Technical White Paper

Asphalt Pavement – Micro-sampling and Micro-extraction Methods

Fundamental Properties of Asphalts and Modified Asphalts III Product: FP 09

March 2015

Prepared for
Federal Highway Administration
Contract No. DTFH61-07-D-00005

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INTRODUCTION

Background

When investigating hot and warm mix asphalt pavement performance and distress, the observed distress can provide clues about the failure mode, but often in order to properly diagnosis the distress or failure, it is necessary to sample and test the existing pavement. Pavement sampling is traditionally performed by coring. Common core diameters are 100 and 150 mm, but smaller or larger diameter cores can be collected. Cores smaller than 100 mm diameter are typically used for depth checks, cores larger than 150 mm diameter are used in special test applications.

After coring, laboratory testing typically consists of determining:

1. The volumetric and rheological properties of the core,
2. Recovery of the core asphalt, and
3. Chemical and rheological characterization of the recovered asphalt.

In some cases, the primary interest is in characterizing the recovered asphalt. However, a sticking point with characterizing recovered asphalt in terms of low temperature rheology using the bending beam rheometer (BBR) is that it takes a substantial amount of recovered asphalt. A BBR beam requires approximately 15 g, and usually several beams are needed. A 25 mm slice from a 150 mm diameter core will provide roughly 50 g of recovered asphalt. If slices thinner than 25 mm are required, for example to evaluate the oxidation gradient or just the extent of oxidation in the top 12 mm, then additional cores slices are required. The extra time and effort and copious amounts of organic solvent to extract sufficient asphalt for BBR often limits its application in characterizing the low temperature rheology of recovered asphalt. In terms of the amount of solvent: to recover all the asphalt from a core that is 50 mm thick and 150 mm in diameter would require roughly 12 liters (12,000 mL), whereas to recover the asphalt from a micro-sample weighing 150 mg would require about 100 mL.

During the Strategic Highway Research Program (SHRP), DSR and parallel plate geometry was considered for characterizing asphalt low temperature properties, but the method wasn't selected because it was recognized that DSR mechanical measurements at temperatures below about 5°C produced substantial compliance errors in the absolute values of the dynamic moduli.(1)

The limiting temperature of about -5°C for DSR parallel plate measurements due to instrument compliance has been resolved by measuring the instrument compliance and performing appropriate corrections to the data.(2,3) Reliable DSR measurements can now be obtained on asphalt samples at temperatures as low as -40°C. The method allows the use of 4 and 8 mm diameter parallel plates at low temperature.

During the initial method development of the low temperature DSR method, 4 mm diameter plates were used and the method was referred to as "4-mm DSR." Later, 8 mm diameter plates were included in the method. Although the method allows both 4 and 8 mm diameter plates, the method will be referred to hereinafter as simply “4-mm DSR.”

The 4-mm DSR method requires only 25 mg of material (in practice about 150 mg is necessary in order to allow sample trimming, etc.), which is several orders of magnitude less than the amount required to fabricate a BBR beam. Also, no specimen pre-molding is needed and a relatively low temperature (60 ~ 70°C) is required to load the samples into the rheometer. BBR m-value and creep stiffness S(t) are estimated through a correlation with 4-mm DSR developed by Sui et al.(4)

When performing a pavement investigation, and the primary interest is in characterizing the recovered asphalt rheology including the low temperature rheology, the 4-mm DSR represents a breakthrough in sampling requirements that makes simpler, small scale sampling and extraction of asphalt pavement feasible.

Definition of Micro-sampling and Micro-extraction

The method to collect small-scale samples, which will be referred to hereinafter as “micro-sampling,” involves using a rotary hammer drill, masonry bit, and dust collection system. Micro-sampling refers to a sample size of roughly 200 g of pavement. A draft method in AASHTO format is provided in the Appendix.

Micro-extraction refers to recovery of roughly 10 g of asphalt from about 200 g of pavement. The micro-extraction process involves many of the standard extraction procedures performed under AASHTO T319 only in a simpler smaller-scale manner requiring substantially less solvent.

The micro-sampling and extraction scheme is illustrated in figure 1.

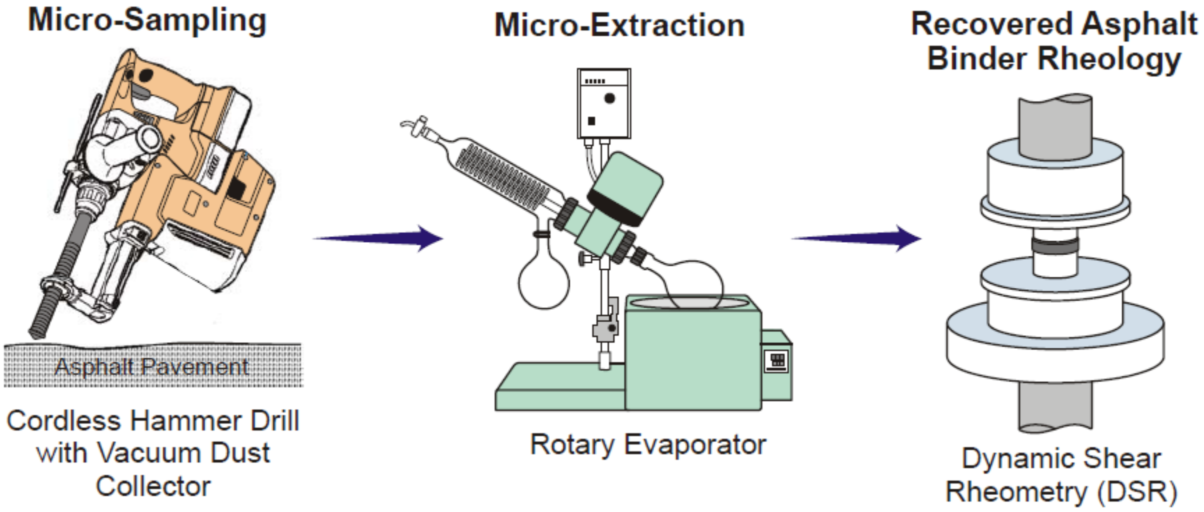

Figure 1. Illustration. Micro-sampling and extraction scheme.

