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JHRP-84-3

DEVELOPMENT OF A RAPID TEST METHOD
FOR ASPHALT CONTENT DETERMINATION
IN HOT MIX PAVING MIXTURES

FINAL REPORT

Jorge Javier Martinez Chavez

PURDUE UNIVERSITY

JOINT HIGHWAY RESEARCH PROJECT

JHRP-84-3

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DEVELOPMENT OF A RAPID TEST METHOD FOR ASPHALT
CONTENT DETERMINATION IN HOT-MIX PAVING MIXTURES

TO: H. L. Michael, Director
Joint Highway Research Project

January 10, 1984

FROM: D. N. Winslow, Research Associate
Joint Highway Research Project

Project: C-36-6FF

File: 2-4-32

The Final Report attached is submitted on the HPR Part II Research Study titled "Development of a Rapid Test Method for Asphalt Content of Paving Mixtures". The Report has a similar title, "Development of a Rapid Test Method for Asphalt Determination in Hot-Mix Paving Mixtures". Mr. J. J. Martinez Chavez has authored the report and performed the study under my direction.

The objective of the Study to develop a rapid test method has been fulfilled and is fully presented in the Report. As a patent application is in process on the method the Report must be treated as Confidential at the present time and until you are notified that classification has been removed.

The Report is submitted for presentation to the JHRP Board and for review by DOH and FHWA.

The developments presented in the Report should be of great value to DOH and all agencies using hot-mix paving mixtures. The test method developed is economical, rapid, simple to perform and reasonably accurate - much better in all these characteristics than currently used methods.

Respectfully submitted,

D. N. Winslow
Research Associate

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Final Report

DEVELOPMENT OF A RAPID TEST METHOD FOR ASPHALT
CONTENT DETERMINATION IN HOT-MIX PAVING MIXTURES

by

Jorge Javier Martinez Chavez

Joint Highway Research Project

Project No.: C-36-6FF

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16. Abstract <p>A rapid test method was developed for the determination of asphalt cement content in hot-mix bituminous paving mixtures. It is based on the extraction of asphalt cement from mixtures with trichloroethylene and subsequent measurement of the transmittance of light through the extracted solution.</p> <p>A good correlation was found between the results obtained using the rapid test and those obtained using the standard test (ASTM D-2172, Method E1) for samples tested in the field at asphalt mix plants.</p> <p>The test uses a portable spectrophotometer and a metal can for extraction. The equipment needed for the test costs about \$300.00 and is highly portable. The asphalt content can be determined in less than ten minutes.</p> <p>The possibility of using the rapid test on materials containing emulsified asphalt, slag aggregate, unusually high amounts of fine material (<#200 sieve) and recycled material was also studied. The test appears to be easily adaptable to emulsions and slag aggregate. It is unaffected by normal amounts of fine material, but may have to be modified for mixtures with high fines contents. More work is needed before it can be used on recycled mixes.</p>			
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HIGHLIGHT SUMMARY

Martinez Chavez, Jorge Javier. M.S., Purdue University, December 1983. Development of a Rapid Test Method For Asphalt Content Determination in Hot-Mix Paving Mixtures. Major Professor: Douglas N. Winslow..

A rapid test method was developed for the determination of asphalt cement content in hot-mix bituminous paving mixtures. It is based on the extraction of asphalt cement from mixtures with trichloroethylene and subsequent measurement of the transmittance of light through the extracted solution.

A good correlation was found between the results obtained using the rapid test and those obtained using the standard test (ASTM D-2172, Method E1) for samples tested in the field at asphalt mix plants.

The test uses a portable spectrophotometer and a metal can for extraction. The equipment needed for the test costs about \$300.00 and is highly portable. The asphalt content can be determined in less than ten minutes.

The possibility of using the rapid test on materials containing emulsified asphalt, slag aggregate, unusually high amounts of fine material (<#200 sieve) and recycled material was also studied. The test appears to be easily

adaptable to emulsions and slag aggregate. It is unaffected by normal amounts of fine material, but may have to be modified for mixtures with high fines contents. More work is needed before it can be used on recycled mixes.

INTRODUCTION

Quality control tests are frequently performed on bituminous mixtures produced at an asphalt paving plant. Among these tests, one determines the asphalt content of the plant's product. It is generally felt that the asphalt content is the most critical mix parameter determining the performance of the mixture. A survey found that, by a wide margin, an asphalt content test was felt to be the most important test to be performed on a highway material prior to its placement at the construction site (1).

In Indiana, an asphalt content determination is made once for every 1,000 tons of paving mixture that is produced. This test requires approximately one hour to complete. Large, modern plants are able to produce at least several hundred tons of mixture in one hour. If an error or malfunction occurs, and the plant is producing material with the wrong asphalt content, this will not be discovered until the asphalt content test is completed. Thus, a minimum of several hundred tons can be produced before some corrective action is indicated. In order to prevent this, it is necessary to have a more rapid asphalt content test method that will give a result in a shorter length of time and one that

can be repeated on a more frequent schedule.

There are currently several methods for determining the asphalt content of material at a plant. One is an extraction procedure. This is the test method used by the State's inspectors (2). There are several variations to this type of test but all share the same essential aspects. A weighed amount of bituminous mixture is combined with sufficient solvent to remove essentially all of the asphalt cement. The residual aggregate is then dried and weighed and the asphalt content is obtained from the differences in these weights. This technique requires approximately one hour, and a considerable amount of apparatus.

There are also several variations of a displacement-type test (3,4). These all measure the volume of the non-porous portion of a weighed sample of the paving mixture by immersion in a liquid. This volume, and the associated weight, plus a knowledge of the density of the aggregate portion (and that of the asphalt for greater accuracy) allows the calculation of the asphalt content. These methods also require approximately one hour.

Another type of asphalt content test is based on the moderation, or loss in energy, of neutrons that pass through a paving mixture (5). It has been established that their moderation is, among other factors, proportional to the asphalt content of the mixture. Tests using nuclear gages

can be performed in about four minutes (according to the manufacturer). However, they require recalibration whenever any of the materials in the mix are changed. Nuclear gages are also expensive (approximately \$7,500), and have the disadvantage of containing a radioactive source, for the production of neutrons, that requires radiological safety measures.

These currently available test methods all suffer from one or more of several drawbacks: slowness, complexity and an increased chance of experimental error on the part of the inspector, expensive equipment, and necessity for frequent recalibration.

The primary objective of this research was to develop a test method for determining the asphalt content of hot-mix paving mixtures that would be both faster and simpler than the methods in existence, that also uses less expensive equipment than these methods, and that uses portable instruments for easy field testing. A secondary objective was to try extending the fast method to mixes containing slag aggregate, emulsified asphalts, and to recycled material.

The test method that has been developed is based on the rapid extraction of the asphalt cement from the mixture with trichloroethylene, and subsequent measurement of the transmittance of light through the extracted solution. The

percentage of asphalt in the original mixture is obtained from an established calibration curve of asphalt content in the mix vs. light transmission through the solvent-asphalt solution.

PRELIMINARY RESEARCH

The general scheme of the new test method is to rapidly extract the asphalt from a weighed quantity of bituminous mix using a standard quantity of solvent. Then, a rapid test is performed on the extracted asphalt-solvent solution to determine its asphalt content and, hence, the asphalt content of the mix. The extraction is performed by vigorous agitation of the mixture-solvent combination in a closed container.

A number of preliminary questions had to be answered during the development of the new test method. These included: the optimum extraction solvent, the best solution testing method, etc. What follows is a summary of this preliminary research. Complete details of each question are given in the appropriate appendices.

Solvent Selection

The first step in the research was the selection of the solvent that was both the safest, and had the best solubility characteristics. Five different solvents were considered: methylene chloride, trichloroethylene, perchloroethylene, kerosene, and 1,1,1 trichloroethane. The

rate of dissolution for the five different solvents was determined by mixing asphalt and solvent at time zero, and measuring the decrease in the transmittance of light through the solutions as time passed. This work was repeated with three different asphalt cements and each solvent. Appendix A describes this work.

