

Techniques to Fingerprint Construction Materials (R06B)

Techniques to Fingerprint Construction Materials (R06B)

TO Pam Hutton, SHRP2 Implementation Manager

COPY Sam Rosenblum, Program Manager
Kate Kurgan, Product Lead

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PREPARED BY Maria Chrysochoou, Subject Matter Expert

REGARDING Implementation Close Out of SHRP2 Product: Techniques to Fingerprint Construction Materials (R06B)

Overview

The goal of this Implementation Assistance Program (IAP) was to determine if two spectroscopic techniques met “proof of concept” for selected materials used in transportation construction and were ready for national implementation. The two techniques were X-ray Fluorescence (XRF) and Fourier Transform Infrared (FTIR) spectroscopy. The following three State highway agencies were selected for IAP funding to participate in the evaluation: Tennessee Department of Transportation (TDOT) to implement XRF only for several materials, Maine Department of Transportation (Maine DOT), and Alabama Department of Transportation (ALDOT) to implement both XRF and FTIR for selected materials.

This summary report includes the following sections:

- Overview of Product Activity - Executive Summary
- Output (Deliverables)
- Outcomes
- Benefits
- Appendix A – List of Key Agency Participants
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- Appendix C – Webinar Agendas
- Appendix D – Peer Exchange Agenda and Participants List
- Appendix E – IAP State Agency Evaluation Reports Summary

Overview of Product Activity - Executive Summary

Two spectroscopic techniques were selected for this Proof of Concept IAP, XRF and FTIR spectroscopy, and specifically test the portable equipment available in the market for quality assurance/quality control (QA/QC) of materials used in construction projects of interest to state highways agencies. The R06B IAP was initiated in 2016 as a part of second Strategic Highway Research Program (SHRP2) Solutions Round 7 with a request for applications from highway agencies in April 2016. Seven agency applications were received and reviewed. The following are the three agencies selected for the program:

- TDOT was awarded \$250,000 to conduct an evaluation of portable XRF for a variety of materials including thermoplastics, aggregates and glass beads.
- Maine DOT was awarded \$250,000 to evaluate both XRF for analysis of chloride in concrete, steel grade, paints, glass beads, and presence of recycled engine oil bottoms (REOB) in asphalt; and FTIR to evaluate the composition of asphalt binders.
- ALDOT was awarded \$250,000 to evaluate XRF for field analysis of thermoplastics and FTIR for assessment of the presence of Recycled Asphalt Pavement (RAP) and Recycled Asphalt Shingles (RAS) in hot asphalt mixes.

Output (Deliverables)

This R06B product evaluation was broken into eight tasks. The following summarizes the output from each task and documents related to these tasks are located on the following SHRP2 webpage:

shrp2.transportation.org/Pages/R06B.aspx

- Task 1 Showcase

The showcase was held on November 1-2, 2016 at the Connecticut Advanced Pavement Laboratory. The 32 participants included representatives of the three IAP agencies, seven additional state agencies interested in R06B, four technology vendors, and members of the R06B team from Federal Highway Administration (FHWA), American Association of State Highway and Transportation Officials (AASHTO), and Jacobs Engineering Group Inc. (Jacobs). The 1.5-day agenda was divided into three components: overview of the technologies, agency experience, and laboratory equipment demonstrations. A report of the showcase is available on the SHRP2 R06B webpage.

- Task 2 Agency Work Plans

Each awarded IAP agency prepared a work plan to describe how they intended to execute their Proof of Concept evaluation. All three agencies developed work plans to evaluate the XRF technology and two of the agencies (ALDOT and Maine DOT) evaluated the FTIR technology. The primary tasks of each work plan were to obtain the equipment and receive training; identify target materials for testing, develop and execute laboratory-based protocols; develop field-based protocols and site for field testing; evaluate the laboratory versus the field results; and report their findings. Each IAP agency prepared a summary report of their Proof of Concept evaluation and findings. Anyone interested in an individual IAP state's report can contact Steve Cooper, FHWA SHRP2 Product Lead, or the IAP agency's point-of-contact listed in Appendix A.

- Task 3 Equipment Procurement

AASHTO worked with the subject matter expert (SME) to spec and procure XRF and FTIR equipment for the three IAP states. AASHTO procured 3 XRFs (one for each state) and 2 FTIR (one for ALDOT and one for Maine DOT).

- Task 4 Marketing and Communications

A key deliverable of R06B is the Primer, which provides interested agencies with background on both technologies in terms of key principles, equipment and applications for construction materials. The documents relied on material prepared during the R06B research phase, updated with information acquired during this Proof of Concept phase. The Primer is available on the SHRP2 R06B webpage under the Related Materials and Information heading.

