MEETING SUMMARY

SHRP2 TECHNIQUES TO FINGERPRINT CONSTRUCTION MATERIALS (R06B) Peer Exchange, Nashville, TN

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Purpose

This Peer Exchange was intended to provide state transportation agencies and vendors an opportunity to hear experiences and lessons learned from the 3 IAP states and others using the technologies developed through the *Techniques to Fingerprint Construction Materials* (R06B) product, in the frame of the second Strategic Highway Research Program (SHRP2). Participants discussed 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 SHRP2 effort is coming to a close so the discussion included potential next steps post-SHRP2 and future plans of several participating states.

Attendees

States participating in the SHRP2 Implementation Assistance Program (IAP) for R06B attended the event (Tennessee, Alabama and Maine), as well as 9 other states (Florida, Connecticut, Minnesota, New Jersey, Iowa, Missouri, Massachusetts, Illinois, and Montana) interested in learning more about the R06B technologies. (See Appendix B for a full list of participants, both present and remote.)

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.

The peer exchange achieved its intended purpose. The first day was devoted to hearing from Subject Matter Expert (SME) Maria Chrysochoou, IAP State DOT's (Maine, Tennessee and Alabama) and FHWA (Terry Arnold) as well as getting input from the non IAP states (Florida, Connecticut, Minnesota, New Jersey, Iowa, Missouri, Massachusetts, Illinois, and Montana) in attendance. The IAP states provided their perspective on the results of their deployment efforts on both XRF and FTIR. Progress has been made to utilize these technologies and all indications seem to predict that more support will help promote their use. Both Maria and Terry provided more detailed explanations of how the technologies work and how to use them in the lab. The non-IAP states talked about their efforts using these technologies and

implementing their use in the lab and in the field. The second day provided a summary of Day 1 and continued discussions with Pennsylvania DOT. Then, all the participants spent an hour in the lab overviewing the equipment that TDOT uses. Finally, the morning concluded with a brainstorming exercise to get input from the participants on what the next steps should be to further promote the use of the technologies. Many ideas were generated, and four immediate items were provided in response to the question: "Is there anything you feel strongly FHWA should jump on in the next year?"

1. Support another Peer Exchange.
2. Visit Turner Fairbanks to obtain support and exchange ideas with the FHWA personnel.
3. Support continued talk – particularly through formation of a user group.
4. FHWA consider after acceptance of libraries.

Notes from the Peer Exchange

Danny Lane, TDOT, Steve Cooper, FHWA, Kate Kurgan, AASHTO: *Welcoming Remarks*
Danny welcomed everyone. Tennessee is excited about what other states are doing and ready to take this technology to the next level.

Steve Cooper thanked everyone for attending and reminded them that this Task Order ends Feb 2019. He encouraged the attendees to consider what the group would be interested in pursuing in the future, post-SHRP2. Questions to consider include: what state DOT's list as needs, what types of communication will keep these moving, and what steps should be taken to coordinate and aid more states to get involved. He welcomed everyone to feel free to ask questions and make comments.

Kate Kurgan gave a brief review of the SHRP2 program. Looking for innovative real-world solutions to 'get in and get out'. She reviewed the agenda and encouraged everyone to ask questions and participate!

Maria Chrysochoou, UCONN: Introduction of *Techniques to Fingerprint Construction Materials* (R06B)
Maria began her tutorial explaining that the principles of XRF technology, how it works, the types of XRF equipment and the procedure to use handheld decides to obtain XRF readings as a list of elemental concentrations. Maria also explained some of the nuances of XRF, for example it is a technology that measures a specific thickness of a material and also that the build-in calibrations may not work accurately for other materials due to matrix effects. Thus, calibration using standards of similar composition to the material of interest should always be performed when testing a new type of matrix.
The Advantages of using XRF include:

- Pre-calibrated for a wide range of elements
- 1-2-minute testing time
- Little or no sample prep required, depending on the material
- No maintenance required—costs only associated with equipment acquisition ($25-$40K)
- Several applications possible (more bang for your buck)

The Limitations with using XRF include:

- Built in calibrations only work for certain material types – development of material-specific calibration often needed
- Does not work for light elements (no organic materials)

Maria then described the principles of FTIR technology. Three types of FTIR include: Laboratory transmission FTIR that is suitable for gas and liquid analysis, typically not solids, Diffuse Reflectance (DRIFT) accessory: ATR accessory to probe granular samples, and Lab- or field-based Attenuated Total Reflectance (ATR) spectrometer.

Interpretation of FTIR spectra relies on the comparison between the peaks observed in the spectrum of a pure compound with the spectrum of a mixture. The FTIR doesn't see materials but groups of molecules. Different materials can share the same bonds so that assignment of a peak to a particular material is not always straight forward; -you need to connect the peak with other observations.
Some basic tips on how to do this include: create a library of reference spectra for specific materials, have a general idea of what you are looking for, use existing databases to narrow down the type of functional group you are looking for, use complementary techniques to corroborate your suspicions.
Spectral Libraries Maria listed include:

- Vendors may provide some
- https://www.lib.utexas.edu/chem/info/spectra.html comprehensive list of online libraries
- NIST database (16,000 compounds): https://srdata.nist.gov/gateway/gateway?property=IR+spectra and http://webbook.nist.gov/chemistry/vib-ser.html to search by vibration energies
- EPA database (organic contaminants, solvents): https://www3.epa.gov/ttnemc01/ftir/refnam.html
- Free library for organic compounds, searchable by wavenumber: http://sdbs.db.aist.go.jp/sdbs/cgi-bin/cre_index.cgi
- Commercial libraries such as http://www.fdmspectra.com/

The Advantages using FTIR include:

- Little to no sample preparation required (caveat: your job 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;
- No maintenance or operation costs, cost only capital investment.

