

Standard Title Page - Report on Federally Funded Project

1. Report No.: FHWA/VTRC 06-R18	2. Government Accession No.:	3. Recipient's Catalog No.:	
4. Title and Subtitle: Detection of Polymer Modifiers in Asphalt Binder		5. Report Date: January 2006	
		6. Performing Organization Code	
7. Author(s): Stacey Diefenderfer		8. Performing Organization Report No.: VTRC 06-R18	
9. Performing Organization and Address Virginia Transportation Research Council 530 Edgemont Road Charlottesville, VA 22903		10. Work Unit No. (TRAIS)	
		11. Contract or Grant No. UPC 00075399	
12. Sponsoring Agencies' Name and Address Virginia Department of Transportation FHWA 1401 E. Broad Street P.O. Box 10249 Richmond, VA 23219 Richmond, VA 23240		13. Type of Report and Period Covered Final	
		14. Sponsoring Agency Code	
15. Supplementary Notes			
<p>16. Abstract</p> <p>This study addressed the evaluation of alternative test methods to identify the presence of polymer modifiers in performance-graded binders for the purpose of quality assurance. A method of identification is presented in AASHTO T302, Polymer Content of Polymer-Modified Emulsions and Asphalt Binders, that uses Fourier transform infrared (FTIR) spectroscopy to evaluate the constituent elements in binders or emulsions. With proper calibration, output from FTIR can be used to determine the presence and approximate content of polymers in an asphalt binder. AASHTO T301, Elastic Recovery Test of Bituminous Materials by Means of a Ductilometer, offers an alternative method to determine the presence of polymer by evaluating the elasticity of the binder.</p> <p>Samples of binder were collected from contractor tanks and tested in accordance with AASHTO T301 and AASHTO T302. The performance grade was verified in accordance with AASHTO M320. Test results were evaluated to identify calibration needs, test variability, and choice of preferred methodologies for adoption into the quality assurance program. Results of the study identified the use of either FTIR analysis or elastic recovery as a timesaving alternative to full-fledged performance grading in the initial investigation of concerns about the presence of polymer. Both methods identified binders containing varying polymer contents with no instances of false positive identification. However, based on the results of this study, neither method is suitable to determine binder grade.</p> <p>The investigator recommends that the elastic recovery and FTIR analysis be incorporated as quality assurance tests to verify the presence of polymer in mixtures that specify the use of polymer-modified asphalt binders. Following this, the frequency of quality assurance sampling of polymer modified binders should be increased to ensure that inferior material is not being used in premium mixtures. Further, AASHTO T301 should be adopted in place of Virginia Test Method 104 for use with unaged binders.</p> <p>Incorporating the use of elastic recovery testing and FTIR spectroscopy as alternatives to performance grading will benefit VDOT by allowing increased quality assurance testing of premium asphalt mixtures. This will result in minimizing VDOT's risk of acceptance of inferior material and maximizing the benefits of using premium materials. Typically, performance grading is performed once per month on one binder sample from each active grade of binder in a VDOT district. This is estimated to result in testing less than 5% of the binder lots used in any district during a typical month. Although neither elastic recovery testing nor FTIR spectroscopy was shown conclusively to determine binder grade, almost all PG 76-22 binders shipped into Virginia contain polymer modifiers. Thus, the detection of the polymer is a first level indicator for quality assurance. The potential cost of these tests is approximately \$200 per test for elastic recovery and approximately \$120 per test for FTIR spectroscopy. Overall, the increased testing is expected to result in improved pavement quality by reducing the acceptance of inferior material.</p>			
17 Key Words polymer modified asphalt binders, elastic recovery, Fourier transform infrared spectroscopy, performance grading, quality assurance		18. Distribution Statement No restrictions. This document is available to the public through NTIS, Springfield, VA 22161.	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 18	22. Price

FINAL REPORT
DETECTION OF POLYMER MODIFIERS IN ASPHALT BINDER

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(A Cooperative Organization Sponsored Jointly by the
Virginia Department of Transportation and
the University of Virginia)

In Cooperation with the U.S. Department of Transportation
Federal Highway Administration

Charlottesville, Virginia

January 2006
VTRC 06-R18

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ABSTRACT

This study addressed the evaluation of alternative test methods to identify the presence of polymer modifiers in performance-graded binders for the purpose of quality assurance. A method of identification is presented in AASHTO T302, Polymer Content of Polymer-Modified Emulsions and Asphalt Binders, which uses Fourier transform infrared (FTIR) spectroscopy to evaluate the constituent elements in binders or emulsions. With proper calibration, output from FTIR can be used to determine the presence and approximate content of polymers in an asphalt binder. AASHTO T301, Elastic Recovery Test of Bituminous Materials by Means of a Ductilometer, offers an alternative method to determine the presence of polymer by evaluating the elasticity of the binder.

Samples of binder were collected from contractor tanks and tested in accordance with AASHTO T301 and AASHTO T302. The performance grade was verified in accordance with AASHTO M320. Test results were evaluated to identify calibration needs, test variability, and choice of preferred methodologies for adoption into the quality assurance program. Results of the study identified the use of either FTIR analysis or elastic recovery as a timesaving alternative to full-fledged performance grading in the initial investigation of concerns about the presence of polymer. Both methods identified binders containing varying polymer contents with no instances of false positive identification. However, based on the results of this study, neither method is suitable to determine binder grade.