A marketing plan and awareness resources were developed by AASHTO to support the information exchange on the R06B technologies. The resources include a website, technical brochures, and a fact sheet. These resources are available on the SHRP2 R06B webpage under the Related Materials and Information heading.

- Task 5 Education

Over the 3-year period of this IAP effort, education and outreach efforts included two technical webinars, a peer exchange, and one presentation to interested groups. There were separate 90-minute technical webinars for XRF on August 16, 2018 and FTIR on March 27, 2019. The online attendance for XRF was 67 and for FTIR was 42. The recorded sessions are available on the SHRP2 R06B webpage under the Presentations and Webinars heading.

The peer exchange was hosted by TDOT on September 25 and 26, 2018. On September 25, 2018, the three IAP states presented their findings, followed by a presentation by Terry Arnold of FHWA and presentations of additional states using the technologies. On August 2, 2018, the entire group outlined steps to advance R06B implementation and visited the TDOT laboratories for a demonstration of the equipment and applications. The peer exchange report is available on the SHRP2 R06B webpage under the Presentations and Webinars heading.

On August 9, 2018, Joe Kerstetter of TDOT and Rick Bradbury of Maine DOT provided a presentation of the project to the AASHTO committee on Materials and Pavement meeting. The slides are also available on the R06B webpage.

- Task 6 Technical Support

The SME provided technical support to the IAP agencies for the laboratory development and field evaluation of each technology. The support included two SME visits to each IAP states: TDOT in September 2017 and June 2018; ALDOT in November 2017 and June 2018; and Maine DOT in October 2017 and June 2018. During the first visit, the SME demonstrated the use of the equipment, discussed the development of protocols for sample preparation and testing and participated in a first round of testing materials. During the second visit, results from the laboratory and the field were discussed, as well as steps forward with particular materials and the techniques overall for further adoption.

The SME provided additional technical support to the IAP states with emails and phone calls on an as needed basis during the lifetime of the project, including preparation of work plans and final reports.

- Task 7 Final report (this report)

- Task 8 Project Management

To support the successful accomplishment of the tasks summarized herein, the R06B management team, SME, and the three DOTs had scheduled meetings over the 3-year IAP period.

The R06 management team teleconferenced monthly and included individuals from FHWA, AASHTO, and Jacobs. The kick-off meeting was held on March 1, 2016. Each meeting the SME provided an overview of recent accomplishments and plans for future activities. The group would discuss the activities and recommend changes as needed.

The SME coordinated quarterly technical meetings with the three IAP states, as needed, to review the agencies' progress, provide direction for future activities, and discuss technical or administrative issues. There were four teleconferences starting in July 2017 and continuing until October 2018.

Outcomes

- State Feedback; Change in Practice

Overall, XRF was found to be a highly useful equipment with multiple applications across several materials. All three IAP states observed the need for careful and consistent sample preparation, as well as understanding of the need for calibration curves specific to the material under consideration, using appropriate standards. The need for consistent sample preparation that involves particle size reduction and preparation of homogeneous pellets renders the field adoption of the equipment more difficult than originally anticipated. Still, the ease of use, with no need for chemicals renders the XRF an attractive alternative for several materials and tests that DOTs currently use (for example, testing of glass beads, chloride content of concrete, and quality of aggregate).

The adoption of FTIR for identification of polymers as asphalt additives is a well-established application that will continue in the future. The evaluation of RAP and RAS presence using this technology was not ready for field use at this time. Additional research and development of FTIR for asphalt-related applications is required for wider use of the technology.

- Action and Adoption Plans

All three IAP states have both portable XRF and FTIR equipment acquired through the project or already available to them.

TDOT plans to adopt XRF for testing of glass beads and surface aggregates, as well as explore the Maine DOT-developed application for chloride content in concrete. TDOT also plans to further investigate the use of XRF for thermoplastics. While FTIR evaluation was not included in the TDOT work plan for this product, TDOT independently acquired a handheld FTIR and is investigating its use for field evaluation of polymer additives in asphalt and other applications.

Maine DOT plans to adopt XRF for chloride evaluation of concrete to replace the current titration-based technique. Maine DOT also plans to deploy it in the field for QA/QC of steel rebar. Maine DOT is interested in expanding the use of XRF across several applications, such as detecting recycled engine oil bottoms (REOB) in asphalt and measuring the thickness of galvanized coatings in railings. Maine DOT also evaluated the use of FTIR to characterize asphalt binders and determine their source; it is currently building a library of spectra to use as a baseline against which field samples can be compared in the future.

ALDOT did not reach the stage of full adoption for XRF use to analyze the TiO₂ content in thermoplastics, but plans to work on additional development of the method. For FTIR, ALDOT will continue to use the FTIR for identification of polymer additives in asphalt, but did not involve any plans for new or additional uses through this project, as the proof-of-concept for evaluation of RAP and RAS in asphalt was not successful at this stage.