The solvents that dissolved the asphalt cements in the shortest time were: trichloroethylene and methylene chloride. Trichloroethylene was selected for use in the rapid test because of its higher boiling point (87°C). A higher boiling point should decrease the concentration of its toxic vapors in the air.

Extraction Procedure

Once the solvent was selected, it was necessary to develop a satisfactory, and rapid, extraction method. The chosen method is one in which known quantities of mixture and solvent are placed in a container that is then closed and shaken vigorously. Details of the extraction container are given in Appendix B.

Another variable that was considered was the extent of shaking for adequate extraction. Several mixtures were shaken for varying lengths of time, and the asphalt content of the extract was measured as a function of shake-time. This established that a surprisingly brief shake was sufficient.

Indiana specifications currently call for a sample mass in the range of 1500-2500 grams for the standard extraction test. In conformance with this, a 2000 gram sample size was chosen for the rapid test. This selection was also based on the size of an extraction container that would be convenient. After some solubility tests, it was found that 2000 ml of trichloroethylene was the required amount of solvent for optimum extraction of the asphalt cement in the paving mixtures. Hence, 2000 ml of solvent was used for the rapid test.

Extract Testing

Four different techniques for rapidly determining the asphalt content of the extract were tried. All of the methods depend upon measuring some property of the extracted solution that is associated with the amount of asphalt present in the solution. In this phase of the preliminary research, asphalt-solvent solutions of known asphalt contents were prepared, and each solution was subjected to the four different techniques. The measured property was then correlated with the known asphalt content of the solution.

The four different solution properties tested were:

1. Specific Gravity (see Appendix C).
2. Viscosity (see Appendix D).
3. Refractive Index (see Appendix E).
4. Absorbance of Light (see Appendix F).

The efficacy of each method in predicting the original asphalt content of the mixture was based on speed, cost of equipment, sensitivity and simplicity. The optimum technique was found to be absorbance of light through the solutions.

Variation in Asphalt Cements and Aggregates
and Development of Calibration Curve

The potential effect of differing asphalts was also investigated. Different crude oil sources and processing methods yield somewhat different asphalt cements. These differences may alter the correlation between the absorbance of light of the extract and the original asphalt content. Therefore, hot-mix paving mixtures were prepared in the lab using different asphalt contents (from 2% to 9%), different types and sources of asphalt cement (AP-5 from Texaco, Lawrenceville, Indiana; AC-20 from Amoco, Whiting, Indiana; and, AC-20 from Asphalt Materials Co., Indianapolis, Indiana), different aggregate gradations : #9 binder (1/2 inch maximum size) and #11 surface (3/8 inch maximum size), and different aggregate types (gravel and crushed stone), in

order to find possible interfering factors (see Appendix G). The terms #9 binder and #11 surface are the Indiana State Department of Highways designations for materials with a maximum size aggregate of 1/2 and 3/8 inches respectively. A calibration curve (absorbance of light vs. asphalt content of the mixture) was obtained for each type of asphalt cement and for each type of mixture (see Appendix G).

All the curves showed essentially the same correlation between the asphalt content of the mixture and the absorbance of light. The results were so close that it was not felt necessary to have a different calibration curve for each type of mixture, type of aggregate and asphalt cement. For this reason, all the values were used to obtain a single calibration curve that was used for determining the asphalt content of all the subsequent samples. This plot, which is a straight line for absorbance vs. asphalt content or an exponential curve for transmittance vs. asphalt content, was obtained by a least squares fit of the values of absorbance of light and asphalt content. This curve is called the standard calibration curve and is shown in Figures 1 and 2.

It is, of course, possible that other asphalt cements, or aggregates, will not coincide with this calibration curve. Such mixtures will require a separate curve which is easily generated with a few laboratory measurements. A calibration curve can be obtained using Beer's Law with at least three points. It is recommended that, if the required

asphalt content for the type of mixture to be tested is known, the points should be close to that asphalt content to increase the accuracy of the calibration curve.

The results of the experiments that have been summarized above form the basis for the test method that is presented here. An efficient solvent was identified and fast and simple means for extracting and analyzing the asphalt cement were developed. These were then applied to a variety of typical mixtures to develop a calibration curve. Other procedures might yield an equally good calibration curve. However, the ones that were selected have the virtues of being both simple and inexpensive. The test method is presented in a step-by-step fashion in the next section.

Figure 1. Standard Calibration Curve. Transmittance of Light (%) vs. Asphalt Content (%)

Figure 2. Standard Calibration Curve. Absorbance of Light vs. Asphalt Content (%)

RAPID ASPHALT CONTENT TEST METHOD PROCEDURE

This section covers apparatus, materials and procedures for determining the asphalt content of hot-mix paving mixtures. Extraction of the asphalt cement is carried out with trichloroethylene. The percentage of transmitted light through the extracted solution is measured and correlated with the asphalt content of the mixture using the standard calibration curve shown in Figure 1.

I. EQUIPMENT

1. Balance or balances having an accuracy of at least 0.01% of the sample mass.
2. Hach spectrophotometer Model DR-100 with good quality cells that can be read to 1% of Transmittance of light (Figure 3)
3. Aluminum extraction container (see Appendix B)
4. Pipet, 1 ml, with pipet filling bulb^{*}
5. Pipet, 100 ml, with pipet filling bulb^{*}
6. Volumetric flask, 2000 ml

7. Small flask with a volume of at least 100 ml.

* Note: dispensing pipets may be substituted for these items.

II. REAGENTS

Trichloroethylene, technical grade. NOTE - This solvent is toxic and should be used only under a hood or in a well-ventilated area. The maximum acceptable concentration of vapor for an 8 hour exposure is reported to be 100 ppm. It should also be pipetted with a pipet filling bulb or a dispensing pipet.

III. PROCEDURE

1. A representative sample of the mixture (approx. 8 kg) shall be placed in a large, flat metal pan. If the mixture is not sufficiently soft to separate with a spatula, it should be warmed in an oven at approximately 230°F (110°C) until it can be handled.
2. Divide the sample into four approximately equal parts. Weigh 2 kg into the extraction container.
3. Add 2000 ml of trichloroethylene to the extraction container, place the rubber O-ring in the groove, and place the lid on the container. Put the metal ring around the extraction container and secure the clamp.

4. Close the bleed valve before starting the extraction procedure.
5. Hold the container, by the metal ring, with both hands, and invert it. Agitate it with a twisting movement for several seconds. Then bring it back to the normal, 'cover up' position. Repeat this operation eight times without a pause.
6. Place the extraction container under the hood and open the bleed valve to release the pressure. This should take no more than ten seconds.
7. Repeat the shaking process seven more times.
8. Again place the extraction container under the hood and open the bleed valve to release the pressure.
9. Remove the metal ring and the lid.
10. Take a sample of the extracted asphalt solution with the 1 ml pipet one inch below the surface of the liquid and put it in the small flask.
11. Add 100 ml of trichloroethylene to the flask using the 100 ml pipet and mix thoroughly.
12. Transfer some of the diluted solution to a spectrophotometer cell, and rinse it twice with the solution. Then fill it with the solution that is to be tested.

13. Fill another cell with pure trichloroethylene to be used as a blank.
14. Adjust the spectrophotometer to 0% transmission with the light path blocked, and to 100% transmission with the pure solvent. Repeat the adjusting operation at least twice.
15. Place the cell with the sample solution in the spectrophotometer and measure the transmittance of light.

IV. CALCULATION

Find the percentage of asphalt in the original mixture by entering the calibration curve, Figure 1, with the measured transmittance and reading the corresponding asphalt content from the other axis.

V. ACCURACY

There are not enough data available to predict the precision of the test method. This question was not explicitly examined in this research. However, when 2-3 duplicate samples were prepared, the measured asphalt contents rarely differed by more than 0.1%. And, they never differed by more than 0.2%. The Hach spectrophotometer can be read to the nearest 1% transmittance. A change in the transmittance of light of a solution by 1% corresponds roughly to a change in the measured asphalt content of 0.1%.

