

The **WRI**te *Toolkit 2.0*

**For Asphalt & Petroleum Coal Products
and Materials Insights**

Micro Field Sampling and
Extraction

+

Universal Simple Aging Test
(USAT)

+

4mm Dynamic Shear
Rheology

=

Fast, Easy Testing of Hot, Cold
and Warm Paving, Roofing,
Emulsion and Sealant Binders

ExpliFit™ Software = Usable
and Economical Data

SAR-Asphaltene Determinator
(SAR-AD™) = "Fingerprint" of
Asphalt, Crude Oil, Coal
Hydrocarbons

Automated Flocculation
Titrimeter = Compatibility
Prediction, Assessment of
Additive Efficiency

Many More

*Insight =
Savings*

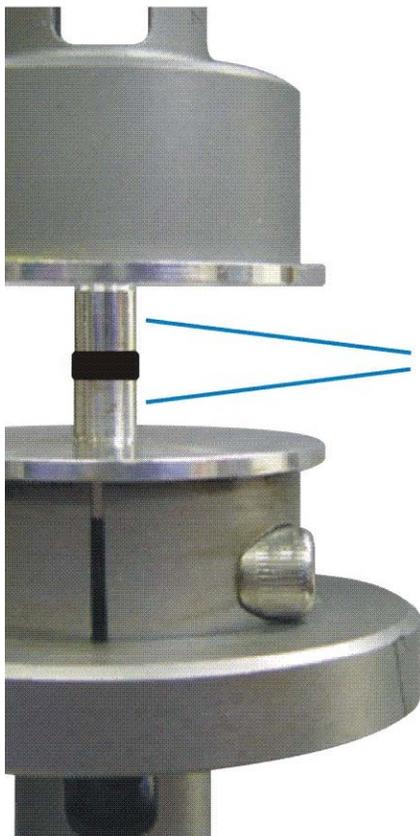
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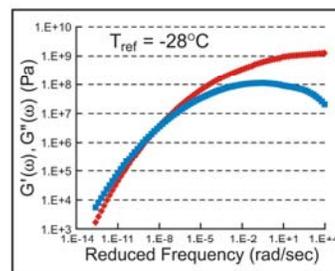
New from WRI

Western Research Institute (WRI), one of the world's foremost leaders in asphalt, petroleum and coal research, brings new tools for unprecedented insight into materials (asphalts, crude oils, additives, mixes, coal fractions, etc.) for highway construction, roofing applications, refinery operations, blending, forensics, mitigation, process optimization and new technology applications. WRI led Fundamental Properties, Asphalt Research Consortium (ARC) and Heavy Oil and Asphalt Industry Research Consortium teams have delivered more than 100 new tools to the asphalt and petroleum industries. These tools include analytical test methods, performance test methods, material models and correlations, laboratory best practices and software for understanding and designing pavement and roofing materials, binders, additives and predicting long term performance in the field or in real life applications.

To experience the latest in asphalt research first-hand, plan to attend WRI's Petersen Asphalt Research Conference.



4mm Diameter Plate Dynamic Shear Rheology (DSR) and other new methods for faster, easier asphalt testing, specification, and selection



Rheology

Low and Intermediate Temperature

- Asphalt
- Modified Binder
- Emulsion Residue
- Crack Sealant



Specifications

Asphalt

- AASHTO T 313
- AASHTO R 49-09
- Emulsion Residue
- Crack Sealant



Universal Simple Aging Test

Short and Long-Term

- Asphalt
- Modified Binder
- Warm Mix
- Emulsion Residue



Field Sampling

- Hammer Drill
- Rapid Sampling
- Easy Extraction

1 Small-Sample Dynamic Shear Rheometry (4mm DSR)

Before WRI developed 4mm dynamic shear rheology (DSR), rheometer compliance errors made it impossible to collect reliable data at low temperatures using standard plate geometries for specification testing. The 4mm DSR is changing tests and specifications used for asphalt binders.

WRI applied the methodology for DSR instrument compliance correction to asphalt low-temperature property measurements using small parallel plate geometry. DSR tests can now be conducted at low temperatures with approximately 25 mg of sample per test, which is about 500 times less material than needed for traditional bending beam rheology (BBR) testing.