- State Organizational Structure Change; New Role Designations

The use of the XRF and FTIR as diagnostic tools will be incorporated into the existing material laboratories of the three IAP states. The state agencies may explore the adoption of the equipment by field inspectors, and the acquisition of additional portable XRF equipment for distribution in regional offices.

- Other Recommendations for Future Activities and/or Programs

All three IAP states, as well as other states that participated in the peer exchange, voiced a strong desire to form a working group towards the more widespread implementation and advancement of both technologies. There is a strong need for established procedures that are specific to every material, and a need for relevant standards that can be used for the particular application. The National Institute for Standards and Technology (NIST) may play an important role in the development of standards particular to these applications. Turner Fairbanks at FHWA may also be a key partner in creating universal standards and methods for some of these applications.

The states also voiced a need for detailed documentation supporting the particular application for each material, especially in the form of instructional videos. Each application has nuances with respect to sample preparation and equipment calibration that have to be captured for proper implementation, and vendor manuals or training are insufficient to prepare future users.

Benefits

- Expectations versus Actual Implementation

The proof of concept value of the XRF technology was demonstrated for several materials and is demonstrated by the plans of at least two states (Tennessee and Maine) to fully adopt it as the preferred method for glass bead, surface aggregate, concrete chloride, and steel rebar testing in the laboratory and in the field. Other applications, such as the evaluation of the TiO₂ content in thermoplastics that were investigated by TDOT and ALDOT, were found to be less ready for field use but still hold promise with additional development. A widespread adoption for XRF across multiple state agencies would promote the development of much needed material-specific standards and testing procedures.

The proof of concept value of the FTIR technology was less evident, given that the technology requires more in depth knowledge of chemistry, and has limitations in terms of detection limits and specificity to the analyzed material. The use of the FTIR to determine the presence of RAP and RAS in asphalt mixes was not feasible at this time. However, the technique may provide insights into asphalt properties and presence of admixtures, especially if more widespread use leads to the development of a comprehensive reference database that is specific to asphalt and construction materials.

- Quantification of Gain

No quantitative study was performed by the states in terms of the monetary gain of using the technology. However, it was noted during the peer exchange, and in the individual reports, that the XRF technology has considerable potential to decrease testing costs and enhance laboratory safety, as it replaces wet chemistry methods that employ acids and hazardous chemicals. Testing of glass beads for lead (Pb) and arsenic (As), and testing of concrete for chloride content are examples of two such chemical-intensive methods.

Another important gain that was noted by the states for both the XRF and FTIR is their value as deterrent; if there is a quick, nondestructive method to test materials in the field, contractors and manufacturers are less likely to attempt compromising their quality. The development of standards and widespread use of the technologies will substantially enhance this effect in the long term.

Appendix A - List of Key Agency Participants

<i>Tennessee Department of Transportation</i>		
Brian K. Egan, P.E. Director Materials and Tests Division		
Danny Lane Assistant Director Research and Product Evaluation Materials and Tests Division		
Michael J. Doran, P.E. Assistant Director-Laboratory Manager Materials & Tests Division		
Joseph Kerstetter, PE Transportation Project Specialist - SR Laboratory Supervisor Materials and Tests Division		
Joe Simon Transportation Project Specialist - SR Research and Product Evaluation Materials & Tests Division		
Jimmy Scales Jr., E.I. Graduate Transportation Associate Materials & Tests Division Research and Product Evaluation		
<i>Maine Department of Transportation</i>		
Name	Title	Role
Mark Alley	Laboratory Testing Engineer	Overall management of XRF and FTIR testing including personnel, equipment, and supplies; training; and data management
Derek Nener-Plante	Asphalt Pavement Engineer	Asphalt SME; experimental design, data analysis
Bruce Niles	Chemistry Laboratory Supervisor	Radiation safety; laboratory testing
James Robinson	Freeport Laboratory Supervisor	Coordinated XRF testing – Freeport
Caroline Nguembu-Tagne	Laboratory Technician	XRF testing – Freeport
John Clark	Laboratory Technician	XRF testing – Bangor
Casey Nash	Assistant Engineer	FTIR testing; data management/analysis
Ryan Vose	Independent Assurance	Onsite XRF testing
<i>Alabama Department of Transportation</i>		
Name	Title	Role
Lyndi Blackburn	Special Projects Engineer	Project Oversight and Management
Andre Jenkins	Chemical Testing Section Manager	XRF testing lead
Torey Bell	Liquid Asphalt Engineer	FTIR testing lead

Appendix B - Showcase Agenda and Participants List



Agenda
SHRP2 Technology Showcase
Techniques to Fingerprint Construction Materials (R06B)
 November 1-2, 2016