Figure 3. Hach Spectrophotometer Model DR-100

FIELD TESTS AND DISCUSSION OF RESULTS

The new test method was evaluated by testing asphalt mixtures, in the field, at mix plants. In order to do so, several asphalt plants located in the vicinity of West Lafayette, Indiana were visited. The plants were selected solely because they were located nearby. They were visited when they were producing mixtures for the State. This was done so that direct comparisons could be made between the State's current test method and the new, faster one. This work was done during the Fall of 1982 and the Summer of 1983.

Production rates at the plants were such that the State inspector rarely took more than one sample per day. This also meant that the new method could only be tried once a day since the object was to obtain a direct comparison of the two methods. Furthermore, field testing was restricted to asphalt cement mixtures. Emulsified asphalt mixtures were not tested. This combination of circumstances meant that relatively few comparisons were possible.

The process of testing was as follows. A sample (approximately 8 kg.) was taken directly from a delivery truck by the State inspector. It was quartered by the

inspector. One part of the sample (1500- 2500 grams) was tested by the inspector using the State's standard extraction method (ASTM D-2172, Method EI) using methylene chloride and a vacuum extractor. Another part of the sample (exactly 2000 grams) was tested by the author using the rapid test method. The difference in testing time was appreciable; the standard test took 45-60 minutes; the rapid test took no more than 10 minutes, and frequently less. The results obtained are shown in Table 1.

The design asphalt content is the expected value in the mix. However, this value may not be the value for the mix sample owing to variations in the plant operation. The results obtained by the State inspector also may not be the true content because of sampling and testing errors. Furthermore, on some occasions, it was observed that the inspector was not following the ASTM method as regards the drying of the extracted aggregate. Finally, the values obtained with the new method are also subject to sampling and experimental errors. Therefore, it is impossible to say which, if any, of the columns in Table 1 represents the true asphalt content.

The values in Table 1 are generally well correlated with the values expected from the mix design, and with those obtained by the State inspector. However, there are some values that are significantly different (values differing by more than $\pm 0.4\%$) from the design asphalt content and/or the

Table 1. Field Test Results

MIXTURE TYPE	DESIGN ASPHALT CONTENT (%)	ASPHALT CONTENT BY STANDARD TEST (%)	ASPHALT CONTENT BY RAPID TEST (%)
#5 Base			
	4.3	4.0	4.0
	4.3	4.0	4.2
	4.3	4.5	4.2
#5 D BASE			
	4.5	4.6	4.5
	4.5	4.8	4.6
	4.5	4.7	5.3
	4.5	4.5	4.3
#9 BINDER			
	4.8	4.9	5.3
	4.8	4.7	5.3
	4.8	4.5	4.6
	4.8	4.4	4.3
	4.8	5.0	4.3
#11 BINDER			
	4.9	5.6	5.6
	4.9	4.9	5.0
	5.0	5.3	5.0
	5.0	4.9	4.8
#11 SURFACE			
	6.1	5.9	6.2
	6.1	5.8	6.0
	6.1	6.5	6.4
	6.0	6.1	5.6

State test result. There is no obvious reason for these different results. They might have been caused by errors in measuring the volume of the extracted solution (1 ml) which seems to be the most critical step in the procedure. Or, they may merely represent scatter that one might expect in a completely new test method. The values in Table 1 are the first 20 times that this test method has ever been used outside of a laboratory.

However, the new method's results seem sufficiently well correlated with the other estimates of the asphalt content to indicate that the rapid test method has considerable merit and promise. Further, the values were obtained in approximately one tenth the time of the standard test. Thus, in an actual testing situation, a second test could be run quickly in the event of a disparate result from a first test. This rapid re-testing is not possible with the current test method.

It seems clear from the above that additional testing by State inspectors is both desirable and justifiable. The aim of this testing should be to obtain a large set of simultaneous results from both testing methods. For this purpose extreme care should be exercised to insure that the ASTM method is followed exactly.

DISCUSSION OF EXTENSIONS OF THE RAPID TEST METHOD

The rapid test method was developed specifically to test hot-mix bituminous mixtures made with asphalt cement and natural aggregates. However, the possible extension of the test to other classes of mixtures was superficially investigated. Specifically, the other classes that were considered were:

1. Hot mixtures using emulsified asphalt.
2. Mixtures containing slag aggregate.
3. Mixtures containing an unusually high percentage of fine material (<#200 sieve).
4. Recycled mixtures.

Emulsified Asphalt Mixtures

The rapid test method did not work when the hot mixtures were prepared with asphalt emulsions. The extract contained some binder that didn't dissolve. The transmittance of light through the solutions gave results that were in error. The reason seems to be that the asphalt emulsions used were not completely miscible in pure trichloroethylene solvent. Apparently, the emulsifying agent caused the difficulty.

Further tests were carried out, similar to those mentioned in Appendix A, using asphalt emulsions and a modified solvent.

The special solvent was a mixture of trichloroethylene and ethyl alcohol. It was tried with varying proportions of the two components. The results showed that a mixture of 25% ethyl alcohol and 75% trichloroethylene (by volume) appeared to render the emulsions completely soluble. Based on these findings it seems probable that a calibration curve for asphalt emulsion mixtures could be developed using the modified solvent. However, such a curve was not developed in this investigation.

Slag Aggregate Mixtures

When slag aggregate is used as aggregate in the preparation of paving mixtures, the slag tends to absorb more asphalt cement than in the case when natural aggregates are used. Therefore, the process of extraction of the asphalt cement from this material should be more difficult. In using the rapid test, it was thought that when slag aggregate was used, the extraction process would take longer. Several tests were performed with two different materials: a blast furnace slag and one from a basic oxygen process. The former is typically more porous than the latter. Typical mixes were prepared using 50% slag aggregate and 50% sand, with asphalt contents of 5.8, 6.3, 6.8, 7.3 and 7.8% for the blast furnace slag mixtures and 4.5, 5.0, 5.5, 6.0 and 6.5% for basic oxygen process slag mixtures. It was found that increasing the number of shakes of the asphalt extraction container from 15 to 30 produced an adequate extraction, and that the standard calibration curve (Figure 1) was applicable to these mixtures.

Fine Material in Mixtures

The finest portion of a mixture's aggregate (<#200 sieve) can cause the new test method to give an invalid result if it interferes with the light transmittance of the extracted solutions. If fine particles remain in suspension, the transmittance of the solution-suspension will be lowered and the test will yield an apparent asphalt content that is too high.

If this interfering factor is present, it can be detected by an increase in transmittance as time passes and the fines settle. This was never observed with the mixtures reported here. All of the mixtures that were tested met the Indiana gradation specifications that call for a maximum of 4% sub-#200 material (Appendix G).

This lack of interference is apparently because the particles have time to settle in both the extraction container and in the dilution flask. Samples are taken near the top of the liquid in both containers, and this region would be the first to become clear of particles.

The question of interference was studied explicatedly with the following experiment. Silt that passed the #200 sieve was added to pure trichloroethylene in the same proportions as would exist when testing a mix containing 4% fines. The solvent and silt were then thoroughly dispersed in a mixer normally used for dispersing soils (ASTM D-422). Some of the suspension was drawn off, while the mixer was in operation, and immediately placed in

the spectrophotometer. The instrument was set to read 100% transmittance with the pure solvent. With the suspension, it read 92% 15 seconds after the cell was placed in the instrument. This value gradually rose to 95% after 21 minutes and reached 100% only after 120 minutes.

The above indicates that fine material can interfere with the results. However, this test was an extreme case in which the fines were vigorously dispersed, and in which no settlement occurred prior testing. In a real mixture the fines are never so well dispersed, and are likely to be present as larger aggregations that settle much more quickly. (Settlement speed is proportional to the square of particle size). Further, the test method has, naturally, several points during which settlement does occur.

Because of these differences, the fines in the mixtures did not interfere with the test results for mixtures containing normal amounts of fines (within specifications). This is an additional advantage of the new test method over the standard extraction methods. In these latter methods, fines can be washed away and tallied as if they were asphalt.

