This study was conducted under Federal Highway Administration project HPR-1-18(178). The opinions and findings expressed or implied are ours. The contents do not necessarily reflect the official views or policy of the U.S. Department of Transportation.

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Publication of this paper sponsored by Committee on Characteristics of Bituminous Materials.

Evaluating Asphaltene Settling Test and Relating Results to Physical Properties of Paving Asphalts

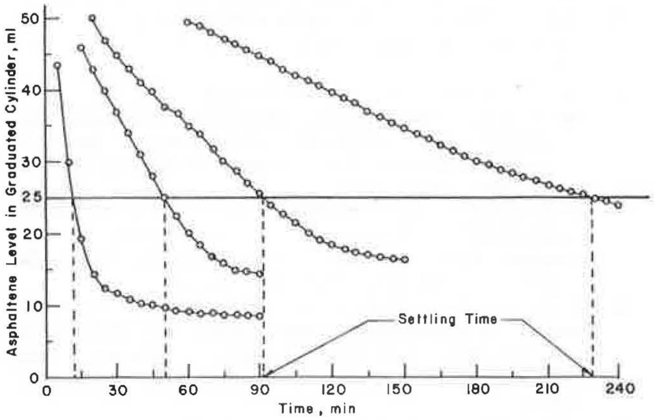
FREDDY L. ROBERTS AND THOMAS W. KENNEDY

The asphaltene settling test was designed to measure the compatibility of the components of an asphalt cement by measuring the settling rate of the asphaltene for a mixture of asphalt in hexane. The results are reported as the length of time required for the asphaltenes to settle out of a solution of hexane. The main premise on which the settling test is based is that the asphaltenes with the longer settling times are better dispersed in the hexane-maltene phase and that longer settling times indicate that the mixtures have more compatible components. The purposes of this study were (a) to evaluate the settling test in terms of test repeatability and the effect of varying selected parameters of the procedure on test results, (b) to determine the settling time of a series of asphalt cement specimens in order to investigate potential relationships between settling time and asphalt characteristics, and (c) to evaluate the effectiveness of asphalt modifiers such as softening agents and antistripping agents. Based on the results, it is felt that additional work is required if the test is to have a practical value. Nevertheless, the findings indicate that (a) the test has a fair repeatability; the coefficients of variation of settling times for 262 asphalts ranged from 2.8 to 9.4 percent; (b) the settling time of asphalt is sensitive to the test temperature; (c) no well-defined relationship was found between settling time and specification-type asphalt characteristics (penetration, viscosity, specific gravity, flash point); (d) the major factor that affected test results was the asphalt producer; and (e) the addition of antistripping agents reduced the settling time with respect to the virgin asphalt, whereas the effect of adding a softening agent was inconsistent, although there was a tendency for reduced settling times. A modified procedure to simplify the procedure, to reduce the time and cost of performing the test, and to improve its repeatability is presented.

The objective of the study summarized in this paper was to evaluate the asphaltene settling test, which has been suggested as a means to rapidly evaluate asphalt durability and the compatibility of asphalts and to determine how effective asphalt-softening agents, which have been proposed for use in recycled asphalt mixtures, are in redistributing the molecular agglomerates present in aged asphalts.

The test is based on previous work (1,2) describing relationships between asphaltenes and durability that led to the development of the settling test, which, according to Plancher and others (3), was developed by Hoiberg and Suhaka for the Asphalt Roofing Manufacturers Association. Later modifications by Plancher [comments in review of report by Kennedy and Lin (4)] adapted the test for paving-

Figure 1. Typical asphaltene settling curves.



grade asphalts (3). The test measures the relative degree of dispersion of asphaltenes of paving asphalts when the asphalts are dissolved in hexane. This dispersion is considered to be a measure of the compatibility of the asphalt components, which is important to asphalt chemistry (3). Test results are reported as a settling time or settling rate of the asphaltene in the solution of asphalt and hexane.

Based on the results reported by Plancher and Petersen at the Laramie Energy Technology Center (LETC) and by others, the test has begun to be used or to be considered for use by various agencies and groups. Plancher and others (3) reported several advantages to this test: (a) good repeatability and high sensitivity to changes in asphalt composition and (b) applicability to virgin, recovered or aged, modified, and blended asphalts. Modified asphalts are asphalts that have been mixed with modifiers, such as asphalt-softening or rejuvenating agents.

