MEETING SUMMARY

SHRP2 TECHNIQUES TO FINGERPRINT CONSTRUCTION MATERIALS (R06B) SHOWCASE, STORRS, CT

TO	Steve Cooper, Pam Hutton, Kate Kurgan,
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LOCATION	Connecticut Advanced Pavement Laboratory, Storrs, CT

Purpose

This Showcase and Technology Demonstration was intended to provide state transportation agencies and vendors an opportunity to learn about the technologies offered through *Techniques to Fingerprint Construction Materials* (R06B), which was developed through the second Strategic Highway Research Program (SHRP2). Participants attended demonstrations for X-ray Fluorescence (XRF), which measures elemental concentrations in the test materials, and for Fourier Transform Infrared (FTIR) spectroscopy, which identifies the qualitative composition of materials using characteristic vibrational frequencies. The goal was for participants to see how these non-destructive testing (NDT) technologies work with portable devices, how they can benefit state agencies, and to understand how their peers plan to use them. The event also offered participants an opportunity to work with AASHTO to decide which equipment to purchase for their agency's implementation assistance projects.

Attendees

States participating in the SHRP2 Implementation Assistance Program (IAP) for R06B attended the event (Tennessee, Alabama and Maine), as well as 14 other states interested in learning more about the R06B technologies. (See Appendix D for a full list of attendees.) Five equipment vendors, including three XRF and two FTIR manufacturers, also participated in the showcase.

Executive Summary

Through *Techniques to Fingerprint Construction Materials* (R06B), two technologies were identified that could make significant advances in performing Quality Assurance/Quality Control (QA/QC) testing of construction materials. The technologies are XRF and FTIR spectroscopy. Alabama, Maine and Tennessee received Proof of Concept awards through the IAP, administered by the Federal Highway Administration (FHWA) and the American Association of State Highway and Transportation Officials (AASHTO), to test out the technologies in their respective states. A two-day Showcase and Technology Demonstration was held at the Connecticut Advanced Pavement Laboratory in Storrs, Conn. to demonstrate use of the R06B technologies and provide states with the opportunity to exchange information with their peers. All three IAP and 14 other states participated in the showcase.

Overall, the showcase achieved its intended purpose. On the first day AASHTO's Kate Kurgan and FHWA's Steve Cooper opened the event with information on the SHRP2 program. The subject matter expert (SME), Dr. Maria Chrysochoou of UCONN, and Terry Arnold of FHWA's Turner Fairbanks provided in depth presentations on the technology principles and applications for construction materials. Two vendors, Thermo Scientific and Agilent Technologies, also gave presentations on applications for the technologies. Finally, a roundtable discussion was held with representatives of three states, Tennessee, Alabama and Vermont, who provided their perspective and experiences with spectroscopic techniques. On day two, participants observed a hands-on demonstration of both NDT systems by five vendors, two for FTIR (Bruker and Agilent) and three for XRF (Bruker, Olympus and Thermo Scientific). In order to ensure everyone had enough time with the equipment, the demonstrations were done in small groups in rotating sessions. Participants seemed to value the equipment demonstrations. The FHWA's Terry Arnold was clearly impressed by the sensitivity of the handheld XRF equipment to detect lighter elements.

Following the demonstrations, the state attendees went through the technology comparison tables created by the SME, Maria Chrysochoou and provided their opinions (see Appendix A for the XRF Equipment Comparison Table and Appendix B for the FTIR Equipment Comparison Table). After lunch, the IAP states and a few others interested in the conversation remained for a special discussion of the IAP work plans. The discussion addressed questions regarding specifics of each plan and input on what equipment each state would prefer to use. A general schedule moving forward was outlined and quarterly meetings with the states will begin in January.

Some attendees suggested combining some of the technical trainings so that more than one state could be trained on the equipment at one time. The details and interpretation of how this might be adapted within scope of work and budget will be further discussed.

It's evident from the summary evaluations that all participants left the showcase with a much greater understanding of the R06B technologies. For example, only 53 percent of participants said they had moderate to extensive knowledge of both the XRF and FTIR technologies prior to attending the showcase. However, after attending the showcase that number jumped to 100 percent for the XRF technology and 92 percent for the FTIR technology. In addition, 100 percent of attendees who completed the evaluation felt the showcase content was moderately to extremely effective, 93 percent felt the presentations from the state agencies were moderately to extremely effective, and 100 percent felt the presentations from the vendors were moderately to extremely effective. However, one attendee expressed that the showcase felt rushed for time with not enough time spent on the content. Overall, it seemed attendees were pleased with the showcase and felt it was valuable. To view all of the responses and comments from the evaluations, see Appendix C.