Chemical and Rheological Characterization of Micro-extracted Asphalt

With the development of 4-mm DSR, it is now possible to collect micro-samples from the pavement and fully characterize the low temperature linear viscoelastic properties of the recovered asphalt. The intermediate and high temperature rheological properties can also be determined with DSR using 8 and 25 mm parallel plate geometry.

In terms of chemical characterization, micro-samples provide sufficient asphalt for chemical tests, such as:

- Fourier transform infrared spectroscopy (~200 mg)
- Gel permeation chromatography (~200 mg)
- Differential scanning calorimetry (~200 mg)
- SARA separations (~500 mg)

EXPERIMENTAL

Liquid cell infrared analysis was performed in the transmission mode with a 1.0 mm solution cell with 50 mg bitumen per 1 mL carbon disulfide. Thirty-two scans were co-added on a Perkin Elmer Spectrum 100 operated at a resolution of 4 cm^{-1}.

Dynamic shear tests were performed with a Malvern Kinexus controlled stress dynamic shear rheometer, and a TA Instruments ARES controlled strain dynamic shear rheometer.

RESULTS AND DISCUSSION

Micro-sampling

First Field Trial

The first attempt at micro-sampling by WRI was on a HMA/WMA project in Yellowstone National Park in 2007. The National Park Service would not allow WRI to collect cores (100 mm or 150 mm diameter) for aesthetic reasons, but did allow us to drill small holes with a rotary hammer drill and masonry bit as shown in figure 2. In this first trial a12.7 mm (0.5 in.) rotary hammer drill and 12.7 mm (0.5 in.) diameter masonry bit were used.

This first attempt at sampling with a rotary hammer drill and masonry bit appeared to work relatively well. The masonry bit completely pulverized the pavement and the powder from each hole was simply swept up into a plastic bag.

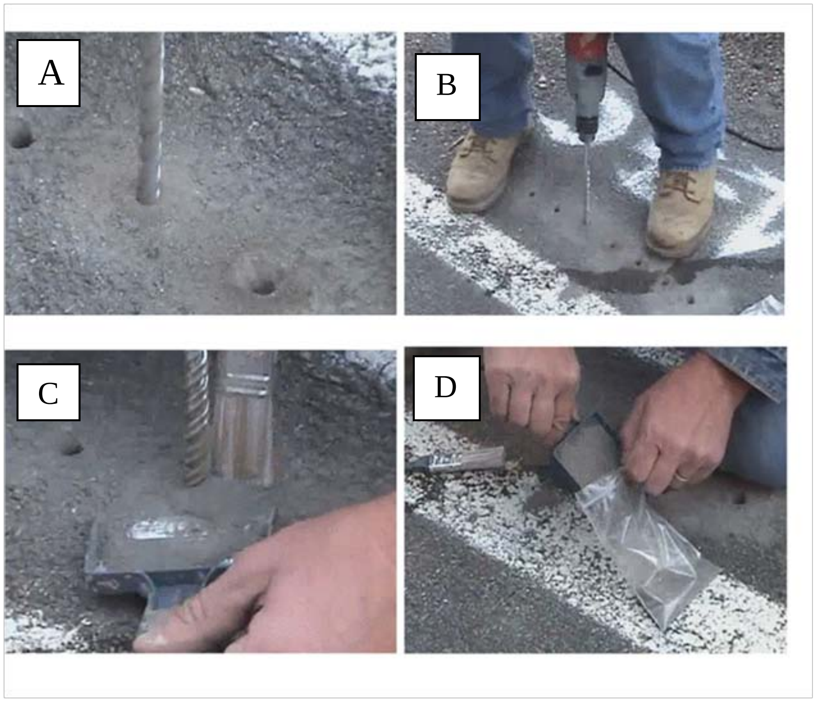

Figure 2. Photo. Micro-sampling. First attempt in the field on the Federal Lands, Yellowstone National Park HMA/WMA project constructed in 2007. A: close up drilling into asphalt pavement with a 12.7 mm masonry bit; B: expanded view of drilling; C: collecting the pulverized pavement; D: storing the pulverized pavement in a plastic bag.

Although this first attempt appeared to work reasonably well, there were a couple of concerns. The collection method of sweeping the powder off the road with a paint brush was not entirely satisfactory as there was concern that the sample was being contaminated with surface material.

Also, the drill bit, after a few minutes of drilling, was relatively warm to the touch, but did not appear to be excessively overheated. The asphalt pavement was significantly easier to drill than concrete, and that helped keep the bit temperature down. The temperature of the bit also appeared to be a function of the drill speed and pressure and how often the bit was withdrawn from the hole to keep its flutes clear.

The conclusion of this first trial was that this method of micro-sampling works, but several issues had to be addressed:

- Contamination of the sample from surface material.
- Potential oxidation of the pavement material during drilling.
- The pulverized pavement consists of very fine aggregate particles partially or fully coated with a thin film of asphalt. These thin films may be susceptible to oxidation even at relatively low temperature (30°C +).

Contamination of the Sample from Surface Material

As discussed in the previous section, the collection of the sample using a paint brush and miniature dust pan was not entirely satisfactory because of concerns the sample was being contaminated by material from the surface of the pavement. During the service life of asphalt pavement, the surface is subjected to high temperature and ultraviolet radiation which causes an inordinate amount of oxidative aging to occur at the surface compared to the underlying pavement. Using a paint brush to sweep the surface could cause a disproportionate amount of this surface material to be included in the sample.