The investigator recommends that the elastic recovery and FTIR analysis be incorporated as quality assurance tests to verify the presence of polymer in mixtures that specify the use of polymer-modified asphalt binders. Following this, the frequency of quality assurance sampling of polymer modified binders should be increased to ensure that inferior material is not being used in premium mixtures. Further, AASHTO T301 should be adopted in place of Virginia Test Method 104 for use with unaged binders.

Incorporating the use of elastic recovery testing and FTIR spectroscopy as alternatives to performance grading will benefit the Virginia Department of Transportation (VDOT) by allowing increased quality assurance testing of premium asphalt mixtures. This will result in minimizing VDOT's risk of acceptance of inferior material and maximizing the benefits of using premium materials. Typically, performance grading is performed once per month on one binder sample from each active grade of binder in a VDOT district. This is estimated to result in testing less than 5% of the binder lots used in any district during a typical month. If the typical binder failure rate is applied to the approximately 250,000 tons of stone matrix asphalt (SMA) produced with PG 76-22 binder placed yearly, the potential failing tonnage due to incorrect binder use is 45,000 tons, resulting in a cost to VDOT of \$675,000 over the cost of material produced with PG 70-22 binder. Although neither elastic recovery testing nor FTIR spectroscopy was shown conclusively to determine binder grade, almost all PG 76-22 binders shipped into Virginia contain polymer modifiers. Thus, the detection of the polymer is a first level indicator for quality assurance. The potential cost of these tests is approximately \$200 per test for elastic recovery and approximately \$120 per test for FTIR spectroscopy. Overall, the increased testing is expected to result in improved pavement quality by reducing the acceptance of inferior material.

FINAL REPORT

DETECTION OF POLYMER MODIFIERS IN ASPHALT BINDER

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INTRODUCTION

Polymer-modified asphalt binders are used by the Virginia Department of Transportation (VDOT) in several standard paving mixtures. Generally, some PG 70-22 and most PG 76-22 binders currently used in Virginia are modified to provide the required physical properties. Typical modifiers for these binders are styrene-butadiene (SB) or styrene-butadiene-styrene (SBS) type modifiers. Specifications require that neat binders with polymer modification be used for particular premium asphalt mixtures. Currently, binders are accepted with manufacturer-provided certificates of analysis for each lot of material shipped into Virginia. For verification purposes, VDOT also performs testing on quality assurance samples from both the manufacturer and end-user/contractor. However, testing of samples from the end user/contractor is limited by the availability of equipment and personnel, which can result in the acceptance of substandard material.

Most verification testing is performed by VDOT on duplicate quality assurance samples taken in accordance with each manufacturer's quality control plan. As an additional quality assurance tool, additional samples are taken from contractors' storage tanks for grading. Contractor tank sampling is specified to occur at the rate of one sample per grade in use within each VDOT district per year, although sampling typically is performed monthly during the active construction season. Ideally, this rate would be increased to at least a monthly sample from any active tanks used by each contractor. At this time, equipment and personnel constraints do not allow the rate to be increased. This leads to occasional discrepancies between the binder grade specified for projects and that actually used. Usually, this is due to misidentification of tank contents, but it has also been found to be due to the co-mingling of different grade binders in storage tanks. The issue with allowing binders other than those specified to be used is related to both performance of the resulting hot-mix asphalt (HMA) and cost, as HMA produced with modified binder costs significantly more than that produced using standard neat binder.

Both elastic recovery and Fourier transform infrared (FTIR) spectroscopy have been widely used to evaluate polymer-modified binders. Chen and Lin (2000) found that elastic recovery provided a good means by which to separate polymer-modified and neat binders. Elastic recovery testing is specified as a requirement for modified binders by a number of states (Asphalt Institute, 2005). These state specifications may use either AASHTO T301, Elastic Recovery Test of Bituminous Materials by Means of a Ductilometer, or ASTM D6084 as a baseline standard testing procedure with numerous variations. Differences include requirements on unaged binder or rolling thin-film oven (RTFO) residue, temperatures (10°C or 25°C), elongation lengths (10 or 20 cm), time to cutting (immediately or 5 min), and minimum

recovery. In addition, the Northeast Asphalt User/Producer Group presented a memorandum introducing a proposed elastic recovery protocol for acceptance of elastomeric polymer-modified binders that uses the AASHTO T301 method for elastic recovery of original material and recommends varying minimum recovery levels for different binder grades. Virginia currently specifies that binders used in particular mixtures must meet elastic recovery requirements for RTFO residue (VDOT, 2002). Testing is performed on RTFO residue at a temperature of 25°C. The specimen is pulled at a rate of 5 cm/min to an elongation of 10 cm, then cut and left undisturbed for 60 min. Details of the procedure are given in Virginia Test Method 104 (VDOT, 2005).

FTIR spectroscopy has been used to evaluate polymers in polymer-modified asphalt binders. He and Button (1991) developed a laboratory procedure to determine the polyethylene content of Novophalt®, which was successful; however, the primary disadvantage of the method was found to be the time required to develop the calibration curves necessary for quantitative analysis. Curtis et al. (1995) used FTIR analysis to quantify the amounts of ethylene vinyl acetate and SBS polymers and styrene butadiene rubber (SBR) latex in different asphalts. Lu et al. (1999) used FTIR spectroscopy to identify the presence of phase separation of modified binders. Molenaar et al. (2004) reported that FTIR spectroscopy was used to determine the presence of polymers in polymer-modified binders but was found unsuitable for quantitative analysis without the availability of calibration curves. The Texas Department of Transportation (2005) has a test specification for determining the percentages of polymer additive (Tex-533-C) that includes the generation of calibration curves. Alabama requires FTIR traces showing the styrene and butadiene peaks to be submitted annually or as polymer supplies change as part of the quality control plan from asphalt suppliers (Alabama Department of Transportation, 2005).

