AN EVALUATION OF THE ASPHALTENE SETTLING TEST

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SUMMARY REPORT 253-2(S)

SUMMARY OF

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Foreword

Research Report 253-2 summarizes the findings and conclusions of an evaluation of the asphaltene settling test. It is the second in a series of reports which describe work performed for Research Project 3-9-79-253, "Moisture Effects on Asphalt Mixtures."

Introduction

The Asphaltene Settling Test (AST) was suggested by the Laramie Energy Technology Center (LETC) as a means for (1) rapidly evaluating asphalt durability and the compatibility of asphalts and (2) determining how effective asphalt softening agents proposed for use in recycled asphalt mixtures are in redistributing the molecular agglomerates present in aged asphalts. The test is based on previous work (Refs 1 and 2) describing relationships between asphaltenes and durability that led to the development of the settling test which, according to Plancher et al (Ref 3), was developed by Hoiberg and Suhaka for the Asphalt Roofing Manufacturers Association.

Later modifications by Plancher adapted the test for paving grade asphalts (Ref 4). 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 represent a measure of compatibility of the asphalt components, which is important to asphalt chemistry (Ref 3).

Test results are reported as a settling time or settling rate of the asphaltene in the mixture of asphalt and hexane. The longer the settling time or the slower the settling rate, 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 (Ref 3).

Objectives

The general objectives of this study were to investigate the test by determining the repeatability of the test, the effects and importance of various test variables, the relationship between asphaltene settling time and asphalt characteristics, and the effect of commonly used modifiers on test results.

Experimental Program

To achieve the objectives of the report, the experimental program was designed to evaluate several factors of the test itself plus the repeatability of test results.

Repeatability

Ten tests on each of seven asphalts, representing five producers, were conducted simultaneously by one operator. In addition, a second operator conducted a similar series of ten tests on each of five of the above asphalts. To evaluate time effects, eight asphalts from six producers were tested ten times over a ten-day period.

Test Variables

(1) *Amount of Asphalt.* The original test method specified that the asphalt be measured to the nearest 0.0001 gram at 2.0000 grams. To determine if this degree of accuracy was required, three specimens from four asphalts with viscosity grades of AC-10 and AC-20 were tested with quantities of asphalt weighing 1.9000, 1.9900, 2.0100, and 2.1000 grams.

(2) *Asphalt Preparation Temperatures.* Since heating the asphalt from room temperature to 80°C (180°F) greatly facilitates obtaining a precise amount of asphalt, it was desirable to determine if heating the asphalt affected test results. For two asphalts, one specimen was tested after each reheating, up to ten times, and 20 asphalts were heated one time and one specimen tested.

(3) *Test Temperature.* Duplicate specimen tests were performed to evaluate the effect of testing at temperatures of 20°C (68°F) and 24°C (75°F) on test results. Ten asphalts (from four producers) with a viscosity range from AC-3 to AC-20 were tested.

Relationship to Asphalt Characteristics

Two hundred and sixty-two virgin asphalts were tested to determine their asphaltene settling times. Possible relationships between settling time and the asphalt specification properties were investigated. Properties such as viscosity, penetration, specific gravity, and flash point were supplied by the Texas State Department of Highways and Public Transportation (DHT).

Effects of Asphalt Modifiers

(1) *Anti-stripping Agents.* One specimen was prepared for each of three asphalts and was then mixed with each of 14 different anti-stripping agents. The amount of anti-stripping agent was fixed at 10 percent of the weight of the asphalt.

(2) *Asphalt Softening Agents.* Two series of tests were performed to determine the effect of asphalt

softening agents on the settling times of virgin, artificially aged, and extracted asphalts. In one series settling times were determined for artificially aged asphalts to which three different asphalt softening agents were added after specified periods of aging (6, 24, 48 hours). The two quantities of asphalt softening agents used in these tests were 1 and 15 percent of the weight of the asphalt. In the second series, settling times were determined for one specimen for each of five extracted asphalts with and without the addition of four asphalt softening agents. The amount of asphalt softening agent was 15 percent of the weight of the asphalt.

Conclusions

Repeatability and Operators

(1) The repeatability of the test values was fair and it was essentially the same for different operators and over a period of time.

(2) A definite operator effect was detected with one of the operators consistently obtaining shorter settling times. This was attributed to the techniques used in transferring the mixture from the stirring flask to the graduated cylinder.

Test Variables

(3) The amount of asphalt selected for study in the mixtures had no practical effect on the settling times in this study.

(4) Heating the asphalt during mixture preparation did not affect the test results nor did repeated heating have an effect. Nevertheless, heating of the asphalt should be minimized.

(5) Testing temperature produced a significant effect on the asphaltene settling time: the settling times were shorter at higher temperatures.

Relationship to Asphalt Characteristics

(6) The settling time varied significantly with producer.

(7) No relationships were found between the settling time and asphalt specification characteristics.

Effects of Asphalt Modifiers

(8) Settling times decreased when 10 percent anti-stripping agent was added to the virgin asphalt.

(9) The settling time of asphalt increased as the aging time increased.

(10) No well-defined relationship existed between settling time and percent softening agent for either the virgin or the artificially-aged asphalts; however, the settling time tended to decrease when 15 percent softening agent was added.

(11) The asphalt softening agents were not equally effective as asphaltene dispersants for the extracted asphalts in the study. This agreed with the findings previously reported by Plancher et al.

Recommendations

Test Procedure

(1) The asphalt should weigh 2.00 grams with an accuracy of ± 0.01 gram when the asphalt-hexane mixture is prepared.

(2) The asphalt should not be heated during mixture preparation.

(3) The testing temperature should be closely controlled, which will probably require a temperature chamber or water bath.

(4) The operator timing should start 45 seconds after stirring is complete. The transfer of the asphalt-hexane mixture should be completed within 45 seconds, preferably sooner.

Research

(5) 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.

(6) 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 other rheologic properties not included in this study.

Utilization of Results

The test is quite easy to conduct and quantitative test values can be obtained. Nevertheless it is felt that a great deal of additional work would be required before the test could have any practical value to practicing engineers. The complexity of the asphaltene, maltene, and hexane mixtures and the interaction of the components and their characteristics would appear to have a very definite effect on the test results and their meaning. Additional work will be required before the test can be used routinely.

References

1. Mertens, E. W., "Predicting Weatherability of Coating Grade Asphalts from Asphaltene Characteristics," *Bulletin 250,* ASTM, 1960, pp 40-44.
2. Greenfield, S. H., and J. R. Wright, "Four Methods for Predicting the Durability of Roofing Asphalts," *Materials Research and Standards,* Vol 2, 1962, pp 738-745.
3. Plancher, H., A. J. Hoiberg, S. C. Suhaka, and J. C. Petersen, "A Settling Test to Evaluate the Relative Degree of Dispersion of Asphaltenes," *Proceedings,* Association of Asphalt Paving Technologists, Vol 48, 1979, pp 351-374.
4. Plancher, H., private communication dated June 5, 1981; comments on Research Report No. 253-2.

KEY WORDS: asphaltene settling time, extracted asphalt, asphaltene dispersant, hexane-maltene phase, laboratory asphalt aging, asphalt modifiers, anti-stripping agent, asphalt softening agent, penetration, viscosity.

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The contents of this report reflect the views of the authors, who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the Federal Highway Administration. This report does not constitute a standard, specification, or regulation.

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