# EVALUATION OF ASPHALT CEMENT EXTRACTION AND RECOVERY METHODS

FINAL REPORT

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ABSTRACT

This study was concerned with the quality of recovered asphalt cement which may be attributable to the method used for extraction or to the Abson recovery. Variables of these procedures such as the time an asphalt cement was exposed to extraction solvent, the total quantity of asphalt cement recovered, the total quantity of extraction solvent, and both the type and quantity of fines in the extracted mix samples were isolated to determine their influence on tested asphalt cement properties. Also examined was the testing variation within one operator and between two operators. Findings with respect to the above aims have led to a recommendation that recovery of asphalt from solution by the Abson method (AASHTO T 170-73) should be strictly adhered to, except that the extraction from the aggregate-asphalt mixture should be in accordance only with Method A of AASHTO T 164-76.

## METRIC CONVERSION FACTORS*

| To Convert from | To | Multiply by |
|---|---|---|
| | **Length** | |
| foot | meter (m) | 0.3048 |
| inch | millimeter (mm) | 25.4 |
| yard | meter (m) | 0.9144 |
| mile (statute) | kilometer (km) | 1.609 |
| | **Area** | |
| square foot | square meter ($m^2$) | 0.0929 |
| square inch | square centimeter ($cm^2$) | 6.451 |
| square yard | square meter ($m^2$) | 0.8361 |
| | **Volume (Capacity)** | |
| cubic foot | cubic meter ($m^3$) | 0.02832 |
| gallon (U.S. liquid)** | cubic meter ($m^3$) | 0.003785 |
| gallon (Can. liquid)** | cubic meter ($m^3$) | 0.004546 |
| ounce (U.S. liquid) | cubic centimeter ($cm^3$) | 29.57 |
| | **Mass** | |
| ounce-mass (avdp) | gram (g) | 28.35 |
| pound-mass (avdp) | kilogram (kg) | 0.4536 |
| ton (metric) | kilogram (kg) | 1000 |
| ton (short, 2000 lbm) | kilogram (kg) | 907.2 |
| | **Mass per Volume** | |
| pound-mass/cubic foot | kilogram/cubic meter ($kg/m^3$) | 16.02 |
| pound-mass/cubic yard | kilogram/cubic meter ($kg/m^3$) | 0.5933 |
| pound-mass/gallon (U.S.)** | kilogram/cubic meter ($kg/m^3$) | 119.8 |
| pound-mass/gallon (Can.)** | kilogram/cubic meter ($kg/m^3$) | 99.78 |
| | **Temperature** | |
| deg Celsius (C) | kelvin (K) | $t_K=(t_C+273.15)$ |
| deg Fahrenheit (F) | kelvin (K) | $t_K=(t_F+459.67)/1.8$ |
| deg Fahrenheit (F) | deg Celsius (C) | $t_C=(t_F-32)/1.8$ |

*The reference source for information on SI units and more exact conversion factors is "Metric Practice Guide" ASTM E 380.

**One U.S. gallon equals 0.8327 Canadian gallon.

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# IMPLEMENTATION

The Department has reacted to the findings herein by implementing the recommendation of the study calling for specific procedures to be followed in the recovery of asphalt cement. Such implementation will technically assist the Department in its on-going recycling program and in its plant certification process.

# INTRODUCTION

The Department's Research and Development Section has recently been involved with extensive analyses of recovered asphalt cement properties. These analyses were undertaken in preparation of the implementation of the state's recycling program and the institution of a plant re-certification program.

Two of the essential data acquisitions involved with recycling asphaltic concrete are the quantity and quality of the existing asphalt cement. No rational mix design can be developed until these two characteristics of the binder are determined and optimized. Either of the two AASHTO methods utilized by this state for extracting asphalt cement from a mix (AASHTO T-164, Methods A and B) will yield a tolerable comparison as far as quantity of asphalt is concerned. However, if these methods are used interchangeably with the Abson recovery (AASHTO T-170), the quality of the recovered asphalt cement may differ significantly. This difference may be more pronounced with highly aged cements, typical of those to be recycled. As the proper addition rate of rejuvenator is determined from these recovered asphalt properties, it is felt that any differences in such properties associated with the method of extraction or with the Abson recovery should be investigated.

Additionally, as part of the state's plant re-certification program, asphalt cement recovered from a plant-produced mix must meet a maximum viscosity index specification. A disparity in test results on the recovered asphalts was occasionally found when the different extraction methods were used interchangeably with the Abson recovery. While the plants were re-sampled on these occasions in fairness to the contractors, the need for an investigation of these disparities was evident.

This study was concerned with the quality of recovered asphalt cement which may be attributable to the method used for extraction or to the Abson recovery. Variables of these procedures such as the time an asphalt cement was exposed to extraction solvent, the total quantity of asphalt cement recovered, the total quantity of extraction solvent, and both the type and quantity of fines in the extracted mix samples were isolated to determine their influence on tested asphalt cement properties. Also examined was the testing variation within one operator and between two operators.

## OBJECTIVE AND SCOPE

The objective of this study was to observe any difference in the quality of asphalt cement attributable to the method used for extraction or to the Abson recovery. As such, the scope focused on the isolation of components of the extraction and recovery processes, specifically through the following aims:

I. determine the effect of time in reagent-grade trichloroethylene upon aged and unaged asphalt cements;

II. determine the effect of a reduction of the quantity of asphalt cement in solution in the Abson recoveries;

III. determine the testing variation within one operator and between two operators;

IV. determine the effect of quantity of solvent in the primary distillation of method AASHTO T-170; and,

V. determine the effect of fines in hot-mix samples upon Abson recovered asphalt cement properties.

## METHODOLOGY

Sufficient quantity of viscosity grade AC-30 asphalt cement from six different Louisiana asphalt sources was used in the study. Viscosity measurements were made on each source of asphalt, so as to have a reference when comparing subsequent test results for accuracy. Additionally, a portion of each source's quantity was artificially aged to various states of oxidation in laboratory forced-air ovens to generate a quantity of aged asphalt; viscosity measurements were made on each source of these aged asphalts. Table 1 contains the viscosity test results for the six original AC-30 asphalts and the six laboratory aged asphalts.

TABLE 1

REFERENCE VISCOSITY DATA

| Asphalt Source | Original AC-30 Viscosity* | Lab-aged Viscosity* |
|---|---|---|
| E | 2,522 | 35,718 |
| S | 2,997 | 86,848 |
| C | 2,986 | 44,986 |
| M | 3,169 | 39,043 |
| L | 3,209 | 59,666 |
| T | 3,235 | 173,998 |

*Absolute viscosity @ 140°F, poises.