The Limitations of using FTIR include:

- It is only applicable to functional groups that have molecular vibrations in the IR region, i.e. polar, asymmetric, covalent bonds.
- FTIR detects polar functional groups, not entire molecules, and as such the observed peaks are not necessarily unique to a single compound.
- Water molecules have very strong IR active vibrations, which can easily obscure IR active vibrations of other molecules when water presence is substantial, such as in aqueous solutions.

Questions:
Danny asked for future extensive training for DOTs as some don't have chemists on board.

IAP State Updates

TDOT – Joe Kerstetter

Tennessee has gained significant technical expertise from the SHRP2 program. The technical information and support has been fantastic. Tennessee had just purchased their first XRF when they were chosen for this program.
XRF uses: Silica and Calcium Carbonate in Limestone aggregate
Titanium in Thermoplastic (without a staff chemist they would like to use XRF for this)
Glass beads -lead, arsenic (MAP 21 tasks us to restrict lead and arsenic but the testing is expensive)
REOB & PPA in binder?

Glass beads: Currently required to test every lot of glass beads with the standard EPA test; they are hoping to do in-house testing to lower the costs. XRF can be used with the built in calibrations for this purpose. The boughta suite of Chinese standards that were soils dosed with heavy metals, ran them and saw that the built in calibrations accurately predicted heavy metal concentrations. In testing they used several methods of preparing glass beads: powder, pellets, and as is, but found that the preparation did not affect the result. Fused pellets are not analyzed accurately by energy-dispersive equipment such as the handheld.

Silica in Limestone test: The XRF can do it, but the data must be compared with the standards that span the range of silica concentrations in aggregates. TDOT utilized standards from both NIST CCRL to produce a workable calibration curve and this worked well

Sample preparation was also found to have significant influence on the results. In this case, in situ measurements were taken along with crushed samples and pressed pellets. Pressed pellet has the least deviation and highest accuracy. Because of this it is unlikely that XRF can be used as a true field method for this application However, it can be used in regional labs, in conjunction with the BPN results that have been used for years. The natural variability of aggregate has always to be taken into account, requiring multiple analyses to get representative results.

Calcined Bauxite – AI 203 – Calcined bauxite requires measurements of high Al concentrations above 90%. An attempt to prepare a calibration curve using Russian standards showed that the response was non-linear in that range and thus the handheld is not suitable for this application.

Titanium in Thermoplastic -This test appeared to be easy but it was not. It is necessary to calibrate for titanium. Standards were supplied by manufacturers at 7 different concentrations. The four curves showed similar slopes but different offsets and this could not be explained; likely, they were matrix effects because every manufacturer uses different binders. Terry Arnold at FHWA is working on developing standards from raw materials and further investigate this effect.

REOBs and PPA in Asphalt Binder -By working backward Tennessee was able to verify this worked. Able to verify and read from XRFs.

When FTIR really works!
In September of 2015, TDOT purchased two FTIR instruments for use in the Lab and Field. They have since created libraries for QPL products such as Texture Paint and Anti-Stripping Additives. Originally they collected fingerprints for an Asphalt Binder Sample library and have delved into research into some forensic analysis of Asphalt Cores. Using their analysis of binder in asphalt core they successfully caught a paving job using the wrong grade of binder. TDOT had suspicions that a contractor may have used the wrong grade of Binder based on sample testing. The job called for a PG 76-22, and the DSR %Recovery was about 75% too low indicating it was a PG 64-22. TDOT Field Ops personal questioned whether this PG 64-22 made it into the roadway. Field Operations went out and took some cores of the asphalt in question. The Asphalt Lab sawed off the layer of asphalt that needed to be verified. Then they did an asphalt solvent extraction on the mix from the road. Next they spun the resultant solvent and binder to force the dust to bottom as if they were about to run an Abson Recovery test. Instead of completing the Abson Recovery, instead they did an evaporation of the Trichloroethylene in an oven. The resulting spectra showed that the Trichloroethylene does not obscure the Polymer peak. The resulting spectra showed that **none** of the samples had any polymer content at all.

Tennessee is looking to work with other materials in the future.
Tennessee intends to follow Maine using XRF as a rapid test for Chloride Content of Bridge Decks. They will look at Sulphur content of Acid producing rock and soil and try to minimize costly third party testing. They will continue to library Binders using FTIR and will expand to include RTFO and PAV samples to look into aging effects of Binder and Polymer.

This is fantastic technology – it doesn't do everything, but it does more than we could do before.

Lyndi Blackburn – Alabama DOT

Lyndi presented some results and lessons learned from the Alabama experience.
Alabama tried to use FTIR to evaluate the degree of oxidation in Recycled Asphalt Pavement (RAP) and Recycled Asphalt Shingles (RAS). They planned to do this using the carbonyl peak in the FTIR spectrum that correlates with the level of oxidation in the material. They created a calibration curve using pure asphalt binder that was artificially aged in a PAV for several time frames. The calibration curve showed promising results, with the peak intensity increasing proportionally with time. However, when they attempted to evaluate spectra from field samples it was determined that the carbonyl peak was high even in fresh samples. This was because the field samples were modified with polymer. Thus, polymer-modified asphalt binder does not show the same IR behavior as the non-modified. Thus, it was

determined that this application required much additional research that was beyond the scope of the proof-of-concept scope of work.