Notes from the Showcase Presentations

DAY 1: VENDOR AND IAP STATE PRESENTATIONS

Welcoming Remarks

Steve Cooper, FHWA and Kate Kurgan, AASHTO

-We are implementing 79 projects in 37 states. Implemented more than $130M of funding to 99 entities. 63 products and 430 projects underway.
-After this showcase, we will have a report and post PowerPoint presentations to AASHTO SHRP2 website.
-Currently, we are working on a Primer to increase awareness of how XRF and FTIR can be used to test construction materials in a nondestructive manner. The Primer is in draft form right now.
-AASHTO is working on marketing/communications plan on how to get word out on this product to DOTs. There will be videographers on site to capture interviews for future marketing.
-Hands on assistance from AASHTO, FHWA makes SHRP2 different from most other federal programs.

Maria Chrysochoou, UCONN: Introduction of *Techniques to Fingerprint Construction Materials* (R06B)

-Initial R06B project evaluated several spectroscopic techniques, then narrowed down to XRF and FTIR.
-The challenge is to test materials quickly – portable spectroscopy in the field – test and receive results quickly or even instantly. Some of this works but not always and there are some challenges to be aware of.
-Spectroscopy relies on the same principle as a prism. It utilizes light, which interacts with the material and is re-emitted as light with different properties. It is necessary to know what type of radiation is employed and what interaction takes place to be able to interpret the emitted radiation.
-Radiation interacts with the material, is emitted and captured by a detector to produce the spectrum data. The results should tell what material is being tested and what the properties are.
-XRF and FTIR employ light on opposites of the frequency spectrum, XRF is high frequency and FTIR low frequency.
-The goal of employing spectroscopy is to select technologies that work on a variety of materials.
-Physics of XRF analysis:

- X-rays interact with the electrons around the atom nucleus that are arranged in circular orbits called shells that have names K, L, M etc.
- Electrons are excited by the X-rays and this results in re-emission of secondary X-rays. The energy of these secondary X-rays depends on the shell of the electron and on the type of atom. Thus, the emitted energy is characteristic of the atom and the shell and is named accordingly (for example Ka line of Zn)
- The spectrum is used to identify the elements based on their characteristic energies and the intensity of the emitted energy is used for quantitative analysis.

-XRF testing procedure:

- The sample and the X-ray source come into contact. X-rays are emitted, interact with the sample and captured by a detector. An internal calibration is used to report elements and associated concentration. Portable units can be used in a point-and-shoot mode for materials in situ.
- Select calibration, focus on sample, push the button, and the results are projected on a PDA screen as a table. End users don't see the calibrations which renders the use of the equipment easy but can hinder interpretation of results in some cases.

-XRF calibration issues:

- Typically minimum of two calibrations for different range of concentrations. May have specific requirements for additional calibrations for example liquid or other matrices.
- Not always easy to navigate the options as you can choose a calibration without really knowing what it means.
- In certain cases you may need to come up with your own calibration curve.

-Key Points:

- Measurement time – the longer the measurement time, the higher the precision – typically one to five minutes.
- Instruments will not give you an upper level of concentration – you may be out of calibration level and not know it.
- Concentrations of other elements present – sometimes multiple elements will obscure neighboring elements present. Software is available to deal with this but the user must be aware.

-Detection limits: the lighter the element the higher the LODs. Refer to testing time and matrix.
-Penetration depth: How far are the X-rays going in? Where are they measuring? Your materials have a finite depth so you need to know where your measurement is coming from. X-rays will go farther in water than in a silica matrix. What you're testing influences where you are testing. Moisture content has an impact and will change overall response. Instruments are highly sophisticated but depend on operator's knowledge and interpretation.
-Also have to think of penetration depth of emission (coming back out of material)
-Can't change penetration depth on hand held devices.

- For example - Wet paint on asphalt. You have to realize the things you measure are not coming from the same place – heavier elements like lead will be detected even if it's below the surface layer of paint you are measuring.

-Potential Applications:

- QC for epoxies, thermoplastics and traffic paints (draft AASHTO developed by R06B, American Society for Testing and Materials (ASTM) standard exists D5381)
- Detection of Pb and As in glass beads
- Detection of Pb and other contaminants in bridge paints, soils or any other materials
- Detection of motor oil admixtures in asphalt on the basis of trace metal content (e.g. Zn)
- Detection of lime in asphalt
- QA/QC of Portland cement and aggregates
- Analysis of alloys and steel metal grades

-Standard Methods

- SW-846 Test Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment
- AASHTO TP 106 Standard Method Of Test For Determination Of Heavy Metal Content Of Glass Beads Using X-Ray Fluorescence (XRF)
- AASHTO M 85 Chemical (Oxide) Analysis by X-Ray Fluorescence (XRF)
- AASHTO T 105-14 Standard Method of Test for Chemical Analysis of Hydraulic Cement (ASTM Designation: C 114-11be1)

-XRF Advantages:

- Automatic Reading – No analysis experience required (with caveats)
- On-site readings possible for multiple elements = decrease number of required tests
- One to five minute testing time
- Little or no sample preparation required
- No maintenance required – costs only associated with equipment acquisition

Christian Balotescu, Thermo: Major XRF Application Areas:

- Rebar Testing in Bridges – Identifying grades of steel
- Measurement of Corrosive Elements
- Cement/Grout Inspection
- Good for Magnesium and elements with higher atomic number.
- Detection of lead in paint – very effective.
- Detection of Contaminants in Soils near Bridges.
- Highway Markers – lead and arsenic in glass beads
- Recycling Existing Structures
- Highway Sound Barriers – galvanized coating thickness.
- Portable XRF in Transportation – Snowplows.

Maria Chrysochoou, UCONN: Fourier Transform Infrared (FTIR) spectroscopy overview and potential applications for construction materials.

-Infrared radiation has lower energy then X-rays.
-FTIR is best for testing asphalt, polymers, and organic materials.
-FTIR detects bonds between atoms.

- Bonds absorb energy and dance. They stretch and bend in different ways. Infra-red vibrations can be measured. We are detecting the movement of molecules that only dance at certain frequencies and using the measured frequencies to identify materials.
- Single element bonds don't vibrate– nitrogen and oxygen will not measure because they don't have a different partner to create a bond.

- Quantitative analysis can be set up by relating peaks areas to known concentrations – but this doesn't work in the presence of additional compounds.

-Two main types of equipment.

- Solids need ATR or DRIFT accessory – single base equipment with accessories that are interchangeable for different samples.
- ATR – Beam touches surface of sample – lower penetration depth than XRF – ATR is a strictly surface method because of lower radiation energy.
- Diffuse Reflectance (DRIFT) FTIR – ATR such a tiny surface it is hard to get representative spectrum of the entire material. DRIFTs probe multiple surfaces for more representative results.

-Three main types of crystals

- IRE Diamond is very robust
- Zinc selenite is research grade.
- Germanium is not as hard as diamond – can be damaged.

-FTIR is complicated for interpretation. What you are seeing in your spectrum is not materials (not asphalt, polymer) but the vibration of functional groups of molecules. You see these in many combinations and of multiple bonds. Ultimately, qualitative analysis is matching the configuration of peaks with particular materials. "This peak is a double carbon-bond and the overall spectrum appears like latex."

- Must establish a library of reference spectra for specific material types. States can work together for this.
- Must have general idea of what you are looking for. Narrow your idea of what you are looking for.
- Use existing databases to narrow down type of functional groups.
- Use complementary techniques to corroborate your suspicions.

-Spectral Libraries

- University of Texas at Austin comprehensive library of Chemical Spectra and Spectral Data: https://www.lib.utexas.edu/chem/info/spectra.html
- National Institute of Standards and Technology Database (16,000 compounds): https://srdata.nist.gov/gateway/gateway?property=IR+spectra
- EPA FTIR Spectra Database (organic contaminants, solvents): https://www3.epa.gov/ttnemc01/ftir/refnam.html
- Spectral Library for Organic Compounds (searchable by wavenumber): http://sdbs.db.aist.go.jp/sdbs/cgi-bin/cre_index.cgi
- Fiveash Data Management FTIR and Raman Spectral Libraries: http://www.fdmspectra.com/

-Potential Application for Construction Materials.

- Evaluation of RAP/RAS properties
- Detection of lime in asphalt
- Identification of additives in asphalt binders (e.g. polymers)
- Identification of additives in Portland Cement Concrete (accelerators, retarders)
- Has promise and can be developed further.

-Advantages:

- Little to no sample preparation required (caveat: your job is to make sure sample is representative)
- Actual testing time is 3-5 min.
- Applicable for wide range of materials, including organic materials that are insensitive to X-Ray-based methods
- Little maintenance or operation costs

Questions on XRF and FTIR:

-Health aspects?

- Both portable devices are rugged.
- XRF needs OSHA certification for use.

-Does an agency need to specify that contractors need to use this technology?

- There are other ways, but typically contracts will say you need to use X technique/lab method and measure to specific standards.

-In comparison to XRF inside lab which takes a long time on metals –

- XRF gives us better results in shorter time.
- Use XRF for screening.

-Can you use this technology in court from a challenge by the contractor? Not if you do not have an approved specification. You will need to verify with an acceptable method to verify.
-Can you control the depth of penetration of the X-rays? Not with the handheld models.
-Are the specifications and material available for these technologies? Yes, Maria indicated you can download material from the sites referenced in the presentations.
-Can you come up with a threshold to detect REOB or ground rubber? Terry Arnold indicated you can.
-Alabama has an XRF. Do you use a backup method to check it? Yes, we created a curve but need to have a way to verify it.

