2:1	99.41	0.01	0.37	0.05			0.21	0.01	0.0098	0.0006		
	2:1	99.26	0.01	0.50	0.05			0.24	0.01	0.0082	0.0006		
	2:1	99.43	0.01	0.33	0.04			0.24	0.01	0.0091	0.0006		
	2:1	99.38	0.01	0.41	0.05	0.41	0.00	0.20	0.01	0.0083	0.0006		
	3:1	99.09	0.01	0.62	0.06			0.29	0.01	0.0079	0.0006		
	3:1	99.07	0.01	0.64	0.06			0.28	0.01	0.0087	0.0006		
	3:1	99.03	0.01	0.70	0.06			0.26	0.01	0.0092	0.0006		
	3:1	99.00	0.01	0.73	0.06			0.26	0.01	0.0089	0.0006		
	3:1	99.05	0.01	0.68	0.06	0.67	0.00	0.25	0.01	0.0088	0.0006		
	1:2	99.77	0.00	0.11	0.03			0.11	0.01	0.0059	0.0005		
	1:2	99.88	0.00					0.12	0.01	0.0055	0.0005		
	1:2	99.72	0.00	0.15	0.03			0.13	0.01	0.0057	0.0005		
	1:2	99.74	0.00	0.14	0.03			0.11	0.01	0.0058	0.0005		
	1:2	99.69	0.00	0.19	0.03	0.15	0.00	0.12	0.01	0.0061	0.0005		
	1:3	99.91	0.00					0.09	0.01	0.0043	0.0005		
	1:3	99.90	0.00					0.10	0.01	0.005	0.0005		
	1:3	99.76	0.00	0.13	0.03			0.10	0.01	0.0051	0.0005		
	1:3	99.82	0.00	0.09	0.03			0.08	0.01	0.0039	0.0004		
	1:3	99.81	0.00	0.09	0.03	0.10	0.00	0.10	0.01	0.004	0.0004		
Ennis latex white	1:1	98.81	0.01	1.03	0.07			0.15	0.01	0.0094	0.0006		
	1:1	98.89	0.01	0.96	0.07			0.15	0.01	0.0071	0.0005		
	1:1	98.86	0.01	0.97	0.07			0.16	0.01	0.0085	0.0006		
	1:1	98.94	0.01	0.89	0.06			0.16	0.01	0.0077	0.0006		
	1:1	98.79	0.02	1.05	0.07	0.98	0.00	0.15	0.01	0.0091	0.0006		
	1:1	98.98	0.01	0.86	0.06			0.15	0.01	0.0082	0.0006		
	1:1	98.83	0.01	1.01	0.07			0.15	0.01	0.0079	0.0006		
	1:1	98.83	0.01	1.00	0.07			0.16	0.01	0.0081	0.0006		
	1:1	98.90	0.01	0.94	0.07			0.15	0.01	0.0082	0.0006		
	1:1	98.74	0.02	1.09	0.07	0.98	0.00	0.15	0.01	0.0094	0.0006		
	1:1	98.93	0.01	0.91	0.06			0.15	0.01	0.009	0.0006		
	1:1	98.86	0.01	0.98	0.07			0.15	0.01	0.0085	0.0006		