Even though a considerable amount of development work has been done with the test, it has not otherwise been used extensively or been applied to a large number of asphalts. The primary objective of this study is to evaluate the asphaltene settling test and determine the factors that affect it.

TEST DESCRIPTION AND EXPERIMENTAL PROGRAM

According to Plancher and others (3), asphalt has traditionally been considered to be composed of oils, asphaltenes, and resins, but it is more correctly a mixture of complex molecular structures ranging from paraffinic types to highly condensed ring structures, with varying degrees of aromaticity, substitution, and heteroatom content. It was also suggested that asphalt compatibility is related to the interaction of molecular components and that changes in the asphalt components could affect the compatibility of the asphalt mixture, which in turn could affect its performance characteristics. Some of the factors that could affect component compatibility are original composition, feedstock blending, aging, asphalt modifiers, and the adsorption characteristics of the mineral aggregates.

The asphaltene settling test was developed to measure the compatibility of the components of an asphalt cement by measuring the asphaltene settling rate for a mixture of asphalt and hexane. Typical settling-rate relationships are shown in Figure 1. Because many of the relationships related to particles settling from a solution were linear over a reasonable period, the time required for the asphaltenes to settle to the 25-ml level in a graduated cylinder was adopted as the settling time. The slower the settling rate or the longer the settling time, the more compatible the asphalt components. This measure of compatibility is based on the premise that asphaltenes with longer settling times are better dispersed in the hexane-maltene phase than those with shorter settling times (3). Plancher also noted that the test results could be affected by the quantity, size, shape, and density of the asphaltene particles and by the viscosity and density of the hexane-maltene phase. Likewise, the effect of changes in test variables, such as quantity of asphalt, test temperature, etc., has not been systematically studied to any extent.

Aged asphalts generally have longer settling times than virgin asphalts. This increase in settling time is partly attributed to increased viscosity of the hexane-maltene solution, charged particles, changes in asphaltene geometry with increased aging levels (spherical to platelet), density, etc., and therefore it should not be construed that aged asphalts are more compatible or durable simply because they have longer settling times. According to Plancher, caution should be exercised when cross-comparing asphalt systems.

OBJECTIVES

This study was developed to evaluate the asphaltene settling test. This evaluation included determining the repeatability for (a) tests conducted simultaneously by the same operator or at the same time by two different operators, (b) tests conducted over a period of time by the same operator, and (c) tests conducted by different laboratories (asphalt cements have been sent to LETC, but results are not available at this time) and the effect of variations of the following parameters on test results: (a) amount of asphalt, (b) asphalt preparation temperature, and (c) test temperature. An additional objective was to use the test to investigate (a) the relationship between the asphaltene settling time and asphalt characteristics for various producers and (b) the effect of commonly used additives or modifiers, such as antistripping agents and asphalt-softening agents, on test results.

TEST METHOD

The asphaltene settling test proposed by LETC involves the following steps:

1. Dissolve 2.0000 g of asphalt cement in 50 ml of n-hexane by stirring the mixture for 16-24 h at 20°C (a stirring time of 20 h was established for use in this and in subsequent studies),
2. Transfer the digested mixture into a 50-ml graduated cylinder, and
3. Read the asphaltene level in the graduated cylinder at 5-min intervals and record.

The asphalt settling time is defined as the time in minutes required for the asphaltene meniscus to descend to the 25-ml level in the 50-ml graduated cylinder (3). Additional details of the test and required equipment are contained in a report by Kennedy and Lin (4).

MATERIALS

Materials included in the study were virgin asphalt cements, asphalts extracted from pavements, antistripping agents, and asphalt-softening agents. The antistripping and softening agents were included in order to determine the effect of these additives on the settling times.

Except for the extracted asphalts, the asphalt cements were identified by using an alphanumeric

Table 1. Summary of repeatability of asphaltene settling test: simultaneous tests.