Yangia Wang, Agilent Technologies: Major FTIR Application Areas

- HMA/RAP
- Ground Tire Rubber Quantitation
- Thermally Degraded Concrete

Terry Arnold, FHWA: FHWA Chemistry Lab Experiences Benefits and Limitations of the Technologies-Methods and Applications Developed

-Terry explained various FTIR Spectrums on various tests – to show how to determine elements in mixes.
-Oxidized Asphalt – how do your account for aggregate in RAP? Can you extract the binder, filter it, solvent recovery to remove aggregate (but XRF will show tiny amounts left behind)? You have to purify the asphalt.

- Be careful you know what you are looking at when looking at RAP. Western Research Institute has done a lot of research on it.
- Turner Fairbanks has done extensive research and is quite good at analyzing data.

-What is lime? – Many kinds of lime… Ca(OH)2 we are talking about Calcium Hydroxide.
Limestone is Calcium Carbonate or Calcite CaCO3 (Home Depot), dolomitic limestone is a mixture CaMg (CO3)2
-Turner Fairbanks has 1500 asphalts measured from around the country. (Some 10-25% REOB)
-They have a round robin group working to determine the test bed of known REOBs.
-REOB Calculator – helps you recognize if you have recycled engine oil bottoms.
Next Steps:

- Use your imagination! (Engineers need to be chemists too)
- Measured bolts, asphalt samples and other materials.

-Portable equipment of both technologies seven years ago was not performing well so it didn't make the grade for the research. *Terry was impressed with the Showcase on Wednesday and the results from the current portable equipment.*

Maria Chrysochoou (Moderator): Agency Experiences Roundtable Discussion: Challenges and needs for nondestructive materials testing

- State experiences with spectroscopic techniques
- Benefits and limitations
- Wish list for materials testing

ALDOT, Andre Jenkins

-Alabama has a new XRF to assist them with measuring ultra-low sulfur content in diesel fuel. Found an online method to analyze diesel fuel. The method required some things we didn't have but the XRF worked as far as we know. Began to create standards from lab work, then compared with the XRF and found the numbers were same.

-The lab includes two FTIR with ATR attachments, although the ALDOT methods don't require the ATR but may in the future. All lab equipment, no portable so far. Using dissolved asphalt samples in trichloroethylene. They have standards for each type of asphalt that are needed.
-Every time a different asphalt is presented the lab must start over. The same asphalt from the same producer can yield different results every time. All binders are also tested.
-Alabama's first Advantage 330 XRF was purchased in 2000. May need alternate power supply at 20keV which also could affect the calibration process. Unfortunately, they accidentally oxidized the tube by losing the helium purge but don't plan to make that mistake again.
-There is too much work to have time for vendors to come and approve results.

- Theoretically the lab approves asphalt before it's laid down. The procedure is to get asphalt approved before using it. The reality is often the lab is catching up with the contractors. This can lead to QA/QC results that suppliers will be cautious on.

-XRF is applied for many uses. The TX method determines titanium dioxide in thermoplastics.
Total petroleum hydrocarbons in soils is the main reason Alabama tests the way they do. Need to test TPH soils at the site instead of back at the lab. Smaller resources and more need to have these.

- Tried to save time by using new equipment but lack of staff is a problem. New equipment is really nice. It is smaller and easy to use.
- Due to state budget issues the lab has half the staff with the same work load so this equipment helps save time and effort.
- Analysis of materials for the entire state depends on this lab.

-XRF and FTIR allow the Alabama lab to keep up with quality analysis much faster.

TDOT, Joseph Kerstetter

-Tennessee has had a Premier XRF from Australia for 14 years.

- Tests cement, fly ash, limestone, it is a time savings.
- Roughly $40-$50K new.
- Perlex at Turner Fairbanks is about $85,000.
- The equipment is very sensitive.
- XRF equipment needs more standard reference documents for standard matrixes.

-FTIR equipment (handheld portable) has only been used for a year.

- The lab has a knowledge gap to fill.
- The equipment is touchy and you have to know what you are looking for.

-Chemistry will tell you what engineering will not.
-Findings bring up questions for the lab to process.

- They did not want to hold a manufacturer to a standard with outstanding questions.
- Clear need to create a knowledge base – without chemists it is challenging to train engineers to be chemists.

-Get SBS polymers and play with them in the FTIR, play with each asphalt blend.
-Cement, paint samples, wood products for preservatives, reduction of chemical use.

VTrans, Jerry McMahan

-Virginia's lab has had a hand held XRF Bruker S2 Ranger for two years.

- Great sensitivity, has standard-less curves, some matrices are accurate but not all.
- Sodium content in Portland cement is accurate upon testing. It saves time instead of using wet chemistry tests.
- Looking for a more standard reference for practices. Looking to set up their own standards.
- Fly ash and cement calibrations are different.
- Definitely saves time.