However, experimenters who are testing mixtures that contain larger amounts of fines should check for possible interference before using the test as presented here. Checking is easily done by observing the transmittance of an extracted solution as a function of time. It should not increase. If it does, the method

will need to be modified by filtering or to encourage settlement.

Recycled Mixtures

Recycled mixtures may contain old, weathered asphalt, foreign material that a roadway has picked up in service, and rejuvenating additives. Any of these has the potential to interfere with either the extraction process or the light transmittance or both.

Several determinations of asphalt content using the rapid test were performed in recycled mixtures. In all cases, the diluted extracted solution contained black particles in suspension. The composition of these particles was not determined. However, they settled much more slowly than would typical, inorganic, mineral matter. These suspensions were darker; therefore the transmittance of light was lower. Consequently, the asphalt content results obtained were higher than the expected values as determined by the standard extraction test.

It may be possible to eliminate the interference in extracts from recycled mixtures by filtering with a non-absorbing, polymeric filter, or by encouraging sedimentation. It may also be possible to develop a different calibration curve for these materials. These points were not examined in the study, except to observe that a Teflon filter did produce an extract with a generally greater transmittance. However, testing recycled materials is an increasingly important operation, and more research on this question is needed.

IMPLEMENTATION OF THE RAPID TEST METHOD

The rapid test, as it exists now, appears ready for immediate use for testing conventional hot-mix paving mixtures containing either normal aggregates, or slag. The test is not ready, currently, for application to mixes containing emulsified asphalts, or to recycled mixtures. A user would have to assemble the necessary apparatus: conventional glassware and balance, a can for the extraction, and a suitable spectrophotometer. The test procedure given on pages 13-16 could then be followed.

The method can be applied either at the mix plant, or in the field at the placement site. In the later situation, it might be necessary to have the means available for warming the mix if it has cooled excessively. Because of its simplicity, and low cost, the test is particularly well suited for use by smaller organizations such as cities and counties for testing mixtures that they use.

The calibration curve that is presented here was developed using three different asphalts. It is not known to what extent other binders, from other sources, might effect it. A user would be well advised to make several test mixes, as described in Appendix G, using the asphalt cement in question. These tests will either verify the validity of the existing curve, or provide the user with a new curve that is valid for the mixtures to be tested.

CONCLUSIONS

1. The new, rapid test method can correctly determine the binder content of hot-mix paving mixtures made with asphalt cement.
2. Equipment for the test method is less expensive (approximately \$300.00) than existing methods and it can be used in the field without a laboratory.
3. The test method is unaffected by the presence of the normal amounts of fine material used in Indiana.
4. The test method can be easily modified to work with slag coarse aggregate.
5. The rapid test method appears extendable to mixes using emulsified asphalt.
6. Special calibration curves for unusual circumstances can be easily generated using Beer's Law.
7. More work is necessary to extend the test method to recycled mixtures.

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APPENDICES

APPENDIX A

SOLVENT SELECTION TEST

Tests were performed in order to select the solvent with the fastest rate of solubility and the best safety properties out of five different solvents that were considered as potential candidates for the new test method. The tests consisted of measuring the transmittance of light through solvent-asphalt solutions as a function of time. In this way, the solvents showed different dissolving powers at short times (1-10 min).

The solubility test was carried out by dissolving 0.5 grams of asphalt cement with 150 ml of solvent using a mechanical stirrer; the rate of dissolution was determined by measuring the decrease in transmitted light through the solutions at different times: 0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 5.0, and 10.0 min. The instrument used was a Turner spectrophotometer Model 350 set at 670 nm.

Each of the three asphalt cements used in the research project was tested in each of the solvents.

Five different solvents were tested. They were:

Kerosene (KER)	b.p. 175-325 °C
Methylene chloride (MC)	b.p. 40 °C
Perchloroethylene (PCE)	b.p. 121 °C
1,1,1 Trichloroethane (TC)	b.p. 74 °C
Trichloroethylene (TCE)	b.p. 87 °C

All the results are shown in Tables A1, A2 and A3. These values are the average of two separate tests carried out with the same solvent-asphalt cement pair.

Figures A1, A2 and A3 show the results in graphical form. The best solvents are methylene chloride and trichloroethylene. They dissolved the asphalt samples at faster rates than the other solvents. Although these two solvents have almost the same rate of solubility, and both of them are toxic to some degree (6,7), trichloroethylene was selected for use in the rapid test because of its higher boiling point (87 °C). A higher boiling point should decrease the concentration of toxic vapor in the air. However, it should be noted that this solvent should be used only under a hood or in a well ventilated area (2).

Table A1. Solubility Test Using Asphalt AP-5 from Texaco

Time (min)	KER %T	PCE %T	TC %T	MC %T	TCE %T
0.0	100.0	100.0	100.0	100.0	100.0
0.5	74.0	53.5	54.0	25.5	34.0
1.0	64.0	25.5	31.0	11.0	15.0
1.5	55.0	14.5	21.0	7.0	8.5
2.0	47.0	10.0	14.0	5.5	6.0
2.5	39.0	8.0	10.0	5.0	5.0
3.0	25.0	7.0	7.0	4.0	5.0
5.0	10.0	6.5	4.0	3.0	4.5
10.0	6.0	5.5	2.0	2.0	4.0

Table A2. Solubility Test Using Asphalt AC-20
from Asphalt Materials Co.

Time (min)	KER %T	PCE %T	TC %T	MC %T	TCE %T
0.0	100.0	100.0	100.0	100.0	100.0
0.5	70.0	20.0	45.0	22.0	15.0
1.0	54.5	9.0	27.0	7.0	5.0
1.5	43.0	5.0	17.5	3.0	2.5
2.0	34.0	3.0	12.0	3.0	2.0
2.5	27.5	2.0	8.5	1.0	1.5
3.0	22.5	2.0	7.0	1.0	1.0
5.0	13.5	1.5	2.0	1.0	1.0
10.0	4.0	1.5	1.0	0.5	1.0

Table A3. Solubility Test Using Asphalt AC-20 from Amoco

Time (min)	KER IT	PCE IT	TC IT	MC IT	TCE IT
0					
0.5	100.0				
1.0	70.0	100.0	100.0	100.0	100.0
1.5	54.0	50.0	57.5	15.5	23.0
2.0	41.0	27.5	34.0	5.5	7.0
2.5	32.0	15.0	21.0	4.0	3.5
3.0	25.5	9.5	14.0	3.5	3.0
5.0	20.5	6.0	10.0	3.0	3.0
10.0	11.0	4.5	7.0	3.0	2.5
	5.0	2.0	3.5	2.0	2.5
		2.0	1.0	1.5	1.0

Figure A1. Solubility Test Using Asphalt AP-5 from Texaco

Figure A2. Solubility Test Using Asphalt AC-20 from Asphalt Materials Co.

Figure A3. Solubility Test Using Asphalt AC-20 From Amoco

APPENDIX B

ASPHALT EXTRACTION CONTAINER

The extraction container should be made of a durable material, as light as possible, easy to handle, resistant to wear (and the solvent), and it should be easy to seal securely. The size of the container should be such that, after being filled with the testing mixture and the solvent, there is enough space to allow good mixing. Based on these requirements, the asphalt testing container was fabricated with the following specifications:

MATERIAL :	ALUMINUM (Alcoa Aluminum 6061 T6)
CAPACITY :	6,864 ml (1.81 gal)
HEIGHT :	21.27 cm (8.375 in)
DIAMETER :	int. 20.27 cm (7.981 in)
	ext. 20.59 cm (8.106 in)
WEIGHT :	5,696 g (12.56 lb)

The aluminum used to fabricate the container was resistant to wear. However, other types of aluminum might be equally good. The rubber O-ring was made of Viton. This material is not affected by the solvent.

This extraction container was designed in such a way that it could be easily handled and shaken with two hands. It has a bleed valve in order to release the pressure inside the container. It is shown in Figure B1.

Figure B1. Asphalt Extraction Container

APPENDIX C

SPECIFIC GRAVITY OF SOLUTIONS

Specific gravity is the mass of a unit volume of a substance, or solution, divided by the mass of an equal volume of water. The specific gravity of asphalt solutions should decrease with an increase of asphalt cement concentration (S.G.= 1.0000) in the solution if the solvent is trichloroethylene (S.G.=1.4560). If this decrease follows a sensible trend, it might be possible to use this property for the rapid test.