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
	1:1	98.99	0.01	0.83	0.06			0.17	0.01	0.0084	0.0006		
	1:1	98.79	0.02	1.06	0.07			0.15	0.01	0.0082	0.0006		
	1:1	98.83	0.01	1.00	0.07	0.96	0.00	0.15	0.01	0.0084	0.0006		
	2:1	97.50	0.03	2.31	0.10			0.18	0.01	0.0133	0.0007		
	2:1	97.59	0.03	2.25	0.10			0.14	0.01	0.0142	0.0007		
	2:1	97.65	0.03	2.19	0.10			0.16	0.01	0.0134	0.0007		
	2:1	97.78	0.03	2.06	0.09			0.14	0.01	0.0133	0.0007		
	2:1	97.75	0.03	2.09	0.10	2.18	0.00	0.15	0.01	0.0121	0.0006		
	3:1	95.90	0.05	3.99	0.13			0.09	0.01	0.0164	0.0007		
	3:1	95.63	0.05	4.26	0.13			0.09	0.01	0.0153	0.0007		
	3:1	95.68	0.05	4.21	0.13			0.09	0.01	0.0149	0.0007		
	3:1	95.54	0.05	4.36	0.13			0.08	0.01	0.0143	0.0007		
	3:1	95.49	0.06	4.40	0.14	4.25	0.00	0.09	0.01	0.0155	0.0007		
	1:2	99.44	0.01	0.44	0.04			0.12	0.01	0.0058	0.0005		
	1:2	99.31	0.01	0.56	0.05			0.13	0.01	0.0051	0.0005		
	1:2	99.39	0.01	0.49	0.05			0.11	0.01	0.0053	0.0005		
	1:2	99.24	0.01	0.65	0.05			0.10	0.01	0.0054	0.0005		
	1:2	99.37	0.01	0.48	0.05	0.53	0.00	0.14	0.01	0.0048	0.0005		
	1:3	99.57	0.00	0.32	0.04			0.10	0.01	0.0054	0.0005		
	1:3	99.59	0.00	0.31	0.04			0.09	0.01	0.005	0.0005		
	1:3	99.58	0.00	0.31	0.04			0.11	0.01	0.005	0.0005		
	1:3	99.58	0.00	0.32	0.04			0.10	0.01	0.0052	0.0005		
	1:3	99.66	0.00	0.25	0.03	0.30	0.00	0.09	0.01	0.0054	0.0005		
Ennis latex paint yellow	1:1	99.39	0.01	0.40	0.05			0.20	0.01	0.0046	0.0005	0.0022	0.0007
	1:1	99.41	0.01	0.37	0.04			0.21	0.01	0.0057	0.0005	0.0021	0.0007
	1:1	99.32	0.01	0.46	0.05			0.20	0.01	0.0057	0.0005	0.0022	0.0007
	1:1	99.37	0.01	0.41	0.05			0.21	0.01	0.005	0.0005		
	1:1	99.29	0.01	0.44	0.05	0.42	0.00	0.26	0.01	0.0047	0.0005	0.0028	0.0007
	2:1	98.92	0.01	0.83	0.06			0.25	0.01	0.0077	0.0005	0.0025	0.0007
	2:1	98.99	0.01	0.76	0.06			0.24	0.01	0.0071	0.0006	0.0027	0.0007
	2:1	98.92	0.01	0.80	0.06			0.26	0.01	0.0074	0.0006		
	2:1	98.88	0.01	0.84	0.06			0.27	0.01	0.0067	0.0006	0.0029	0.0008

Paint Sample	Paint: Water Ratio	LE	LE +/-	Ti	Ti +/-	Avg	Stdev	Fe	Fe +/-	Zr	Zr +/-	Pb	Pb +/-
	2:1	98.80	0.02	0.94	0.07	0.83	0.00	0.25	0.01	0.0074	0.0006	0.0026	0.0007
	3:1	98.35	0.02	1.35	0.08			0.28	0.01	0.0088	0.0006	0.0033	0.0008
	3:1	98.36	0.02	1.27	0.07			0.36	0.01	0.008	0.0006	0.0043	0.0008
	3:1	98.26	0.02	1.46	0.08			0.27	0.01	0.008	0.0006	0.0047	0.0009
	3:1	98.37	0.02	1.34	0.08			0.27	0.01	0.0098	0.0006	0.0037	0.0008
	3:1	98.36	0.02	1.32	0.08	1.35	0.00	0.31	0.01	0.0086	0.0006	0.0031	0.0008
	1:2	99.56	0.01	0.27	0.04			0.16	0.01	0.0051	0.0005		
	1:2	99.64	0.00	0.20	0.03			0.15	0.01	0.0053	0.0005		
	1:2	99.65	0.00	0.19	0.03			0.16	0.01	0.004	0.0005		
	1:2	99.57	0.01	0.28	0.04			0.15	0.01	0.0044	0.0005		
	1:2	99.65	0.00	0.20	0.03	0.23	0.00	0.14	0.01	0.0046	0.0005		
	1:3	99.70	0.00	0.15	0.03			0.15	0.01	0.0049	0.0005		
	1:3	99.72	0.00	0.15	0.03			0.13	0.01	0.0041	0.0004		
	1:3	99.74	0.00	0.12	0.03			0.13	0.01	0.0052	0.0005	0.0018	0.0006
	1:3	99.69	0.00	0.17	0.03			0.14	0.01	0.0053	0.0005		
	1:3	99.64	0.00	0.21	0.03	0.16	0.00	0.14	0.01	0.0049	0.0005		

Table C.7. Lab XRF Results of Liquid Paint Samples Received by ME DOT (All Data in wt.%)