To resolve this issue, a venturi collection system was developed as shown in figure 3. The venturi system is a centrifugal collector using cyclonic action to separate dust particles from the gas stream. The dust/gas stream enters at an angle and is spun rapidly. The centrifugal force created by the circular flow throws the dust particles toward the wall of the cyclone. After striking the wall, these particles fall into a collector bottle located underneath the venturi. A shop vacuum is used to draw the particles into the venturi.

An alternative method to the venturi system is to use a rotary hammer drill with a dust collection system as illustrated in figure 1. There are several rotary hammer drills with integrated dust collection systems commercially available, including cordless versions. The dust containers on these systems are typically large enough to collect a micro-sample (200 g ±), and the dust collected in the containers is easily removed.

Figure 3. Photo. Micro-sampling with a venturi collector system.

Maximum Temperature of Masonry Bit and Pulverized Asphalt Pavement under Sustained Drilling

Figure 4 summarizes the results of an experiment to measure the maximum bit and powder temperatures under sustained rotary hammer drilling of an asphalt pavement core. The sustained drilling was for a period of approximately 5 minutes. Maximum bit and powder temperatures of 105°C and 65°C, respectively, were observed. These temperatures are not particularly high in terms of causing unwanted oxidation to occur during the drilling process.

In this experiment, the rapid non-contact collection systems discussed in the previous section were not used, so the temperature of the bit and powder could be easily measured with a temperature gun infrared thermometer. The reported temperatures from this experiment will be higher than when using vacuum collection systems due to the reduced contact time between the powder and the warm drill bit with these systems.

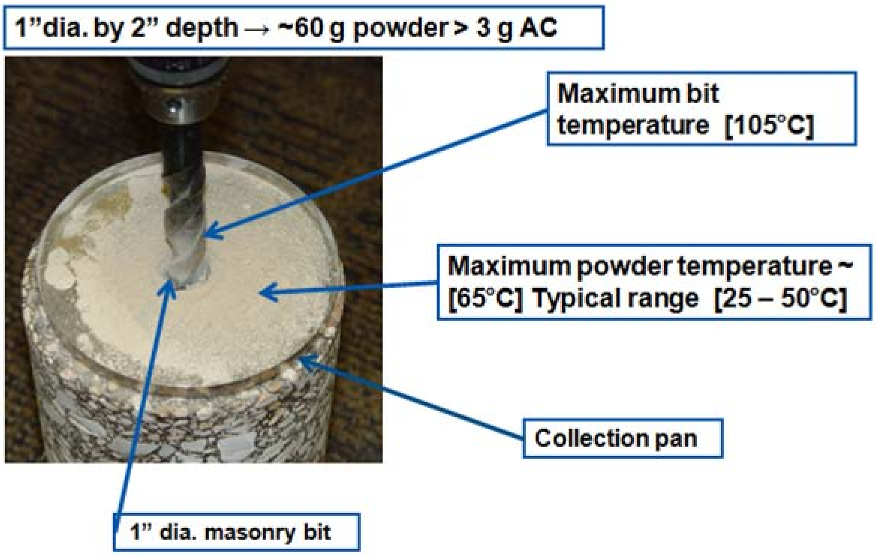

Figure 4. Photo. Micro-field sampling, temperatures during sustained drilling.

Oxidation during Rotary Hammer Drilling - IR Spectra and Rheology

Two experiments were conducted to evaluate the extent of oxidation from the drilling process. The first experiment just involved infrared spectrometry. The second experiment involved infrared spectrometry and rheology.

First Experiment (oxidation during drilling)

In the first experiment, the infrared spectra of recovered asphalt from rotary hammer drilling and an asphalt pavement core slice were compared. The drilling was performed from the top down with a 22.2 mm (7/8 in.) masonry bit to a depth of 25 mm. The powder from the drilling was collected with a metal pan similar to the one shown in figure 4. After drilling, the top 25 mm of the core was removed using a wet saw and an intact piece of the slice weighing approximately several hundred grams was used to recover the binder.

The asphalt binder material from the drilling powder and slice were extracted using an 85:15 (v/v) mixture of toluene and 95% ethyl alcohol. After extraction, the solutions were centrifuged to remove the fine aggregate material. The solvent was removed using a rotary evaporator with an argon gas purge to eliminate the exposure of the asphalt material to oxygen during solvent removal. The final stage of solvent removal was accomplished using reduced pressure and an elevated temperature. Infrared spectroscopy was used to confirm the removal of solvent.

The IR spectra are compared in figure 5. The carbonyl moiety is unchanged between the drilling and wet saw, which strongly suggests that drilling is not causing significant oxidation.