Aim I - Effect of Time in Solvent

In the past, Louisiana had always made use of AASHTO T-164, Method B, as its procedure for extraction prior to Abson recovery. It was not uncommon for this extraction, using reagent-grade trichloroethylene, to be incomplete after the initial eight hours of refluxing. This was the case primarily when dealing with asphaltic concrete samples from roadways where some aging of the binder had occurred, rather than from fresh hot mix samples from current plant production. In some cases, the total time an asphalt cement was in contact with the solvent was 44 hours (soaking overnight prior to reflux operations, refluxing during the next day's normal working hours, and soaking overnight prior to the following morning's Abson recovery). It was not until a recent need to extract and recover a large number of samples within a short period of time that the centrifuge alternate for extraction, AASHTO T-164, Method A, was tried. Using the centrifuge method, extractions can be completed and recoveries performed within an eight-hour work day.

Aim I of this study was to identify what effect a prolonged dilution in solvent had upon any of Louisiana's six sources of asphalt cement. Times were chosen to coincide with what was considered to be maximum exposure (44 hours, using reflux), normal exposure (20 hours, using reflux), and minimum exposure (5 hours, using centrifuge). For each of the six sources of asphalt cement, 75 grams of both the original and oven-aged binder were added to separate Abson recovery flasks and diluted with 125 ml. of reagent-grade trichloroethylene yielding a 200-ml. solution for the Abson recovery. Triplicate samples were prepared for each of the three time exposures to be studied. In total, 108 samples were prepared; six sources, two levels of viscosity within each source, three time exposures for each level of viscosity and three replicates for each time exposure. It is pointed out that only one gentle heating of material was necessary for each viscosity level within any asphalt source; the nine asphalt samples so represented (three time exposures and three replicas) were all poured into their separate flasks at one time. The subsequent addition of the solvent to each flask was then scheduled to allow for the proper time exposure for each sample.

Aim II - Effect of Reduced Sample Size

The Abson procedure calls for a minimum of 75 grams of asphalt cement to be recovered. Occasionally, small quantities of asphaltic concrete are submitted for analysis (i.e., two 4-inch cores from the wearing course of a roadway) which, when extracted, contain less than the 75-gram minimum.

Aim II of this study was to examine what effect a reduced quantity of recovered asphalt had upon any subsequent viscosity determinations. It was decided to limit the scope of this phase to only the oven-aged asphalts from each of the six previously mentioned asphalt sources, the belief being that the occurrence of small sample quantities was isolated to cored specimens from aged roadways. In this phase, 50 grams of each aged asphalt was dissolved in 150 ml. of reagent-grade trichloroethylene. The schedule of sample preparation was such that each asphalt was in solution for five hours, inclusive of recovery time. Similar to the previous aim, triplicate samples were prepared and tested yielding a total of 18 Abson recovered viscosity results (six sources of aged asphalt with three replicas each, all 50-gram quantities of asphalt and all exposed in solvent for five hours). These 18 results allow interaction analysis with 18 of the 108 results attained in Aim I whenever 75 grams of aged asphalt were tested in triplicate with five-hour exposure to solvent.

Aim III - Effect of Operator

The source of the asphalt cement was not considered to be a variable which would influence the effect either within or between operators. As such, only asphalt source E was used for this phase of the study. Two operators, both previously familiar with the Abson procedure, each performed nine recoveries on 75 grams of the original AC-30 asphalt and nine recoveries on 75 grams of the aged asphalt (three replicas after 5 hours, 20 hours and 44 hours dilution in solvent). Additionally, operator effect was considered when the procedure sample size was reduced from 75 grams to 50 grams; here, two operators each performed six recoveries on aged asphalt exposed for five hours in solvent.

Aim IV - Effect of Solvent Quantity

When using AASHTO T-164, Method A, for centrifugal extraction of asphalt cement prior to any subsequent Abson recovery, the quantity of reagent-grade trichloroethylene necessary to completely wash clean a mix may vary. With new mix, two washes are sufficient (approximately 900 ml.); with older mix, four and even six washes are necessary (approximately 1800 ml. and 2700 ml., respectively). The quantity of effluent (solvent plus dissolved asphalt cement) generated in the extraction process is then reduced to approximately 200 ml. by any primary distillation method before beginning the AASHTO T-170 Abson recovery procedure.

Aim IV of this study was to examine what effect a different quantity of extraction solvent had upon the recovered asphalt viscosity determinations, the larger quantity necessitating a longer time under the heat of a primary distillation procedure. In this phase, only the oven-aged asphalts from all six sources were used, the belief being that the main cause for a greater quantity of solvent is the age of the binder being extracted. Duplicate sets of 75 grams of asphalt from each source were dissolved in three separate quantities of reagent-grade trichloroethylene solvent (900 ml., 1800 ml. and 2700 ml.). All 36 samples prepared as above were allowed to stand in solution for 1-1/2 hours prior to initiating the primary distillation and subsequent Abson recovery.

Aim V - Effect of Mineral Fines

The Abson recovery procedure specifies centrifuging all extraction effluent prior to the distillation recovery to eliminate any error (high viscosity and/or low penetration) caused by an ash content in the recovered asphalt cement. In addition to the above possible error, it was believed that the quantity or the type of fines contained in a mix, even if centrifuged from the effluent, might cause other errors. Specifically, a high level of fines might increase the time required for extraction. This increase in time in a centrifuge-type extraction is directly related to the need for an increase in the number of washings; this effect has previously been isolated in Aim IV. However, any increase in time in a reflux-type extraction, caused by a clogging of the filter papers, will result in a longer exposure of the asphalt cement to the heat required with this type extraction. The type of fines could also influence the recovered asphalt cement properties if any chemical or physical (gradation, absorption, etc.) reactions occurred.

Aim V of this study examined two different types of mineral filler fines (cement stack dust and fly ash), each added at two levels (2% and 12%, by weight) to a lab-prepared mix, using source S original AC-30 asphalt cement. Other than the type and quantity of fines, the prepared mixes were identical--1200 grams at 7.5% asphalt, yielding 90 grams of recovered asphalt cement. All mixes were prepared at 260°F and allowed to cool to ambient for 24 hours prior to initiating the extraction procedure. The viscosity of the asphalt cement prior to mix fabrication was tested to be 3,075 poises. These four mix combinations of fines type and quantity were extracted in duplicate by both the centrifuge method and the reflux method. A total of 16 Abson recoveries were involved in this phase.