Producer and Specimen No.	Operator	Asphaltene Settling Time (min) Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9	Test 10	Extremes (min)	Range (min)	Mean (min)	SD (min)	Coefficient of Variation (%)
A9	1	30.0	28.2	30.0	29.6	28.7	29.3	28.1	29.0	27.6	28.2	27.6-30.0	2.4	28.9	0.84	3.0
	2	30.6	28.7	30.7	31.0	26.6	28.6		27.5		27.9	26.6-31.0	4.4	28.9	1.64	5.7
B15	1	33.5	34.3	30.4	30.5	29.2	33.9	34.8	34.2	36.4	35.6	29.2-36.4	7.2	33.3	2.41	7.2
	2	32.4	31.2	31.2	31.2	29.1	31.4	26.3	31.5	33.7	30.5	26.3-33.7	7.4	30.9	1.99	6.4
C20	1	32.7	31.1	28.8	28.0	26.6	27.8	23.6	30.0	31.2	30.9	23.6-32.7	9.1	29.1	2.69	9.2
	2	26.5	29.2	27.2	27.0	25.5	25.5	24.9	28.0	25.8	26.0	24.9-29.0	4.1	26.6	1.31	4.9
D4	1	59.0	62.6	53.8	53.7	57.6	51.0	57.6	60.0	65.0	59.0	51.0-65.0	14.1	57.9	4.24	7.3
	2	53.9	65.0	53.0	49.0	49.0	55.2		52.7	55.4	58.0	49.0-65.0	16.0	54.6	4.87	8.9
D5	1	60.0	62.6	57.6	59.5	62.2	62.6	54.0	62.6	62.5	62.2	54.0-62.6	8.6	60.6	2.88	4.8
	2	60.6	57.7	54.0	45.0	55.0	56.0		56.7	62.5	62.0	45.0-62.5	17.5	56.6	5.31	9.4
A249	1	24.1	21.7	22.5	22.5	22.2	21.1	22.0	21.1			21.1-24.1	3.0	22.2	0.96	4.3
H245	1	98.5	98.4	104.1	104.1	97.5	104.1	102.7	102.0	98.2		97.5-104.1	6.6	101.1	2.87	2.8

Note: Test temperature = 20° C.

Table 2. Summary of repeatability of asphaltene settling test: daily runs.

Producer and Specimen No.	Asphaltene Settling Time (min) Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8	Day 9	Day 10	Extremes (min)	Range (min)	Mean (min)	SD (min)	Coefficient of Variation (%)
A9	28.8	28.7	31.5	29.4	29.4	28.6	29.1	29.4	29.9	30.4	28.6-31.5	2.9	29.5	0.89	3.0
A53	23.6	24.9	25.0	25.0	24.1	22.2	22.8	22.8	23.7	23.1	22.2-25.0	2.8	23.7	1.01	4.3
B15	30.0	30.0	32.6	34.8	33.3	31.3	30.9	31.5	34.0	30.7	30.0-34.8	4.8	31.9	1.68	5.3
C20	30.5	27.3	28.4	29.0	28.9	28.7	32.5	28.0	27.1	28.0	27.1-32.5	5.4	28.8	1.61	5.6
D4	59.7	56.3	58.4	61.8	69.0	57.3	62.0	60.0	58.9	62.3	56.3-69.0	12.7	60.6	3.58	6.9
D5	60.5	60.2	66.2	60.0	58.0	62.2	59.5	61.4	63.2	57.3	57.3-66.2	8.9	60.9	2.58	4.2
G178	73.5	78.0	75.0	79.6	69.7	72.5	71.4	72.7	77.2	77.5	69.7-79.6	9.9	74.7	3.25	4.4
P170	126.3	130.4	126.9	121.8	127.7	125.0	130.0	131.0	125.5	133.6	121.8-133.6	11.8	127.8	3.46	2.7

Note: Test temperature = 20° C.

code in which a letter represented the producer and a number represented a sample of asphalt from that producer. For antistripping agents, the producer identification letter was preceded by an A and for softening agents an R preceded the producer identification. The extracted asphalts were identified as EE. These asphalts were extracted in 1980 by using trichloroethylene.

Tests were performed on 262 virgin asphalts and 5 samples of an extracted asphalt that were obtained from the Texas State Department of Highways and Public Transportation (DHT). Virgin asphalts were secured from 16 different producers in Texas and Oklahoma and included grades that ranged from AC-3 to AC-20.

The two categories of additives or modifiers used were antistripping agents and asphalt-softening agents. Fourteen types of antistripping agents provided by seven producers and five types of asphalt-softening agents provided by five producers were included in the test program.