The extraction procedure uses a sample of 2 kg of bituminous mixture and 2,000 ml of trichloroethylene. Solutions for specific gravity testing were prepared by dissolving appropriate amounts of asphalt cement in solvent. The mixtures were dissolved with a mechanical stirrer until all the asphalt cement was in solution. This operation took between 10 and 15 min. All three asphalt cements were tested.

Once a solution was completely dissolved, it was poured into a graduated cylinder and the specific gravity was measured with a hydrometer. The specific gravities were measured with a hydrometer at intervals of 10 minutes. Readings

were taken until a stable value was obtained. The results are given in Tables C1, C2 and C3 and shown in Figures C1, C2 and C3.

The solutions were prepared as follows:

Asphalt Content of Mixture (%)	Grams of Asphalt in 2 kg of Mixture	Grams of Asphalt for test	ml of TCE
2.0	40	10.0	500
2.5	50	12.5	500
3.0	60	15.0	500
3.5	70	17.5	500
4.0	80	20.0	500
4.5	90	22.5	500
5.0	100	25.0	500
5.5	110	27.5	500
6.0	120	30.0	500
6.5	130	32.5	500
7.0	140	35.0	500

Table C1. Specific Gravity of Asphalt Solutions
Using Asphalt AP-5 from Texaco

Time (min)	Specific Gravity	Time (min)	Specific Gravity	Time (min)	Specific Gravity
2.0 %		2.5%		3.0 %	
0	1.4500	0	1.4485	0	1.4460
10	1.4495	10	1.4480	10	1.4455
20	1.4495	20	1.4475	20	1.4450
30	1.4495	30	1.4475	30	1.4450
3.5 %		4.0 %		4.5 %	
0	1.4435	0	1.4420	0	1.4420
10	1.4435	10	1.4415	10	1.4410
20	1.4430	20	1.4410	20	1.4400
30	1.4430	30	1.4410	30	1.4400
5.0 %		5.5 %		6.0 %	
0	1.4385	0	1.4365	0	1.4350
10	1.4380	10	1.4360	10	1.4340
20	1.4375	20	1.4360	20	1.4335
30	1.4375	30	1.4355	30	1.4335
		40	1.4355		
6.5 %		7.0 %			
0	1.4315	0	1.4300		
10	1.4310	10	1.4295		
20	1.4310	20	1.4295		
30	1.4310	30	1.4295		

Table C2. Specific Gravity of Asphalt Solutions
Using Asphalt AC-20 from Asphalt Materials Co.

Time (min)	Specific Gravity	Time (min)	Specific Gravity	Time (min)	Specific Gravity
2.0 %		2.5 %		3.0 %	
0	1.4525	0	1.4495	0	1.4475
10	1.4520	10	1.4500	10	1.4470
20	1.4515	20	1.4500	20	1.4470
30	1.4515	30	1.4500	30	1.4470
3.5 %		4.0 %		4.5 %	
0	1.4455	0	1.4435	0	1.4385
10	1.4450	10	1.4430	10	1.4385
20	1.4450	20	1.4430	20	1.4380
30	1.4450	30	1.4430	30	1.4380
5.0 %		5.5 %		6.0 %	
0	1.4375	0	1.4345	0	1.4320
10	1.4375	10	1.4345	10	1.4320
20	1.4375	20	1.4340	20	1.4320
30	1.4370	30	1.4340	30	1.4320
40	1.4370				
6.5 %		7.0 %			
0	1.4310	0	1.4285		
10	1.4305	10	1.4275		
20	1.4300	20	1.4270		
30	1.4295	30	1.4265		
40	1.4295	40	1.4265		

Table C3. Specific Gravity of Asphalt Solutions
Using Asphalt AC-20 from Amoco

Time (min)	Specific Gravity	Time (min)	Specific Gravity	Time (min)	Specific Gravity
2.0 %		2.5%		3.0 %	
0	1.4515	0	1.4485	0	1.4470
10	1.4510	10	1.4485	10	1.4465
20	1.4510	20	1.4485	20	1.4465
30	1.4510	30	1.4485	30	1.4465
3.5 %		4.0 %		4.5 %	
0	1.4445	0	1.4430	0	1.4405
10	1.4440	10	1.4425	10	1.4405
20	1.4440	20	1.4425	20	1.4405
30	1.4440	30	1.4420	30	1.4405
		40	1.4420		
5.0 %		5.5 %		6.0 %	
0	1.4385	0	1.4360	0	1.4350
10	1.4385	10	1.4360	10	1.4350
20	1.4385	20	1.4355	20	1.4350
30	1.4385	30	1.4355	30	1.4350
6.5 %		7.0 %			
0	1.4330	0	1.4310		
10	1.4325	10	1.4305		
20	1.4325	20	1.4300		
30	1.4320	30	1.4300

Figure C1. Specific Gravity of Asphalt Solutions
Using Asphalt AP-5 from Texaco

Figure C2. Specific Gravity of Asphalt Solutions
Using Asphalt AC-20 from Asphalt Materials Co.

Figure C3. Specific Gravity of Asphalt Solutions
Using Asphalt AC-20 from Amoco

These results indicate a good relationship between the asphalt content of the solutions and their specific gravities. However, the specific gravities changed with time. This is an adverse factor in running a rapid test. It was felt that these changes might be due to slight temperature changes, and that this effect might be eliminated if each specific gravity reading was normalized by dividing it by the specific gravity of the pure solvent at the same temperature.

Additional tests were run in which the specific gravity was measured in solutions with 2%, 5% and 7% asphalt contents at three different temperatures : 36°F (2°C), 70°F (21°C) and 91°F (33°C). In each test, the specific gravity was measured simultaneously in the asphalt solution and in the pure solvent. The readings were taken at ten minutes intervals. The results are given in Tables C4, C5 and C6 and in Figures C4, C5 and C6.

Table C4. Specific Gravity of Asphalt Solutions and Pure Solvent (TCE) Using Asphalt AC-20 from Asphalt Materials Co. Temperature 36°F (2°C)

% Asphalt Content	Time (min)	Specific Gravity of Solution	Specific Gravity of Solvent	Ratio
2.0	0	1.4745	1.4875	0.9913
	10	1.4765	1.4880	0.9923
	20	1.4780	1.4900	0.9919
	30	1.4780	1.4910	0.9913
	40	1.4800	1.4895	0.9936
	50	1.4805	1.4905	0.9933
5.0	0	1.4615	1.4875	0.9825
	10	1.4630	1.4880	0.9832
	20	1.4640	1.4900	0.9826
	30	1.4655	1.4910	0.9829
	40	1.4660	1.4895	0.9842
	50	1.4665	1.4905	0.9839
7.0	0	1.4535	1.4875	0.9771
	10	1.4550	1.4880	0.9778
	20	1.4560	1.4900	0.9772
	30	1.4570	1.4910	0.9772
	40	1.4580	1.4895	0.9789
	50	1.4580	1.4905	0.9782

Table C5. Specific Gravity of Asphalt Solutions and Pure Solvent (TCE) Using Asphalt AC-20 from Asphalt Materials Co. Temperature 70°F (21°C)

% Asphalt Content	Time (min)	Specific Gravity of Solution	Specific Gravity of Solvent	Ratio
2.0	0	1.4495	1.4600	0.9928
	10	1.4495	1.4595	0.9931
	20	1.4495	1.4595	0.9931
	30	1.4495	1.4595	0.9931
5.0	0	1.4385	1.4595	0.9856
	10	1.4375	1.4595	0.9849
	20	1.4375	1.4595	0.9849
	30	1.4370	1.4595	0.9846
	40	1.4370	1.4595	0.9846
7.0	0	1.4300	1.4595	0.9798
	10	1.4295	1.4595	0.9795
	20	1.4295	1.4595	0.9795
	30	1.4295	1.4595	0.9795

Table C6. Specific Gravity of Asphalt Solutions and Pure Solvent (TCE) Using Asphalt AC-20 from Asphalt Materials Co. Temperature 91°F (33°C)

% Asphalt Content	Time (min)	Specific Gravity of Solution *	Specific Gravity of Solvent	Ratio
2.0	0	1.40	1.4230	0.9830
	10	1.40	1.4220	0.9845
	20	1.40	1.4220	0.9845
	30	1.40	1.4220	0.9845
5.0	0	1.39	1.4230	0.9768
	10	1.39	1.4220	0.9775
	20	1.39	1.4220	0.9775
	30	1.39	1.4220	0.9775
7.0	0	1.38	1.4230	0.9698
	10	1.38	1.4220	0.9705
	20	1.38	1.4220	0.9705
	30	1.38	1.4220	0.9705

*These measurements were obtained with a less accurate hydrometer.