-Virginia had questions regarding paint measurements. VDOT uses a standard-less process for paint testing. PennDOT runs XRF for paint on a standard-less program.

- The calibration has no standards.
- Virginia has made some lead standards but haven't used them.
- They take screen bid samples every spring. High counts are sent to a third person lab.

-Asphalt requires a clean standard (asphalt is so varied – you can run it 10 times and get different numbers).
-FTIR and XRF instruments need to create a knowledge base so the more people are using it and adding info; will help us use the equipment better and get clearer results.
-Virginia staff are currently novices with FTIR.
-The FTIR Bruker alpha has been used for five months at VDOT.

- Primary use is to check binder additives.
- Non-approved.
- New issues with new knowledge base.

Day 2: EQUIPMENT DEMONSTRATION

The day began in the lab with all five vendors providing demonstrations of their equipment. Small groups of participants rotated through half hour presentations. The participants then gathered for a discussion of their impressions of the vendors' products and their thoughts moving forward.

-Kate Kurgan from AASHTO said that for the three proof of concept states AASHTO would like to buy the technology the states prefer – if there is extra we want to know what the other states are interested in too.
-Pros/Cons: what did you see and what do you like?

- Alabama (Lindy Blackburn): The technology from AASHTO – do they have to be provided from the same company for each of the three teams? There has to be a clear rational for each purchase – whether the state or AASHTO.
- Olympus was a favored XRF although all of them have the same qualities.
- The FTIR price comparison did not seem to the participants to equate to a better quality product.

-AASHTO is buying one FTIR (for Alabama) and two XRFs
The states recommended the Bruker FTIR.
For XRF - Alabama and Tennessee like the Olympus Vanta (not realizing there was a price increase). Maria suggested that being the newest technology there may be a learning curve to use it.
-Michele from Iowa thought the Thermo XRF was amazing.

DAY 2: WORK PLAN DISCUSSION

Kevin Chesnik, ARA: Remarks about Work Plans

-Kevin walked thru the Work Plan Template and the Schedule Spreadsheet. The effort was to help the three states provide more detail in their work plans.
-AASHTO's job is to make sure things get done according to plan and budget – no surprises. This helps AASHTO budget time to support as needed.
-Technical details help teams understand where they are at and the scope of what it takes to get to what they want.
-Tennessee brought up the issue of choice of materials to test and regulation barriers.

- Titanium Dioxide levels in Thermoplastic Pavement Markings. (White Thermo Titanium 10 percent min) works well with the product.
- Silica Dioxide, Lead, and Arsenic levels in Glass Beads. (Silica 60 percent min, Lead and Arsenic less than 200 ppm each) was chosen because it is time consuming to ask bead manufacturers to test their glass beads (and somewhat unreliable).

- Silica Dioxide and Calcium Carbonate in Surface Quality Aggregate. (Type I Silica 40 percent min and Calcium 32 percent maximum; Type II Silica 30 percent min; Type III Silica 20 percent min; Type IV Silica 10 percent min) is our largest test material so it would be helpful to be able to test in the field.

State Work Plan Presentations

Alabama:

FTIR

-No existing in-field procedures for testing stock piles. Want to test piles for oxidation.

-Want to develop something totally new and useable for other states in the future.

-Sample sizes will be developed in the lab in process. Will have to play with a lot of sources to determine ability to test RAP and RAS (shingles are the first priority). Alabama allows for 5 percent shingles with huge variability among piles. For years they used 3 percent for years without issues. 5 percent causes a lot of problems.

-Breaking down the timeframe will help write bullets that make sense.

-Can FTIR be used to measure oxidation? Yes. Harder with unknown samples.

- Library development would be key – AL will target areas of the state (three regions), bring back shingles and rap bags and begin to build the library.
- What is the oxidation level I can live with? Shingles have oxidized liquid – so you can use 42 percent virgin liquids… shingles are a pain to work with for quality control.
- Shingles are useable but there is a need to be smarter on how to use them and how much to use.
- How will you come up with the acceptable levels? Still thinking about – using this technology to measure and characterize shingle piles to develop a specification for contractors to use and to mix.
- 35 percent recycle but over 25 percent needed extractions. Measurements are all over the board.
- Perceptions are so varied with respect to RAP properties.
- Need a better way to characterize our RAP and RAS piles.
- Starting with a single source – begin to develop a base and move forward from there.

XRF

- Similar – but there is already a 10 percent specification for TiO2 in thermoplastics. One major producer of thermoplastics is willing to work with the state, Ozark.
- There is no reason to recalibrate the XRF if you can adapt the settings for your uses. If you don't have to recalibrate – why would you – more work than you realize.

-Kevin was not sure all this can happen for $250K.