# RESULTS

## Aim I - Effect of Time in Solvent

The 108 recovery and subsequent viscosity test results associated with this phase are reported in Tables 2 and 3. Table 2 contains the 54 results attained when recovering the original viscosity from all six asphalt sources at three time exposures in solvent and with three replicate results for each time exposure. Table 3 contains 54 similar results attained when recovering the lab-aged viscosity.

## Aim II - Effect of Reduced Sample Size

The 18 recovery and subsequent viscosity test results associated with the 50-gram asphalt sample size of this phase are reported in Table 4. Table 4 contains triplicate recovered viscosity values for each of the six asphalt sources when 50 grams of lab-aged asphalt was exposed to the extraction solvent for five hours. For comparison purposes, the 18 test results using a 75-gram sample size, previously generated under Aim I when identical variables were employed, are reported in Table 5.

## Aim III - Effect of Operator

The 48 recovery and subsequent viscosity test results associated with this phase are reported in Tables 6, 7 and 8. Tables 6 and 7 both contain results obtained when 75 grams of asphalt were recovered; Table 6 using original asphalt cement and Table 7 using lab-aged asphalt cement. Table 8 contains results obtained when 50 grams of asphalt were recovered. The asphalt cement used in this phase was from source E.

TABLE 2

EFFECT OF TIME IN SOLVENT (ORIGINAL ASPHALTS)*

| Asphalt Source E (2,522 poises) | | | Asphalt Source S (2,997 poises) | | | Asphalt Source C (2,986 poises) | | |
|---|---|---|---|---|---|---|---|---|
| 5 hours | 20 hours | 44 hours | 5 hours | 20 hours | 44 hours | 5 hours | 20 hours | 44 hours |
| 2,526 | 2,544 | 2,747 | 3,107 | 3,011 | 3,978 | 1,804 | 1,644 | 1,976 |
| 2,523 | 2,393 | 2,773 | 3,405 | 3,639 | 3,954 | 1,698 | 1,845 | 1,829 |
| 2,384 | 2,382 | 2,420 | 3,782 | 3,720 | 4,018 | 1,948 | 2,049 | 1,865 |

| Asphalt Source M (3,169 poises) | | | Asphalt Source L (3,209 poises) | | | Asphalt Source T (3,235 poises) | | |
|---|---|---|---|---|---|---|---|---|
| 5 hours | 20 hours | 44 hours | 5 hours | 20 hours | 44 hours | 5 hours | 20 hours | 44 hours |
| 3,012 | 3,142 | 3,214 | 3,559 | 3,497 | 3,780 | 3,210 | 3,371 | 3,666 |
| 3,193 | 2,941 | 2,892 | 3,819 | 3,350 | 3,558 | 3,686 | 3,425 | 3,587 |
| 2,689 | 2,767 | 2,922 | 3,420 | 3,279 | 2,953 | 3,454 | 3,622 | 3,964 |

*Results shown are recovered absolute viscosities @ 140°F (poises).

TABLE 3

EFFECT OF TIME IN SOLVENT (LAB-AGED ASPHALTS)*

| Asphalt Source E (35,718 poises) | | | Asphalt Source S (86,848 poises) | | | Asphalt Source C (44,986 poises) | | |
|---|---|---|---|---|---|---|---|---|
| 5 hours | 20 hours | 44 hours | 5 hours | 20 hours | 44 hours | 5 hours | 20 hours | 44 hours |
| 31,954 | 23,733 | 33,544 | 87,909 | 85,491 | 94,084 | 27,004 | 31,258 | 33,253 |
| 25,789 | 26,480 | 32,874 | 84,436 | 100,954 | 112,527 | 30,611 | 29,490 | 32,265 |
| 28,845 | 28,769 | 25,576 | 92,849 | 82,260 | 97,043 | 36,036 | 35,356 | 36,004 |

| Asphalt Source M (39,043 poises) | | | Asphalt Source L (59,666 poises) | | | Asphalt Source T (173,998 poises) | | |
|---|---|---|---|---|---|---|---|---|
| 5 hours | 20 hours | 44 hours | 5 hours | 20 hours | 44 hours | 5 hours | 20 hours | 44 hours |
| 33,952 | 42,294 | 45,069 | 72,663 | 60,622 | 74,728 | 139,028 | 124,946 | 198,403 |
| 36,598 | 35,200 | 37,042 | 62,019 | 52,899 | 55,165 | 157,641 | 148,925 | 179,683 |
| 31,904 | 30,772 | 36,217 | 49,103 | 49,097 | 53,403 | 160,557 | 157,893 | 151,228 |

*Results shown are recovered absolute viscosities @ 140°F (poises).

TABLE 4

RECOVERY OF 50-GRAM ASPHALT QUANTITY*

| Asphalt Source E (35,718 poises) | Asphalt Source S (86,848 poises) | Asphalt Source C (44,986 poises) |
|---|---|---|
| 32,543 | 96,909 | 38,132 |
| 29,094 | 114,104 | 42,897 |
| 28,023 | 108,567 | 39,032 |

| Asphalt Source M (39,043 poises) | Asphalt Source L (59,666 poises) | Asphalt Source T (173,998 poises) |
|---|---|---|
| 39,261 | 59,828 | 229,267 |
| 33,310 | 59,434 | 215,728 |
| 36,649 | 58,417 | 231,742 |

*Recovered absolute viscosities @ 140°F (poises).

TABLE 5

RECOVERY OF 75-GRAM ASPHALT QUANTITY*

| Asphalt Source E (35,718 poises) | Asphalt Source S (86,848 poises) | Asphalt Source C (44,986 poises) |
|---|---|---|
| 31,954 | 87,909 | 27,004 |
| 25,789 | 84,436 | 30,611 |
| 28,845 | 92,849 | 36,036 |

| Asphalt Source M (39,043 poises) | Asphalt Source L (59,666 poises) | Asphalt Source T (173,998 poises) |
|---|---|---|
| 33,952 | 72,663 | 139,028 |
| 36,598 | 62,019 | 157,641 |
| 31,904 | 49,103 | 160,557 |

*Recovered absolute viscosities @ 140°F (poises).

TABLE 6

OPERATOR EFFECT - 75 GRAMS, ORIGINAL ASPHALT*

Operator 1

| 5 Hours | 20 Hours | 44 Hours |
|---|---|---|
| 2,526 | 2,544 | 2,747 |
| 2,523 | 2,393 | 2,773 |
| 2,384 | 2,382 | 2,420 |

Operator 2

| 5 Hours | 20 Hours | 44 Hours |
|---|---|---|
| 2,134 | 2,541 | 2,772 |
| 2,492 | 2,330 | 2,593 |
| 2,383 | 2,374 | 2,592 |

*Recovered absolute viscosities @ 140°F (poises) when original viscosity (2,522 poises) Source E asphalt was dissolved in solvent for three time exposures.