ANALYSIS AND DISCUSSION OF TEST RESULTS

The primary objectives were to determine the repeatability of the test, the effects and importance of modifying various test parameters on test results, the relationship between asphaltene settling time and asphalt cement characteristics, and the effect of commonly used modifiers on the settling times of virgin asphalt.

Repeatability Tests

Two types of repeatability tests were conducted: (a) repeatability of simultaneous tests by the same operator or different operators and (b) repeatability of tests over a period of time by the same operator. Approximately 10 settling tests were conducted simultaneously by one operator on seven asphalts and by a second operator on five asphalts. In addition, 10 replicated tests on eight asphalts were conducted over a period of 10 days by one operator. The resulting settling times are summarized in Tables 1 and 2.

As can be seen, the range for the settling times obtained for the various test series varied from 2.4 to 14.0 min for operator 1 and from 4.1 to 17.5 min for operator 2. The corresponding values of the standard deviation were 0.84-4.24 for operator 1 and 1.31-5.31 for operator 2. Since the mean settling times varied significantly for the various test series, coefficients of variation were calculated. These coefficients varied from 2.8 to 9.2 percent for operator 1 and from 4.9 to 9.4 for operator 2. Thus, the repeatability was fair and appeared to be essentially the same for the two operators.

A comparison of the mean values, however, indicates that operator 2 generally obtained shorter settling times. This variation probably is significant statistically; however, it is not of practical significance since the range of values for any given test series was much larger than the difference between operators. This difference could be attributed to sampling techniques, temperature control, and rate of digestion, i.e., stirring rate. However, due to the consistency of the difference, it is felt that the more probable cause is the time required to transfer the solution to the graduated cylinder at the beginning of the timing process. It is therefore recommended that the timing of the test start 20 s after stirring ends.

In addition, consideration should be given to eliminating the 10-ml hexane wash since it is felt that the amount of residue left in the flask probably would not affect the test results but that the change in mixture concentration in the upper portion of the cylinder could quite easily affect them. The washing process definitely adds to the time required for transfer and in fact is responsible for the largest portion of the transfer time.

The settling times obtained from the series of tests conducted over a period of time are summarized in Table 2. These data do not indicate any consistent changes with time. The range and standard deviations varied from 2.8 to 12.7 and from 0.89 to

3.58, respectively. The coefficients of variation varied from 2.7 to 5.9 percent, which is consistent with the variation of values obtained by simultaneous multiple tests conducted by the same operator. These results suggest that well-trained operators can probably repeat the test over a period of time and also that they can repeat it at any given time.

Figure 2. Relationship between amount of asphalt in mixture and settling time.

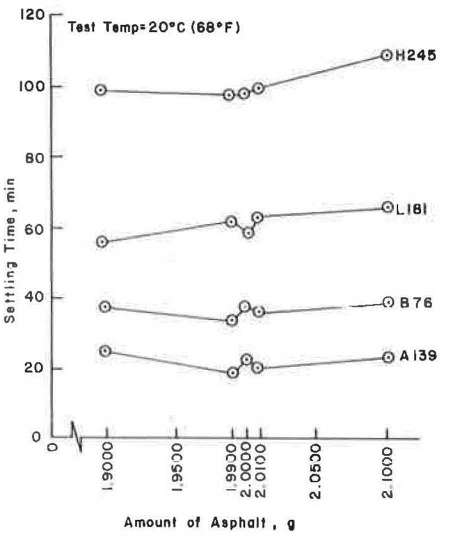


Figure 3. Effect on settling time produced by heating asphalt cement at time of weighing.

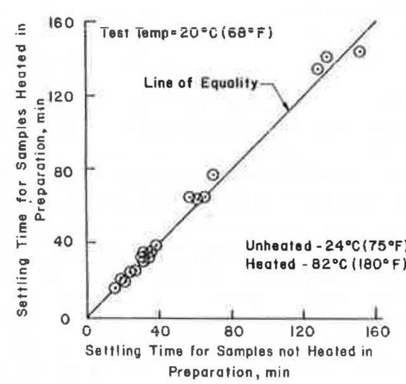
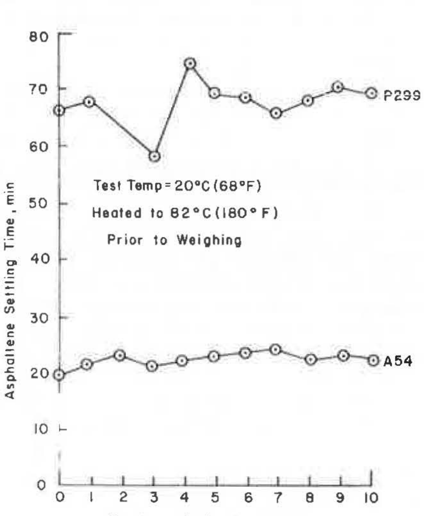