This reduced sample size has allowed the development of new applications that were previously too expensive, time consuming, impractical or impossible. These applications include low-temperature testing of asphalt emulsion residues, crack sealants, roofing asphalts, field extracted aged binders, faster testing at pavement or roofing conditions, asphalt blending studies, and pavement forensic studies.

The 4mm plate DSR method provides a potential low-temperature specification test for paving and roofing grade binders, emulsion residues, crack sealants, and other “soft” or thermally sensitive materials. Of interest to producers, contractors, DOTs and suppliers seeking to characterize the performance of these materials, the 4mm plate DSR method provides direct measurement of mechanical properties at or near in-use temperatures, and full temperature range master curves can be created without complex conversion from creep to relaxation. When compared to BBR and 8mm DSR test methods, the 4mm plate DSR shows great promise.

Current AASHTO specification for DSR (T315) has been red-lined to include 4mm DSR, under review by a Binder Expert Task Force, and similarly an ASTM version is also under development. FHWA HRT-15-053 describes 4mm DSR.

New testing tools are made possible by 4mm DSR, including micro field sampling, micro extraction, emulsion residue testing method, and fast micro-aging (USAT). The 4mm DSR is being practiced in many stakeholder research laboratories, and offered by most DSR manufacturers.



4mm Dynamic Shear Rheology makes low-temperature testing possible.

2 Micro Field Sampling and Micro Extraction

This simple, small-scale method uses a hammer drill with a vacuum dust collector to sample field pavement for laboratory extraction and analysis. With the development of 4mm DSR and the use of 25 mm DSR, small amounts of asphalt binder are sufficient to evaluate low-, intermediate- and high-temperature rheology. Thus, large-scale coring and extraction methods are less necessary. The micro-extraction process dramatically reduces the amount of solvent required to extract the asphalt, significantly reducing cost and exposure to solvent fumes. State DOTs, contractors, and researchers can benefit from Micro Field Sampling and Extraction to perform pavement aging.



← **Micro Field Sampling vs. resource-intensive coring** →

FHWA HRT-15-051 describes micro-sampling which has been used in many field projects both in the US and in Europe.



3 Universal Simple Aging Test (USAT)

WRI has developed a fast thin film (such as 300 μm) aging as an alternative to standard Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel (PAV). The technique, known as the Universal Simple Aging Test (USAT), was presented at the 5th EE Congress in 2012 and won the top award poster at TRB 2013*. The USAT is described in FHWA HRT-15-054.

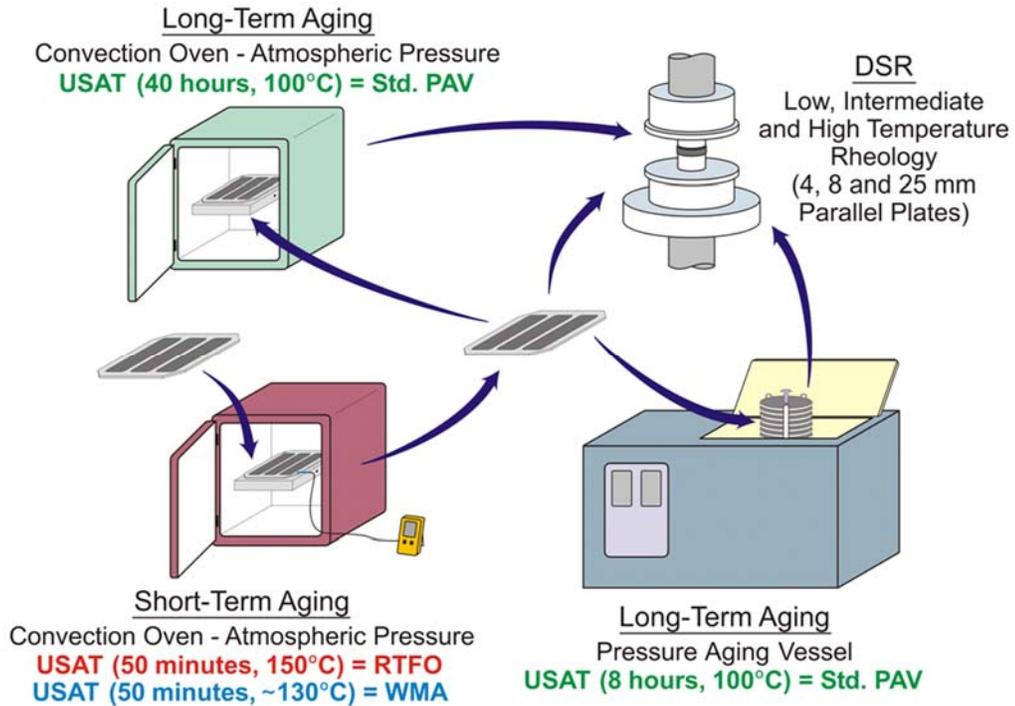
The USAT provides a small-sample approach to quickly characterize the oxidative aging behavior of asphalts, polymer-modified asphalts, emulsion residues, recycled asphalts and other recovered paving or roofing materials. It can also be used to simulate the aging during plant hot and warm-mixing.