TABLE 7

OPERATOR EFFECT - 75 GRAMS, AGED ASPHALT*

Operator 1

| 5 Hours | 20 Hours | 44 Hours |
|---|---|---|
| 31,954 | 23,733 | 33,544 |
| 25,789 | 26,480 | 32,874 |
| 28,845 | 28,769 | 25,576 |

Operator 2

| 5 Hours | 20 Hours | 44 Hours |
|---|---|---|
| 30,571 | 24,601 | 30,733 |
| 25,306 | 25,882 | 27,926 |
| 28,945 | 28,957 | 23,378 |

*Recovered absolute viscosities @ 140°F (poises) when lab-aged viscosity (35,718 poises) Source E asphalt was dissolved in solvent for three time exposures.

TABLE 8

OPERATOR EFFECT - 50 GRAMS, AGED ASPHALT*

| Operator 1 | Operator 2 |
|---|---|
| 36,219 | 32,543 |
| 32,833 | 29,094 |
| 32,306 | 28,023 |
| 29,871 | 30,214 |
| 34,323 | 28,468 |
| 31,611 | 30,617 |

*Recovered absolute viscosities @ 140°F (poises) when lab-aged viscosity (35,718 poises) Source E asphalt was dissolved in solvent for five hours.

Aim IV - Effect of Solvent Quantity

The 36 recovery and subsequent viscosity test results associated with this phase are reported in Table 9. The twelve samples prepared with any one of the three solvent levels were all allowed to soak in solvent for 1-1/2 hours prior to initiating a primary distillation. As such, the following testing times apply:

| Solvent Quantity | Testing Time |
|---|---|
| 900 ml. | 1-1/2 hours soak + 2 hours primary and Abson |
| 1,800 ml. | 1-1/2 hours soak + 3 hours primary and Abson |
| 2,700 ml. | 1-1/2 hours soak + 4 hours primary and Abson |

Aim V - Effect of Mineral Fines

The 16 recovery and subsequent viscosity test results associated with this phase are reported in Table 10. Included in the table is the testing time, from the beginning of the extraction procedure to the completion of the Abson recovery. One of the results, shown and noted in the table, was eliminated for analysis purposes as its recovered viscosity was unexplainedly in obvious error.

TABLE 9

EFFECT OF SOLVENT QUANTITY*

| Asphalt Source | Quantity of Extraction Solvent 900 ml. | 1800 ml. | 2700 ml. |
|---|---|---|---|
| E (35,718 poises) | 34,545 | 33,027 | 36,380 |
| | 35,261 | 36,358 | 40,394 |
| S (86,848 poises) | 111,369 | 109,846 | 111,030 |
| | 104,297 | 115,667 | 109,306 |
| C (44,986 poises) | 40,319 | 45,435 | 41,660 |
| | 41,211 | 44,228 | 54,493 |
| M (39,043 poises) | 36,483 | 44,435 | 44,460 |
| | 37,910 | 38,723 | 38,781 |
| L (59,666 poises) | 85,258 | 92,068 | 73,483 |
| | 72,616 | 84,081 | 71,029 |
| T (173,998 poises) | 259,071 | 149,784 | 134,142 |
| | 253,296 | 243,196 | 174,127 |

*Recovered absolute viscosities @ 140°F (poises) when 75 grams of asphalt were recovered.

TABLE 10

EFFECT OF MINERAL FINES

| Filler Type | Quantity (by Wt. of Mix) | Extraction Procedure | Test Time* | Recovered Viscosity @ 140°F (poises) |
|---|---|---|---|---|
| Fly Ash | 2% | Centrifuge | 4 hours | 4,246 |
| Fly Ash | 2% | Centrifuge | 4 hours | 4,410 |
| Fly Ash | 12% | Centrifuge | 6 hours | 4,338 |
| Fly Ash | 12% | Centrifuge | 6 hours | 4,667 |
| Fly Ash | 2% | Reflux | 7 hours | 3,540 |
| Fly Ash | 2% | Reflux | 7 hours | 3,814 |
| Fly Ash | 12% | Reflux | 73 hours | 6,314 |
| Fly Ash | 12% | Reflux | 26 hours | 5,186 |
| Cement Dust | 2% | Centrifuge | 4 hours | 4,198 |
| Cement Dust | 2% | Centrifuge | 4 hours | 4,471 |
| Cement Dust | 12% | Centrifuge | 6 hours | 4,518 |
| Cement Dust | 12% | Centrifuge | 6 hours | 4,825 |
| Cement Dust | 2% | Reflux | 7 hours | 3,546 |
| Cement Dust | 2% | Reflux | 7 hours | 3,596 |
| Cement Dust | 12% | Reflux | 31 hours | 5,324 |
| ~~Cement Dust~~ | ~~12%~~ | ~~Reflux~~ | ~~30 hours~~ | ~~1,964~~** |

*Time from beginning of extraction to completion of Abson recovery. Reflux times are divided as follows:

7 hours -- 6 hours under heat of reflux + 1 hour Abson

26 hours -- 9 hours under heat of reflux + 16 hours soak + 1 hour Abson

31 hours -- 14 hours under heat of reflux + 16 hours soak + 1 hour Abson

73 hours -- 8 hours under heat of reflux + 64 hours soak + 1 hour Abson

**Result considered an outlier; not included in analysis.

# DISCUSSION OF RESULTS

For each of the five aims of this study, the results reported in Tables 2 through 10 were compared by statistical analysis of variance procedures (ANOVA). This analysis rests on a separation of the variance of all the observations into parts, each part measuring variability attributable to some specific source, e.g., to internal variation of the several populations examined, to variations from one population to another, or to variations between interactions of the populations. The basic study objective concerned testing the "null" hypothesis that all the populations identified in any specific Aim of the study had equal means. The ANOVA procedure grouped data, by rows and columns, into the populations to be examined. The hypothesis of equal means (no difference between row and/or column means) is accepted if the observed data means are close; if they are significantly dispersed, we reject the hypothesis. The test of significance in the ANOVA analysis uses the F distribution, ratioing the sum of squares and degrees of freedom for the various population means to the sum of squares and degrees of freedom within the specific populations. In essence, the F statistic is comparing the variability noted between the mean of any population and the overall mean to the variability noted between the data within any mean.