Storrs, Connecticut
 Hosted by the Connecticut Advanced Pavement Laboratory

Tuesday, November 1, 2016
 CAPLAB Seminar Room

Time	Topic	Speakers
1:00 – 1:15PM	Welcome	Steve Cooper, FHWA Kate Kurgan, AASHTO
1:15-2:00PM	Introduction of the Techniques to Fingerprint Construction Materials (R06B) <ul style="list-style-type: none"> X-ray Fluorescence (XRF) overview and potential applications for construction materials 	Maria Chrysochoou, UCONN Cristian Balotescu, Thermo
2:00-2:30PM	Introduction of the Techniques to Fingerprint Construction Materials (R06B) <ul style="list-style-type: none"> Fourier Transform Infrared (FTIR) spectroscopy overview and potential applications for construction materials 	Maria Chrysochoou, UCONN
2:30-3:30PM	FHWA Chemistry Laboratory Experiences <ul style="list-style-type: none"> Methods and applications developed Benefits and limitations of the technologies 	Terry Arnold, FHWA

3:30-5:00PM	Round-Table Discussion: Challenges and needs for non-destructive materials testing <ul style="list-style-type: none"> • State experiences with spectroscopic techniques • Benefits and limitations • Wish list for materials testing 	ALDOT, Andre Jenkins TDOT, Joseph Kerstetter VTrans, Jerry McMahan Moderator: Maria Chrysochoou
5:00PM	Adjourn for the day	

Wednesday, November 2, 2016 CAPLAB Main Laboratory		
8:00AM – 11:45PM	Demonstration of Both Technologies in the laboratory 5 stations (3 XRF and 2 FTIR) Break-out groups with 5-6 participants, approximately 45 minutes per station	Thermo Scientific Bruker Olympus Agilent
11:45AM-12:00PM	Final Questions & Closing Comments	Maria Chrysochoou, AASHTO, FHWA
12:00PM	Showcase Adjourns	
1:00PM-4:00PM	R06B Implementation Assistance Program Recipients - Individual Workplan meeting	Maria Chrysochoou, UCONN
	Adjourn for the day	

Roundtable Discussion format

Moderator will ask a series of questions and each of the panelists will respond. The discussion will last 90 min and the last 20-30 min will be dedicated to Q&A from the audience and open discussion. Each question will thus be about 10 min in total, with 3 min for each panelist. Questions include:

1. What spectroscopic techniques are you using in your laboratory and when did you start implementing those? What kind of materials are you testing? How many people are currently using them?
2. What was the motivation behind adopting these techniques?
3. What are the benefits you have seen so far and what are your goals for the future?
4. What are obstacles you have encountered in use of the techniques?
5. What resources do you feel could enhance the application of spectroscopic techniques in your laboratory?
6. What materials testing needs do you have that are not currently being met by available experimental techniques?

Attendee list

First Name	Last Name	Title	Organization/Agency
Mark	Alley	Laboratory Testing Engineer	Maine DOT
Terry	Arnold	Senior Research Chemist	FHWA
Christian	Balotescu	Senior Sales Engineer	Thermo Scientific Niton
Michelle	Barger	Chemist	Iowa DOT
Torey	Bell	CEG	ALDOT
Beran	Black	Supervising Laboratory Scientist	New Hampshire DOT
Lyndi	Blackburn	Asst. State Materials & Tests Engineer	ALDOT
Denis	Boisvert	Chief of Materials Technology	New Hampshire DOT
Richard	Bradbury	Director of Materials Testing	Maine DOT
Jennifer	Caban	Sales and Applications Support Specialist	Olympus
Kevin	Chesnik	Principal Engineer	ARA
Maria	Chrysochoou	Associate Professor	University of Connecticut
Stephen	Cooper	SHRP2 R06B Product Lead	FHWA
Andre' L.	Jenkins, Sr.	Alabama Chemical Testing Section Manager	ALDOT
Marc	Joyal	Engineering Technician IV	New Hampshire DOT
Joseph	Kerstetter	Research and New Products Engineer	TDOT
Kate	Kurgan	Associate Program Manager SHRP2 Implementation	AASHTO
James	Mahoney	Executive Director	CT Transportation Institute
Jonathan	Margalit	Business Development Mgr.	Thermo Fisher Scientific
Keegan	McHose	Molecular Product Specialist	Agilent Technologies
Jerry	McMahan	Chemist	Vermont DOT
Fred	Morris	Sales Manager	Bruker Optics
Bruce	Niles	Chemistry Laboratory Supervisor	Maine DOT
Rick	Rainville	Sales Representative	Bruker
Greg	Sholar	State Bituminous Engineer	Florida DOT
Jozsef	Simon	Transportation Project Specialist - SR	TDOT
Joshua	Smith	Ph.D.	Agilent Technologies