Alabama intended to use the portable XRF to measure TiO2 levels in white thermoplastic traffic marking materials shortly after placement or at the site of placement. TiO2 is a substance used as a white pigment in thermoplastic traffic markings used on state and federally funded roadways. TiO2 is exceptionally efficient in scattering light, which when added to thermoplastic compounds increases the brilliance of the material. It maintains its whiteness when wet, which also enhances the wet visibility of thermoplastic materials. By determining and monitoring the actual amounts of TiO2 used in thermoplastic materials placed on state roadways, ALDOT hoped to validate the required amounts of TiO2 being used and better assure the overall quality of Alabama roadways. The minimum amount of TiO2 required by the State of Alabama is 10% by weight. Lesser amounts contribute to elevated safety concerns to roadway users by decreasing the overall visibility of the white lines marking the roadway limits. This level of quality assurance and verification will also insure that costs are in alignment with the quantity of product required by specification.

They found the Laboratory XRF and Portable XRF had discrepancies and in fact, the handheld seemed to have more accurate readings. They used an Olympus Vanta C Series Model DTMA 10073. The manufacturer did not provide the promised standards in powder form, but Tennessee stepped in and supplied some standards. The 3 methods of standard preparation are loose pellets, melted on top, and melted pellets. They evaluated 5 projects and made over 100 readings. One observation was that drop on glass beads caused discrepancy in field testing and wasn't accounted for in the calibration. The revised plan is a sampling mode in the field testing powder (while still in the bags) and using melted sample collected in pans (TDOT sampling protocol). They plan to get independent measurement of the TiO2 Content of those samples. Terry is providing calibration numbers for this effort.

Questions:
Pennsylvania asked regarding testing directly in the field if it was known when thermoplastics are applied if the thickness is being measured (there is a gauge that measures thickness, but construction people don't always use it). Lyndi responded that they do. Contractors refused to measure the center line due to safety concerns with oncoming traffic in the adjacent lane. This leaves having retro measurements on edge lines.
A question was raised about the panel samples from the TDOT method and interference of glass beads with the test but they can be flipped and tested without glass bead layer on top.

Maine – Derek Nener-Plante

The Goals of Maine's R06B work included: to maximize non-destructive testing, to reduce test time and cost and to reduce incorporation of out-of-spec material into DOT work. For XRF they wanted to test: chlorides in bridge deck cores, titanium in traffic paint, REOB in PG Binder, stainless steel rebar, glass beads for lead and arsenic, and to determine the presence of RAS in HMA. For FTIR they were testing: the presence of polymer in asphalt and creating an asphalt binder library. They found there were significant benefits to NDT testing including saving time, lower cost, reducing wet chemistry testing, (no chemist on staff), and reducing the incorporation of out of spec material into DOT work.

Chloride – Maine compared the results of a traditional wet chemistry method, titration, with the results of the XRF. Greater errors came with greater chloride contents. They could never get 0 with titration as there is never a complete absence of chlorides while the XRF has a detection limit below which residual chloride cannot be measured. In most cases the XRF and Titration were within the Titration range of error. There needs to be further work with known chloride values but overall the XRF provides sufficient accuracy to determine unacceptable chloride contents. In retrospect, a year ago, Maine didn't know they could use this to test titration but now they are able to quibble about .01 statistical differences that may not be that practical. They asked the question is it possible for 2 chemists to repeat this? Alabama asked Maine to please make sure repeatability is included in the AASHTO standards.

Maine wanted to determine if they could point and click on a piece of concrete to get accurate results or if they really needed pellets from pulverized cores. Core testing was found to have higher variability because the area of

measurement is not representative of the entire material; for example, the operator has to make sure to avoid aggregate. Pellets from cores provided excellent correlation.

Conclusions from study of bridge deck cores include: pellets of pulverized material are superior to surface readings of slices, no binding agent is required, now in process of testing lab-prepared reference samples, now in process of validating correlation with independent split-sample comparisons. The outcomes have been valuable and could have forensic value given a bit more thought. While not yet ready to replace the statewide testing, Maine believes they could use it as a "definite fail" if not a "go-no/go" test.

XRF RAS – RAS is allowed in Maine's maintenance mix on low-priority mixes. Maine tested different RAS and mixed RAP/RAS piles to see if they could use an element as a marker for RAS presence and percentage. The results showed that the material variability is too high and also that the trace metals would be too low to detect once RAS became diluted in HMA. This application was not further pursued.

FTIR – Primarily PGAB binder verification gets both FTIR and XRF testing. Maine is adding these tests to the standard suite and building a library. They banned REOB but have a very limited binder supply. They use the same supplier for over 90% of the mix but that supplier changes its supply often using an open market. The real application would be looking for polymer and others on the road.

Challenges: Maine's challenges going forward include the further verification for chloride content and determining how to get a known amount. They want to determine how to fabricate that sample. They will need to develop internal procedures for chloride content determination and FTIR PGAB data analysis. Another goal is to look into galvanized coating thickness. Long term, they want to improve verifying manufactured materials such as rebar and guard rail with XRF. They are currently using the Thermo equipment.

Maine is looking for feedback for getting standards for chloride content. FHWA isn't sure what their involvement will be next, but Turner Fairbanks might help particularly to determine how to fabricate testing with pieces with known chloride.

Terry Arnold, FHWA Turner Fairbanks, "Know More Before You Pour"
XRF can find multiple elements at one time.
Theoretically you can measure lime in a stone.
You need to know what you are looking for before you test.
FTIR is useless with water. There is a tech brief on how to find REOB in your asphalt on Turner Fairbank web page.
https://www.fhwa.dot.gov/publications/research/infrastructure/pavements/18043/18043.pdf
Table in Public Roads – on status of REOB per state – last March 2018.
https://highways.dot.gov/sites/fhwa.dot.gov/files/publications/public-roads/3d-issue/2018-spring/index.html
https://www.fhwa.dot.gov/publications/publicroads/17sept/03.cfm
Ground Tire Rubber composition
Important Spectroscopy Rules

- Be sure you know what you are looking at (or you will make wrong conclusions)
- Spectrometers are DUMB – you have to tell it what you know – Calibration
- Spectrometry is Very Powerful – Can be extremely useful in Forensic Investigations
- It Does Not Do Everything