## Aim I - Effect of Time in Solvent

As previously mentioned, two tables contain the testing results for this aim of the study: Table 2 for the original viscosity asphalts and Table 3 for the lab-aged viscosity asphalts.

Table 11 is the computer printout of the analysis of the original viscosity asphalt data in Table 2. The computer "model" assigned the data, by rows and columns, to the populations of asphalt source and time in solvent. As was expected, the analysis shows the asphalt source to be highly significant (probability of a greater

# TABLE 11

## ANALYSIS OF TIME IN SOLVENT (ORIGINAL ASPHALTS)

DEPENDENT VARIABLE: VISC

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE | F VALUE | PR > F | R-SQUARE | C.V. |
|---|---|---|---|---|---|---|---|
| MODEL | 17 | 23.09516548 | 1.35853915 | 28.29 | 0.0001 | 0.930354 | 7.3078 |
| ERROR | 36 | 1.72889867 | 0.04802496 | | STD DEV* | | VISC MEAN* |
| CORRECTED TOTAL | 53 | 24.82406415 | | | 0.21914599 | | 2.99881481 |

| SOURCE | DF | TYPE I SS | F VALUE | PR > F | DF | TYPE IV SS | F VALUE | PR > F |
|---|---|---|---|---|---|---|---|---|
| SRCE | 5 | 22.18895281 | 92.41 | 0.0001 | 5 | 22.18895281 | 92.41 | 0.0001 |
| TIME | 2 | 0.38352515 | 3.99 | 0.0272 | 2 | 0.38352515 | 3.99 | 0.0272 |
| SRCE*TIME | 10 | 0.52268752 | 1.09 | 0.3964 | 10 | 0.52268752 | 1.09 | 0.3964 |

*Units = poises x $10^{-3}$

TABLE 12

MEAN RESULTS OF TIME IN SOLVENT (ORIGINAL ASPHALTS)

MEANS

| SRCE | N | VISC* |
|---|---|---|
| C | 9 | 1.85088889 |
| E | 9 | 2.52133333 |
| L | 9 | 3.46833333 |
| M | 9 | 2.97466667 |
| S | 9 | 3.62377778 |
| T | 9 | 3.55388889 |

| TIME | N | VISC* |
|---|---|---|
| 1 | 18 | 2.95661111 |
| 2 | 18 | 2.92338889 |
| 3 | 18 | 3.11644444 |

| SRCE | TIME | N | VISC* |
|---|---|---|---|
| C | 1 | 3 | 1.81666667 |
| C | 2 | 3 | 1.84600000 |
| C | 3 | 3 | 1.89000000 |
| E | 1 | 3 | 2.47766667 |
| E | 2 | 3 | 2.43966667 |
| E | 3 | 3 | 2.64566667 |
| L | 1 | 3 | 3.59933333 |
| L | 2 | 3 | 3.37533333 |
| L | 3 | 3 | 3.43033333 |
| M | 1 | 3 | 2.96466667 |
| M | 2 | 3 | 2.95000000 |
| M | 3 | 3 | 3.00933333 |
| S | 1 | 3 | 3.43133333 |
| S | 2 | 3 | 3.45566667 |
| S | 3 | 3 | 3.98333233 |
| T | 1 | 3 | 3.45000000 |
| T | 2 | 3 | 3.47266667 |
| T | 3 | 3 | 3.73900000 |

*Units = poises x $10^{-3}$

F = 0.0001). This is visually evident from the computer mean results as tabulated in Table 12, where the mean recovered viscosities differ widely between the six asphalt sources. What is important to note in Table 11 is that time in solvent is a significant factor, given these particular six asphalt sources (probability of a greater F = 0.0272). This level of significance ($\alpha$ = 0.0272) implies that there is only a 2.72% probability of error in rejecting the "null" hypothesis, namely, that the average Abson recovered viscosity is not dependent on the time an asphalt remains dissolved in solvent. It should be pointed out, however, that the magnitude of difference, at least for the six asphalt sources this study examined, is small as is shown in Table 12 (the mean recovered viscosities for any of the three time exposures differs by less than 200 poises).

An interesting observation can be made in the case of one particular source of asphalt, source C. Both from the raw data in Table 2 and from the mean results of Table 12, a noticeable "softening" occurs when this asphalt source is recovered. That is, consistently lower recovered viscosities were found (1851 poises, average) than were prepared (2986 poises). No explanation can be offered other than a chemical composition change occurs when this asphalt source is exposed to reagent-grade trichloroethylene.

Table 13 is the computer printout of the analysis of the lab-aged viscosity asphalt data in Table 3. Again the computer "model" assigned the data, by rows and columns, to the populations of asphalt source and time in solvent. As was to be expected, the analysis shows the asphalt source to be highly significant; easily evident from the computer mean results tabulated in Table 14. What is important to note in Table 13 is that, once again, time in solvent is a significant factor (probability of a greater F = 0.0085). In fact, time in solvent is more significant with these aged asphalts than it was with the original asphalts; the magnitude of difference in average recovered viscosities between the three levels of time exposure now spans approximately 10,000 poises.

TABLE 13

ANALYSIS OF TIME IN SOLVENT (LAB-AGED ASPHALTS)

DEPENDENT VARIABLE: VISC

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE | F VALUE | PR > F | R-SQUARE | C.V. |
|---|---|---|---|---|---|---|---|
| MODEL | 17 | 115118.02944987 | 6771.64879117 | 73.77 | 0.0001 | 0.972094 | 14.1230 |
| ERROR | 36 | 3304.67011667 | 91.79639213 | | STD DEV* | | VISC MEAN* |
| CORRECTED TOTAL | 53 | 118422.69956654 | | | 9.58104337 | | 67.83990741 |

| SOURCE | DF | TYPE I SS | F VALUE | PR > F | DF | TYPE IV SS | F VALUE | PR > F |
|---|---|---|---|---|---|---|---|---|
| SRCE | 5 | 112930.56183676 | 246.05 | 0.0001 | 5 | 112930.56183676 | 246.05 | 0.0001 |
| TIME | 2 | 1002.37174670 | 5.46 | 0.0085 | 2 | 1002.37174670 | 5.46 | 0.0085 |
| SRCE*TIME | 10 | 1185.09586641 | 1.29 | 0.2721 | 10 | 1185.09586641 | 1.29 | 0.2721 |

*Units = poises x $10^{-3}$

The same observation as was previously made with respect to a noticeable "softening" when asphalt source C is recovered can be made here also. Both from the raw data in Table 3 and from the mean results of Table 14, consistently lower recovered viscosities were found (32,364 poises) than were prepared (44,986 poises).