PURPOSE AND SCOPE

In an effort to increase the rate of testing of polymer-modified binders, two test methods were evaluated to determine their sensitivity to the presence of modifiers: elastic recovery (AASHTO T301) and FTIR spectroscopy (AASHTO T302, Polymer Content of Polymer-Modified Emulsions and Asphalt Binders). These methods were chosen as attractive alternatives to performance grading as they require minimal time to perform and equipment and trained personnel were readily available. The main objective of this study was to validate the use of these tests as verification and investigation tools in quality assurance of polymer-modified binders.

METHODS

Materials

Binders were sampled from contractor storage tanks for evaluation. Sampling occurred when paving was performed under contracts that required modified PG 76-22 binders. Samples

were collected in pairs, with one sample being tested for performance grading and the other used for elastic recovery and FTIR spectroscopy testing.

Calibration standards for the FTIR analysis were made from polymer-loaded binder and neat base binders supplied by one producer. The polymer-loaded binder was a PG 64-22 base binder containing 17% polymer by weight. Standards were made using both PG 64-22 and PG 70-22 neat bases and mixed to contain approximately 1%, 3%, and 5% polymer by weight. Standards were prepared by heating the neat base binder and polymer-loaded binder until sufficiently fluid to pour. The polymer-loaded binder was added to the base binder such that the desired concentration by mass of polymer was obtained. Standards were then heated to a temperature of approximately 185°C and blended using a high shear mixer in accordance with the manufacturer's recommendations. Mixing was initiated at a speed of approximately 500 RPM and increased gradually to a speed of approximately 1200 RPM. After 2 hours of mixing, samples were taken every 15 min and tested using the dynamic shear rheometer until the measured $G^*/\sin \delta$ for consecutive samples varied by less than 5% to indicate homogeneous dispersion of the polymer. Standards were evaluated using the FTIR analysis and elastic recovery methods and were performance graded.

Performance Grading

Grading was performed in accordance with AASHTO M320 to verify that all tested binder met the PG 76-22 criteria. Testing included rotational viscosity of the original binder at 135°C and 163°C; dynamic shear modulus and phase angle of the original binder, RTFO residue, and pressure aging vessel (PAV) residue; mass loss; and creep compliance and slope of the PAV residue.

FTIR Spectroscopy

A Nicolet 510PO Fourier transform infrared spectrometer was used for this study. Binder samples were dissolved in methylene chloride solvent. A few drops of each methylene chloride solution were applied to sodium chloride windows to form a thin film of binder. Infrared spectra were then run on the sample of binder. Each spectrum encompassed 32 scans with a range of 1800 to 600 cm^{-1} wavenumbers and a resolution of 4 cm^{-1} . The peak values at 965 and 1375 cm^{-1} were used to calculate the absorbance ratio. The peak value at 1375 cm^{-1} is indicative of the base asphalt absorbance, and the peak value at 975 cm^{-1} is indicative of the SB or SBS absorbance. Sets of five samples were tested initially to evaluate test variability; this number was reduced to three replicates once the range of acceptable variability was established.

Elastic Recovery

Elastic recovery was performed in accordance with AASHTO T301. Testing was performed on unaged binder at a temperature of 25°C. After each specimen was prepared, it was equilibrated for 90 min prior to testing, then pulled at a rate of 5 cm/min to an elongation of 20

cm. Specimens were then held in the elongated position for 5 min prior to being cut; after cutting, the specimens were allowed to recover for 60 min. Finally, the specimens were retracted until the severed ends just met and the final measurement was taken. Samples of each binder were tested in two sets of three to evaluate within and between set variability. Three binders were tested as six individual specimens to evaluate variability attributable to testing.

RESULTS AND DISCUSSION

The relationship between FTIR analysis and elastic recovery results was first examined to determine how comparable responses from the differing methods were. Figure 1 shows the elastic recovery plotted against the peak absorbance ratio determined from FTIR analysis. It is interesting to note the level of correlation between the two measurements, since the methods of measurement are dependent on different physical responses and the specimen quantities required are quite different.