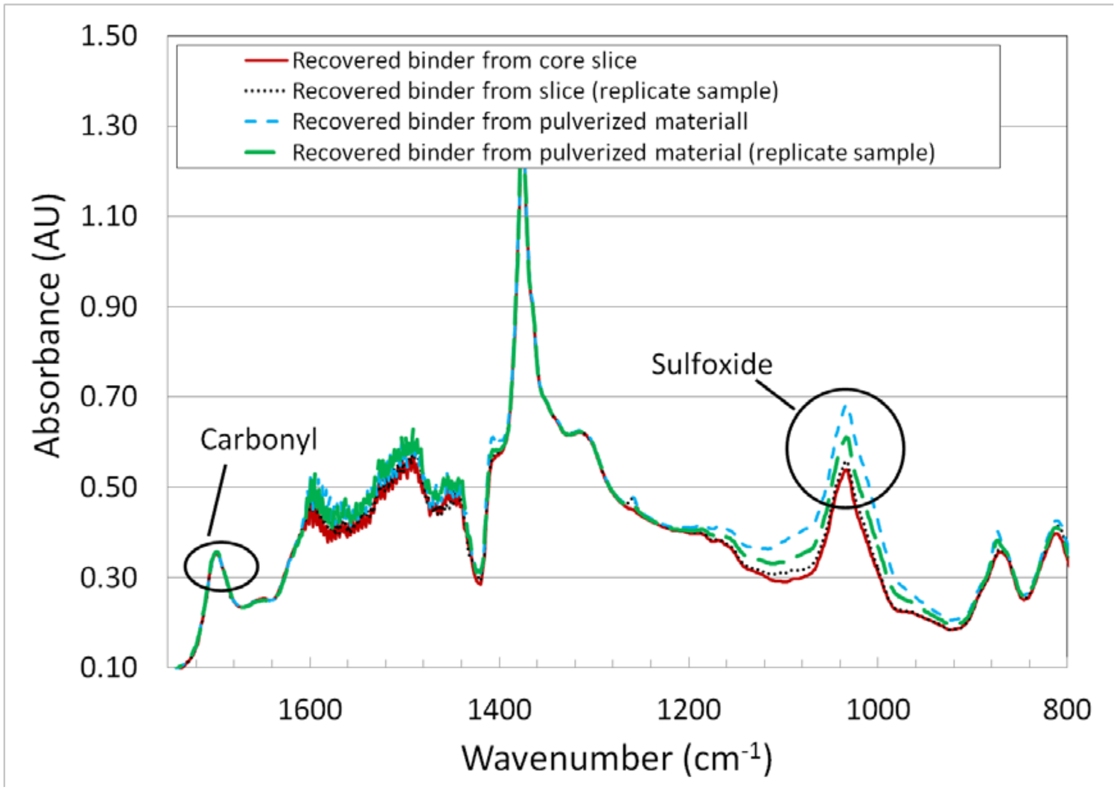

Figure 5. Graph. First experiment: Comparison of two samples obtained by drilling a core and two samples obtained from the same core by slicing with a wet saw.

In terms of the sulfoxide functional group, there was some concern with the relatively modest variation of the sulfoxide functional group absorbance between the powder and slice spectra. However, the sulfur chemistry of asphalt is complex and poorly understood, and several explanations for the variance are possible:

- Variation in sulfoxide in the recovered binders during the extraction process could be related to the surface area to total asphalt.
- If the drilling operation caused additional oxidation, it is unlikely additional sulfoxides would be produced without producing carbonyl in the highly oxidized core.
- Finally, the results may be simply due to non-homogeneity in the sample.

It appears the drilling process did not oxidize the asphalt in terms of carbonyl production, and the increase in sulfoxide is not necessarily attributable to the drilling process for reasons described above. This conclusion was verified in the second experiment discussed below.

Second Experiment (oxidation during drilling)

The second experiment was similar to the first experiment except the rheology of the recovered binder was examined in addition to infrared spectroscopy. Also, a venturi collector system as shown in figure 3 was used to collect the sample rather than a metal pan as shown in figure 4.

Both the IR spectra and rheology (G* master curves) as shown in figures 6 and 7 respectively, show that samples collected with the rotary hammer drill and venturi collection system are essentially equivalent to the top slice from the same core.

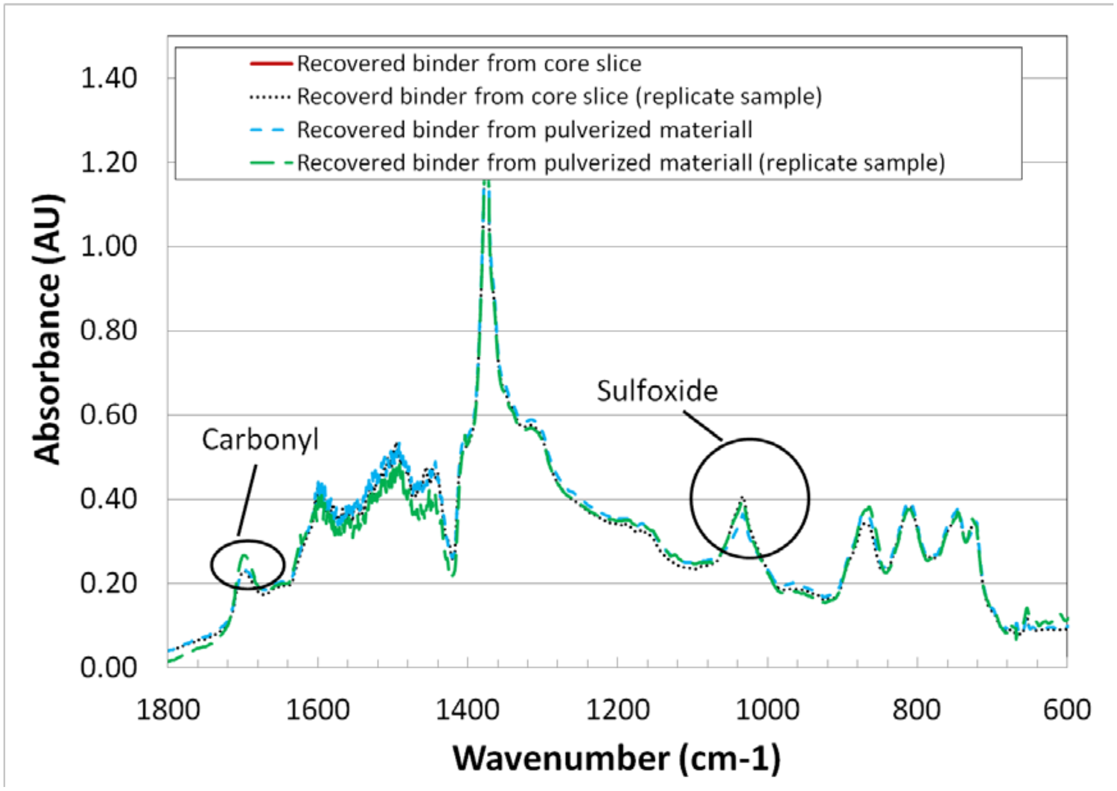

Figure 6. Graph. Comparison IR spectra: Recovered binder from a core slice versus using rotary hammer drill with a venturi collector.