Experiences of Other States

Connecticut – David Howley

Currently CDOT has no equipment or experience. David shared that participating he has learned more than he knew coming into this event. CDOT is looking at chemistry (but they have no chemists now). They have an issue with pyrrhotite in concrete foundations -now we are considering getting back into chemistry. (If pyrite or pyrrhotite are present in the aggregate material used to make concrete, the material itself can be compromised. If water and air get

into the concrete through small cracks and holes, the iron sulfides inside can begin to break down, cracking the concrete and allowing more air and water in, causing even more damage.) The state still has a materials lab but they are just not testing with chemistry. UConn is now investing internal funds because pyrrhotite is naturally occurring in mass aggregate that over time disintegrates. They are using combo of XRF and other methods to narrow down amounts of reactive pyrrhotite. State legislatures are working on it as well – it's a huge issue for home owners as well as structural owners.
Discussion Points

- NDT tools are nice to have but states need to be able to justify the money and clearly show the purpose of the tests and that they are getting the data necessary to get work done. Maine is replacing a process with one that is cheaper. Peer research and other grants may be useful for money to obtain equipment.
- It's helpful to find issue you need to deal with then cross reference with equipment. The hard part is agreeing there is an issue. Work with local university to get a project underway, prove it worked with something small and show how it can be expanded.
- Many states are currently accepting materials on certificates.
- Pennsylvania also find veins of pyrite in the middle of projects and has to manipulate plans to avoid them and contain them to keep them away from water run-off.
- East Tennessee is also finding veins of pyrite material and is starting to look at this as well as sulfur content. If found, it has to be encapsulated. Middle TN has pavement currently with rust coming thru.

Massachusetts – Maggie McDonald

Terry Arnold came to help Massachusetts use the FTIR as they were able to purchase both XRF and FTIR equipment (desktop pieces) thru a research study on REOB project started in January 2018. Right now, they are using these machines for research and building a library. They are gaining knowledge and better understanding although not analyzing data as they would like. They are anchoring the FTIR, warm mix, binder, and agents as part of a library so eventually they will have a foundation of information to use toward whatever applications they want to do. Currently they have used XRF for binder but would love to use it for glass beads which require a calibration curve so they are open to any suggestions anyone else has.
Calibration will be different for different machines. Tennessee bought Chinese standards that were silicates dosed with heavy metals.

Florida – Cassady Allen

For many years now, Florida areas with road problems have brought samples to the lab for testing to find where the problems started. The state lab is divided into bituminous, pavement, structures, and all exist in different buildings. Often the staff of the bituminous lab will send samples to chemistry lab only to return with sheets of data, of which they were not particularly concerned with what came out. The chemistry lab is now short staffed with no time for testing our stuff for forensics. Cassady is the chemist who works with engineers and they discussed 2 years ago if the equipment could also be available in the bituminous lab. Instead of buying more desk top units, they decided to purchase portable equipment that could also be used in the field. Now they have a portable unit, calibrated in the lab, and send engineers on the road with explicit directions to shoot samples so all is well. A feasibility study showed they couldn't afford both portable units, so they purchased the FTIR. The Bruker unit was a learning curve. They are now analyzing polymers, liquid asphalt, and almost anything else that comes into the labs.

There have been some problems with polymers and they are still in process of creating calibration curves. Florida has multiple sources of materials and they are still developing master calibration curves. They treat modified different from non-modified, high polymer binders different than normal binders. Although they are still learning, they are using FTIR daily. One downside is that currently they cannot take it on the road. Although they have everything to load it on the truck but it's only good for liquid materials. When trying to use it for other things, it picks up too much noise to identify peaks. The goal is to take it to the field so they are still working on it. Another goal is to develop guideline and analysis measures for the producers as performance measures. The lab is looking to move forward with ICP in REOB.
Discussion Points

- This can be used as an extra security check such as when detecting polymer.
- Standardize practices within your DOTs.

- Maine found that binder testing is well and good, but they also have other tests to get there. The power here is to come up with a procedure to test what was laid down only an hour ago. Are we using this to verify they aren't ripping us off? Every approach to quality assurance is different including using certificates to get involved on site in the plant. Even if you are in the plant you may not know what is in the mix but having staff at the plant can be useful even when it's really the contractor's responsibility.
- Depending on whether you are in Quality Assurance mode or forensics mode it's useful to let the plant know you are checking and you have the ability to analyze the mix.
- Consider if changes to the methodology will have the same output. Sometimes it's not changing what we do but having a new tool to use. Occasionally there is a question, even from the contractor, who is not sure of his own mix, and now there is a tool to follow up even if not necessarily a forensic problem or a certification but just a daily problem. This is just one more tool to look at potential issues quickly.
- A lot of time is spent on asphalt but with glass beads, rebar and other materials, the concept is still the same. For those who need to sell it to leadership consider relating it to one of the big three materials where the most money is spent, plus throw the safety aspect into your plea such as the result of slippery roads. As this grows, states will learn new things and need to talk to each other and share experiences.
- One big factor is that many people are unaware of these technologiesand they zone out when data is presented to them. We think it's a great technology and there is some discussion on more FTIRs for districts but there is still a need to get it to the field.
- Do you have the DRIFT? No, just ATR.
- All DOTs are not created equal and many would love to have a chemistry lab like Florida does. Pavement is our biggest asset and every state needs to know what is in theirs. As more states come on board, more mass is created, and it will require specifications that can be supported by AASHTO and FHWA. 4-C has task groups working on this in parallel with SHRP2.
- Pennsylvania mentioned that standards for IR are valuable. This is often a required test, but not often in deep enough detail so that everyone is doing it the same way. There need to be general characteristics of an IR scan if everyone wants to see the same thing. NTPEP hasn't provided that level of detail so it is different from one state to another and lacks clear starting parameters. They are trying to institute a list of general requirements for scans. XRF will also need a general requirement list for consistent scans.