Jennifer	Smoker	Consultant	Jacobs
Yanqia	Wang	Application Engineer	Agilent Technologies
Brenda	Waters	Chemist III	Pennsylvania DOT
Zina	Zadoroshnaya	Principle Engineer Materials	New Jersey DOT

Appendix C - Webinar Agendas

Techniques Fingerprint Construction Materials – X-ray Fluorescence Applications SHRP2 R06B

Webinar AGENDA

August 22, 2018

Start Time: 2.00 p.m. (Eastern Time)

Outcome: Participants will possess a common knowledge of the principles of X-ray Fluorescence technology and its applications for common construction materials

Objectives:

- Review the principles of X-ray Fluorescence use for construction materials
- Provide an overview of promising applications and lessons learned for specific construction materials

Agenda (1.0 hours):

1. Welcome and Introductions Overview & Goal of SHRP2 Program Round 7
(5 min.) Kurgan/Cooper
2. R06B Research Product; Overview of XRF
 - a. PowerPoint Presentation Summary of R06 objectives and principles of XRF technology. (10 min.) Chrysochoou
3. XRF applications and lessons learned: (40 min.) State DOT representatives
 - Steel rebar, Pb in bridge paint, chloride in concrete (Maine DOT)
 - Aggregates, Bauxite, Glass beads, Thermoplastics (TDOT)
4. Questions/Answers (10 min) State DOTs

Techniques Fingerprint Construction Materials – Fourier Transform Infrared (FTIR) Spectroscopy Applications

SHRP2 R06B

Webinar AGENDA –March 27, 2019

Start Time: 1 pm to 2:30 pm EST

Outcome: Participants will possess a common knowledge of the principles of Fourier-Transform Infrared technology and its applications for common construction materials

Objectives:

- To review the principles of FTIR use for construction materials
- To present State DOT FTIR Experiences
- Provide an overview of promising applications and lessons learned for specific construction materials

Agenda (1.5 hours):

5. Welcome and Introductions
 - Overview & Goal of SHRP2 Program Rd 7 **(5 min.) Kurgan/Cooper**
6. R06B Research Product; Overview of FTIR
 - a. PowerPoint Presentation Summary of R06B objectives and principles of FTIR technology. **(10 min.) Chrysochoou**
7. IAP state lessons learned: **(60 min.) State DOT Presenters**
 - FDOT’s latest uses of FTIR **(Cassady Allen - FDOT)**
 - Baseline characterization of asphalt binders **(Derek Nener-Plante –Maine DOT)**
 - Use of product libraries for QA and Forensic analysis of asphalt binders **(Joseph Kerstetter - TDOT)**

Questions/Answers **(15 min.) State DOT’s**

Appendix D - Peer Exchange Agenda and Participants List



Agenda SHRP2 R06B <i>Techniques to Fingerprint Construction Materials</i> Peer Exchange Nashville, Tennessee		
Hosted by the Tennessee Department of Transportation TDOT REGION THREE OFFICE 6601 Centennial Blvd Nashville, Tennessee 37243		
DAY 1 – Wednesday, September 26, 2018		
Time	Topic/Presentation	Speaker
8:30 AM – 9:00 AM	Welcoming Remarks	
	<ul style="list-style-type: none"> TDOT FHWA AASHTO SME (Subject Matter Expert) (Meeting Goals, Agenda Review, Introductions) 	<i>Danny Lane, TDOT</i> <i>Steve Cooper, FHWA</i> <i>Kate Kurgan, AASHTO</i> <i>Maria Chrysochoou, University of Connecticut</i>
9:00 AM – 9:30 AM	Overview of XRF and FTIR Technologies for Construction Materials <ul style="list-style-type: none"> Background Limitations Enhancements, etc. 	<i>Maria Chrysochoou</i>