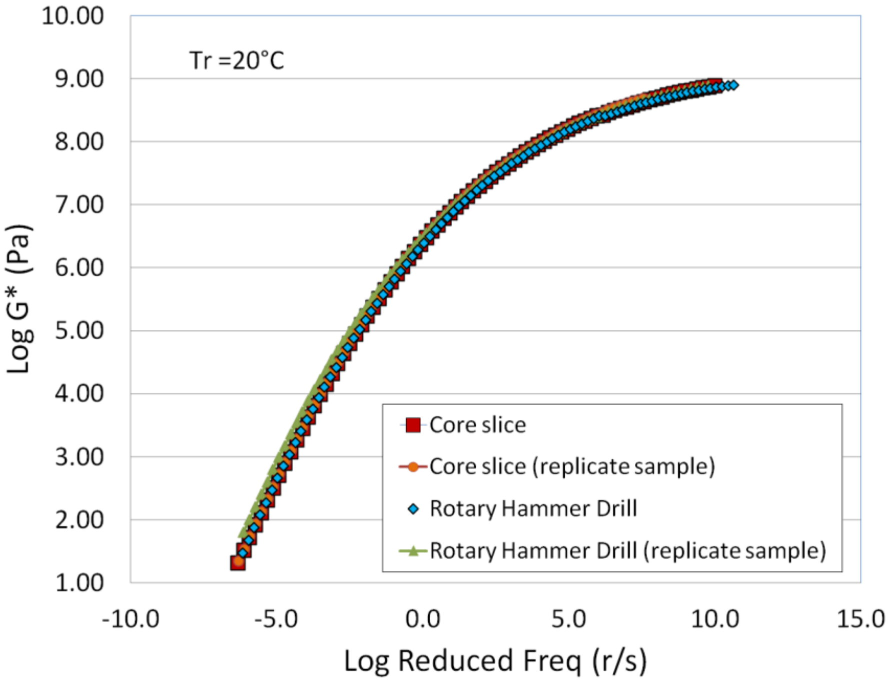

Figure 7. Graph. Comparison of the complex shear modulus: Recovered binder from a core slice versus using a rotary hammer drill with a venturi collector.

Micro-extraction

The micro-extraction method as proposed here is a straightforward, simple modification of AASHTO T 319: Standard Method of Test for Quantitative Extraction and Recovery of Asphalt Binder from Asphalt Mixtures. A draft method in AASHTO format is provided in the Appendix. T319 describes a procedure for the extraction and recovery of asphalt binder from asphalt mixtures that has a minimal effect on the physical and chemical properties of the asphalt binder recovered. The method is intended for use when the physical or chemical properties of the recovered asphalt binder are to be determined.

T319 can also be used to determine the quantity of asphalt binder in the HMA or RAP. However, due to the small quantity of sample involved, the micro-extraction process is not appropriate for determining the quantity of asphalt binder in either HMA or RAP.

The current AASHTO T 319 is somewhat tedious, and equipment intensive. The method uses an extraction vessel that was developed during the Strategic Highway Research Program (SHRP). The equivalent method to T319 in ASTM is D6847-02 *Standard Test Method for Quantitative Extraction and Recovery of Asphalt Binder from Asphalt Mixtures* was withdrawn in 2010 because there were too few labs performing it to establish required interlaboratory precision and bias.

The micro-extraction concept allows the same solvents to remove the asphalt from the aggregate as T319, but there are a number of proposed departures from T319 that dramatically simplify and reduce exposure to harmful, potentially carcinogenic, fumes during the extraction process. The most important departure from T319 is the substantial reduction in the amount of solvent required.

CONCLUSIONS

The micro-sampling and micro-extraction methods described in this report have been shown to be viable alternatives to standard coring and extraction. Micro-sampling involves drilling the pavement to the desired depth using a cordless hammer drill and 24.4 mm (1 in.) masonry bit then collecting the pulverized material using a vacuum integral to the drill. The method requires only a few minutes and only one drill operator. The asphalt is then recovered from the pulverized material using a micro-extraction method.

Benefits include:

- Improved safety during pavement sampling by reducing the time the sampling crew and traffic personnel are exposed to traffic
- Improved safety in the laboratory by substantially reducing the amount of organic solvent required and exposure of laboratory technicians to harmful, carcinogenic fumes from the solvent.
- A simpler, faster method to collect pavement samples for forensic studies to determine what's causing a particular distress such as fatigue cracking, raveling, etc. and what is the best rehabilitation strategy.
- Improved extraction results since the micro-sample is pulverized and removing the asphalt faster than conventional core extractions and less asphalt is left behind on the aggregate.
- Reduced cost by reducing the time to perform sample collection in the field and the time to recover the binder in the laboratory.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the Federal Highway Administration, U.S. Department of Transportation, for financial support of this project under contract no. DTFH61-07D-00005.

DISCLAIMER

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APPENDIX

Standard Method of Test for

Standard Practice for Micro-sampling Asphalt Pavement

AASHTO Designation: T XXX-13

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1. SCOPE

1.1. This practice describes a procedure for removal of a micro-sample (200 ± 20 g) of pulverized asphalt pavement for extraction and recovery of the binder for laboratory testing. It is intended for use when the physical or chemical properties of the recovered asphalt are to be determined. It is not intended to determine the quantity of asphalt binder or the gradation of the recovered aggregate. This method is applicable to HMA or WMA pavement.