Figure 4. Relationship between settling time and number of heating cycles prior to weighing asphalt cement.



Samples of the asphalt were sent to LETC in order to develop between-laboratory comparisons; however, their results were not available at this time.

Test Parameters

Three test parameters, two associated with mixture preparation and the other with testing temperature, were evaluated to determine the effect of changes in the parameters on test results.

Mixture Preparation

The amount of asphalt and the effect of heating the asphalt during the weighing operation are important with respect to the time required for sample preparation. Thus, the effects of changes in these two parameters were evaluated.

Amount of Asphalt

A series of tests on asphalts that exhibited mean settling times ranging from 23 to 98 min was conducted to determine the effects and importance of variations in the amount of asphalt on test results. Mixtures containing 1.9000, 1.9900, 2.0000, 2.0100, and 2.1000 g of asphalt were prepared and tested. The resulting settling times are shown in Figure 2.

No consistent relationship was found between the amount of asphalt used in the mixture and the settling time. At or near 2.000 g there appears to be a significant hump in the relationship. This inconsistency is attributed to the short range over which these three tests were plotted, which exaggerates the variation. In fact, the magnitude of the effect is less than the range of values that could be expected with replicated tests (Tables 1 and 2). It therefore is apparent that the amount of asphalt within the limits evaluated is not particularly important. Thus, to facilitate sample preparation in production operations but at the same time to provide reasonable control, it is recommended that the asphalt be weighed to 2.00 g with an accuracy of ±0.01 g.

Heating the Asphalt

Heating the asphalt to 82°C to facilitate weighing the required amount of asphalt to be used in the partial solution of asphalt cement and hexane did not produce any consistent effect on the settling times nor did repeated heatings have an effect (Figures 3 and 4). Erratic results were observed in the repeated heating tests on asphalt cement P299 (Figure 4), which could have been the result of many testing or sampling variations. Since the accuracy of weighing has been reduced and since the asphalt properties could be adversely affected, it is recommended that the asphalt cement not be heated to facilitate weighing.

Test Temperature

Ten asphalts that have a wide range of settling times were selected and tested at temperatures of 24 and 20°C. The resulting settling times are shown in Figure 5. The asphaltene settling times at 20°C were substantially higher than those at 24°C. The effect is large enough to require control of the test temperature. Water baths or controlled-environment chambers are probably required and justified.

Relationship to Asphalt Characteristics

A limited analysis was conducted to investigate

potential relationships between settling time and physical specification properties supplied by the Texas DHT for 55 asphalt cements. Properties analyzed were viscosity, penetration, specific gravity, and flash point. Asphaltene settling times were determined for each virgin asphalt and the results compared with the physical properties of the asphalt cements contained in the report by Kennedy and Lin (4).

No relationships between asphaltene settling time and these physical properties of the asphalt cements were found to exist. This result was not unexpected since many of the physical properties are controlled during production and are only indirectly related to the composition of the asphalt. Examples of the nature of the relationships involving viscosity and penetration are shown in Figures 6 and 7. According to Plancher, settling times, however, may be related to other rheological properties or characteristics.

The only factor found to affect settling time was the asphalt producer, which probably is related to composition. The means and ranges of settling times for the 16 producers located in Texas and Oklahoma are summarized in Figure 8. The variation of mean settling time between producers ranges from 16.2 to 235.0 min. Within the observation period of 132 h, the asphalt cement from producer E had no observed settling time and was considered to have an infinite settling time.

Large variations in settling time for some producers are evident; e.g., producers C, G, M, and N had coefficients of variation in excess of 30 percent. Such variation could be related to changes in crude, blending, etc.; however, there is no evidence available from this study to support or reject such a hypothesis.