The USAT performs short-term RTFO equivalent aging in 50 minutes. Even more significant, USAT performs long-term aging PAV equivalent aging in 8 hours: a 12 hour time saving compared to standard PAV, and potentially 24 hour saving when considering a longer 40hr PAV. By combining the USAT with 4mm DSR, low-temperature rheology and specification parameters, such as m-value and creep stiffness, and the new ΔT_c relaxation parameter, can be determined on samples of limited quantity. Each USAT 300 μm thick plate produces 3g of aged bitumen. The thickness is adjustable.

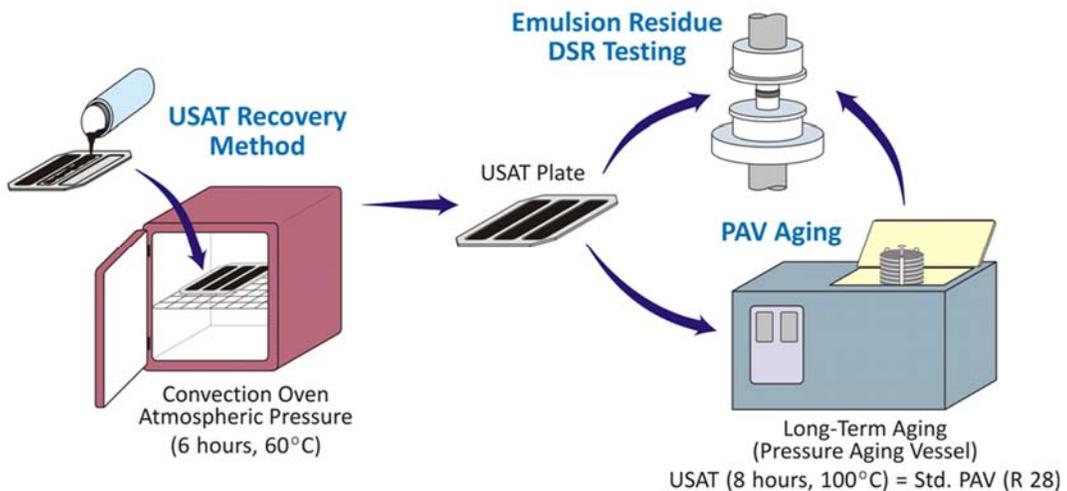


Farrar, M.J., S.L. Salmans, J.P. Planche, "Recovery and Laboratory Testing of Asphalt Emulsion Residue: Application of Simple Aging Test and 4mm Dynamic Shear Rheometry."

USAT Aging and 4mm Rheology Testing for HMA and WMA



USAT Emulsion Residue Recovery, Aging and 4mm Rheology Testing



Advantages of the USAT Recovery Method over AASHTO PP 72-11 Method B

- No silicone mat, no wet film applicator
- More uniform residue surface and thickness (300 µm)
- USAT plate can be placed directly in the PAV
- PAV time is reduced from 20 to 8 hours

* Farrar, M.J., R.W. Grimes, C. Sui, J.P. Planche, S.C. Huang, T. F. Turner, R. Glaser, *Thin Film Oxidative Aging and Low Temperature Performance Grading Using Small Plate Dynamic Shear Rheometry: An Alternative to Standard RTFO, PAV, and BBR*, presented at the Eurasphalt & Eurobitume Congress, Istanbul, Turkey, June 13-15 2012.