Paint Sample	LE	LE +/-	Ti	Ti +/-	Fe	Fe +/-	Cu	Cu +/-	Zn	Zn +/-	Mo	Mo +/-
ME DOT Corothane 8/8/13	91.04	0.08	8.88	0.14	0.01	0.00	0.0203	0.002	ND		0.0028	0.0006
ME DOT Corothane 8/8/13	90.98	0.08	8.91	0.14	0.03	0.00	0.0176	0.002	0.0084	0.0011	0.0035	0.0006
ME DOT Corothane 8/8/13	91.23	0.07	8.68	0.13	0.02	0.00	0.0204	0.0021	0.0105	0.0012	0.0028	0.0005
ME Spec Waterborne TP - white	93.48	0.09	6.42	0.17	0.09	0.01	ND		ND		ND	
ME Spec Waterborne TP - white	93.28	0.09	6.63	0.17	0.08	0.01	ND		ND		ND	
ME Spec Waterborne TP - white	93.43	0.08	6.45	0.17	0.11	0.01	ND		ND		ND	
ME Spec Waterborne TP - yellow	97.83	0.03	1.67	0.09	0.50	0.02	ND		ND		ND	
ME Spec Waterborne TP - yellow	97.84	0.03	1.66	0.09	0.50	0.02	ND		ND		ND	
ME Spec Waterborne TP - yellow	97.94	0.03	1.52	0.09	0.54	0.02	ND		ND		ND	

Table C.8. XRF Results from Field Tests (All Data in wt.%)

Location in Connecticut		Ti content (%)	
		White	Yellow
Exit 66 - I95, Westbrook		12.71	3.44
		12.89	4.25
		11.25	3.99
		12.14	3.51
	avg.	12.25	3.80
	s.d.	0.74	0.39
Exit 64 - I95, Westbrook		11.75	2.08
		12.17	2.90
		11.43	2.32
	avg.	11.78	2.43
	s.d.	0.37	0.42
Route 154, Old Saybrook		7.80	
		10.79	
		9.04	
	avg.	9.21	
	s.d.	1.50	
Exit 9 - Route 9, Higganum		22.19	2.83
		26.11	2.75
		19.93	2.26
		18.57	3.22
	avg.	21.70	2.76
	s.d.	3.30	0.40

Table C.9. XRF Results from Field Tests of Paints in Maine (All Data in wt.%)

Element	Laboratory				Field	
	Liquid Paint Ti (%)	s.d.	Painted Ti (%) Block	s.d.	Painted Bridge (%)	s.d.
Ti	8.82	0.13	2.67	2.67	9.21	0.29
Fe	0.02	0.01	13.30	13.30	17.83	3.68
Zn	0.01	0.01	ND	-	34.20	4.85
Pb	ND	-	ND	-	0.02	0.01

Table C.10. XRF Results from Field Tests of White and Yellow Lines in Maine (All Data in wt.%)

	Ti content (%)	
	White	Yellow
	7.36	1.86
	7.47	1.93
	7.89	1.82
Average	7.57	1.87
SD	0.28	0.06

APPENDIX D

Generic Implementation Plans

The concluding task of this project is to develop a road map for the adoption of the proposed methods by SHAs. This road map includes a step-by-step outline of the activities and processes that should be undertaken to facilitate change in the philosophy of QA/QC testing and develop familiarity with spectroscopic testing that is fundamentally different in nature from most field QA/QC processes currently used. The experience from collaboration with two DOTs indicated that of three proposed methods, only two are currently viable for nationwide adoption by SHAs. Specifically, the proposed procedure of fingerprinting chemical admixtures by PCC did not raise interest in DOE materials engineers, but was considered a method more suitable for private vendors or concrete mixing plants. Thus, while the method is valid and can be implemented, it will not be further discussed in the proposed implementation plan because the target audience does not include SHAs. The plans for each of the remaining two methods, XRF for paints and ATR for RAP testing, will be discussed separately because of their different nature and maturity stage.

Implementation plan for XRF testing of traffic and other paints

The following steps are proposed for adoption of the method by an SHA:

1. Assemble an implementation team to develop and follow through the implementation plan.
2. Conduct market research to identify the vendor and purchase the equipment.
3. Conduct staff training on the use of XRF instruments in accordance with OSHA and other state regulations.
4. Conduct staff training in accordance to both AASHTO method and equipment-specific instructions.
5. Develop a database of the laboratory-tested standard samples for the range of state-approved materials.
6. Review existing QA/QC procedures and develop a plan for transition or integration of the XRF method into a state QA/QC program.
7. Conduct in situ pilot-testing phase of the new method.

Following is the detailed description of each step.

Step 1. Assemble Implementation Team

It is recommended that an SHA should assemble a technical committee that will oversee all steps, develop a specific plan and timeline, and coordinate all necessary steps for successful implementation. It is envisioned that this committee will include mainly DOT materials engineers and QA/QC specialists, and potentially a representative from FHWA. They are recommended to undertake the following activities:

a. Assign the specific responsibilities for all DOT personnel involved with XRF method implementation and future use.
b. Develop an approach and establish a schedule for completing the implementation steps in hand.
c. Deliver the necessary training to all personnel involved with XRF-related testing.
d. Hold regular meetings to keep all informed of the progress in all XRF-related activities.
e. Determine potential funding sources and coordinate with FHWA activities that may promote the project.