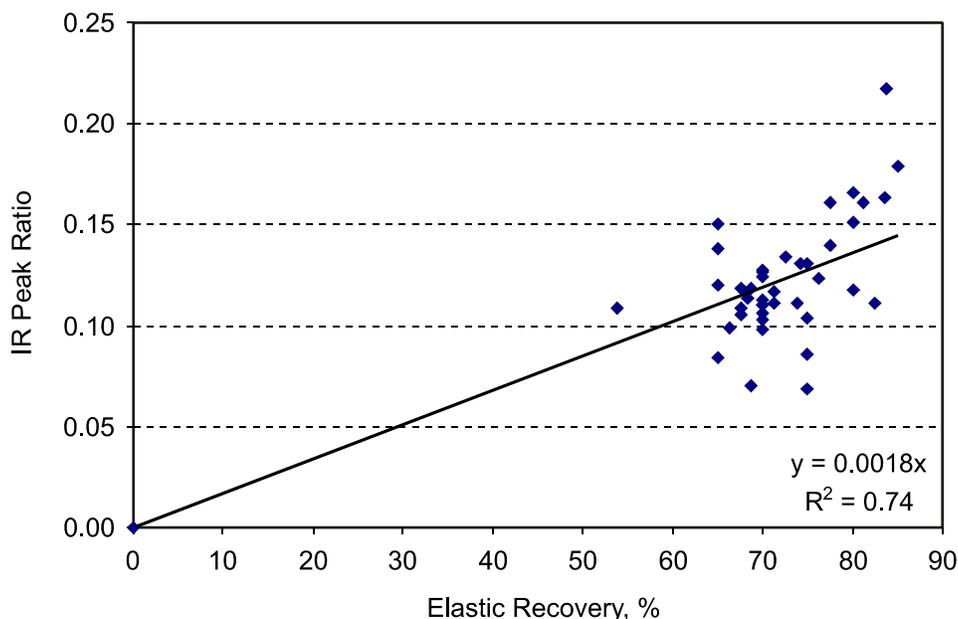


Figure 1. Comparison of Responses for IR Analysis and Elastic Recovery

Calibration curves were compiled for the FTIR analysis and elastic recovery testing using material supplied from one producer. This was done to determine the sensitivity of each method to polymer content and to evaluate the effect of the base binder grade on the test response. Figure 2 indicates that FTIR spectroscopy is highly sensitive to polymer content and that the peak ratio is a linear function of the polymer content. Figure 3 illustrates the elastic recovery response as a function of the polymer content. This figure indicates that elastic recovery and polymer content are less strongly related (by having lesser values of R^2) than in the case of the FTIR spectroscopy, although there is still a robust relationship. Exponential and power relationships were investigated as well but were found to be less robust in fitting the data than was the linear relationship. In Figures 2 and 3, the base binder grade is shown to have had a