1.2. The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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

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2. REFERENCED DOCUMENTS

2.1. *AASHTO Standards*:

2.1.1. AASHTO T XXX-12 Standard Method of Test for Determining the Low Temperature Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).

2.1.2. AASHTO TXXXX-13 Standard Method of Test for Qualitative Micro-Extraction and Recovery of Asphalt Binder from Asphalt Mixtures.

2.2. *ASTM Standards*

2.2.1. ASTM D3665 – 12 Standard Practice for Random Sampling of Construction Materials.

3. TERMINOLOGY

3.1. *Definitions*:

3.1.1. *Micro-sample* **—** Pulverized asphalt pavement, sample of ~ 200 g or less.

3.1.2. *Micro-extraction***—**Extraction and recovery of asphalt binder from a micro-sample.

Note 1—Typically, 200 g of pulverized asphalt pavement will produce about 10 g of asphalt depending on the percent asphalt in the mixture. Dense graded HMA or WMA is typically between 4.5 and 6.5% asphalt.

4. SIGNIFICANCE AND USE

4.1. The Samples obtained in accordance with the procedure given in this practice may be used to extract and recover the asphalt binder for further testing such as the low, intermediate, and high temperature SHRP Performance Grade specification parameters m-value, creep stiffness, $G^* \sin \delta$, and $G^*/ \sin \delta$ as well as chemical composition by e.g. infrared absorption (IR) analyses.

5. APPARATUS

5.1. *25-mm (1 inch) diameter masonry bit*

5.2. *Hammer drill with vacuum attachment*

5.3. *300-mL (10 oz) metal tins with lids*—for sample storage

5.4. *Cooler*—for storing samples

5.5. *Freezer*—for long term sample storage

6. MATERIALS

6.1. Dry ice or other cooling material

7. HAZARDS

7.1. Eye protection should be worn when operating the hammer drill.

8. PROCEDURE

8.1. Select the location on the pavement to be sampled and the sampling depth. Brush the surface with a stiff paint brush to remove dust and other debris. Avoid areas with obvious contamination.

8.2. Determine the number of drills required. For example if the selected sampling depth is 50-mm (~ 2”) then four drill holes to a depth of 50-mm (2”) will be required to obtain roughly 200 g ± of pulverized pavement (assuming an approximate in-place density of 2-g/cm^3).

8.3. After drilling the required number of holes to obtain a 200 g ± sample remove the pulverized material from the vacuum storage on the drill to a metal tin with lid. Or in accordance with Note 3, when collecting surface samples i.e. ~10-mm (3/8”) depth simply sweep the pulverized material onto a small dust pan (or a stiff paper) and transfer into a metal tin.

Note 2—When removing the pulverized material from the vacuum storage on the drill it is necessary to remove the filter and brush out the material between the filter baffles. Use a small (1” wide) nylon brush. Typically about 80% of the pulverized material is visible in the container and about 20% is on the filter.

Note 3—When sampling the surface to a depth of approximately 10-mm (3/8”), it is not necessary to use the vacuum to collect the sample, instead the pulverized material can simply be swept up using a small (1” wide) nylon brush, and then placed in the tin.

8.4. Place the metal tin in a cooler with dry ice or other suitable cooling material. Ice is not suitable due to the possibility of contaminating the samples with water from the melting ice. The purpose of keeping the sample cool is to prevent further oxidation of the asphalt during transport.

8.5. After transporting the samples back to the laboratory, place the samples in a freezer until solvent extraction.

9. REPORT

9.1. Report the detailed location where the pavement sampling occurred.

Standard Method of Test for

Qualitative Micro-extraction and Recovery of Asphalt Binder from Asphalt Mixtures

AASHTO Designation: T XXX-13

1. SCOPE

1.1. This test method describes a procedure for the extraction and recovery of asphalt binder from a small amount of asphalt mixture (< 200 g). It is intended for use when the physical or chemical properties of the recovered asphalt are to be determined. It is not intended to determine the quantity of asphalt binder or the gradation of the recovered aggregate because the samples are too small and do not represent the aggregate gradation. This method is applicable to HMA sampled from the pavement, RAP sampled from the pavement or stockpile, HMA plant production, or laboratory fabricated HMA, and recycled asphalt shingles (RAS).

1.2. The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standards:*

2.2.1. AASHTO T XXX-12 Standard Method of Test for Determining the Low Temperature Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

2.2. *ASTM Standards:*

2.2.1. ASTM D92 - 12b Standard Test Method for Flash and Fire Points by Cleveland Open Cup Tester

3. TERMINOLOGY

3.1. *Definitions*:

3.1.1. *Micro-sample*—Asphalt mixture sample less than 200 g.

3.1.2. *Micro-extraction*—Extraction and recovery of asphalt binder from a micro-sample.

Note 1—In general, 200 g of asphalt mixture will produce about 10 g of asphalt depending on the percent asphalt in the mixture. Dense graded HMA is typically between 4.5 and 6.5% asphalt.

3.1.3. *Rotary Evaporator*—also referred to as a "rotovap" is used to remove solvent from mixtures. The solvent is removed under vacuum and is trapped by a condenser. The flask is rotated during the evaporation process to increase the exposed surface area which increases the evaporation rate.

3.1.4. *Bumping*—occurs during rotary evaporation when the contents of the rotary evaporator flask are drawn up into the condenser.