Step 2. Conduct Market Research and Purchase Equipment

The purchase of the XRF equipment is a critical step in proceeding with all subsequent steps. Several vendors provide portable XRF. It is recommended that the implementation team reviews available equipment and its specifications in comparison with the particular DOT needs (accuracy, calibration range, etc.) and proceeds with procuring the selected equipment according to the specific DOT procedures.

Step 3. Conduct Training for X-ray Safety

This training is independent of the specific XRF equipment and is necessary for any operator of X-ray devices. The regulations on ionizing radiation may be found here: https://www.osha.gov/SLTC/radiationionizing/ (accessed February 16, 2014). Additionally, X-ray sources are subjected to regulations that vary from state to state. The operating staff should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization who is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring X-ray equipment at regular intervals.

Step 4. Conduct Equipment-Specific Training

In addition to the general safety training required for operators of X-ray equipment, additional training should be provided to all personnel who will handle the XRF in the lab and in the field. While the operation of XRF equipment is generally straightforward, users should receive standard training with regard to safety, equipment operation, sample handling, software operation and data storage and handling. The proposed AASHTO method includes details to this end and may be used as a guide. The implementation team should review the method and devise a testing protocol, if deemed necessary.

Step 5. Development of Material Standards

This is a critical step in the implementation process. The use of portable XRF in the field as QA/QC method requires that appropriate standards are developed in the lab that can serve as basis of comparison with the field results. It is recommended that the SHAs do not rely on manufacturer data on chemical composition to serve as standards. This is because such data typically provide only broad ranges of concentrations, but also because the method has nuances depending on the matrix and the testing conditions, so that a bulk chemistry determined by some other method may not be reflected accurately under field testing conditions. For example, the water content and film thickness of paint both substantially influence the measured Ti concentrations. Thus, it is recommended that relevant standards are developed for each case; for example, for Ti content in bulk paint and in paint strips on asphalt or bridges. This process should be conducted for all state-approved materials that are expected to be tested and the relevant standards should be published prior to the wide implementation of the method.

Step 6. Develop Integration Plan for Existing and New QA/QC Procedures

This is another important step, given the uncertainty that typically arises during transitions periods. While this step should take place concurrently with Step 5, it is described here separately in order to clearly delineate the actions and highlight the importance of these activities. The purpose of this step is to understand the relationship between the results of the existing and the new testing procedures in order to produce a standard of quality that is consistent with the pre-existing ones. Lab testing procedures that relate the existing and new processes should be undertaken and published, in order to ensure transparency in the regulatory and QA/QC process.

Step 7. Conduct Pilot-Testing Phase

It is recommended that the method should be piloted for a given period (e.g., 1–3 months) prior to full-scale implementation, in order to familiarize all personnel with the testing procedures and remove any glitches identified. Method evaluation processes (e.g. questionnaires for staff and potentially for contractors) should be implemented during this stage.

It is estimated that approximately 12–18 months are necessary to complete all aforementioned steps. This will largely depend on the number of materials for which standards will be developed, but here it is assumed that only traffic paint testing will be adopted initially. The projected timeline is shown in Table D1.

Table D1. Projected Timeline for Implementation of XRF for Traffic Paints

Implementation Step	Months 1–3	Months 4–6	Months 7–9	Months 10–12	Months 12–18
1. Assemble implementation team	▨				
2. Equipment purchase	▨				
3. Conduct X-ray training		▨			
4. Conduct equipment training		▨			
5. Develop lab-based standards		▨	▨	▨	▨
6. Develop integration plan			▨	▨	▨
7. Pilot test					▨

Implementation Plan for ATR-FTIR Testing of RAP

While the implementation steps are generally similar to the ones described for XRF, there are also some significant differences. One of the main differences is that there are currently no QA/QC procedures or standards for the moisture content and degree of oxidation in RAP, and that there are widely differing opinions with regard to the role of these parameters in asphalt quality. However, it is assumed that implementation will signify commitment of the SHA to utilize this method, at the very least for research purposes, even if QA/QC standards are ultimately not enforced. For this reason, the following steps are proposed:

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 a preliminary strategic plan with regard to RAP use and objectives of ATR testing.
4. Develop a sampling and testing plan to fulfill testing objectives.
5. Conduct market research to identify the vendor and purchase the equipment.
6. Conduct staff training according to both AASHTO method and equipment-specific instructions.
7. Implement the sampling and testing plan.
8. Evaluate results and review the strategic plan for RAP use and testing.
9. Conduct pilot-testing for field application of developed standards and/or testing objectives.