Minnesota – Jason Krogeman

Minnesota divides materials into 2 groups, mainly bituminous binders (black and sticky) and everything else.
They have used XRF for some time and work with FTIR for epoxy regulation and sometimes forensics. Minnesota purchased both the handheld XRF and FTIR 18 months ago. With the XRF they started galvanization testing alum and zinc and helping the Geotech staff identify rocks. They want to do more binder testing with FTIR to build a library.
Discussion Points

- What happens when a state builds up a bank of data on a binder then it changes out of nowhere, then what? Figure out why? Then what? What do you do about it? Do you not accept it? A: Use primary tests to verify, go back to FTIR to look at qualitative issues. It's not a performance test but it says "something has changed" and it changes every year. When finger printing construction materials, the fingerprints only work in comparison with something else and it's that comparison that leads in a specific direction. It may not show what to reject but provides a jump start to lead to the next step and it provides one more piece of the puzzle.
- MnDOT had extra funding after a 2-year cycle so now they are trying to get away from the AA method of galvanization of metals for a point and shoot method.
- Geotechs find random stuff and need to confirm they know what they have. This is an opportunity to spot check to see if more in-depth testing is required.
- Pennsylvania fingerprints a lot of things so if the method is accurate enough to pick up a change, this throws up a flag. If you have multiple tests they can provide trigger points to go back and check where there are questions. Where changes are evident it is possible to show when it was approved this was the fingerprint. It isn't necessary to say what is wrong, but that it has changed so the burden of proof is to show whatever is being provided now works just as well or better.

- Take caution when using fingerprinting as it makes sense but know the limitations. XRF doesn't see most of what is in thermoplastics. This gives only one piece of the puzzle. There may not be time to build the whole puzzle so take care about jumping to conclusions. Picking and choosing testing can mean there is a possibility that what was not tested may be where the change lays.
- Need to teach our staff how to collect the fingerprints and must have a process by which to train them.
- Rick Bradbury was suggested to survey the COMP. Maria will send the original survey from the beginning of the research to Rick who will send to the COMP members for input.

New Jersey – Darshan Patel (provided slides too)

New Jersey is currently using both FTIR and XRF for testing some construction materials. Using FTIR, we are testing Steel and Traffic Paints, and using XRF, we are testing Portland and blended cement, pozzolan, fly ash, slag, patching materials and glass beads.

FTIR helps New Jersey to achieve its goals for quality and color uniformity of all paint system. Currently New Jersey is using Rutgers IR Test Method to create standard paint libraries. This method saves fingerprints as standards for each approved paint. The evaluation of field sample is based on NJDOT's Correlation Threshold (CT). For acceptance of all field samples, NJDOT's Correlation Threshold (CT) requirement is 98 percent. If Correlation Coefficient (CC) is greater or equal to Correction Threshold (CT), then the sample passes. If Correlation Coefficient (CC) is less than Correlation Threshold (CT), then the sample fails. The benefits of using Rutgers IR Test Method are identifying paint contamination, sample non-uniformity, incorrect sample markings and change in paint formulation.

Thursday Highlights – Kevin Chesnik

- Each state does this in their own way but there is a common outcome everyone is working toward.
- There is documented procedure that shows what people are doing.
- The question for state DOT's remains, how to replace present procedure with new technology.
- Others are using this technology outside of SHRP2 now.
- Many people have different amounts of resources.
- Think about specific FTIR and XRF applications for your state.

Pennsylvania – David Kuniega

Pennsylvania has a lab with 4 staff and $250,000 worth of forensic equipment for cement and asphalts. Their XRF is used for cement compounds, the XRD equipment is used for compounds and crystals structures with aggregates. There is very little interaction between the chemistry lab and asphalt lab and only in the past few years because of REOB have they worked together. There is no library built as of yet.

Pennsylvania often accepts certifications and when they investigated hand held equipment (6-8 years ago) it was with many reservations. They needed alloy confirmation that the handhelds could not support as well as the table top equipment. Older handheld models did not perform well. One big issue is which corrosion on bolts on the back of signs were too small for the handhelds to accurately measure. Technology seems to have improved but you still have to consider the amount of preparation that is involved.

Discussion Points

- Maine had pointed out that there are traditional chemistry techniques and preparation before running analysis for a reason. Materials need to be in a format for the equipment to be able to see. Also, it is necessary to take the material in the state it's in and weed out other interferences. Just shooting a handheld into a RAP pile, directly into the material won't work.
- There is a whole world of possibilities in fingerprinting including routine quality assurance and forensics. Yet each looks at different things. You have to have targets and know to look for specific things. In order to move this technology, it is necessary to decide what to look at. Derek and Lyndi's examples are good, to look at specific elements such as chloride. States can do this. The materials being analyzed are compounds including

both organic and inorganic and XRF works for inorganic while FTIR works for organic materials. Understand the limitations and keep in mind that the test is looking at one small slice.