- RAP and RAS are a huge project. Field validation is done in-house and should not be too far from sites.
- It would be beneficial if AASHTO would buy the equipment – as the state can't sole source and have to bid it.
- State will not pay $70K but liked the Bruker. FHWA will support both types of technology for Alabama.

-AASHTO will coordinate when to purchase and the budget.

-Equipment delivery date is approximately January 2017.

-OSHA certification requirements?

- Equipment agreement between AASHTO and the DOT – do we need to add the certification requirement?
- If the lab has an XRF it is already certified. Tell state public safety department the lab is going to handle it well and with qualified people.
- Need certified people to use the technology.
- Vendor may in fact send a letter to the state safety radiation office.

Maine

-XRF –Maine's original plan for use was:

- Traffic paint titanium dioxide levels.
- Glass beads, and stainless steel rebar for strength.
- Proper alloys in stainless steel.

-Decommissioned atomic absorption – outsourcing chemical cement testing to a private lab.

-A portable XRF would decrease what has to be sent out of state.
-Desire to test suppliers.
-Bridge decks with chloride content in cores.
-Environmental department is excited to use it as well.
-Forensics on premature pavement with high copper contents – possibly RAS? Want to do baseline testing on RAS sources and then screen mixes for RAS additions.
-Originally focused solely on XRF. We would like to now consider possibilities with FTIR. Maine does not have FTIR but they do have a staff person skilled in FTIR for analysis.
The contract provides the flexibility to drop a few test materials and try an FTIR instead.

Maria Chrysochoou, UCONN: ***Closing Comments***
A general schedule moving forward includes:

- January 1 – obtain equipment.
- Quarterly conference call with states – set up for January.
- Training – end of March /early April.
- April 9-16, April 30 week, could be a joint training in Alabama.*
- Data collection in May.
- Specific training with Maria probably over the summer when data is available.
- Final drafts due towards end of November. Send a final copy to Kevin for review.

-States requested Terry Arnold from FHWA Turner Fairbanks be invited to as many events as possible.

*Maria suggested a peer exchange among states for a training on technology with similar binders. It might piggy back on what each state is doing separately. Alabama is willing to host. Peer training is within their budget. Maria can make two trips to each state to provide technical assistance.

Appendix A – Equipment Comparison Table XRF

Appendix B – Equipment Comparison Table FTIR

Appendix C – Evaluations

Appendix D – Attendee List

Appendix E - Agenda

Appendix A – Equipment Comparison Table XRF (at meeting)

	Olympus	Thermo	Bruker
Model	Delta Professional Geochem	Niton Xl3t GOLDD+	S1 Titan 800
X-ray Tube	Ag 4W	Ag 2 W 50 kV	Rh 50 kV
Detector	Standard SDD (165 eV resolution)	SDD (<185 eV resolution)	Fast SDD
Calibration	36 elements (Mg-U)	39 elemen ts (Mg -U) Mining + Soil	4 calibrations included in price (check again on limestone and industrial ones $1K each) Mg-U
Additional calibrations	not necessary, extended range in single mode	available but not necessary	
Docking station	Yes, but small, not equivalent to the workstation	Yes, mobile test stand	
Portable workstation	Not included in price, $2,750 in UCONN quote	available but not necessary, mobile is good enough	Yes, it is $2720 included in the 37K
Additional features	Software allows matrix effect adjustments	helium purge for light element LOD improvement	alloy calibration comes with integrated material libraries
LODs in Si matrix (mg/kg)			
Mg		6000 or 1500 with He purge	1000 or 7000 for different calibrations
As		4 (60 s analysis)	3 (120 s analysis)
Cost (with shipping)	$31,570	$35,115	$37,581
with test stand	$34,320		

Comments:

Olympus

- Can switch back and forth
- New one is about the same price.
- Only one is only a hand held - very rugged. Passed military grade. More geared toward hand held - so specialized in field materials.
- Liked the reporting screen better than the others
- Didn't have a mode for soil - but you could calibrate it yourself.
- Looks at everything every time you are on it.
- Stand will come out in December.
- Only one with single range for calibration

Thermo

- Does have pre radical calibrations.
- Modes are calibrations - has many.

Bruker

- Had a camera option.
- Mining modes/soil modes both give different results for the same materials. If you care about the precise data you need to know more about the calibration.

Overall Comments

- All are the same –it just depends on what you want your screen to look like.
- Warranties are the same terms for each.
- Will likely set up our own stuff once we have the equipment - not as likely to use the vendors.
- All can set up special calibrations - can ask them to do it as well.
- If states are okay maybe we should try them all to test them out. This is proof of concept for technology as well as vendors.
- Focused on these models - they make others as well. Maria chose the high end models because they can do light metals. Lower end technology was excluded.
- Light - Mg Si Al
- Why are there multiple calibrations - how did they do that? Need to follow up with Olympus.
- All three were coming up the same with the same samples.
- States will do their own testing for modes.
- Specifications: Need for paint specs. All three teams have spec requirements in their work.
- Need specs for contractor contracts.
- Will need standard test and spec to apply.