Although no correlations with viscosity were detected previously, the settling times are shown in Figure 8 by viscosity grade for each producer. Within the limits of this study, no relationship could be found between settling time and the producer viscosity grade. For some producers the set-

Figure 5. Effect of test temperature on settling time.

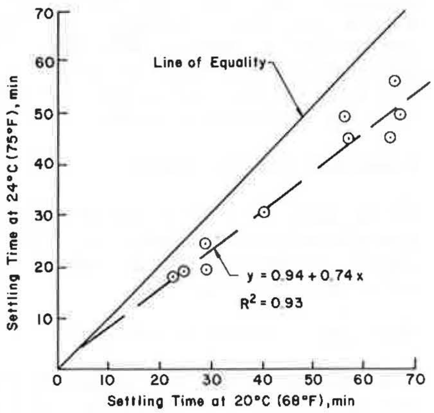


Figure 6. Relationship between viscosity and settling time.

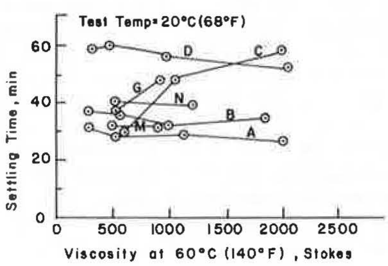


Figure 7. Relationship between penetration and settling time.

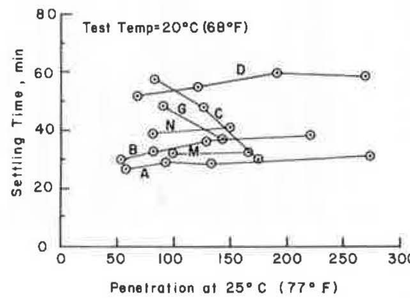


Figure 8. Means and ranges of settling times of various viscosity grades for various producers.

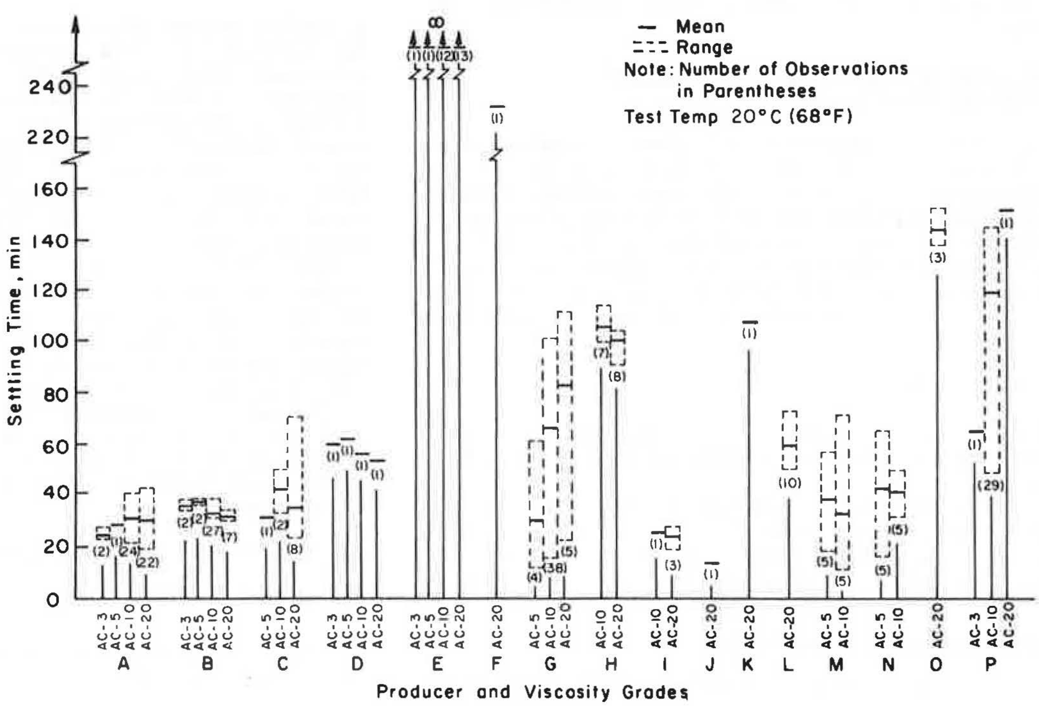


Figure 9. Relationship between settling time and volume of settled asphaltenes.