4 ExpliFit® Correlation Software

Bridges the Gap from Data to Decision

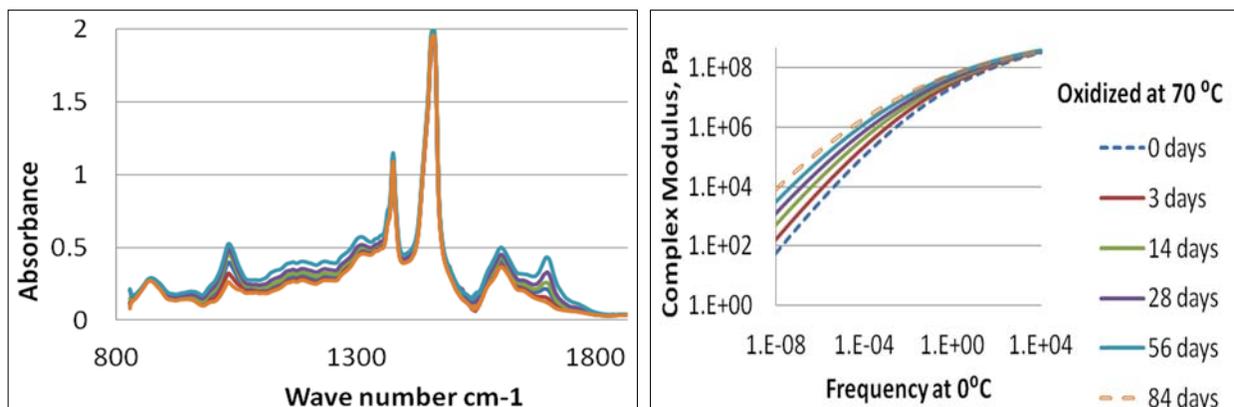
WRI's patent-pending ExpliFit® software program lets correlates between any combination of spectral, chemical or physical data measurements. For example, it helps “decode” spectra and reveals the link to physical data, connecting master curves to infrared spectra or asphalt SAR-AD fractions. The correlation equations can characterize asphalt rheology using only chemical data, to monitor binder changes in asphalt pavement or shingles, determine surface treatment schedules and milling depths...

Makes Data Understandable, Transportable, Economical and Simply... Usable

Unlike other chemometric regression methods (e.g. Partial Least Squares, Artificial Intelligence, and Neural Networks), ExpliFit® clearly defines the variables of importance. Correlations are presented as measured variables in closed-form equations (no complicated latent variables), and the regression equation can be transported outside the program and easily fit to additional data. For statistical validation, independent variables and the numbers of observations are shown in ANOVA tables. Chemical data, which is often faster and more economical to acquire, can now replace greater time and material consuming as well as more expensive physical data collection methods used for asphalt material development.

Insight Made Easy

ExpliFit® uses Microsoft Excel® files for input and output and provides clear, easy-to-read graphics for fit analyses. Users are guided through the process to prevent overfitting data. ExpliFit® can be used in any discipline where chemical or physical data are generated and where relationships between multiple influences are desired. It has a wide range of applications from biochemistry to petroleum product analysis.



ExpliFit® provides a shear modulus master curve of asphalt oxidation (right) from inexpensive infrared measurements.

5 Automated Asphaltene Determinator™

This WRI patented HPLC based system quantitatively separates the asphaltenes from petroleum products into multiple solvent-defined fractions. It is useful for evaluating additives, aging severity, coking, and fouling potential. The Asphaltene Determinator™ (AD) can also be used to identify thermally degraded (visbroken) asphalt bases, airblown asphalts and other unconventional feedstocks and blends.



Some of the world's largest oil companies use WRI's Asphaltene Determinator™ to avoid coking during petroleum refining.