<p>9:30 AM – 10:15 AM</p>	<p>Lessons Learned - TDOT</p> <ul style="list-style-type: none"> • XRF use for QA/QC glass beads, aggregates, cements, thermoplastics • Laboratory and field tests – protocol development • Challenges and path forward 	<p><i>Joe Kerstetter, TDOT</i></p>
<p>10:15 AM – 10:30 AM</p>	<p>Break</p>	
<p>10:45 AM – 11:30 AM</p>	<p>Lessons Learned - ALDOT</p> <ul style="list-style-type: none"> • XRF use for thermoplastics • FTIR use for polymer in asphalt, Rap and RAS testing • Laboratory and field tests– protocol development • Challenges and path forward 	<p><i>Lyndi Blackburn, ALDOT</i></p>
<p>11:30 AM – 12:15 PM</p>	<p>Lessons learned - Maine DOT</p> <ul style="list-style-type: none"> • XRF testing for analysis of <ul style="list-style-type: none"> ○ Chloride in concrete ○ Pb in paint and contaminated media ○ Cements ○ Steel ○ RAS • FTIR testing of polymer in asphalt • Laboratory and field tests– protocol development • Challenges and path forward 	<p><i>Rick Bradbury, Maine DOT</i></p>
<p>12:15 PM – 1:30 PM</p>	<p>Lunch at Nearby Venues</p>	
<p>1:30 PM – 3:00 PM</p>	<p>2018 Experiences Non IAP States & FHWA R&D Program</p> <ul style="list-style-type: none"> • Connecticut • Florida • Minnesota • New Jersey • Pennsylvania • Others on the phone 	<p><i>Terry Arnold, FHWA David Howley, Connecticut DOT Cassady Allen, Florida DOT Jason Krogman, Minnesota DOT Darshan Patel, New Jersey DOT David Kuniega, Pennsylvania DOT</i></p>
<p>3:00 PM- 3:15 PM</p>	<p>Break</p>	
<p>3:15 PM – 4:45 PM</p>	<p>Group Discussion</p>	<p><i>All Participants</i></p>
<p>5:00 PM</p>	<p>Adjourn</p>	
<p>6:00 PM</p>	<p>Optional Group Outing to Downtown Nashville</p>	



Agenda

SHRP2 R06B *Techniques to Fingerprint Construction Materials* Peer Exchange Nashville, Tennessee

Hosted by the Tennessee Department of Transportation
 TDOT REGION THREE OFFICE 6601 Centennial Blvd
 Nashville, Tennessee 37243

DAY 2 – Thursday, September 27, 2018

Time	Topic/Presentation	Speaker
8:00 AM – 8:15 AM	Review Day 1 Highlights	<i>Kevin Chesnik, ARA</i>
8:15 AM – 9:00 AM	Turner-Fairbanks Laboratory Workplan for R&D of Spectroscopic Techniques	<i>Terry Arnold, FHWA</i>
9:00 AM – 10:00 AM	Moving Forward with XRF and FTIR Deployment Efforts <ul style="list-style-type: none"> • Brainstorming • Pooled Fund Research options • Sensitivity Studies - What needs to be evaluated further? • Specification Needs • Quality Acceptance use • Hardware and Software Improvements • Equipment Precision and Accuracy 	<i>All participants</i>
10:00-10:15 AM	Break	
10:15 – 11.45 AM	Hands on Laboratory Session at TDOT <ul style="list-style-type: none"> ▪ XRF demos for glass beads, aggregates etc. ▪ FTIR demo for polymers in asphalt 	<i>TDOT personnel, Terry Arnold, FHWA</i>

<p>11:45 AM – 12:00 PM</p>	<p>Closing Remarks</p>	<p><i>Steve Cooper, FHWA Kate Kurgan, AASHTO Maria Chrysochoou, SME Danny Lane, TDOT</i></p>
<p>12:00 PM</p>	<p>Adjourn and/or Head to Airport</p>	

Participant list

Cassady Allen	FDOT	cassady.allen@dot.state.fl.us
Mark Alley	Maine DOT	mark.alley@maine.gov
Terry Arnold	FHWA (Turner Fairbanks)	Terry.Arnold@dot.gov
Lyndi Blackburn	ALDOT blackburnl@dot.state.al.us	
Rick Bradbury	Maine DOT	Richard.bradbury@maine.gov
Kevin Chesnik	ARA	kchesnik@ara.com
Maria Chrysochoou	University of Connecticut	maria.chrysochoou@uconn.edu
John Clark	Maine DOT	John.T.Clark@maine.gov
Steve Cooper	FHWA- RC	Stephen.J.Cooper@dot.gov
Brian Egan	TDOT brian.egan@tn.gov	
David Howley	CT DOT	David.Howley@ct.gov
Joseph Kerstetter	TDOT Joseph.Kerstetter@tn.gov	
Jason Krogman	Minnesota DOT	jason.krogman@state.mn.us
David Kuniega	Pennsylvania DOT	dkuniega@pa.gov
Kate Kurgan	AASHTO	kkurgan@ashto.org
Danny Lane	TDOT Danny.Lane@tn.gov	
Derick Nener- Plante	MaineDOT	Derek.Nener-Plante@maine.gov
Caroline Nguemba-Tagne	MaineDOT	Caroline.Nguemba-Tagne@maine.gov
Darshan Patel	NJ DOT	DarshanB.Patel@dot.nj.gov
Tony Pope	TDOT tony.pope@tn.gov	
Jimmy Scales	TDOT jimmy.scales@tn.gov	
Jason Selvage	ALDOT selvageg@dot.al.us	
Jozsef Simon	TDOT Joe.T.Simon@tn.gov	
Jennifer Smoker	Jacobs	Jennifer.Smoker@jacobs.com
John Steele	FHWA-TN	John.Steele@dot.gov
Phone Participants		
Todd Bennett	Missouri DOT	Todd.Bennett@modot.mo.gov
Maggie McDonald	Massachusetts DOT	maggie.mcdonald@dot.state.ma.us
Oak Metcalfe	Montana DOT	rmetcalfe@mt.gov
Kelly Morse	Illinois DOT	Kelly.Morse@illinois.gov
Michelle Barger	Iowa Department of Transportation	michelle.barger@dot.iowa.gov
Barry Paye	Wisconsin DOT	barry.paye@dot.wi.gov
Brenda Waters	Pennsylvania DOT	brwaters@pa.gov