Aim II - Effect of Reduced Sample Size

Table 15 presents the computer analysis of the data contained in Tables 4 and 5. The computer "model" assigned the data to the populations of sample size and asphalt source. As was to be expected, the analysis shows the asphalt source to be highly significant, easily evident from the mean results tabulated in Table 16. What is important to note in Table 15 is that sample size is a significant factor, given these particular six asphalt sources (probability of a greater F = 0.0001). This is highly significant and is easily observed in the mean results tabulated in Table 16, where the recovered viscosities with the 50-gram sample size average 16,000 poises greater than with the 75-gram sample size when testing an average prepared viscosity of approximately 75,000 poises.

Aim III - Effect of Operator

Table 17 presents the computer analysis of the data contained in Table 6 where two operators each tested one source of original viscosity asphalt at three time exposures with three replicas for each exposure. The "model" assigned the data to the populations of operator and time in solvent. It can be observed that with this data, from only asphalt source E, time in solvent was somewhat significant. This is consistent with the overall finding, previously discussed under Aim I, that time is significant given all the possible asphalt sources in Louisiana. What is important to note in Table 17 is that the operator was not significant (probability of a greater F = 0.4194). It should be stated that the two operators used in the study were both familiar with the Abson procedure.

## TABLE 14

## MEAN RESULTS OF TIME IN SOLVENT (LAB-AGED ASPHALTS)

MEANS

| SRCE | N | VISC* |
|---|---|---|
| C | 9 | 32.364111 |
| E | 9 | 28.608222 |
| L | 9 | 58.855444 |
| M | 9 | 36.560889 |
| S | 9 | 93.061444 |
| T | 9 | 157.589333 |

| TIME | N | VISC* |
|---|---|---|
| 1 | 18 | 66.0498889 |
| 2 | 18 | 63.6910556 |
| 3 | 18 | 73.7787778 |

| SRCE | TIME | N | VISC* |
|---|---|---|---|
| C | 1 | 3 | 31.217000 |
| C | 2 | 3 | 32.034667 |
| C | 3 | 3 | 33.840667 |
| E | 1 | 3 | 28.862667 |
| E | 2 | 3 | 26.327333 |
| E | 3 | 3 | 30.634667 |
| L | 1 | 3 | 61.261667 |
| L | 2 | 3 | 54.200000 |
| L | 3 | 3 | 61.098667 |
| M | 1 | 3 | 34.151333 |
| M | 2 | 3 | 36.088667 |
| M | 3 | 3 | 39.442667 |
| S | 1 | 3 | 88.393000 |
| S | 2 | 3 | 89.568333 |
| S | 3 | 3 | 101.216000 |
| T | 1 | 3 | 152.406667 |
| T | 2 | 3 | 143.921333 |
| T | 3 | 3 | 176.438000 |

*Units = poises x $10^{-3}$

## TABLE 15

## ANALYSIS OF SAMPLE SIZE

DEPENDENT VARIABLE: VISC

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE | F VALUE | PR > F | R-SQUARE | C.V. |
|---|---|---|---|---|---|---|---|
| MODEL | 11 | 122209.56788675 | 11109.96071698 | 265.60 | 0.0001 | 0.991852 | 8.6819 |
| ERROR | 24 | 1003.91535200 | 41.82980633 | | STD DEV* | | VISC MEAN* |
| CORRECTED TOTAL | 35 | 123213.48323875 | | | 6.46759664 | | 74.49541667 |

| SOURCE | DF | TYPE I SS | F VALUE | PR > F | DF | TYPE IV SS | F VALUE | PR > F |
|---|---|---|---|---|---|---|---|---|
| SMPL | 1 | 2567.76982003 | 61.39 | 0.0001 | 1 | 2567.76982003 | 61.39 | 0.0001 |
| SRCE | 5 | 113554.08398792 | 542.93 | 0.0001 | 5 | 113554.08398792 | 542.93 | 0.0001 |
| SMPL*SRCE | 5 | 6087.71407881 | 29.11 | 0.0001 | 5 | 6087.71407881 | 29.11 | 0.0001 |

*Units = poises x $10^{-3}$

TABLE 16

MEAN RESULTS OF SAMPLE SIZE

MEANS

| SMPL | N | VISC * |
|---|---|---|
| 50 | 18 | 82.9409444 |
| 75 | 18 | 66.0498889 |

| SRCE | N | VISC * |
|---|---|---|
| C | 6 | 35.618667 |
| E | 6 | 29.374667 |
| L | 6 | 60.244000 |
| M | 6 | 35.273000 |
| S | 6 | 97.462333 |
| T | 6 | 188.993833 |

| SMPL | SRCE | N | VISC * |
|---|---|---|---|
| 50 | C | 3 | 40.020333 |
| 50 | E | 3 | 29.886667 |
| 50 | L | 3 | 59.226333 |
| 50 | M | 3 | 36.406667 |
| 50 | S | 3 | 106.526667 |
| 50 | T | 3 | 225.579000 |
| 75 | C | 3 | 31.217000 |
| 75 | E | 3 | 28.862667 |
| 75 | L | 3 | 61.261667 |
| 75 | M | 3 | 34.151333 |
| 75 | S | 3 | 88.398000 |
| 75 | T | 3 | 152.408667 |

*Units = poises x $10^{-3}$

# TABLE 17

## ANALYSIS OF OPERATOR (ORIGINAL ASPHALT)

DEPENDENT VARIABLE: VISC

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE | F VALUE | PR > F | R-SQUARE | C.V. |
|---|---|---|---|---|---|---|---|
| MODEL | 5 | 0.24807894 | 0.04961579 | 2.70 | 0.0737 | 0.529350 | 5.4347 |
| ERROR | 12 | 0.22056933 | 0.01838078 | | STD DEV* | | VISC MEAN* |
| CORRECTED TOTAL | 17 | 0.46864828 | | | 0.13557573 | | 2.49461111 |

| SOURCE | DF | TYPE I SS | F VALUE | PR > F | DF | TYPE IV SS | F VALUE | PR > F |
|---|---|---|---|---|---|---|---|---|
| OPER | 1 | 0.01285339 | 0.70 | 0.4194 | 1 | 0.01285339 | 0.70 | 0.4194 |
| TIME | 2 | 0.21715544 | 5.91 | 0.0164 | 2 | 0.21715544 | 5.91 | 0.0164 |
| OPER*TIME | 2 | 0.01807011 | 0.49 | 0.6235 | 2 | 0.01807011 | 0.49 | 0.6235 |

*Units = poises x $10^{-3}$

A closer look at the statistics generated in Table 17 reveals the fact that the model does not explain all the observed variances ($R^2 = 0.529$). What this says is that the within-group "error" accounts for about as much of the total variance as does the model. In essence, the within-operator variance is large enough to make any between-operator difference insignificant. It is pointed out that the within-operator standard deviation, pooled from the ANOVA data for this particular source asphalt and its average recovered viscosity of 2495 poises, is shown in Table 17 to be 136 poises. This is considered to be quite acceptable, and the fact that this small degree of within-operator variance masks out any between-operator difference implies good reproducibility between operators. The mean recovered viscosity for operator 1 and operator 2 is given in Table 18, with no significance attributable to the difference.