6 Automated Flocculation Titrimeter (AFT)

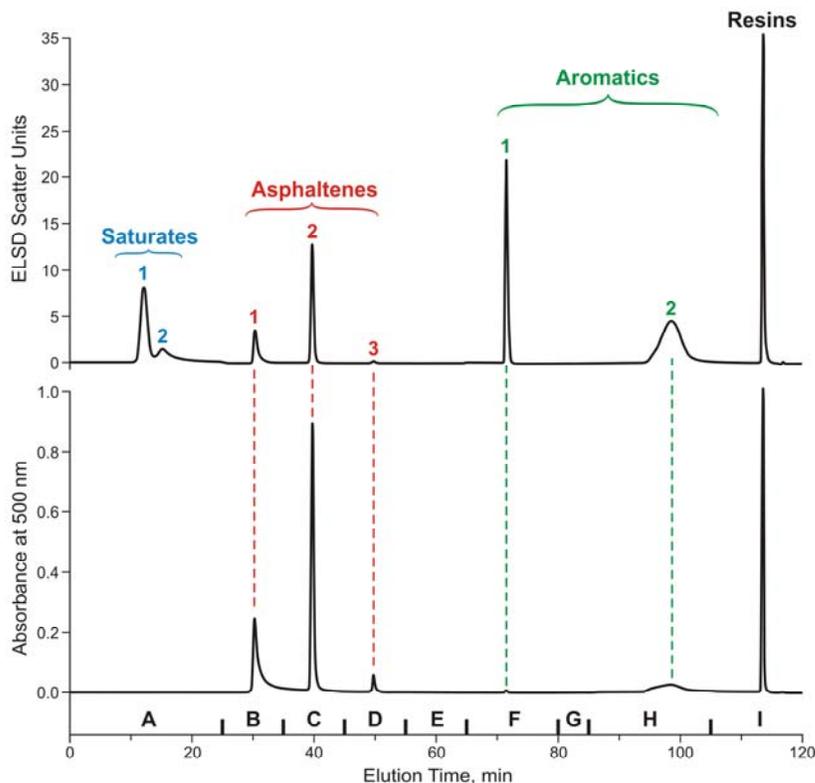
The Automated Flocculation Titrimeter (AFT) developed by WRI, provides a fast and accurate way to collect maltene and asphaltene stability parameters. The data is useful for predicting coking to optimizing yield, to predict or determine blend stability, or gauge the effectiveness of additives. The K47100 AFT is available through Koehler Instruments.

7 Integrated Automated SARA Separation (SAR-AD™)

Traditional chromatographic separations take days and require large amounts of sample and solvent. Combining the Asphaltene Determinator™ (AD) and SAR (saturates, aromatics, and resins) separation provides access to a petroleum product “fingerprint” in less than 2 hours with only milligrams of sample. This new tool provides a way to determine aging, processing and blending guidelines. The patented SAR-AD™ works for paving and roofing asphalt binders, crude oils, aged pavement or shingle binders, blends with additives, and coal fractions. SAR-AD™ is described in FHWA-HRT-15-055.

Integrated SAR-AD™ Tool Provides Even More

- Characterize and select asphalt binders and crude oils to mitigate poor performance due to refining processes or crude oil source variations.
- Provide composition related to mechanical properties and performance.
- Monitor asphalt aging and schedule asphalt pavement surface treatments.
- Combined with AFT (see 7), predict compatibility of aged asphalts (RAP and RAS), polymer-modified asphalts, bio-binder modifiers or rejuvenators, and warm- and cold- mix additives.