- This equipment is encouraged as another layer of checking – first line screening.
- Whether using XRF or FTIR, both need to focus on what you are looking for (not broad spectrums of things).
- With XRF because of the radiation components, states often need to account for every handheld with nuclear gauges.
- One point yet to be discussed is the safety of the equipment. Don't hold the sample in your hand while shooting X-rays. Radiation safety is a real issue. Bear this in mind. Need to train technicians using these. Tracking equipment is important to state inspectors and can create issues for owners.
- DOT materials specifications are much higher than environmental specifications. In environmental work it's all matrixes – the interferences are removed.
- Uniform matrices are not used. Each material will have its own matrix. In the long term, wet preparations will work to remove the noise.
- It's a good idea to look at this, as it will give inspectors a new level of check though not fool proof. Just the fact that we are looking for issues will put people on notice (manufacturers and contractors) and that makes a big difference. It is important to make assumptions that the element you are looking for is that compound – multiple tests on materials is needed along with some level of comfort with making assumptions.
- Darshan has a plethora of tests included for his paint.
- To develop this technology and go wider it also has to go uniformly. Not every material will be tested the same way with IR.
- As REOB work depends on averages and seems rather complicated but is it really doable? Is it possible to write an AASHTO specification with a spreadsheet? Terry's tech brief that has everything in it including examples on how to build a spread sheet. It's just not a format that can be translated into an AASHTO format. COMP standards don't get to the right level and uniformity is necessary in everyone's processes on how we look at this. Sharing data between multiple states will necessitate a higher level of uniformity. Internal DOT finger printing that's limited to a specific state's equipment will not need this level of uniformity.
- It would be helpful to have the manufacturers in this conversation. Maria suggested that maybe the tech brief should be sent to the equipment manufacturers to integrate into the equipment they sell to the DOTs. They would need to take into account the variability of REOBs. There will not be a standard test method in a court of law.
- Rick suggested to focus on a national standard of application of the test and include recommended practices. It was suggested to draft practice with details on how to calibrate and use. Use of XRF overall consideration. This would be very practical.
- One big hurdle that needs to be addressed is standards of application. Terry is writing a tech brief on using handhelds. FHWA doesn't necessarily have to write it – but a combined effort would be helpful. It would be the handbook on how to use handhelds.
- Establish the underlying reason we do what we do.

Illinois - Kelly Morse

Illinois doesn't do much field work and has no handhelds. They have instrument chemists that use XRF and FTIR regularly and they try to control field variables in the lab. Illinois is working on known polymers, calibration curves, and identifying REOB. It is very robust work in the lab, but they are worried about matrix effect controls in the field. How much value is there in what you collect in the lab? They are interested in the idea of showing potential problems that are investigated further in the lab. Kelly is concerned about standards and calibration curves with field materials.

Discussion Points

- A focused approach was suggested, namely to find one thing, eliminate the anomalies in the field, really nail one thing down and prove it first for field instruments.
- Kevin mentioned that Susan Martinovich as the SCOR Chair set aside 5 million dollars of research funds to look for future technology that would impact things 50 years from now.
- Things are progressing in our technology over the last 10 years. The industry knows what we want because of what we are telling them. It's there if they want it.

Missouri - Todd Bennet

Missouri has a physical lab with a handheld XRF for structural specs and a screen tool (large ppe readings result in sending paint to the lab). XRF is used in the lab to test glass beads and thermos plastics as screening tool for heavy metals. FTIR is used for fingerprinting, admixtures, and unknowns. He looks forward to hearing more. Not performance so much as second tier confirmation which can also become a performance measure.

Discussion Points

- Performance measures is where the money is going now.
- Missouri is the one state that makes everything a performance measure.
- Buy the best tools. When you have the money buy the best equipment you can find – it's worth the investment. Don't buy the bare bones – you don't know what you will want to use it for tomorrow.
- Send your spectra to Turner Fairbanks – they can help. Just pick up the phone.

Questions for tomorrow:

- What portable testing can be used to find Pyrrhotite? Is there additional research this group can piggyback on?
- What parallel tracks should be working in tandem? 4-C task groups, etc.
- What are our next steps?
- What are the safety aspects in the process?

Brainstorming Session

Moving Forward with XRF

- Advance calibration standards with material types (steel, glass beads, galvanization on aluminum pipe and guard rail)
- Work with environmental staff with water, soil (footprint with hazardous materials/impacts)
- Standards for chloride testing in concrete – for this to go forward there would have to be centralized program to create this standard. It would not be a single DOT
- Creating XRF standards for different materials
- Expand the number of standards available off the shelf with XRF
- Need for NIST to see the value to sell the standards?
- Different reasons to be checking aggregates – identify and correlate your aggregate sources
- Reach out to Turner Fairbanks for support with advancement
- Practice document/video series (TC3/Volpe) – major things you need to consider for method of analysis, how to set up a calibration
- Procedures for sampling – contamination of sample
- This doesn't fall into one technical subcommittee within AASHTO – probably don't want a standard for each material – but there will probably be unique things to consider about each material
- Need to have calibration standards to produce accurate XRF results for many materials.
- Aggregate peaks will mask some of the things we're looking for in asphalt mix.
- Challenging to find direct applications for project produced materials.
- Getting a hold on calibration standards for materials types, steel, glass beads, galvanization, cement, aluminization, environmental issues – water, soil, lead, residual hazardous materials.
- Standards for chloride testing for concrete. There will have to be a place that creates a standard to calibrate machines otherwise we are using our individual issues. There are high chloride cement standards – but 3-4 in the range you need. You could have about 8 to use.
- Limits to buying standards although they are helpful. True standards have a lot of work involved. Need availability to purchase standards along with what each state is individually using.
- NIST may need to see value in it. It's a daunting task.
- CCL has data to back up numbers – if enough states are involved, we could have enough data to build a more general standard.

- Peer group to answer issue of standards.
- Stainless steel rebar – done.
- Aggregate: different reasons for testing looking for different things. Use it to correlate your aggregate sources. 100 different materials different for every geology.
- ASR technology – looking at change of concentration of solution.
- Both technologies – practice document that covers the entire process – what you need to consider, method of analysis, how to set up calibration, (tech brief).
- Post REOB federal tech brief. (Jen)
- Procedures and policies – also procedures for sampling, contamination of samples.
- One challenge – this doesn't fall into one technical subcommittee at AASHTO – unless we do an individual AASHTO materials sample it doesn't follow procedure.
- It is on a level for a general equipment thing – but each material will have specific issues and a specific subcommittee.
- Having a document is good – but people don't read carefully. Having a video series to demonstrate how to do this and what to consider. TC3 Communication – on demand. NHI not so useful.