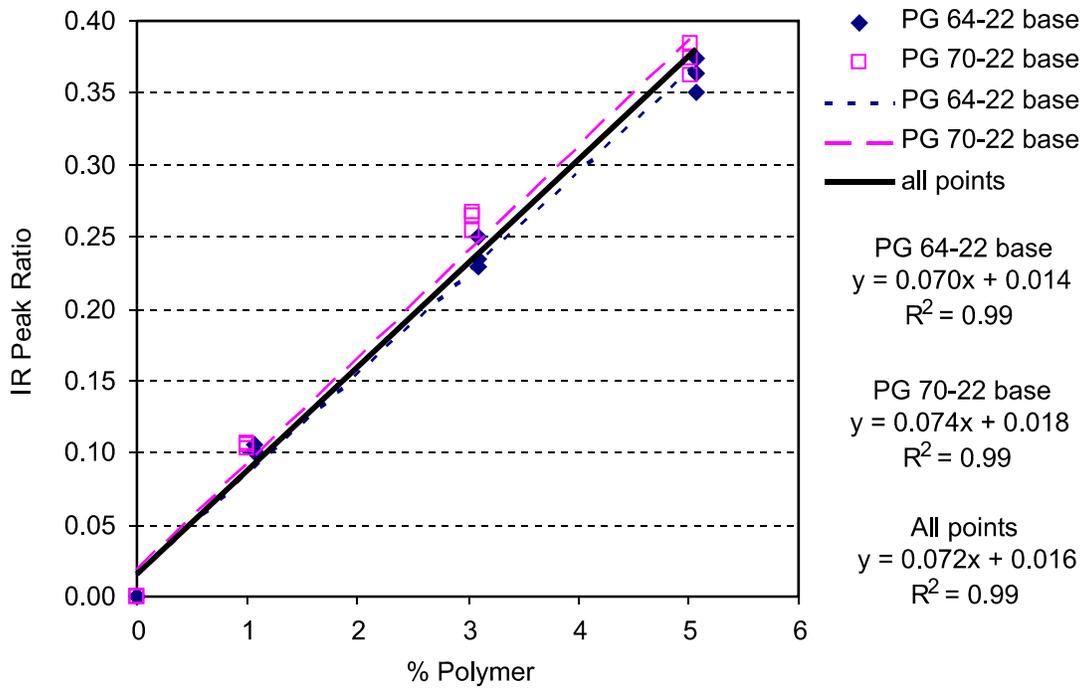


Figure 2. Calibration Curves of Polymer Content for FTIR Analysis

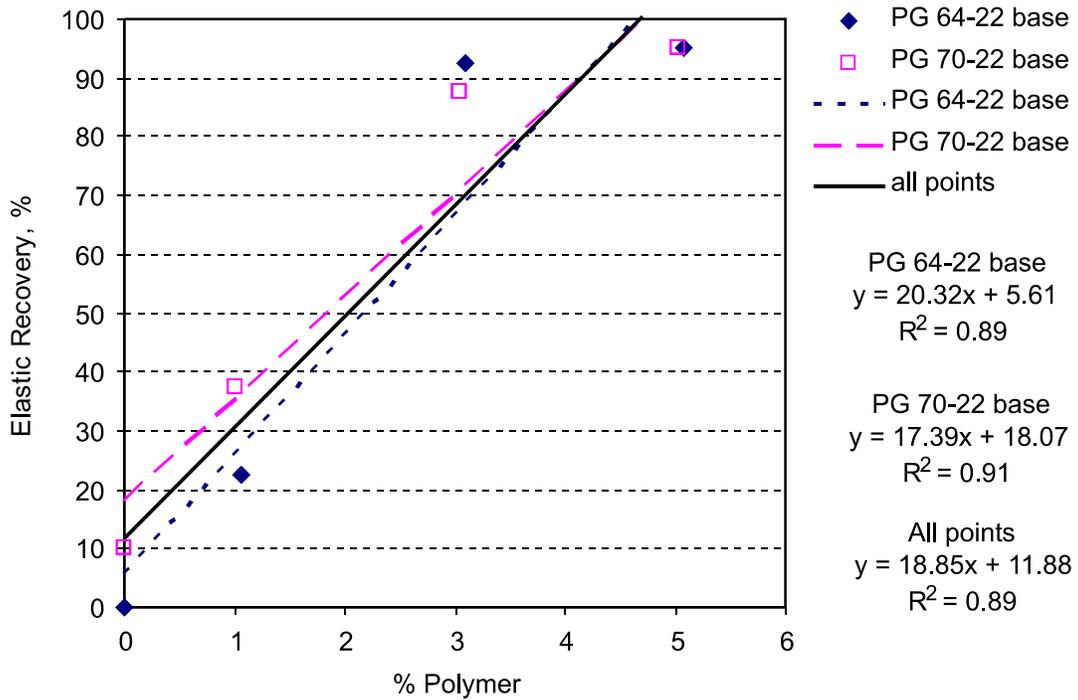


Figure 3. Calibration Curves of Polymer Content for Elastic Recovery

minimal effect on the resulting calibration equations for polymer content. This implies that neither method is sensitive to binder grade as the addition of polymer changes the binder grades considerably.

Calibration points relating the percentage of polymer with the performance grade are shown in Figure 4. This further confirms that the evaluation of the polymer percentage in a binder may not be well correlated with the performance grade. This is due in part because the base binder used in the production of polymer-modified binders is unknown to the accepting agency and impacts the correlation between polymer content and resultant binder grade. Figure 4 indicates that similar polymer concentrations can result in different high temperature grades that are dependent on the initial grade of the base binder. For example, the addition of 1% polymer may also result in a high temperature grade of either 70 or 76 depending on the grade of the base binder. Since VDOT has no indication of the base binder grade, it is not possible to relate the polymer content to the binder grade consistently. In addition, it should be noted that these results are from samples taken from one binder supplier; results from different suppliers are expected to vary considerably, as the necessary polymer contents to meet differing grades are greatly dependent upon the base binder source and production properties.

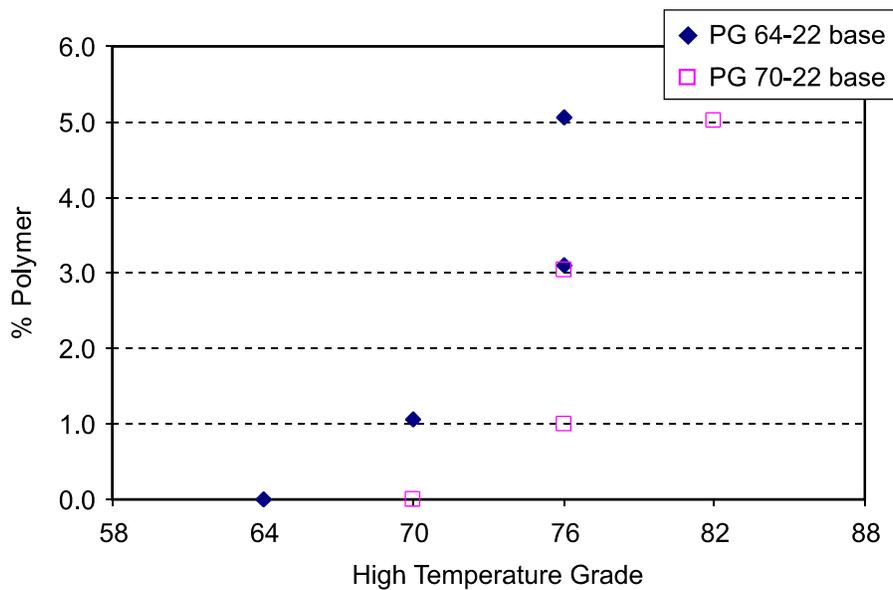


Figure 4. Calibration Points of Polymer Content Versus High Temperature Performance Grade

Variability of Response

Specimens for both FTIR analysis and elastic recovery testing were tested using multiple replicates to evaluate the variability of each test. Neither AASHTO T301 nor AASHTO T302 contains statements of precision or bias, so it was of interest to investigate test repeatability. FTIR specimens were tested using five replicates of each sample for the initial phase of the study and then with sets of three replicates to expedite testing. Figure 5 indicates that the coefficient of variation was minimally affected by the reduction in replicates. The maximum variation seen among FTIR analysis replicates during this study was 11.3%; this was for a single-operator, single-instrument scenario.