Figure C4. Ratio of Specific Gravity of Asphalt Solutions and Pure Solvent (TCE) Using Asphalt AC-20 from Asphalt Materials Co. Temperature 36°F (2°C)

Figure C5. Ratio of Specific Gravity of Asphalt Solutions and Pure Solvent (TCE) Using Asphalt AC-20 from Asphalt Materials Co. Temperature 70°F (21°C)

Figure C6. Ratio of Specific Gravity of Asphalt Solutions and Pure Solvent (TCE) Using Asphalt AC-20 from Asphalt Materials Co. Temperature 91°F (33°C)

These latter results show that normalizing with the pure solvent's specific gravity does not improve the situation. Further, different calibration curves would be needed for different solution temperatures. This is demonstrated in Figure C7. This would be a considerable drawback for a field test method. Therefore, specific gravity measurements were rejected as a possible test for asphalt content in the solutions.

Figure C7. Variation of Specific Gravity with Temperature of Asphalt Solutions

APPENDIX D

VISCOSITY OF SOLUTIONS

Viscosity is the resistance to flow of a fluid, either liquid or gas. A change in the asphalt cement concentration (a highly viscous material) in a solution, should be indicated by a change in the viscosity of the solution. If this change is sufficient to be measured, and to show differences in asphalt concentrations of the solutions, this property might be useful for the rapid test.

The viscosity of different asphalt solutions within a range from 2% to 7% was measured. Only one asphalt cement was tested. The instrument used was a Haake falling ball viscometer Model B. It measures the viscosity of liquids by correlating the time a ball takes to fall a certain distance in a liquid to that liquid's viscosity.

The falling time was measured five times for each solution and the average was used for calculating the viscosity using the manufacturer's empirical equation.

The solutions were prepared as follows:

Asphalt Content of Mixture (%)	Grams of Asphalt in 2 kg of Mixture	Grams of Asphalt for Test	ml of TCE
2.0	40	1.00	50
2.5	50	1.25	50
3.0	60	1.50	50
3.5	70	1.75	50
4.0	80	2.00	50
4.5	90	2.25	50
5.0	100	2.50	50
5.5	110	2.75	50
6.0	120	3.00	50
6.5	130	3.25	50
7.0	140	3.50	50

The results are shown in Table D1. Figures D1 and D2 show that the curve for falling time follows the same trend as the viscosity curve. Therefore, the practical value to use directly would be the falling time; not the viscosity. However, the curves don't show a good relationship between the falling time, or viscosity, and the asphalt content of the solutions. The sensitivity of this property of the solutions is not good enough to measure the asphalt content. The reason is that there is not a sufficiently large change in viscosity with these small changes in the amount of asphalt in the solutions. Therefore, viscosity measurements were rejected for determining the asphalt content of the solutions.

Table D1. Viscosity of Asphalt Solutions Using
Asphalt AC-20 from Asphalt Materials Co.

% of Asphalt Content	Falling Time (sec)	Viscosity (cp)
2.0	129	0.68
2.5	126	0.66
3.0	122	0.64
3.5	132	0.70
4.0	137	0.73
4.5	143	0.76
5.0	141	0.75
5.5	140	0.75
6.0	142	0.76
6.5	142	0.76
7.0	146	0.79

Figure D1. Viscosity of Asphalt Solutions Using Asphalt AC-20 from Asphalt Materials Co.

Figure D2. Falling Times of Asphalt Solutions Using Asphalt AC-20 from Asphalt Materials Co.

APPENDIX E

REFRACTIVE INDEX OF SOLUTIONS

The refractive index of a liquid is the ratio of the velocity of light in a vacuum to the velocity of light in the liquid. It provides a method for identifying a substance, and a method for determining the purity of the substance. Thus, a change in the concentration of an asphalt solution should show a change in the refractive index of the solution. If the change is great enough to correlate the asphalt concentration of the solutions with its refractive index, this property might be useful for the rapid test.

The refractive indices of different asphalt solutions within a range from 2% to 7 % were measured. Only one asphalt cement was tested. The instrument used was a Bausch & Lomb Abbe-3L refractometer. It was necessary to make a dilution because the solutions were too dark to be used in the refractometer. The dilution was 1+9 and it was made after the preparation of the dilutions. A dilution of 1+9 means 1 ml of asphalt solution plus 9 ml of solvent.

The solutions were prepared as follows:

Asphalt Content of Mixture (%)	Grams of Asphalt in 2 kg of Mixture	Grams of Asphalt for Test	ml of TCE
2.0	40	10.0	500
2.5	50	12.5	500
3.0	60	15.0	500
3.5	70	17.5	500
4.0	80	20.0	500
4.5	90	22.5	500
5.0	100	25.0	500
5.5	110	27.5	500
6.0	120	30.0	500
6.5	130	32.5	500
7.0	140	35.0	500

The results are shown in Table E1 and graphically in Figure E1.

Table E1. Refractive Index of Asphalt Solutions Using Asphalt AC-20 from Asphalt Materials Co.

Asphalt Content (%)	Refractive Index
2.0	1.4750
2.5	1.4755
3.0	1.4745
3.5	1.4745
4.0	1.4745
4.5	1.4745
5.0	1.4750
5.5	1.4740
6.0	1.4740
6.5	1.4735
7.0	1.4735

This property was difficult to measure. First, the solutions were too dark to obtain a reading in the refractometer, and it was necessary to dilute them. Furthermore, the volatility of the solvent was a problem; the solutions evaporated from the refractometer rapidly. Readings had to be taken in no more than 15 seconds. The sensitivity of the refractometer was 0.0005 units. The values for the solutions were so close that was not possible to find a good relationship between the refractive index of the solutions and their asphalt content. Hence, refractive index measurements were unsuitable for the rapid test.

Figure E1. Refractive Index of Asphalt Solutions Using Asphalt AC-20 from Asphalt Materials Co.

APPENDIX F

ABSORBANCE OF LIGHT BY SOLUTIONS

The physical property of solutions which is probably the most widely used in quantitative chemical analysis is the ability of materials in solution to absorb light. The general technique of using absorption of light in quantitative analysis is called absorptiometry. When the light utilized is in the ultraviolet, the visible and near the infrared portions of the spectrum, the technique is often referred to as spectrophotometry.

It has been known for some time that, with most materials in solution, the important feature with respect to the light absorption is how many molecules are in the light path, not their exact position in the light path. Thus doubling the concentration of a light-absorbing material has the same effect as doubling the path length.

For a given compound at a fixed wavelength and path length, the absorbance is a linear function of the concentration. Thus a plot of absorbance vs. concentration will be a straight line. This is stated mathematically in Beer's law

$$A = \log \frac{P_0}{P} = \log \frac{1}{T} = abc$$

where:

A = absorbance

P_0 = the amount of light falling on the sample

P = the amount of light transmitted by the sample

T = transmittance

a = the absorptivity of the solution. This is a constant dependent on the wavelength of the incident light and the nature of the absorbing material.

b = length of the light path through the solution, in cm

c = concentration of the absorbing material.

Most spectrophotometers can indicate either absorbance or transmittance of light, although the instruments actually measure the transmittance.