Moving forward with FTIR

- Advance calibration standards with material types (asphalt, paints, epoxies, admixtures).
- Work with environmental staff with organic contaminates in water, soil (footprint with hazardous materials/impacts).
- Expand the number of standards available off the shelf with FTIR.
- Reach out to Turner Fairbanks for support with advancement.
- Practice document/video series (TC3/Volpe) – major things you need to consider for method of analysis, how to set up a calibration.
- Procedures for sampling – contamination of sample.
- This doesn't fall into one technical subcommittee within AASHTO – probably don't want a standard for each material – but there will probably be unique things to consider about each material.
- Creation of a central database for construction materials – would have to be searchable by vendor because there will be variation – CSV file – needs to be unified file for database and each database linked to equipment type. Every equipment produces their individual files X and Y – it could be imported to a general file to marry data files across equipment types.
- Look into existing databases – and develop one specifically for the transportation industry. The RUFF Database – free databases (example: Crystal Sleuth https://www.softpedia.com/get/Science-CAD/CrystalSleuth.shtml) provide aggregates but not sure of others. Turner Fairbanks has a list of Infra-Red databases – not one from transportation so far.
- Combine both technologies in a practice document that covers the entire process – what you need to consider, method of analysis, how to set up calibration, (similar to a tech brief).
- Post REOB Federal tech brief on AASHTO web page. (Jen)
- Document procedures and policies – also procedures for sampling, contamination of samples.
- One challenge – this doesn't fall into one technical subcommittee at AASHTO – unless we do an individual AASHTO materials sample it doesn't follow procedure.
- It is on a level for a general equipment thing – but each material will have specific issues and a specific subcommittee.
- Having a document is good – but people don't read carefully. Having a video series to demonstrate how to do this and what to consider. TC3 Communication – on demand. An NHI course was considered not as useful.

Funding Moving Forward

- IAP states need to spend the remaining SHRP2 funding.

- Pooled funds which require a lead state and supporting funds might be useful for standard development.
- FHWA Turner Fairbanks possible support.
- Videos and training with FHWA support.
- NCHRP problem statements.
- FHWA Every Day Counts initiative.
- AII - AASHTO Innovation Initiative for proven technology.
- State DOT/UTC research

Moving Forward with Champions/Lead States

- Create a peer group regarding XRF standards.
- Create a list of champions from this peer exchange.
- State led pooled fund.
- FHWA led pooled fund.
- FHWA led Peer Exchange to continue the dialogue.
- NTPEP – tracking along the same lines – check with NTPEP leadership and AASHTO liaisons
- AASHTO Committee – COMP.
- Industry user group specific to material.
- User group annual meeting led by FHWA.
- Need to solve sample prep and calibration to get to the field for the technology.
- Survey from 2008 in original research to be sent out to COMP.

Is there anything you feel strongly FHWA should jump on in the next year?

- Support another Peer Exchange.
- Visit Turner Fairbanks to obtain support and exchange ideas with the FHWA personnel.
- Support continued talk – particularly through formation of a user group.
- FHWA consider after acceptance of libraries.

How far have we deviated from original scope of the project?

- Originally the product focused on concrete (very little asphalt), but the goal is still to get to the field.
- Consider revising the goal to expanding field work and saving money in the lab.
- Survey to resend and then report out replies.

Thank you for your participation!

Danny emphatically stated this is not the end of this! We are excited and continue to use this equipment. We'd like to meet again. Kate asked that states please share with us anything you would like to add to our web site.
Steve thanked all participants.

Adjourned

Appendix A – Agenda

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 1 – Wednesday, September 26, 2018

Time	Topic/Presentation	Speaker
8:30 AM – 9:00 AM	**Welcoming Remarks**	
	• TDOT • FHWA • AASHTO • SME (Subject Matter Expert) • (Meeting Goals, Agenda Review, Introductions)	*Danny Lane, TDOT* *Steve Cooper, FHWA* *Kate Kurgan, AASHTO* *Maria Chrysochoou, University of Connecticut*
9:00 AM – 9:30 AM	**Overview of XRF and FTIR Technologies for Construction Materials** • Background • Limitations • Enhancements, etc.	*Maria Chrysochoou*
9:30 AM – 10:15 AM	**Lessons Learned - TDOT** • XRF use for QA/QC glass beads, aggregates, cements, thermoplastics • Lab and field tests – protocol development • Challenges and path forward	*Joe Kerstetter, Tennessee DOT*
10:15 AM – 10:30 AM	**Break**	
10:45 AM – 11:30 AM	**Lessons Learned - ALDOT** • XRF use for thermoplastics • FTIR use for polymer in asphalt, Rap and RAS testing	*Lyndi Blackburn, Alabama DOT*

	• Lab and field tests– protocol development • Challenges and path forward	

11:30 AM – 12:15 PM	**Lessons Learned - Maine DOT**
	• XRF testing for analysis of
	  ○ Chloride in concrete
	  ○ Pb in paint and contaminated media
	  ○ Cements
	  ○ Steel
	  ○ RAS
	• FTIR testing of polymer in asphalt
	• Lab and field tests– protocol development
	• Challenges and path forward
		Rick Bradbury, Maine DOT