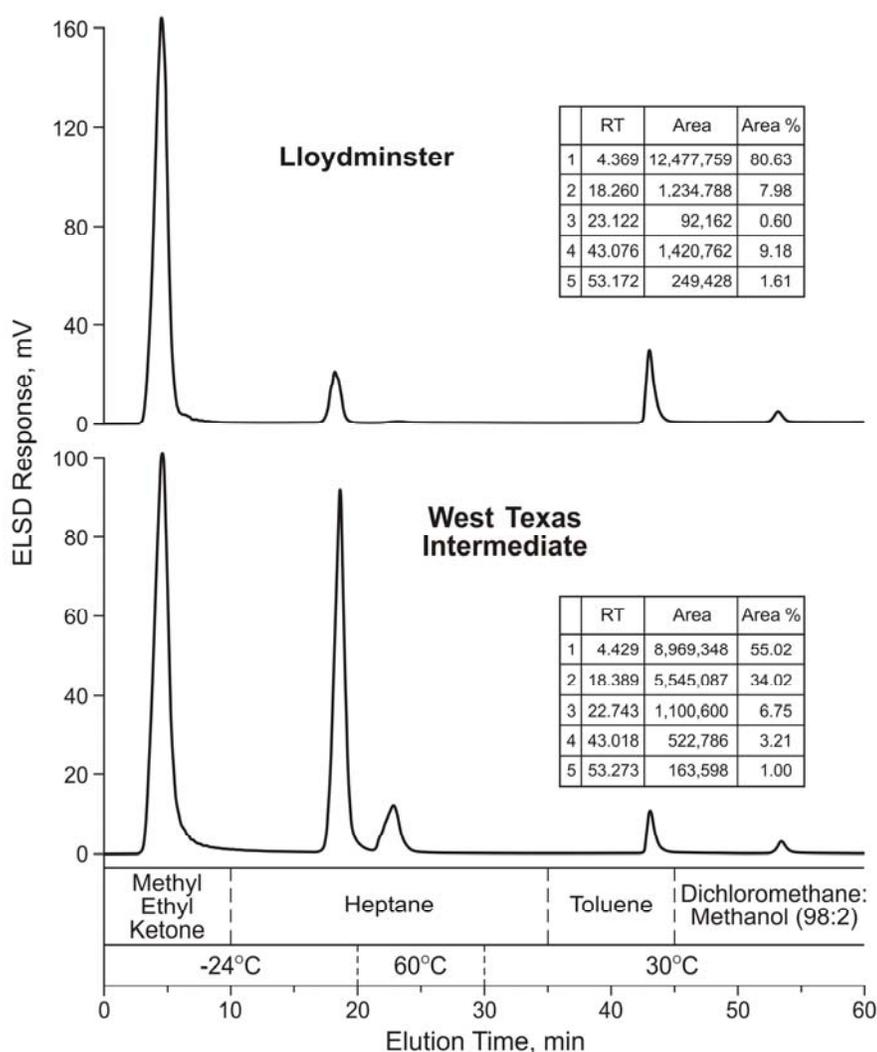


A SAR-AD Separation of an Asphalt Binder

8 Waxphaltene Determinator™

Excess waxes in asphalts may contribute to poor binder cracking and rutting performance as well as to steric and physical hardening.

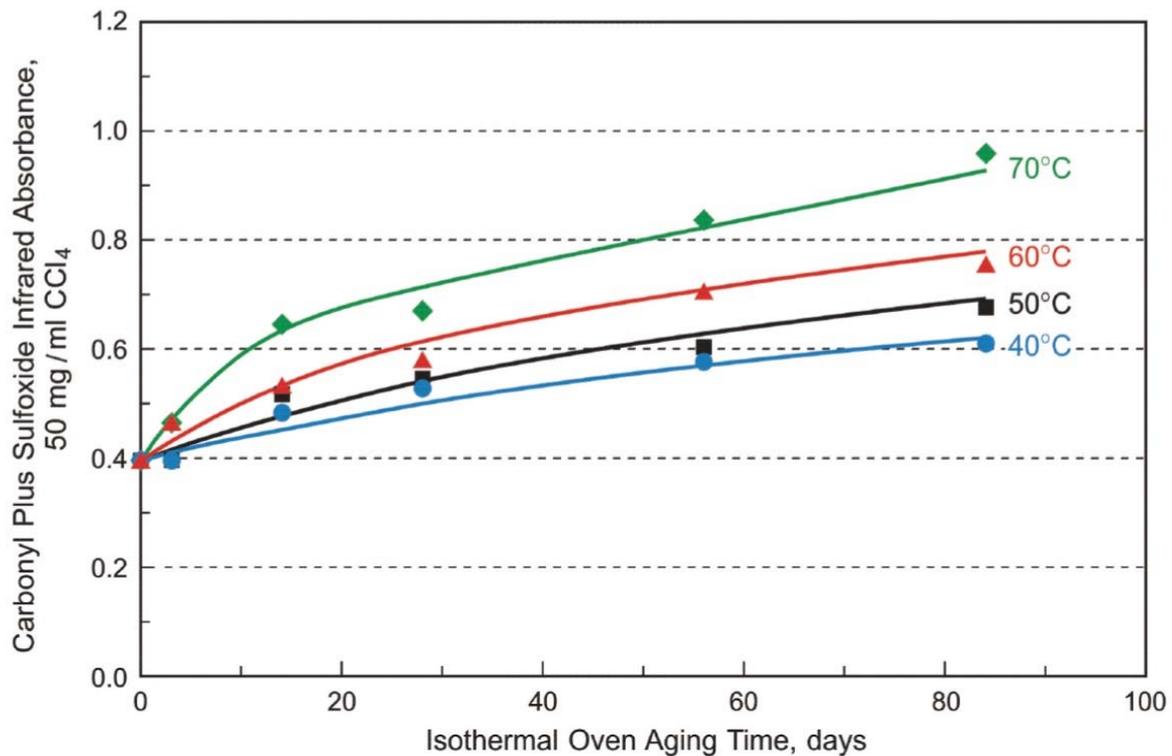
The **Waxphaltene Determinator (WD)** method is a high-performance liquid chromatography based separation, developed by WRI, to rapidly separate waxes and other fractions of asphalts and petroleum materials. For this separation, waxes and other asphalt components are precipitated onto a column at -24°C . A low molecular weight wax enriched fraction is then eluted with heptane while maintaining the column temperature at -24°C . The column is then heated to 60°C to melt the remaining waxes and elute them with the warm heptane. This fraction contains high molecular weight n-paraffin microcrystalline waxes.