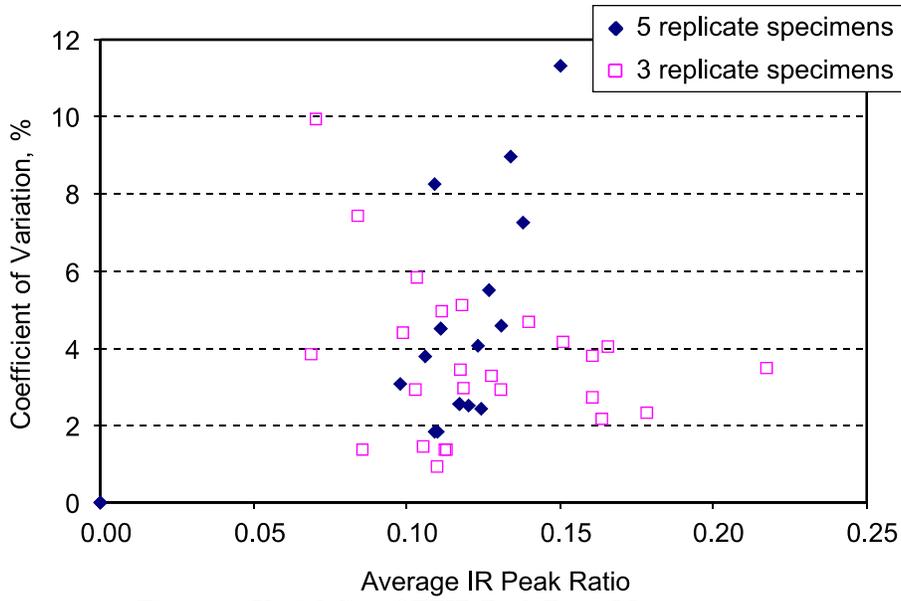


Figure 5. Variability of FTIR Peak Ratio Measurements

Elastic recovery samples were tested as two replicates of three specimens, as the ductilometer could elongate three specimens simultaneously. It was noted that testing of each replicate resulted in a set of three identical results, so to determine if the ductilometer was influencing the response or if the response was truly binder dependent, a small set of three samples was tested using six individual replicates. Each replicate set of the three samples was tested simultaneously. Table 1 shows the results of the individual replicates for the three samples; it can be seen that the ductilometer did not appear to influence the results of each replicate test set, as each binder has different responses. The variability between the sets of replicates was also investigated to determine the optimum number of replicates necessary to achieve a representative recovery value for each binder; this is illustrated in Figure 6. From this figure, it can be seen that the maximum coefficient of variation was 8.4%. This is considerably less than the variation in the FTIR analysis, again for a single-operator, single-instrument scenario.

Table 1. Elastic Recovery Measured For Individual Replicates

Sample	Replicate						Average	Coefficient of Variation
	1	2	3	4	5	6		
A	67.5%	65%	70%	70%	70%	70%	68.75%	3.04
B	75%	72.5%	75%	75%	75%	72.5%	74.17%	1.74
C	70%	65%	65%	70%	70%	70%	68.33%	3.78

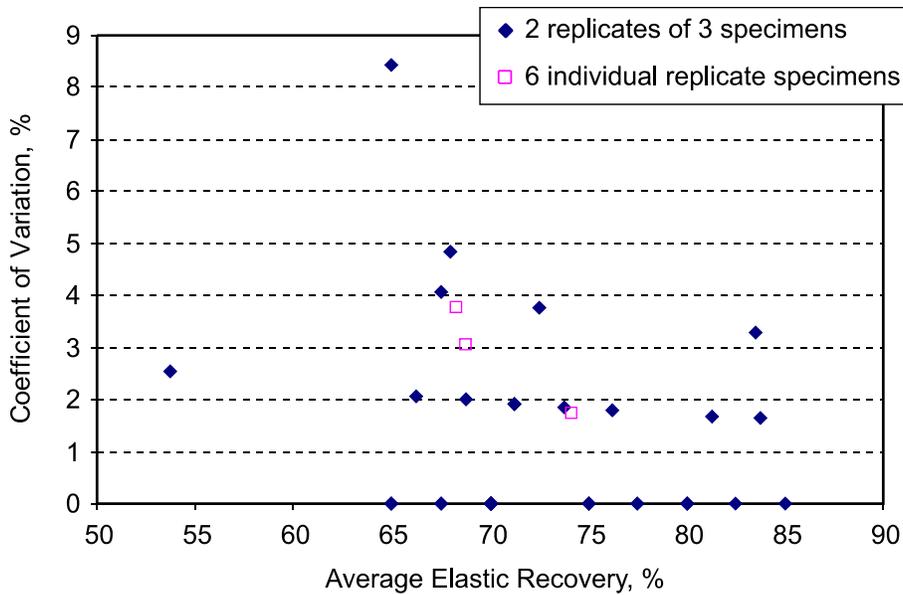


Figure 6. Variability of Elastic Recovery Results

Correlation with Performance Grade

One consideration of this study was the potential use of the FTIR spectroscopy and elastic recovery methods to screen binders for performance grade. To evaluate this potential use, samples were also tested to verify their performance grade. Figures 7 and 8 show the results of the FTIR analysis and elastic recovery testing, respectively, in comparison to the binder high-temperature performance grade. Only the high temperature grade was considered in this case, as it is the grade directly affected by elastomeric polymer modification and because Virginia typically specifies only one low temperature grade (-22°C) except in rare situations.