4. SIGNIFICANCE AND USE

4.1. The paving mixture is extracted with a mixture of toluene and ethanol, or trichloroethylene. The intent of this standard is to extract and recover asphalt binder from an asphalt mixture sample without significantly changing the rheological properties of the recovered binder. The asphalt mixture sample size is limited to a maximum of 200 g (referred to as a micro-sample) which greatly reduces the solvent requirements and exposure of laboratory technicians to harmful organic vapors. The method only recovers about 10 g of asphalt binder. However recent development of 4-mm Dynamic Shear Rheometry (AASHTO T XXX-12), which allows low temperature rheological testing to determine Bending Beam Rheometer (BBR) m-value and creep stiffness, dramatically reduces the amount of binder required. Four-mm DSR only requires about 25 mg of asphalt unlike the BBR which requires about 15 g per beam, and several beams are required.

5. APPARATUS

5.1. *Erlenmeyer flask* —500 mL

5.2. *Round Bottom Recovery Flask*—1000 mL

5.3. *Amber glass bottle*—120 mL, wide-mouth

5.4. *Flow meter*—Gas flow meter, capable of indicating a gas flow up to 1000 mL/min.

5.5. *Manometer or Vacuum Gage*, suitable for measuring the specified vacuum.

5.6. *Rotary Evaporator*—Although the solvent removal can be accomplished with one rotary evaporator (rotovap) equipped with an oil heating bath, two rotovaps are recommended to reduce the time it takes to perform an extraction: (1) a rotovap equipped with an oil heating bath capable of maintaining a temperature of 170°C ±; and (2) a rotovap equipped with a water bath capable of maintaining a temperature of 100°C. The rotovap is illustrated in Figure 1 and should be equipped with a distillation flask, a variable speed motor capable of rotating the distillation flask at a rate of at least 50 rpm, condenser, solvent recovery flask, and heated bath. The rotovap should be capable of holding a recovery flask at a 15° angle. The rotovap setup should include a trap (with dry-ice) placed between the vacuum source and the condenser unit.

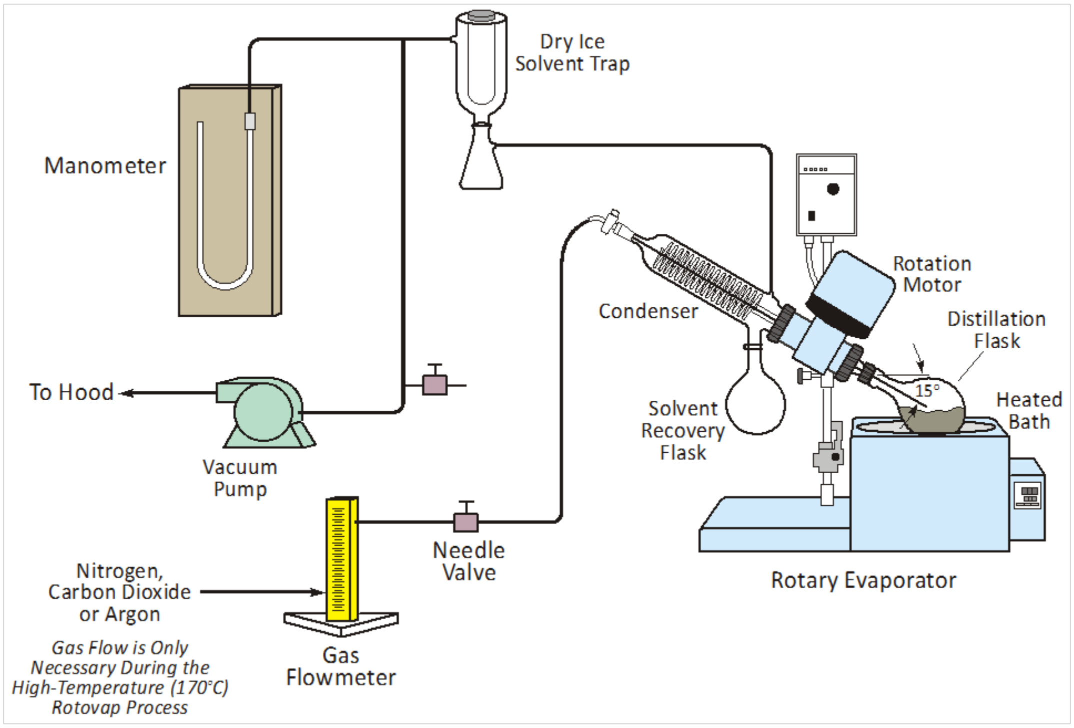

Figure 1—Rotary evaporator and recovery system scheme [Adapted from ASTM D5404 Standard Practice for Recovery of Asphalt from Solution Using the Rotary Evaporator].

5.7. *Centrifuge*—Batch unit capable of exerting a minimum centrifugal force of 770 times gravity.

5.8. *Centrifuge Bottles*—wide mouth glass centrifuge bottles, 100 mL.

5.9. *Thermometric Device*—A built-in temperature measurement device capable of displaying the oil temperatures to the nearest 1°C [2°F].

5.10. *Utilities*

5.10.1. *Vacuum System*, capable of maintaining a vacuum of to within 60.7 kPa [65 mm of Hg] of the desired level up to and including 80 kPa [600 mm of Hg].

5.10.2. *Cooling water source*.

5.11. *Balance,* of suitable capacity meeting the requirements of AASHTO M231, class G2.

5.12. *Glass syringe*—50 mL, 6-inch long 12 gauge flat needle.

5.13. *N-EVAP*—Nitrogen evaporator with water bath.

6. REAGENTS AND MATERIALS

6.1. *Solvents:*

6.1.2. Trichloroethylene, reagent grade.

6.1.3. Toluene/Ethanol—85:15 (v/v) mixture of toluene (reagent grade) and 95% ethanol.

6.2. *Nitrogen, Argon, or Carbon Dioxide Gas—at least 99.95 % pure, in a pressurized tank, with a pressure-reducing regulator valve.*

Note 2—Different flow rates may be required depending on which gas is used.