Following is the detailed description of each step.

Step 1. Assemble Implementation Team

It is recommended that an SHA should assemble a technical committee that will oversee all steps, develop a specific timeline, and coordinate all necessary steps for successful implementation. It is envisioned that this committee will include DOT material engineers and asphalt specialists, as well as representatives of other stakeholders involved in RAP management in the state or at the federal level (FHWA). They are recommended to undertake the following activities:

a. Assign the specific responsibilities for all personnel involved with ATR method implementation and future use.
b. Develop the various plans described below and establish a schedule for completing the implementation steps in hand.
c. Deliver the necessary training to all personnel involved with ATR-related testing.
d. Hold regular meetings to keep all informed of the progress in all ATR-related activities.
e. Determine potential funding sources and coordinate with FHWA activities that may promote the project.

Step 2. Data Compilation for Existing RAP Management Practices

It is recommended that a systematic documentation process for RAP management is installed statewide, if it is not already in place. Data related to the age, storage and utilization practices of produced RAP is necessary to interpret the results of the ATR testing in a larger context. Thus, this is a critical step in order fully benefit from the adoption of this method.

Step 3. Develop Preliminary Strategic Plan with Regard to RAP Use and Objectives of ATR Testing.

This is a key step in the process and should perhaps precede all other steps. ATR testing is essentially a management tool that can help SHAs implement a strategy with regard to the desired properties of RAP-mixed asphalt. Thus, defining such a strategy is necessary to guide the utilization of the test. If the long term goals cannot be clearly defined at the initial stage because of insufficient information, then the preliminary plan should outline the necessary data required in order to develop quality criteria and management objectives and delineate the role of ATR testing in order to obtain the desired dataset.

Step 4. Development of Sampling and Testing Plan

Once the testing objectives have been set, a more specific testing plan should be developed that will specify the number and locations of samples to be collected, taking into account parameters such as seasonal variations and construction schedule. The plan should specify sampling and material handling procedures to facilitate lab testing.

Step 5. Conduct Market Research and Purchase Equipment

Several vendors provide portable ATR FTIR. It is recommended that the implementation team review available equipment and its specifications in comparison with the particular DOT needs (accuracy, calibration range, etc.) and proceed with procuring the selected equipment according to the specific DOT procedures.

Step 6. Conduct Staff Training

Future users should receive standard training with regard to safety, equipment operation, sample handling, software operation, and data storage and handling. The proposed AASHTO method includes details to this end and may be used as a guide in conjunction with the particular equipment manual. The implementation team should review the method and devise a testing protocol, if deemed necessary. Often, vendors provide training that includes data manipulation and interpretation, which is the more involved part of the testing procedure. It is recommended that the implementation team assign a “champion” who will obtain more in-depth training, e.g., by visiting an external lab, and who will be responsible for training all other SHA personnel involved.

Step 7. Implement Sampling and Testing Plan

This step involves the implementation of the testing plan after all prior steps have been completed.

Step 8. Evaluate Results and Review Strategic Plan for RAP Use and Testing

The results of the testing plan should be assembled and reviewed critically in the context of the RAP management objectives set forth in Step 3. The implementation team should evaluate whether the testing objectives have been met and how the results are related to current management practices. Additional testing objectives and modified management practices may be suggested as a result of this evaluation, and the strategic plan will be revised.

Step 9. Conduct Pilot-Testing for Field Application of Developed Standards, Management Practices and/or Testing Objectives

Finally, a pilot testing phase with concurrent testing is proposed in order to test the feasibility and suitability of any proposed practices or standards under field conditions. The completion of this step may result in additional cycles of Steps 8 and 9 to establish a robust management plan for RAP by the SHA.

While it is difficult to anticipate a timeline for the plan because of the number of variables that come into play, a tentative 3-year timetable is proposed in Table D2.

Table D2. Projected Timeline for Implementation of ATR FTIR for RAP Testing

Implementation Step	Months 1–6	Months 7–12	Months 13–18	Months 19–24	Months 25–30	Months 30–36+
1. Assemble implementation team	■					
2. State-of-the-practice data compilation	■					
3. Strategic plan development	■	■				
4. Testing plan development		■				
5. Equipment purchase		■				
6. Staff training		■	■			
7. Sampling plan execution			■	■		
8. Data evaluation and strategic plan revision				■	■	
9. Pilot testing of proposed management objectives						■