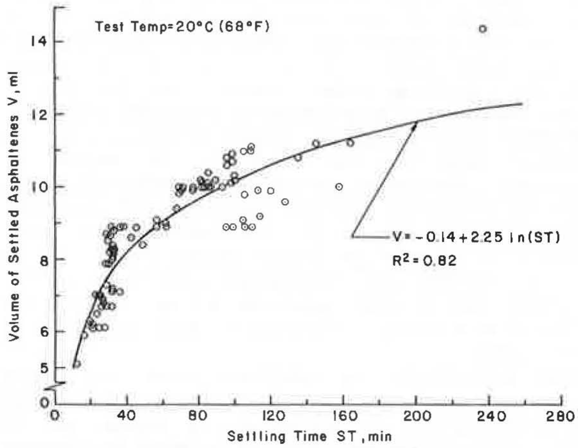
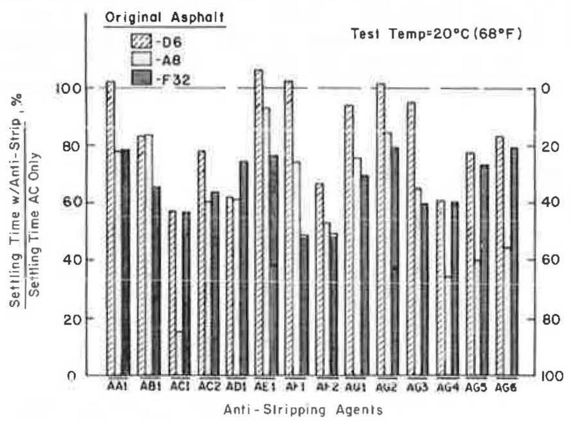


Figure 10. Effect of antistripping agents on settling time for various virgin asphalts.



tling times were essentially the same for all grades, whereas for others large differences occurred.

Relationship Between Settling Time and Volume of Asphaltenes

Since the bulk volume of asphaltenes should influence the settling times, a further analysis was conducted to establish the relationship between asphaltene settling time and the estimated volume of asphaltenes. For this analysis the volume of asphaltenes was defined as the cylinder reading in milliliters after 24 h settling. As shown in Figure 9, the nonlinear equation describing the relationship is

$$V = -0.14 + 2.25\ln(ST) \quad (1)$$

where ST is the asphaltene settling time in minutes and V is the volume of settled asphaltene in milliliters.

The above relationship has a coefficient of determination R^2 of 0.82. A more meaningful relationship with less scatter might be obtained if other factors, such as density of the asphaltenes in the mixture, are considered.

Effects of Asphalt Modifiers

The two types of asphalt modifiers involved in these tests were antistripping agents and asphalt-softening agents.

Antistripping Agents

Three asphalt cements were used to evaluate the effect of 14 different antistripping agents on settling time. Ten percent by weight of asphalt cement of each antistripping agent was added to the asphalt-hexane mixture. The resulting settling times are summarized in Figure 10.

Even though no well-defined relationships existed, the asphaltene settling times decreased when antistripping agents were added, indicating that the presence of the antistripping agents had an effect on test results. It should be noted that antistripping agents AC1, AG4, AG5, and AG6 apparently caused a residue to remain in the flask and on the stirring bar after the transfer of the mixture into the graduated cylinder.

Asphalt-Softening Agents

Tests were conducted to determine the effects of asphalt-softening agents on the settling times of virgin asphalts, artificially aged asphalts, and extracted asphalts.

Virgin Asphalt and Artificially Aged Asphalt

One asphalt cement was selected for artificial aging and subsequent determination of the settling times. The asphalt was artificially aged by placing about 5 g on a flat-bottomed glass pan 3.5 in in diameter in a 121°C oven for a specified period of time. Three different commercial asphalt-softening agents, identified as RB1, RC1, and RD1, were added to 2.0000 g of virgin or aged asphalts in the amounts of 1 and 15 percent and settling times were determined.

The resulting settling times are summarized in Figure 11. It was observed that no well-defined relationship existed between the settling time and the asphalt-softening agent (Figure 11) and that for one asphalt the asphaltene settling time increased with the aging time (Figure 12).