Table 19 presents the computer analysis of the data contained in Table 7 where two operators each tested one source of lab-aged viscosity asphalt at three time exposures with three replicas for each exposure. The "model" assigned the data to the populations of operator and time in solvent. It is important to note from the table that neither the operator nor the time exposure in solvent was significant (operator probability of greater $F = 0.4229$; time probability of greater $F = 0.3530$). This finding is related to the fact that the within-group "error" variance accounts for three times as much of the total variance as does the model ($R^2 = 0.247$). Similar to the previous finding (Table 17) with original viscosity asphalt, the within-operator variance is large enough to make any between-operator and/or time exposure difference insignificant, the mean results for these two populations being shown in Table 20. It is pointed out that the within-operator standard deviation, pooled from the ANOVA data for this particular source asphalt and its average recovered viscosity of 27,992 poises, is shown in Table 19 to be 3200 poises. This precision, as a percentage of the mean, is not as good as it was for the original viscosity asphalt; the coefficient of variation from Table 19 being 11.4%, whereas the coefficient of variation from Table 17 was 5.4%.

TABLE 18

MEAN RESULTS OF OPERATORS (ORIGINAL ASPHALT)

MEANS

| OPER | N | VISC* |
|---|---|---|
| 1 | 9 | 2.52133333 |
| 2 | 9 | 2.46788889 |

| TIME | N | VISC* |
|---|---|---|
| 1 | 6 | 2.40700000 |
| 2 | 6 | 2.42733333 |
| 3 | 6 | 2.64950000 |

| OPER | TIME | N | VISC* |
|---|---|---|---|
| 1 | 1 | 3 | 2.47766667 |
| 1 | 2 | 3 | 2.43966667 |
| 1 | 3 | 3 | 2.64666667 |
| 2 | 1 | 3 | 2.33633333 |
| 2 | 2 | 3 | 2.41500000 |
| 2 | 3 | 3 | 2.65233333 |

*Units = poises x $10^{-3}$

TABLE 19

ANALYSIS OF OPERATOR (LAB-AGED ASPHALT)

DEPENDENT VARIABLE: VISC

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE | F VALUE | PR > F | R-SQUARE | C.V. |
|---|---|---|---|---|---|---|---|
| MODEL | 5 | 40.36720961 | 8.07344192 | 0.79 | 0.5777 | 0.247249 | 11.4325 |
| ERROR | 12 | 122.89837267 | 10.24153106 | | STD DEV* | | VISC MEAN* |
| CORRECTED TOTAL | 17 | 163.26558228 | | | 3.20023922 | | 27.99238889 |

| SOURCE | DF | TYPE I SS | F VALUE | PR > F | DF | TYPE IV SS | F VALUE | PR > F |
|---|---|---|---|---|---|---|---|---|
| OPER | 1 | 7.05001250 | 0.69 | 0.4229 | 1 | 7.05001250 | 0.69 | 0.4229 |
| TIME | 2 | 23.28881478 | 1.14 | 0.3530 | 2 | 23.28881478 | 1.14 | 0.3530 |
| OPER*TIME | 2 | 10.02838233 | 0.49 | 0.6246 | 2 | 10.02838233 | 0.49 | 0.6246 |

*Units = poises x $10^{-3}$

TABLE 20

MEAN RESULTS OF OPERATORS (LAB-AGED ASPHALT)

MEANS

| OPER | N | VISC* |
|---|---|---|
| 1 | 9 | 28.6182222 |
| 2 | 9 | 27.3665556 |

| TIME | N | VISC* |
|---|---|---|
| 1 | 6 | 28.5683333 |
| 2 | 6 | 26.4036667 |
| 3 | 6 | 29.0051667 |

| OPER | TIME | N | VISC* |
|---|---|---|---|
| 1 | 1 | 3 | 28.8626667 |
| 1 | 2 | 3 | 26.3273333 |
| 1 | 3 | 3 | 30.6646667 |
| 2 | 1 | 3 | 28.2740000 |
| 2 | 2 | 3 | 26.4800000 |
| 2 | 3 | 3 | 27.3456667 |

*Units = poises x $10^{-3}$

In the discussion of results for Aim II, Effect of Sample Size, it was pointed out that a reduced sample size (50 grams versus 75 grams) produced significantly different (higher) recovered viscosities. Consequently, no discussion of the results presented in Table 8 will be given except to point out that, by t-test analysis ($t = 2.67$), no significant difference can be attributed to the operator ($t_{.01,10} = 3.17$).

## Aim IV - Effect of Solvent Quantity

Table 21 presents the computer analysis of the data contained in Table 9, excepting the results associated with asphalt source T where the within-group variance was excessively high and would tend to mask out any significance between the remaining data. This elimination of asphalt source T data is deemed valid in that the variance noted between duplicate tests within a group is believed caused partly, if not primarily, by the known lack of applicability of capillary viscometer measurements at the 200,000 poises level. For the remaining 30 test results, the computer "model" assigned the data to the populations of asphalt source and quantity of extraction solvent. Once again, as expected, the analysis shows the asphalt source to be highly significant, easily evident from the mean results tabulated in Table 22. What is important to note in Table 21 is that quantity of extraction solvent is significant at a level of $\alpha = 0.1088$. An inspection of the mean recovered viscosity data in Table 22 for the three levels of extraction quantity would lead this writer to be unwilling to reject the "null" hypothesis of equal means at this level of significance. To overcome the need for more extraction solvent for whatever initially unknown reason is not considered to be practical in light of the magnitude of difference in recovered viscosities associated with the various quantity levels.