Appendix B – Equipment Comparison Table FTIR

	Bruker	Agilent
Type	Benchtop / portable	Handheld
Spectral Range	375-7500 cm-1	600-4500 cm-1
Resolution	better than 2 cm-1	4-16 cm-1
IRE	diamond, Ge or ZnSe	diamond or Ge
DRIFTS	accessory available, not included in price	accessory included in price
other accessories	flow through cell	external and grazing angle reflectance
	heat able plate	
	can be converted to transmission	
software	OPUS, collection and analysis	Micro lab Expert, data analysis, Mobile for collection
Price	21536	71824
	computer excluded from quote	

Comments:

Bruker

- A table top unit and not really a point/click. Not ease of use.
- Sample/lever keeps in place with right amount of pressure.
- Table top needs a ridiculously small sample. But the contact is the same - eye of the equipment is the same in both equipment. Sample size really is the same size in both.
- Supposed to be rugged and can be moved - you just need a table for set up.
- Florida could get by with the Bruker and a chisel and hammer - two for the price of one.
- Battery and wireless to laptop is good.
- Libraries are useless so far - we need to create our own.
- Ability to transfer libraries between machines - seems most adaptable.
- Currently reporting PDFs but working on other forms to download into machines.

Agilent

- On the market for 50-60K
- Warranties are very different.
- Workable in a quarry.

General

- We can interchange the crystals - with different calibration and library - true for both.
- Liked both of them.

Appendix C – Evaluations (condensed)

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

1. What was your subject knowledge level of these NDT techniques *prior to* this showcase?

	1	2	3	4	5	6	7	8	9	10
	(no knowledge)			(moderate knowledge)					(extensive knowledge)	
XRF	1	3	2	1	2	1	2	0	1	0
FTIR	2	2	2	2	1	3	1	0	0	0

2. What was your subject knowledge level of these NDT techniques *after to* this showcase?

	1	2	3	4	5	6	7	8	9	10
XRF	0	0	0	2	1	0	5	4	0	1
FTIR	0	0	1	1	1	3	3	4	0	0

3. How would you rate the effectiveness of the following elements of the showcase?

a. Overall showcase content

1	2	3	4	5	6	7	8	9	10
0	0	0	0	0	0	0	5	3	4

b. Peer presentations on these NDT techniques, successes, and challenges

1	2	3	4	5	6	7	8	9	10
0	0	1	0	0	1	3	2	5	3

c. Manufacturers presentations on equipment and software

1	2	3	4	5	6	7	8	9	10
0	0	0	0	0	1	2	2	5	3

4. Please indicate your level of agreement with the following statements:

a. This showcase provided me with a better understanding of these NDT techniques.

	1	2	3	4	5	6	7	8	9	10
XRF	0	0	0	0	2	1	2	3	4	3
FTIR	0	0	0	0	2	1	1	3	3	3

b. I understand how these NDT techniques can benefit my agency and program.

	1	2	3	4	5	6	7	8	9	10
XRF	0	0	0	0	0	1	4	4	1	2

Rushed for time – minimal content.

	1	2	3	4	5	6	7	8	9	10
FTIR	0	0	0	0	0	2	3	4	1	2

c. I found the format of the showcase encouraged active participation.

1	2	3	4	5	6	7	8	9	10
0	0	0	0	0	0	1	2	4	5

d. My participation in this showcase was worthwhile.

1	2	3	4	5	6	7	8	9	10
0	0	0	0	0	0	1	3	3	5

7. My expectations for what I would learn in the event were met.

1	2	3	4	5	6	7	8	9	10
0	0	0	0	0	0	1	2	5	4

8. The presenters delivered clear information.

1	2	3	4	5	6	7	8	9	10
0	0	0	0	0	0	0	3	5	4

9. What were the most important ideas you learned from the showcase?

The idea from Terry that you should have an idea of what you want to look for, before you go looking.
Possible field applications and future testing possibilities.
Quality of handheld XRF – potentially more useful than I had assumed.
What materials each type of device is best suited for.
Capabilities of equipment. Importance of building libraries.
Company technologies and vendors.
Mechanisms for material identification. Speed and ease of measurement compared with “wet” methods.
Specifics of instrumentation. Potential future applications.
Portability is important, operation is basically similar.
Portable FTIR

10. Are there questions or issues you wished the showcase had addressed that it didn't?

Not that I can think of.
Each individual performance tests and hands on with equipment and the experts helping to interpret data.
More information on detecting polymer and ground tire rubber in asphalt.
Quantitatively analyze for FTIR

11. What else could FHWA do to support you or your agency in learning more about these NDT techniques and innovations?

Get information about these techniques into the hands of state engineers.
How to videos and building libraries of elements.
Workshops like this one.
Provide funding for purchase or loan equipment
Develop master library.
Proper test techniques and new uses.
Provide for more accessories for FTIR
Experience review information.