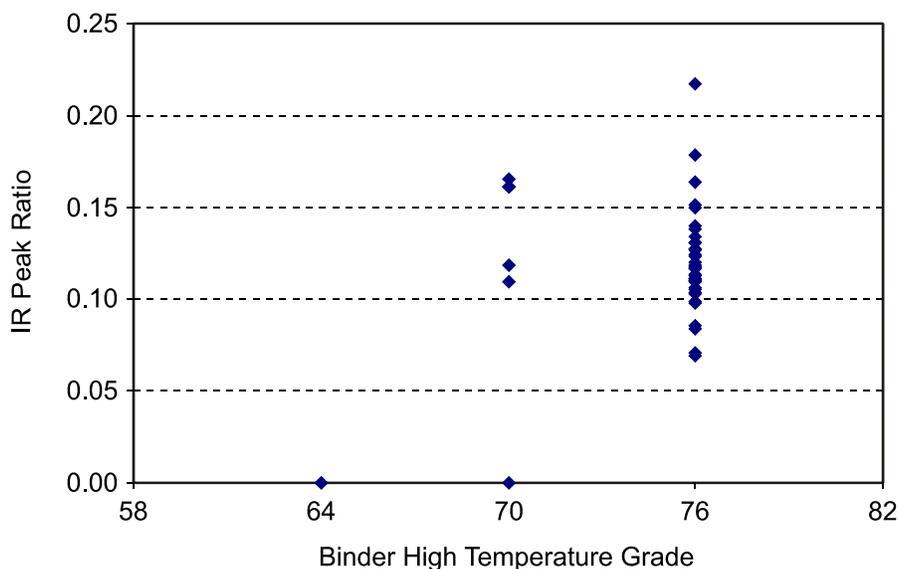


Figure 7. Relationship Between FTIR Peak Ratio Measurements and Binder Grade

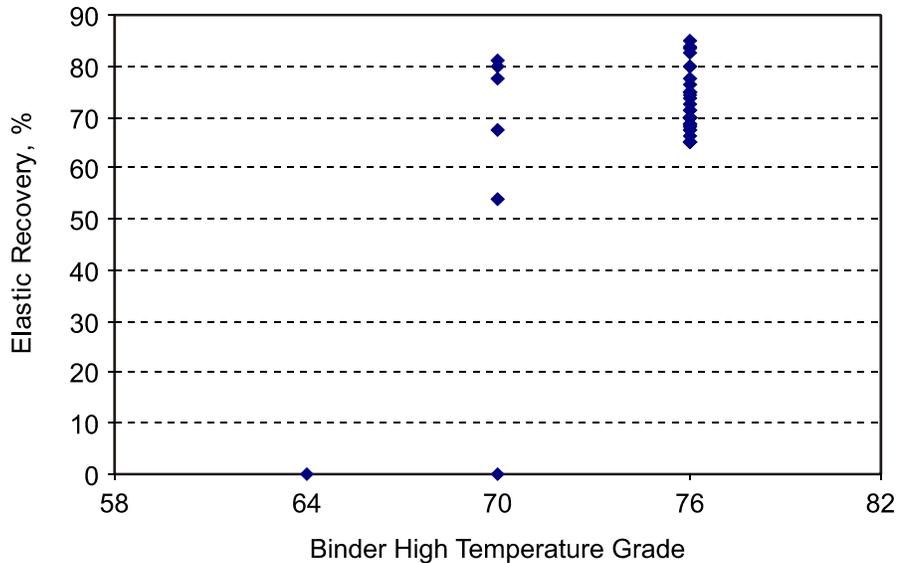


Figure 8. Relationship Between Elastic Recovery Results and Binder Grade

Figures 7 and 8 indicate that neither FTIR analysis nor elastic recovery is sensitive to binder grade. In both figures, there are two data points (one PG 64-22 binder and one PG 70-22 binder) that led to the conclusion of no polymer; these were specifically tested to verify that the tests would not offer false positive results for polymer content. It can also be seen in both figures that the peak values and recovery percentages for the PG 70-22 binders that contained polymer were generally within the range of values seen for the PG 76-22 binders. One important factor to note is that all samples (with the exception of the two samples containing no polymer) were taken from tanks that were supplying PG 76-22 binder; those binders that graded to be PG 70-22 were failing PG 76-22 samples and so were not representative of the typical PG 70-22 samples used in Virginia.

Comparison of Original and RTFO-Aged Binders

A final consideration of this study was to compare briefly the elastic recovery results found for binder in the original condition and that in the rolling thin film oven-aged condition. This comparison was performed to determine if the current Virginia Test Method 104 specification for elastic recovery testing should remain in use or if adoption of a different method using original unaged material should be considered. The benefit of adopting a new method using original material would be a reduced specimen preparation time, as RTFO-aging requires approximately 2 hours of preparation and aging time.

Figure 9 shows the elastic recovery results for six binders tested in the original and RTFO-aged conditions. The figure also displays the high-temperature performance grade for each binder. It can be seen that there is a high degree of correlation between the elastic recovery before and after aging. This indicates that either the original or the RTFO-aged binder can be used in elastic recovery testing.

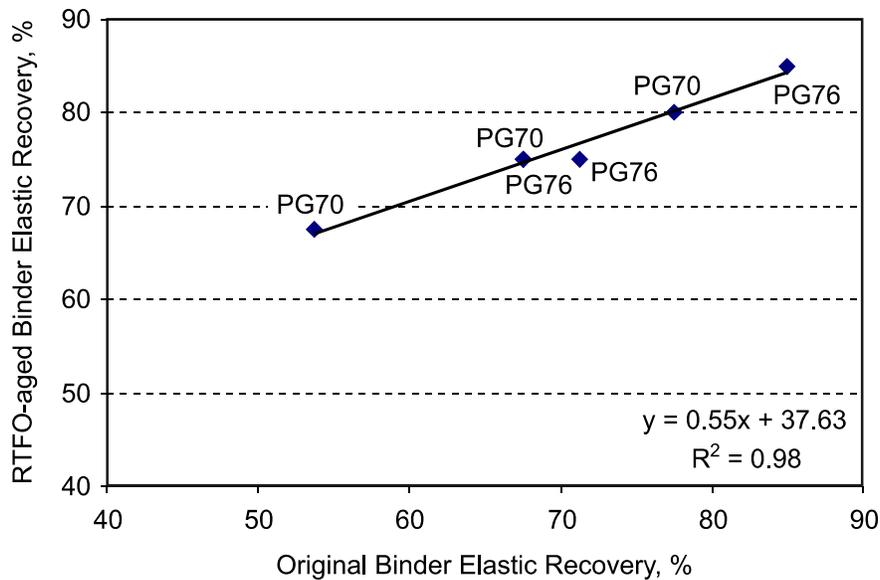


Figure 9. Comparison of Elastic Recovery Results for Original and RTFO-Aged Binders