Reference: JF Schabron, JR Rovani, MM Sanderson, JF Loveridge, L Nyadong, AM McKenna, AG Marshall, "Waxphaltene Determinator Method for Automated Precipitation of Wax and Asphaltene Components", *Energy Fuels*, 2012, 26, 2256-2268.

9 Asphalt Oxidative Aging Chemo-Mechanical Model

WRI developed an oxidative aging model based on a foundation of decades of research by WRI and the asphalt research community. The model represents a significant improvement in the understanding of the oxidative aging phenomenon. It consists of two sub-models: a fundamental-based component for oxidation chemistry (oxidation chemistry rate model) and a semi-empirical component that relates oxidation levels to rheological changes (master curve change model).



The developed chemo-mechanical model is described in FHWA-HRT-15-052 TechBrief.

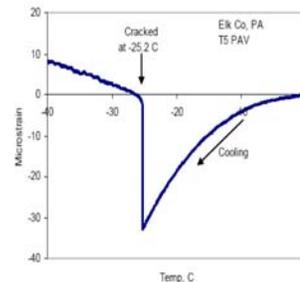
Applications: The model was used in NCHRP project No. 09-54, among other projects, and it provided the basis for the development of loose mix aging kinetics model and aging duration maps for laboratory simulation of long-term aging.

10 Classical Chemical Characterization

- Infrared Spectroscopy (IR) in multiple solutions, ATR and KBr pellets
- UV-visible spectroscopy
- Fluorescence spectroscopy
- Proton, carbon, phosphorous and fluorine NMR
- High temperature Gas Chromatography for determination of alkanes up to C60
- Mass Spectrometry (MS) for quantification and forensic identification
- Differential Scanning Calorimetry (DSC) including modulated DSC, Pressure DSC
- Thermogravimetric Analysis (TGA)
- Atomic Force Microscopy (AFM)
- Optical Microscopy including Fluorescence, Polarized Light ... (pictures of mesophase)
- Total Acid/Base Number (TAN/TBN)
- Water content via Karl Fisher titration
- Size Exclusion Chromatography (SEC, GPC)
- Polymer Quantitation and Characterization in Polymer Modified Binders by IR, SARAD, SEC, Fluorescence spectroscopy

11 Physical Characterization

- Dynamic Shear Rheometer (DSR)
- Bending Beam Rheometer (BBR)
- Binder Superpave Performance Grading and beyond (AASHTO Accredited)
- Multiple Stress Creep Recovery (MSCR) Test
- Rheological Analysis including, but not limited to, Rhea Software
- Standard Rolling Thin-Film Oven (RTFO) & Pressure Aging Vessel (PAV) Procedures
- Universal Simple Aging Test (USAT) for short and long term aging simulation
- Thin-Film Aging Procedures (50-100 microns thick)
- Micro field sampling and Binder extraction and recovery
- Linear Amplitude Sweep Test (LAST)
- Asphalt Binder Cracking Device (ABCD)
- Cigar Tube Stability Test
- Mettler Softening Point (ASTM D3104)
- Oscillating Piston Viscometer (ASTM D7483)
- Brookfield Viscometer
- Du Noüy Ring Tensiometer



Mark Your Calendars for Future

Petersen Asphalt Research Conferences

2020 July 12 - July 15

2021 July 11 - July 14

2022 July 17 - July 20

2023 July 16 - July 19

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