Usually Beer's law is obeyed, and by obtaining a calibration curve for a given solution system, which is a straight line for A vs. concentration or exponential for % T vs. concentration; the concentration of an unknown solution may be obtained from the calibration curve. Therefore, if the asphalt solutions obey Beer's law, a calibration curve might be obtained for use in the rapid test.

In preliminary tests, the transmittance of light was measured for different asphalt-solvent solutions with 2 to 7% asphalt contents. The instrument used was a Turner spectrophotometer Model 350 (set at 670 nm). Two different cells were used with different light path lengths (1 mm and 1 cm).

The solutions were prepared as follows:

Asphalt Content of Mixture (%)	Grams of Asphalt in 2 kg of Mixture	Grams of Asphalt for Test	ml of TCE
2.0	40	1.00	50
2.5	50	1.25	50
3.0	60	1.50	50
3.5	70	1.75	50
4.0	80	2.00	50
4.5	90	2.25	50
5.0	100	2.50	50
5.5	110	2.75	50
6.0	120	3.00	50
6.5	130	3.25	50
7.0	140	3.50	50

These asphalt solutions were too opaque. Therefore, diluted solutions were tested. The dilutions were 1+9 for the small cell (1 mm light path) and 1+99 for the larger cell (1 cm light path). A dilution 1+9 means 1 part of asphalt solution plus 9 parts of trichloroethylene and 1+99 means 1 part of asphalt solution plus 99 parts of trichloroethylene. The objective of using two different sizes of cells was to examine the effect of dilution on the results, because a large dilution might introduce

undesirable errors.

In these, and all following tests, light transmittance was the parameter that was actually recorded. In order to plot the results and get a straight line, the values were transformed to absorbance of light, A , by using Beer's law.

The results shown in Table F1 indicate that these solutions do follow Beer's Law. This is also demonstrated in Figure F1. There is not a great difference between the use of the small cell (dilution 1+9) and the use of the regular cell (dilution 1+99).

Table F1. Absorbance of Light Through Asphalt Solutions
Using Asphalt AC-20 from Asphalt Materials Co.

Asphalt Content	Small Cell (dilution 1+9)		Regular Cell (dilution 1+99)	
	% T	A	% T	A
2.0	72	0.14	69	0.16
2.5	69	0.16	68	0.17
3.0	62	0.21	60	0.22
3.5	59	0.23	58	0.24
4.0	54	0.27	52	0.28
4.5	53	0.28	51	0.29
5.0	47	0.33	45	0.35
5.5	47	0.33	44	0.36
6.0	44	0.36	42	0.38
6.5	40	0.40	41	0.39
7.0	38	0.42	38	0.42

Figure F1. Absorbance of Light Through Asphalt Solutions
Using Asphalt AC-20 from Asphalt Materials Co.

Further tests were undertaken using different dilutions for both cells. These showed that any reasonable combination of dilution and cell size gave the same results. This meant that the more convenient, and much cheaper, large cell could be used in conjunction with a more dilute solution.

The effect of various wavelengths was also studied. Increasing the wavelength from 610 nm to 670 nm slightly decreases the absorbance of the solutions and the sensitivity. For example, the following set of experiments used two different wavelengths: 610 nm (available as a set value on a portable instrument) and 670 nm (the longest wavelength on the Turner spectrophotometer). The effect of different cells was also further investigated. In this case, the solutions were extracted from bituminous mixtures with different asphalt contents (from 2 to 7%) prepared in the lab. Extraction followed the procedure described in RAPID ASPHALT CONTENT TEST METHOD PROCEDURE. The results are shown in Table F2 and Figures F2 and F3. Different dilutions don't effect the absorbance/asphalt correlation. Different wavelengths give different, but equally linear, correlations.

As a result of these tests, the absorbance of light was selected as the method to be used for determining the asphalt content of paving mixtures. A portable spectrophotometer (Hach Model DR-100) that is inexpensive and convenient for field testing was selected. This instrument has

a set wavelength of 610 nm and uses a cell with a 1 cm light path. The selected dilution was 1+100, this means 1 part of asphalt solution plus 100 parts of solvent. This was chosen so that the dilutions could be made with two pipets.

Table F2. Absorbance of Light Through Asphalt Solutions
Extracted from #9 Binder Mixtures

Asphalt Content	Small Cell (dilution 1+9)				Regular Cell (dilution 1+99)			
	670 nm		610 nm		670 nm		610 nm	
	%	% T	A	% T	A	% T	A	% T
2	76	0.12	67	0.17	76	0.12	66	0.18
3	68	0.17	56	0.25	68	0.17	56	0.25
4	62	0.21	49	0.31	60	0.22	46	0.34
5	53	0.28	39	0.41	55	0.26	39	0.41
6	49	0.31	35	0.46	48	0.32	34	0.47
7	44	0.36	28	0.55	44	0.36	28	0.55

Figure F2. Absorbance of Light of Two Different Wavelengths Through Asphalt Solutions Extracted from #9 Binder Mixtures Using a Small cell

Figure F3. Absorbance of Light of Two Different Wavelengths Through Asphalt Solutions Extracted from #9 Binder Mixtures Using a Regular Cell

APPENDIX G

PREPARATION AND TESTING OF HOT ASPHALT PAVING MIXTURES
AND DEVELOPMENT OF CALIBRATION CURVE

Different types of hot asphalt mixtures were prepared in the laboratory with asphalt contents from 2 to 9 %. They were tested according to the developed procedure described in RAPID ASPHALT CONTENT TEST METHOD PROCEDURE.

The objective of testing many different types of asphalt mixtures was to find possible interfering factors that could alter the results. The variables considered were: different types of asphalt cements (AC-20 from Amoco, AC-20 from Asphalt Materials Co., and AP-5 from Texaco), different types of coarse aggregates (gravel and crushed stone), and different mixtures (#9 binder and #11 surface). Table G1 summarizes these variables. The objective was to develop a calibration curve for each combination of variables, and to compare these curves.

The materials used were as follows:

Sand and gravel from Medusa Aggregates Co. Western,
Materials Division, West Lafayette, Indiana

Crushed stone from Delphi Limestone Inc., Delphi, Indiana

Asphalt cement AP-5 from Texaco, Lawrenceville, Indiana

Asphalt cement AC-20 from Amoco, Whiting, Indiana

Asphalt cement AC-20 from Asphalt Materials Co., Indianapolis, Indiana

Table G1. Hot Asphalt Paving Mixtures Designations

		Asphalt AC-20 from Asphalt Materials Co.	Asphalt AC-20 from Amoco	Asphalt AP-5 from Texaco
#9 Binder	Gravel	A-1	B-1	C-1
Mix	Stone	A-2	B-2	C-2
#11 Surface	Gravel	A-3	B-3	C-3
Mix	Stone	A-4	B-4	C-4

The proportioning of the mixes was according to the Indiana State Department of Highways Specifications, section 403.04 (8). The gradation limits for #9 binder and #11 surface mixtures are shown in Tables G2 and G3 and graphically in Figures G1 and G2. The percentage used for each size fraction was the mean value of the gradation limits. The mix formulations are shown in Tables G4 and G5. In each case 2 kg of total mixture were prepared .

The aggregates were heated in an oven at a temperature of 280°F (138°C) for at least 24 hours before mixing. The

asphalt was heated at a temperature of 280°F (138°C) for 4-8 hours. Then it was combined by weight with the aggregate to achieve the desired proportions. The balance used was sensitive to 0.1 grams. The asphalt was added slowly to the aggregate in a mixing bowl. Using a large spoon, the mixing was continued until all the aggregate was completely mixed with the asphalt. This operation took approximately 5 minutes. Then, the sample was transferred into the extraction container. Material left in the mixing bowl was removed by using the 2000 ml of solvent required for the test. In this way, all the material was transferred to the extraction container, and a possible error for asphalt left in the mixing bowl was eliminated. The asphalt cement was then extracted as described in RAPID ASPHALT CONTENT TEST METHOD PROCEDURE.