Appendix E - IAP State Agency Evaluation Reports Summary

Tennessee

Product-specific Output Measures

TDOT successfully deployed the handheld XRF and demonstrated the equipment capabilities and performance both in the laboratory and in the field for a variety of materials.

Proof of Concept Conclusions

TDOT concludes that handheld XRF technology has become advanced enough to be of use for most if not all State Transportation Agencies. For states where WDXRF technology is cost prohibitive, the handheld instrument provides a means of replacing timely wet chemical test methods at a reasonable cost. For states that already have a central laboratory performing XRF analysis, it gives the ability to move the instrument to the field if sample preparation is not an issue for the material in question. It also opens the door to other materials (such as asphalts and liquids) that cannot currently be tested on some WDXRFs because of potential damage to the instrument. In terms of specific materials, TDOT reached the following conclusions:

- The handheld XRF can replicate the WDXRF as far as TDOT's current testing on surface aggregate limestone. The disadvantage is that the handheld XRF still needs the same level of sample preparation as our current method and this removes it as a field test.
- The handheld XRF can be used similarly to limestone on Calcined Bauxite as long as laboratory sample preparation is followed. The only obstacle to this right now is to find more standards that are closer to the minimum 87% requirement. This may be accomplished through further research and/or a round robin type process similar to what NIST and CCRL perform.
- The handheld XRF can analyze Thermoplastic samples for TiO₂ in the field if calibrations can be corrected for the manufacturer. The sampling process is easy to perform in the field before material is placed.
- All sample prep methods evaluated returned accurate results, from this research it has been found that the in-situ samples will be used for verification

Status of Action and Adoption

TDOT's experience with the handheld XRF looks promising for TDOT's materials program. Even though there are obstacles that are intrinsic to each material such as calibrations curves needing to be developed, sample preparation procedures, and general difference in material types being tested, it was found that they can be overcome. Fingerprinting of construction materials is gaining in effort from both state departments of Transportation and Industry. TDOT will continue to implement fingerprinting of construction materials to verify materials and products on the project site and evaluated through the American Association of State Highway Transportation Officials (AASHTO), National Transportation Product Evaluation Program (NTPEP) and AASHTO Product Evaluation Listing (APEL) programs.

Recommendations for Other Future Activities or Programs (by FHWA/AASHTO/Others)

It is recommended that specifications be developed for the general use of XRF and other spectrometry techniques for the fingerprinting of construction materials. These specifications should focus on general principles of spectrometry such as sampling techniques and calibration curves. It is also recommended that states work with FHWA to allow XRF testing to replace expensive United States Environmental Protection Agency test methods to check for heavy metals in glass beads.

Additional Implementation Activities

TDOT will be involved with the provisional test method that was just recently submitted to COMP Subcommittee 2B that details a test method to use XRF to quantify the amount of REOB in asphalt binder. TDOT will test the method and provide comments to the subcommittee and vote on it. TDOT will also continue this research and look into other materials that can be tested such as Chloride in Bridge Decks, PPA in Asphalt Binder, and Sulphur in Acid Producing Soil.

Maine

Product-specific Output Measures

Maine DOT successfully deployed the handheld XRF and demonstrated the equipment capabilities and performance both in the laboratory and in the field for a variety of materials. Maine DOT also used the FTIR in the laboratory to develop a library of binder spectra for future use and expects additional applications to emerge.

Proof-of-Concept Conclusions

The SHRP2 R06B proof-of-concept implementation project was successful in demonstrating the potential applications of portable spectroscopic devices in construction materials testing. Both XRF and FTIR will provide Maine DOT with the ability to produce qualitative and quantitative analysis of a number of materials, including the ability to verify specification compliance for several material properties that the Department did not have the ability to test prior to this project (such as presence of REOB in PG binder). The ability to verify properties such as grade of steel quickly at the project site will reduce the potential for incorporation of nonconforming material being incorporated into the work. Also, XRF has been demonstrated to be a viable replacement for the AASHTO T 260 as a means to determine chloride content of concrete in a manner that reduces test time, environmental impact and cost, while improving technician safety.