6.3. *Oil*—the oil for the heated oil bath should be USP White Oil or Silicone Fluid SWS-101 with flash point above 215°C [420°F] or an equivalent. The flash point is determined in accordance with Test Method D92.

7. HAZARDS

7.1. **Caution—**the solvents listed in Section 6 should be used only under a hood or with an effective surface exhaust system in a well-ventilated area, since they are toxic to various degrees. Consult the current Threshold Limit Concentration Committee of the American Conference of Governmental Industrial Hygienists6 for the current threshold limit values. Wear nitrile gloves and safety glasses.

7.2. Exposure of these solvents or their vapors to high temperatures such as contact with flames, hot glowing surfaces, or electric arcs can produce decomposition products such as hydrogen chloride. Steel drums containing these solvents should be stored in a cool, dry location, kept tightly sealed and opened as infrequently as possible. The hydrogen chloride in decomposed solvent may harden an asphalt during the extraction and recovery test.

8. PROCEDURE

8.1. Place the test sample (maximum 200 g) in the 500 mL Erlenmeyer flask.

Note 3—if necessary, for example when working with a core slice, break the sample into smaller irregular pieces.

8.2. Add sufficient solvent to cover the sample (typically several hundred mL). Stir the solvent/mix for several minutes. Cover the flask with aluminum foil and allow to stand overnight. The next day remove the bulk of the solvent/asphalt solution by decanting into another 500 mL Erlenmeyer flask. Try not to disturb the sediment while decanting.

8.3. Using a glass syringe, place approximately 100 mL aliquots of the solvent/asphalt solution into glass centrifuge tubes, and centrifuge for 30 minutes at 2,200 rpm.

Note 4—it is important to balance the amounts of solution in each tube in the centrifuge to avoid damage to the centrifuge or injury to the operator.

Note 5—the sediment at the bottom of the centrifuge tube after centrifuging can be left in place until after the last centrifuge operation. The sediment can then be removed by adding several mL of solvent to the tube and inserting a thin metal rod into the sediment to loosen it.

8.4. Using a glass syringe, carefully remove the bulk of the solution from the centrifuge tubes and transfer to a 1000 mL round bottom recovery flask.

Note 6—When using a syringe to remove the asphalt/solvent solution where there is sediment in the bottom of the centrifuge tube, position the tip of the syringe needle 1 to 2 mm above the surface of the sediment and slowly draw the solution into the syringe so as not to disturb the sediment.

8.5. Evaporate the solvent in a rotary evaporator (water bath set at 70 °C) until the solution is visually dry. Increase nitrogen, argon or carbon dioxide flow to approximately 600 mL/min to eliminate the exposure of the asphalt material to oxygen during solvent removal. Set the rotational speed to about 45 RPM.

Note 7—To prevent bumping slowly lower the round bottom evaporative flask approximately 40 mm into the water bath, and slowly apply a vacuum of 80.0 ± 0.7 kPa [600 ±5 mm of Hg] below atmospheric pressure.

8.6. Repeat the procedure described in steps 8.2 to thru 8.5, except it is not necessary for the solution in step 8.2 to stand overnight. Instead, allow the solution to stand for approximately 30 minutes, occasionally swirl to agitate, and allow to settle before removing the solvent/asphalt solution. Repeat steps 8.2 to 8.5 until the extraction solvent is slightly yellow or straw colored.

8.7. After the last rotovap is performed in step 8.5, pour approximately 30 mL of solvent into the rotary evaporator flask, swirl to agitate, and transfer the solution to a 120 mL amber colored glass bottle. Continue rinsing using small amounts of solvent ~5 mL each time to remove all the remaining asphalt from the round bottom flask.

Note 8—Typically, it requires roughly 70 mL of solvent to remove the asphalt from the rotary evaporator flask to the glass bottle.

8.8. Place the glass bottle under an N-EVAP and reduce the amount solvent to about 30 mL.

Note 9—Instead of using an N-EVAP to reduce the solvent to approximately 30 mL, a rotovap with a water bath set at 70°C can be used, but care has to be exercised to prevent the solution from bumping. Recommend slowly lowering the bottle into the water bath and slowly increasing the vacuum.

8.9. Evaporate the remaining solvent in a rotary evaporator (oil bath set at 170 °C). Increase nitrogen, argon or carbon dioxide flow to approximately 600 mL/min to eliminate the exposure of the asphalt material to oxygen during solvent removal. To prevent bumping slowly lower the bottle approximately 25 mm into the oil bath, and slowly apply a vacuum of 80.0 ± 0.7 kPa [600 ±5 mm of Hg] below atmospheric pressure. Set the rotational speed to 25 RPM.

Note 10—the maximum depth of the bottle in the oil bath is set at 25 mm to help prevent the bath oil from contaminating the sample by climbing up the side of the bottle while it is rotating.

8.10. Perform the rotary evaporation for a minimum of 1 hour. After 1 hour, if no obvious condensation is occurring on the condenser, maintain the evaporative process for an additional10 minutes.

Note 11—In general, if the rotary evaporative process in 8.10 is followed, the concentration of the solvent remaining will not affect the asphalt rheology. To insure the level of solvent is below the threshold that could affect the asphalt's viscoelastic properties evaluate the solvent concentration using infrared spectroscopy. If the solvent is above the detection threshold of the infrared instrument repeat step 8.10 for 30 minutes. The infrared method is optional.

Note 12—Ash contents of recovered asphalts greater than 1 % may affect the accuracy of rheological test such as DSR.

9. PRECISION AND BIAS

9.1. Repeatability and reproducibility tests have not been established in accordance with standard AASHTO practice.

10. REPORT

10.1. Report source of test sample.