12:15 PM – 1:30 PM	**Lunch at Nearby Venues**	
1:30 PM – 2:15 PM	**FHWA What's In Your Asphalt?**	*Terry Arnold, FHWA*
2:15 PM – 3:00 PM	**2018 Experiences Non IAP States** • Connecticut • Florida • Minnesota • New Jersey • Pennsylvania • Others on the phone	*David Howley, Connecticut DOT* *Cassady Allen, Florida DOT* *Jason Krogman, Minnesota DOT* *Darshan Patel, New Jersey DOT* *David Kuniega, Pennsylvania DOT*
3:00 PM– 3:15 PM	**Break**	
3:15 PM – 4:45 PM	**Continuation of Experiences of Non IAP States and Open Discussion**	*All Participants*
5:00 PM	**Adjourn**	
6:00 PM	**Optional Group Outing to Downtown Nashville**	

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**	*Kevin Chesnik, ARA*
8:15 AM – 9:00 AM	**Turner-Fairbanks Lab Workplan for R&D of Spectroscopic Techniques**	*Terry Arnold, FHWA*
9:00 AM – 10:00 AM	**Moving Forward with XRF and FTIR Deployment Efforts** • 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	*Kevin Chesnik, Facilitator* *All participants*
10:00 AM – 10:15 AM	**Break**	
10:15 AM – 11:45 AM	**Hands on Laboratory Session at TDOT** • XRF demos for glass beads, aggregates etc. • FTIR demo for polymers in asphalt	*TDOT personnel,* *Terry Arnold, FHWA*
11:45 AM – 12:00 PM	**Closing Remarks**	*Steve Cooper, FHWA* *Kate Kurgan, AASHTO* *Maria Chrysochoou, SME* *Danny Lane, TDOT*
12:00 PM	**Adjourn and/or Head to Airport**	

Appendix B – Attendee List

Cassady	Allen	FDOT	cassady.allen@dot.state.fl.us
Mark	Alley	MaineDOT	mark.alley@maine.gov
Terry	Arnold	FHWA (Turner Fairbanks)	Terry.Arnold@dot.gov
Lyndi	Blackburn	Alabama Department of Transportation	blackburnl@dot.state.al.us
Rick	Bradbury	Maine DOT	Richard.bradbury@maine.gov
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Jason	Selvage	Alabama Department of Transportation	selvageg@dot.al.us
Jozsef	Simon	Tennessee Department of Transportation	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 C – Evaluations

Open questions:

9. What were the most important ideas you learned from the peer exchange?
I had no knowledge of this prior to my attendance. The basic overview by Maria was a great introduction. The applications overview from the different DOTs was an eye opener. Important idea: You need an SME to review the data.
What are the states using the NDT techniques for.
The sky is the limit, and can be used for multiple tests on the 2 different types.
Able to know what states are doing and their future plans. How far they search in order to implement new testing methodology.
The discussion/review from other states concerning how they are using the technologies was most beneficial.
Sample prep is important; calibration is critical; high level of training required.
Everyone was having the same issues.
Multiple applications available for XRF.
Areas where testing will and will not work.

10. Are there questions or issues you wished the showcase had addressed that it didn't?
No.
How to engage upper division managers/legislators on the XRF/FTIR concepts and how to apply them in a more widespread fashion.

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

Increase peer exchange events, so other states can know what all the states are doing.
Encourage specifications for these technologies and/or provide funding resources, experts in the field, as well as more peer exchanges.
Presentations.

12. Do you have any ideas or next steps necessary to move this forward in more State DOTs?

Still learning to operate both devices. The more I use the more I will gain knowledge.
Put more states under one roof and give cross training to each other. That will allow each state to learn and easily implement new testing methods.
Publish test methods.
Test is not a go/no-go but used to ID samples with possible issues then conduct additional tests.
Basic documentation for standard method for correct use of XRF & FTIR.
If ARA needs notes, they should record, transcribe and get notes to everyone. Aren't they consultants who get paid to assist?

Rating	Subject knowledge level prior to workshop XRF	Subject knowledge level prior to workshop FTIR	Subject knowledge level after workshop XRF	Subject knowledge level after workshop FTIR	Overal Effective - Content	Overall Effective - peer presentations	Better understanding of XRF	Better understanding of FTIR	XRF Benefits to my agency	FTIR Benefits to my agency	Fostered Active Particiaption	Particiaption Worthwhile	Expectations Met	Delivered clear information
1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
2	2	2	0	0	0	0	0	0	0	0	0	0	0	0
3	1	2	0	0	0	0	0	0	0	0	0	0	0	0
4	2	3	1	2	0	0	1	1	1	1	0	0	0	0
5	5	1	1	1	0	0	0	1	1	1	1	1	1	0
6	0	1	2	1	0	1	2	2	0	1	0	0	0	1
7	1	1	5	5	3	0	5	4	2	2	0	0	0	1
8	3	2	3	2	3	6	2	2	4	3	2	4	1	2
9	0	2	1	2	4	2	1	1	3	3	2	4	7	4
10	0	0	1	1	3	3	3	3	3	3	10	5	1	7
strongly disagree (1-2)	2	2	0	0	0	0	0	0	0	0	0	0	0	0
moderately disagree (3-5)	8	6	2	3	0	0	1	2	2	2	1	1	1	0
moderately agree (6-8)	4	4	10	8	6	7	9	8	6	6	2	5	4	4
strongly agree (9-10)	0	2	2	3	7	5	4	4	6	6	12	8	10	11
sum	14	14	14	14	13	12	14	14	14	14	15	14	15	15
% strongly disagree	14%	14%	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%
% moderately disagree	57%	43%	14%	21%	0%	0%	7%	14%	14%	14%	7%	7%	7%	0%
% moderately agree	29%	29%	71%	57%	46%	58%	64%	57%	43%	43%	13%	36%	27%	27%
% strongly agree	0%	14%	14%	21%	54%	42%	29%	29%	43%	43%	80%	57%	67%	73%