Although these spectroscopic tools have shown great promise, one of Maine DOT’s lessons-learned was the importance of understanding the equipment operation and limitations. Without a clear understanding of the various calibrations, equipment settings, and sample preparation techniques, it is very possible to produce misleading test results. Proper training of those conducting the tests and analyzing the results is critical to successful use of these technologies.

Status of Action and Adoption

The Department has already benefitted from this project in tangible ways. Based on an analysis of the chloride content test data, the current titration process involving hazardous chemicals can be replaced with XRF, resulting in reduced cost and testing time all while increasing technician safety. Also, inspectors will be able to verify that stainless steel rebar meets the correct grade requirements using a rapid, nondestructive test onsite. Verifying the presence of polymer and the absence of REOB in binder will help ensure pavement performance. Expanding the Department’s ability to test materials such as traffic paint and glass beads will help verify specification compliance. Implementation of XRF and FTIR will have immediate benefits, and there will no doubt be additional applications discovered as the technologies are used more widely.

Recommendations for Other Future Activities or Programs

Test standards already exist for some applications of XRF and FTIR, but additional AASHTO test methods will need to be developed.

XRF

1. Develop a QC plan using various standard reference samples and control charts.
2. Obtain additional CCRL reference samples to improve the cement calibration.
3. Provide additional training for Maine DOT’s laboratory technician. Note: scheduled for June 2019.
4. Identify the cause of the observed drift in chloride content results.
 - a. If/when resolved, work with end users of the data to replace T 260 with XRF.
5. Incorporate XRF into the manufactured materials verification testing program for stainless steel rebar, glass beads, cement, and traffic paint.
6. Identify additional uses for XRF to improve service to internal customers. Already identified: soil testing adjacent to bridge rehabilitation projects to detect contamination from lead paint.
7. Work through the AASHTO Committee on Materials and Pavements to develop test standards as needed.

FTIR

1. Continue to build a library of PG binder results.
2. Identify additional uses for FTIR to improve service to internal customers.

Alabama

Product-specific Output Measures

ALDOT attempted to deploy the XRF for QA/QC of thermoplastics using the Ti content in the laboratory and in the field; it also conducted a preliminary laboratory study for the evaluation of the presence of RAP or RAS in asphalt mixes using FTIR.

Proof of Concept Conclusions

ALDOT concluded that neither of the two applications (XRF for thermoplastics or FTIR for RAP/RAS in asphalt) are ready to be implemented on a regular basis in the laboratory or in the field at this time. In terms of the XRF, ALDOT was able to reproduce the results obtained from TDOT for a series of standards provided by thermoplastic manufacturers, in terms of generating a calibration curve for Ti across the concentrations of interest (8 to 12% TiO₂). However, the calibration curves were not identical across manufacturers, and manufacturers were resistant to providing raw materials or further information on the chemical composition of the fillers. ALDOT concludes that there is promise in this application, with additional development needed to understand equipment calibration. Furthermore, implementation in the field indicated that testing directly on the thermoplastic strip after it has been placed on the pavement does not yield comparable results with the laboratory, because of the layer of glass beads that concentrate at the top of the strip. ALDOT also raised concerns about the safety of personnel using the XRF in field conditions to test the strip directly on the pavement.

In terms of the FTIR, ALDOT attempted to investigate the effect of aging and oxidation using the carbonyl (C=O) peak presented in the FTIR spectrum of asphalt binders as indicator. A preliminary study using neat 67-22 binder cured in a Pressured Aging Vessel for up to 7 days showed that there is a general linear relationship between the carbonyl peak area and the amount of aging; however, the slope of the peak presented substantial variability across identical subsamples. Furthermore, evaluation of field samples of 76-22 binder treated with SBS polymer showed that the mere presence of polymer could increase the peak area of carbonyl even in freshly placed binder. Finally, the FTIR analysis of several RAP and RAS samples showed

substantial variability in the carbonyl peak area that could not be readily correlated to the age of the pavement. Thus, ALDOT concludes that there are too many confounding factors to readily use FTIR to infer the presence and quantity of RAP or RAS in asphalt mixes using the carbonyl peak as indicator.

Status of Action and Adoption

ALDOT intends to continue exploring the use of XRF to analyze the Ti content of thermoplastics in collaboration with TDOT and FWHA. ALDOT will also continue to routinely use FTIR to verify the presence of polymers in asphalt binders, a practice it has followed for many years. Currently, ALDOT employs a laboratory-based FTIR instrument that requires binder extraction with trichloroethylene to prepare a sample for analysis. The acquisition of a portable FTIR instrument holds promise for use of the instrument under field conditions, even though this application requires additional development.

Recommendations for Other Future Activities or Programs

ALDOT recommends additional testing and development of standards to test TiO₂ in thermoplastics across different manufacturers.