12. What else could AASHTO do to support you or your agency in learning more about these NDT techniques and innovations?

Get information about these techniques into the hands of state engineers.
How to videos and building libraries of elements.
Workshops like this one. Additional peer technical exchange.
Develop standards
Specifications for steel and traffic paints and ad mixtures.

13. Please provide us with additional comments, feedback, or ideas related to this event or future SHRP2 events:

Having all 5 vendors showcasing their products was a very good idea.
Not crazy about the hotel or room for the meeting.
Very valuable information and discussion.
Very informative. Thank you for your efforts.

Appendix D – Attendee List

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

First Name	Last Name	Title	Organization/Agency	Email Address
Mark	Alley	Laboratory Testing Engineer	Maine DOT	mark.alley@maine.gov
Terry	Arnold	Senior Research Chemist	FHWA	terry.arnold@dot.gov
Christian	Balotescu	Senior Sales Engineer	Thermo Scientific Niton/Tech Measurement Associates	chris@techmeasurement.com
Michelle	Barger	Chemist	Iowa DOT	michelle.barger@dot.iowa.gov
Torey	Bell	CEG	Alabama DOT	bellto@dot.state.al.us
Beran	Black	Supervising Lab Scientist	New Hampshire DOT	bblack@dot.state.nh.us
Lyndi	Blackburn	Asst. State Materials & Tests Engineer	Alabama DOT	blackburnl@dot.state.al.us
Denis	Boisvert	Chief of Materials Technology	New Hampshire DOT	dboisvert@dot.state.nh.us
Richard	Bradbury	Director of Materials Testing	Maine DOT	richard.bradbury@maine.gov
Jennifer	Caban	Sales and Applications Support Specialist	Olympus	jennifer.caban@olympus-ossa.com
Kevin	Chesnik	Principal Engineer	ARA	kchesnik@ara.com
Maria	Chrysochoou	Associate Professor	University of Connecticut	maria.chrysochoou@uconn.edu
Stephen	Cooper	SHRP2 R06B Product Lead	FHWA	stephen.j.cooper@dot.gov
Andre' L.	Jenkins, Sr.	Alabama Chemical Testing Section Manager	Alabama DOT	jenkinsa@dot.state.al.us
Marc	Joyal	Engineering Technician IV	New Hampshire DOT	mjoyal@dot.state.nh.us
Joseph	Kerstetter	Research and New Products Engineer	Tennessee DOT	Joseph.Kerstetter@tn.gov
Kate	Kurgan	Associate Program Manager SHRP2 Implementation	AASHTO	kkurgan@aashto.or
James	Mahoney	Executive Director	CT Transportation Institute	james.mahoney@uconn.edu
Jonathan	Margalit	Business Development Mgr.	Thermo Fisher Scientific	jmargalit@comcast.net
Keegan	McHose	Molecular Product Specialist	Agilent Technologies	keegan.mchose@agilent.com

Jerry	McMahan	Chemist	Vermont DOT	jerry.mcmahan@vermont.gov
Fred	Morris	Sales Manager	Bruker Optics	fred.morris@bruker.com
Bruce	Niles	Chemistry Lab Supervisor	Maine DOT	bruce.niles@maine.gov
Rick	Rainville	Sales Representative	Bruker	rick.rainville@comcast.net
Greg	Sholar	State Bituminous Engineer	Florida DOT	gregory.sholar@dot.state.fl.us
Jozsef	Simon	Transportation Project Specialist - SR	Tennessee DOT	joe.t.simon@tn.gov
Joshua	Smith	Ph.D.	Agilent Technologies	joshua.smith@agilent.com
Jennifer	Smoker	Consultant	CH2M	Jennifer.Smoker@ch2m.com
Yanqia	Wang	Application Engineer	Agilent Technologies	yanqia.wang@agilent.com
Brenda	Waters	Chemist III	Pennsylvania DOT	brwaters@pa.gov
Zina	Zadoroshnaya	Principle Engineer Materials	New Jersey DOT	Zina.Zadoroshnaya@dot.state.nj.us

Appendix E - Agenda

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)** • 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)** • Fourier Transform Infrared (FTIR) spectroscopy overview and potential applications for construction materials	Maria Chrysochoou, UCONN Yangia Wang, Agilent Technologies
2:30-3:30PM	**FHWA Chemistry Lab Experiences** • 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** • 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 lab** 5 stations (3 XRF and 2 FTIR)	Thermo Scientific Bruker Olympus Agilent
	Break-out groups with 5-6 participants, approximately 45 minutes per station	
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 Work plan meeting**	Maria Chrysochoou, UCONN