Table G2. Gradation Limits for #9 Binder Mixtures

Sieve Size	% Passing Spec. Limits	Cumulative Passing (mean) %	Cumulative Retained (mean) %	Grams Retained for each 2000 Grams
3/4 inch	100	100	0	0
1/2 inch	73-91	82	18	360
3/8 inch	40-73	57	43	500
No. 4	34-38	36	64	420
No. 8	18-39	29	71	140
No. 16	12-34	23	77	120
No. 30	5-22	14	86	180
No. 50	1-14	7	93	140
No. 100	0-6	3	97	80
No. 200	0-4	2	98	60

Table G3. Gradation Limits for #11 Surface Mixtures

Sieve Size	% Passing Spec. Limits	Cumulative Passing (mean) %	Cumulative Retained (mean) %	Grams Retained for each 2000 Grams
1/2 inch	100	100	0	0
3/8 inch	85-98	92	8	160
No. 4	50-54	52	48	800
No. 8	30-57	44	56	160
No. 16	20-46	33	67	220
No. 30	8-30	19	81	280
No. 50	2-19	11	89	160
No. 100	0-9	4	96	140
No. 200	0-4	2	98	80

FIGURE G1. GRADATION LIMITS FOR #9 BINDER MIXTURES

FIGURE 62. GRADATION LIMITS FOR #11 SURFACE MIXTURES

Table G4. Job-Mix Formulas for #9 Binder Mixtures

Asphalt Content	2%	3%	4%	5%
sieve size	wt. gms.	wt. gms.	wt. gms.	wt. gms.
1/2 inch	352.8	349.2	345.6	342.0
3/8 inch	490.0	485.0	480.0	475.0
No. 4	411.6	407.4	403.2	399.0
No. 8	137.2	135.8	134.4	133.0
No. 16	117.6	116.4	115.2	114.0
No. 30	176.4	174.6	172.8	171.0
No. 50	137.2	135.8	134.4	133.0
No. 100	78.4	77.6	76.8	76.0
No. 200	58.4	58.2	57.6	57.0
wt. of aggregate (gms.)	1960	1940	1920	1900
wt. of asphalt (gms.)	40	60	80	100
Asphalt Content	6%	7%	8%	9%
sieve size	wt. gms.	wt. gms.	wt. gms.	wt. gms.
1/2 inch	338.4	334.8	331.2	327.6
3/8 inch	470.0	465.0	460.0	455.0
No. 4	394.8	390.6	386.4	382.2
No. 8	131.6	130.2	128.8	127.4
No. 16	112.8	111.6	110.4	109.2
No. 30	169.2	167.4	165.6	163.8
No. 50	131.6	130.2	128.8	127.4
No. 100	75.2	74.4	73.6	72.8
No. 200	56.4	55.8	55.2	54.6
wt. of aggregate (gms.)	1880	1860	1840	1820
wt. of asphalt (gms.)	120	140	160	180

Table G5. Job-Mix Formulas for #11 Surface Mixtures

Asphalt Content	2%	3%	4%	5%
sieve size	wt. gms.	wt. gms.	wt. gms.	wt. gms.
3/8 inch	156.8	155.2	153.6	152.0
No. 4	784.0	776.0	768.0	760.0
No. 8	156.8	155.2	153.6	152.0
No. 16	215.8	213.4	211.2	209.9
No. 30	274.4	271.6	268.8	266.0
No. 50	156.8	155.2	153.6	152.0
No. 100	137.2	135.8	134.4	133.0
No. 200	78.4	77.6	76.8	76.0
wt. of aggregate (gms.)	1960	1940	1920	1900
wt. of asphalt (gms.)	40	60	80	100
Asphalt Content	6%	7%	8%	9%
sieve size	wt. gms.	wt. gms.	wt. gms.	wt. gms.
3/8 inch	150.4	148.8	147.2	145.6
No. 4	752.0	744.0	736.0	728.0
No. 16	206.8	204.6	202.4	200.2
No. 30	263.2	260.4	257.6	254.8
No. 50	150.4	148.8	147.2	145.6
No. 100	131.6	130.2	128.8	127.4
No. 200	75.2	74.4	73.6	72.8
wt. of aggregate (gms.)	1880	1860	1840	1820
wt. of asphalt (gms.)	120	140	160	180

The measured absorbances of the extracted solutions for all laboratory mixes are shown in Tables G6, G7 and G8. These can be shown graphically in two ways: a straight line (absorbance of light vs. asphalt content) or an exponential curve (transmittance of light vs. asphalt content). All the results were obtained by measuring the transmittance and transformed to absorbance of light using Beer's law (see Appendix F). Using the absorbance plot, a straight line was fitted through each set of values by a least squares analysis. These straight lines are shown in Figures G3, G4 and G5. They show essentially the same correlation between the asphalt content of the mixture and the absorbance of light.

The results were so close that it was not felt necessary to have a different calibration curve for each type of mixture and asphalt cement. For this reason, the values for all the different mixtures were combined and a straight line was fitted through them by a least squares analysis. A single calibration curve was obtained and is shown in Figure G6. This curve was used for determining the asphalt content of all the subsequent samples. This plot is an exponential curve for transmittance of light vs. asphalt content (Figure 1) or a straight line for absorbance of light vs. asphalt content (Figure 2).

Table G6. Absorbance of Light for Mixtures
A-1, A-2, A-3 and A-4

Asphalt AC-20 from Asphalt Materials Co.

Asphalt Content	#9 Binder				#11 Surface			
	Gravel		Crushed Stone		Gravel		Crushed Stone	
	A-1		A-2		A-3		A-4	
	%	% T A	% T A	% T A	% T A	% T A	% T A	% T A
2	68	0.17	66	0.18	64	0.19	65	0.19
3	58	0.24	56	0.25	54	0.27	57	0.24
4	50	0.30	48	0.32	45	0.35	49	0.31
5	40	0.40	41	0.39	38	0.42	41	0.39
6	35	0.46	35	0.46	32	0.50	35	0.46
7	30	0.52	32	0.50	30	0.52	31	0.51
8	26	0.59	26	0.59	27	0.57	27	0.57
9	23	0.64	23	0.64	24	0.62	24	0.62

Table G7. Absorbance of Light for Mixtures
B-1, B-2, B-3 and B-4

Asphalt AC-20 from Amoco

Asphalt Content	#9 Binder				#11 Surface			
	Gravel		Crushed Stone		Gravel		Crushed Stone	
	B-1		B-2		B-3		B-4	
	%	% T A	% T A	% T A	% T A	% T A	% T A	% T A
2	66	0.18	66	0.18	66	0.18	69	0.16
3	59	0.23	58	0.24	56	0.25	61	0.22
4	51	0.29	50	0.30	51	0.29	50	0.30
5	44	0.36	45	0.35	46	0.34	40	0.40
6	40	0.40	39	0.41	39	0.41	35	0.46
7	36	0.44	34	0.47	36	0.44	30	0.52
8	32	0.50	30	0.52	33	0.48	27	0.57
9	25	0.60	27	0.57	28	0.55	24	0.62

Table G8. Absorbance of Light for Mixtures
C-1, C-2, C-3 and C-4

Asphalt AP-5 from Texaco

Asphalt Content	#9 Binder				#11 Surface			
	Gravel		Crushed Stone		Gravel		Crushed Stone	
	C-1		C-2		C-3		C-4	
	%	% T A	% T A	% T A	% T A	% T A	% T A	% T A
2	68	0.17	70	0.16	68	0.16	70	0.17
3	59	0.23	64	0.19	60	0.22	60	0.22
4	51	0.29	57	0.24	53	0.28	50	0.30
5	47	0.33	48	0.32	46	0.34	42	0.38
6	40	0.40	40	0.40	37	0.43	39	0.41
7	38	0.42	34	0.47	34	0.47	32	0.50
8	30	0.52	32	0.50	27	0.57	30	0.52
9	28	0.55	30	0.52	25	0.60	26	0.59

Figure G3. Absorbance of Light for Mixtures A-1, A-2, A-3 and A-4

Figure G4. Absorbance of Light for Mixtures B-1, B-2, B-3 and B-4

Figure G5. Absorbance of Light for Mixtures C-1, C-2, C-3 and C-4

Figure G6. Calibration Curve