Extracted Asphalt

A limited amount of extracted asphalt was supplied by District 15, San Antonio. The asphalt was extracted from five cores obtained from five locations on IH10 in District 15. Four different commercial and asphalt-softening agents were selected as modifiers--RB1, RC1, RF1, and RG1--and 15 percent by weight was added to the extracted asphalt. Settling-time ratios for each softening agent are shown in Figure 13. It is assumed that when asphalt modifiers are added to recovered aged asphalt, longer settling times indicate a more compatible system of asphalt and modifiers (comments by Plancher, June 5, 1981). Thus, even though certain asphalt-modifier combinations decreased the settling times, the more compatible mixtures should still be those with the longer settling times.

The ratios of settling time for extracted asphalt with softening agent to initial settling time ranged from 66.6 to 116.0 percent. The more compatible systems are presumed to be those with longer settling times (3).

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

The test is quite easy to conduct and quantative test values can be obtained. Nevertheless, it is

tive as asphaltene dispersants for the extracted asphalts in the study. This agreed with the previously reported findings.

Recommendations

Test Procedure

It is recommended that 2.00 g with an accuracy of ±0.01 g of asphalt cement be used in the preparation of the asphalt cement--hexane mixture. The asphalt cement should not be heated during mixture preparation. The testing temperature for the asphaltene settling test should be closely controlled, which probably requires a temperature chamber or water bath. Timing should start 20 s after stirring has been completed. The transfer of the asphalt-hexane mixture should be completed within 20 s. Consideration should be given to eliminating the 10-ml wash since it changes the mixture concentration in the upper part of the cylinder and is responsible for the major portion of the transfer time. It is also felt that the amount of residue left behind probably is not important.

Research

The significance and meaning of the test results need additional study if the test method is to have practical value. Supplemental tests may be required. Additional research should be conducted to evaluate the relationship between settling time and the performance of pavements. It would also be desirable to investigate the relationship between settling time and basic rheologic properties of asphalt.

ACKNOWLEDGMENT

This investigation was conducted at the Center for Transportation Research, Bureau of Engineering Research, University of Texas at Austin. We would like to thank the sponsors, the Texas Department of Highways and Public Transportation and the Federal Highway Administration, U.S. Department of Transportation.

The contents of this paper reflect our views and we are responsible for the facts and the accuracy of the data presented here. The contents do not necessarily reflect the official views or policies of the Federal Highway Administration and the Texas Department of Highways and Public Transportation. This paper does not constitute a standard, specification, or regulation.

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Publication of this paper sponsored by Committee on Characteristics of Bituminous Materials.

Asphalt Temperature Susceptibility and Its Effect on Pavements

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The objective of this study was to determine the influence of asphalt temperature susceptibility on pavement construction and performance. Review of published information and testing of 16 currently produced asphalt cements indicate that asphalts produced today have the same range of temperature susceptibility as those produced from 1960 to 1973. Data collected from specific refining sources have indicated that the physical properties of asphalt cements from selected refineries have changed with time, whereas asphalt from other refineries shows no statistically significant changes. This appears to be related to the crude from which the asphalt is produced. Results obtained from laboratory tests conducted on material from eight field projects indicate that asphalt cement properties do not correlate well with asphalt concrete tenderness problems observed in the field. However, mixture tests such as the resilient modulus and the indirect tensile test have potential for recognizing tender mixtures and defining mixture temperature susceptibility.

"Asphalt ain't as good as it used to be" (1). This statement is often repeated by field construction and maintenance personnel throughout the United States. Their general belief is that the oil companies are taking the "goodies" out of the asphalt and using them as feedstock for the petrochemical industry. Another widely held belief is that the oil embargo and this country's dependence on foreign crudes have forced the oil companies to use less-than-desirable crudes to manufacture asphalt. In addition, field personnel are convinced that the present asphalt specification tests, which are routinely performed, do not identify the important properties that control field construction and pavement performance.

As evidence of these statements, the field engineers cite a general increase in the occurrence of such problems as placement difficulties (tender mixes), excessive displacement under traffic (low stability), and thermal cracking, raveling, and stripping (water susceptibility) of asphalt concrete pavements. These problems result in shorter service