CONCLUSIONS

- *FTIR spectroscopy and elastic recovery are both suitable methods to verify the presence of polymer.*
- *Both test methods identify binders containing varying percentages of polymer and had no incidence of false positive identification.*
- *Both tests have relatively low variability among test results.* The elastic recovery was more repeatable than FTIR analysis, with the maximum single test coefficient of variability being 8.5% and 11.3%, respectively.
- *High temperature grades of polymer-modified binders are dependent on both the base binder and the polymer content.* Polymer content alone was not sufficient to identify the high-temperature grade.
- *Neither FTIR spectroscopy nor elastic recovery is sensitive to the base binder grade used in the production of calibration standards.*
- *Neither FTIR spectroscopy nor elastic recovery is suitable for identifying the performance grade of binders.* Both methods were unable to distinguish between PG 70-22 and PG 76-22 binders containing polymer. FTIR spectroscopy has the potential to accomplish this task, but extensive calibration is required to account for the combinations of base binders (crude sources) and polymer additives used by binder producers.
- *Elastic recovery results from original binders are well-correlated with elastic recovery results from RTFO-aged binders.*

RECOMMENDATIONS

1. *VDOT's Materials Division should implement the use of elastic recovery and FTIR analysis as quality assurance methods for the verification of polymer in mixtures that specify the use of polymer-modified asphalt binders.* Both methods can be used to provide quality assurance results within 24 hours of receiving sample material, which will result in timely responses to emerging concerns.
2. *VDOT should increase the frequency of quality assurance sampling of polymer-modified binders to ensure that inferior material is not being used in premium mixtures.* Although only performance grading can identify if binders meet the required grade, FTIR analysis and elastic recovery can indicate the presence of polymer required for (M) designated mixtures.
3. *VDOT's Materials Division should adopt AASHTO T301 as the standard used for elastic recovery testing of unaged binders.* This adoption will eliminate Virginia Test Method 104 and reduce the time required for elastic recovery testing by approximately 2 hours.

COSTS AND BENEFITS ASSESSMENT

Typically, performance grading of asphalt binders is performed once per month on one binder sample from each active grade of binder in a VDOT district. This is estimated to result in testing less than 5% of the binder lots used in any district during a typical month. Incorporating the use of elastic recovery testing and FTIR spectroscopy in addition to performance grading will benefit VDOT by allowing increased quality assurance testing of premium asphalt mixtures, such as SMA (PG 76-22). Use of these mixtures typically results in a premium price, although this is thought to be justified by an increase in pavement life and reduction in maintenance costs. An example of the cost difference is seen with stone matrix asphalt (SMA). In 2004 and 2005, the average difference in per ton cost of SMA mixtures produced with PG 70-22 and PG 76-22 binders was approximately \$15, based on the total contract tonnage of SMA let during this time. In 2005, testing of 11 PG 76-22 binder quality assurance samples yielded 2 failures, resulting in a failure rate of 18%. If the binder failure rate is applied to the approximately 250,000 tons of SMA placed yearly, the potential failing tonnage due to incorrect binder use is 45,000 tons yearly. The cost to VDOT for this quantity is \$675,000 over the cost of material produced with PG 70-22 binder. By increasing quality assurance testing, VDOT will minimize its risk of acceptance of and avoid overpayment for inferior material.

Performance grading of a sample consumes 2 workdays, or approximately 16 hours, of personnel and equipment time. Elastic recovery testing on unaged binder consumes approximately 5 hours of equipment and personnel time per test, and FTIR spectroscopy requires approximately 3 hours. If a rate of \$40 per hour for technician and equipment time is assumed, the per test costs for performance grading, elastic recovery testing, and FTIR spectroscopy are, respectively, approximately \$640, \$200, and \$120. Although neither elastic recovery nor FTIR spectroscopy was shown to be able to determine binder grade conclusively, almost all PG 76-22 binders shipped into Virginia contain polymer modifiers. Thus, the detection of the polymer as a

screening test for quality assurance would be of value to VDOT. Upon identification of a suspect binder, performance grading will be performed to verify the need for responsive action. Overall, the increased testing is expected to result in improved pavement quality by reducing the acceptance of inferior material.

Adoption of AASHTO T301 in lieu of Virginia Test Method 104 has the potential to save 2 hours of technician and equipment time currently used in aging material for each set of elastic recovery specimens tested. If the rate of \$40 per hour for technician and equipment time is assumed, each test performed will cost \$80 less than with the use of the current methodology.

ACKNOWLEDGMENTS

The author gratefully acknowledges the efforts of Donald Dodds of the Virginia Transportation Research Council in preparing standards and grading binders and the efforts of Rick Sprissler and Todd Withrow, both of the VDOT Materials Division, in performing FTIR analysis and elastic recovery testing, respectively. In addition, the assistance of Jeff Henderson of VDOT's Salem District Materials Division, Ron Jackson of VDOT's Fredericksburg District Materials Division, Ronnie Seale of VDOT's Northern Virginia District Materials Division, and Phil Chamberlain of VDOT's Richmond District Materials Division in sampling binders is greatly appreciated. The supply of materials from Citgo is also gratefully acknowledged, as is the assistance of Karissa Mooney and Chrissy Skala, both of Citgo.

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