## TABLE 21

## ANALYSIS OF SOLVENT QUANTITY

DEPENDENT VARIABLE: VISC

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE | F VALUE | PR > F | R-SQUARE | C.V. |
|---|---|---|---|---|---|---|---|
| MODEL | 14 | 24986.60816187 | 1784.75772585 | 92.62 | 0.0001 | 0.988565 | 7.0643 |
| ERROR | 15 | 289.03848950 | 19.26923263 | | STD DEV* | | VISC MEAN* |
| CORRECTED TOTAL | 29 | 25275.64665137 | | | 4.38967341 | | 62.13843333 |

| SOURCE | DF | TYPE I SS | F VALUE | PR > F | DF | TYPE IV SS | F VALUE | PR > F |
|---|---|---|---|---|---|---|---|---|
| SRCE | 4 | 24613.35202887 | 319.33 | 0.0001 | 4 | 24613.35202887 | 319.33 | 0.0001 |
| QUAN | 2 | 99.47389047 | 2.58 | 0.1088 | 2 | 99.47389047 | 2.58 | 0.1088 |
| SRCE*QUAN | 8 | 273.78224253 | 1.78 | 0.1607 | 8 | 273.78224253 | 1.78 | 0.1607 |

*Units = poises x $10^{-3}$

TABLE 22

MEAN RESULTS OF SOLVENT QUANTITY

MEANS

| SRCE | N | VISC* |
|---|---|---|
| C | 6 | 44.557667 |
| E | 6 | 35.994167 |
| L | 6 | 79.755833 |
| M | 6 | 40.132000 |
| S | 6 | 110.252500 |

| QUAN | N | VISC* |
|---|---|---|
| 1 | 10 | 59.9269000 |
| 2 | 10 | 61.3868000 |
| 3 | 10 | 62.1016000 |

| SRCE | QUAN | N | VISC* |
|---|---|---|---|
| C | 1 | 2 | 40.765000 |
| C | 2 | 2 | 44.831500 |
| C | 3 | 2 | 48.076500 |
| E | 1 | 2 | 34.903000 |
| E | 2 | 2 | 34.6[illegible]2500 |
| E | 3 | 2 | 36.387000 |
| L | 1 | 2 | 78.937000 |
| L | 2 | 2 | 88.074500 |
| L | 3 | 2 | 72.256000 |
| M | 1 | 2 | 37.196500 |
| M | 2 | 2 | 41.579000 |
| M | 3 | 2 | 41.620500 |
| S | 1 | 2 | 107.833000 |
| S | 2 | 2 | 112.756500 |
| S | 3 | 2 | 110.168000 |

*Units = poises x $10^{-3}$

## Aim V - Effect of Mineral Fines

Table 23 presents the computer analysis of the data contained in Table 10. The computer "model" assigned the data to the populations of filler type, filler quantity and extraction test method. The analysis shows quantity alone to be highly significant ($\alpha = 0.0007$) and its interaction with the test methods to also be highly significant ($\alpha = 0.0023$). An inspection of the mean recovered viscosities in Table 24 (Quant-Test interaction) points out that this significant difference occurs between reflux extraction results rather than between centrifuge extraction results. In essence, the higher level of fines addition did not result in a significant difference in recovered asphalt viscosity when the centrifuge extraction was used, although it increased the quantity of centrifuge extraction solvent needed (see Table 10) and consequently the recovery time. However, the higher level of fines addition did result in a significantly higher recovered viscosity when the reflux extraction was used. This fact is believed directly attributable to the increased time in solvent under heat necessary to reflux extract a high fines content mix through clogging filters. It is pointed out, both in fairness to and agreement with the AASHTO T-170 Abson procedure, that the total test time associated with every one of the reflux recovered, high fines mixes exceeded the specified eight hours.

## General Precision Discussion

Although the objective of this study was to identify any difference in the quality of recovered asphalt cement attributable to isolated variables associated with either the extraction and/or recovery process, a brief statement with respect to precision is felt warranted.

The results presented in this study and their analysis show quite clearly that the precision associated with any Abson recovery is highly dependent upon two factors, namely, the source of the asphalt and the viscosity of the asphalt. Not being able to know in every

TABLE 23

ANALYSIS OF MINERAL FINES

DEPENDENT VARIABLE: VISC

| SOURCE | DF | SUM OF SQUARES | MEAN SQUARE | F VALUE | PR > F | R-SQUARE | C.V. |
|---|---|---|---|---|---|---|---|
| MODEL | 7 | 7.04036490 | 1.00576641 | 8.51 | 0.0057 | 0.894889 | 7.6957 |
| ERROR | 7 | 0.82693750 | 0.11813393 | | STD DEV* | | VISC MEAN* |
| CORRECTED TOTAL | 14 | 7.86730240 | | | 0.34370617 | | 4.46620000 |

| SOURCE | DF | TYPE I SS | F VALUE | PR > F | DF | TYPE IV SS | F VALUE | PR > F |
|---|---|---|---|---|---|---|---|---|
| FILLER | 1 | 0.16522853 | 1.40 | 0.2756 | 1 | 0.02824272 | 0.24 | 0.6398 |
| QUANT | 1 | 3.99585745 | 33.82 | 0.0007 | 1 | 4.18086806 | 35.39 | 0.0006 |
| TEST | 1 | 0.02386519 | 0.20 | 0.6667 | 1 | 0.05238006 | 0.44 | 0.5268 |
| FILLER*QUANT | 1 | 0.02817509 | 0.24 | 0.6402 | 1 | 0.00551250 | 0.05 | 0.8351 |
| FILLER*TEST | 1 | 0.22306511 | 1.89 | 0.2118 | 1 | 0.11123472 | 0.94 | 0.3642 |
| QUANT*TEST | 1 | 2.55243880 | 21.61 | 0.0023 | 1 | 2.44131339 | 20.67 | 0.0026 |
| FILLER*QUANT*TEST | 1 | 0.05173472 | 0.44 | 0.5293 | 1 | 0.05173472 | 0.44 | 0.5293 |

*Units = poises x $10^{-3}$

case, much less control in any case, either of these two factors, a specific precision statement (repeatability) is believed impossible to develop. What is considered possible and useful to Louisiana's efforts, and to a lesser extent to other interested agencies, is a general magnitude of the standard deviation to be expected both from recoveries of new mix (possessing original viscosity asphalts) and from recoveries of old mix (possessing aged viscosity asphalts) The computer ANOVA analysis in Table 11 gives a standard deviation of 219 poises when pooled from data inclusive of all six asphalt sources in their original viscosity condition. This deviation, expressed as a percentage of the mean recovered viscosity, is also reported in the table as a coefficient of variation of 7.3%. It is of interest to note that the coefficient of variation reported in Table 17, when one particular source of original viscosity asphalt was tested, yielded a result of 5.4%. To a similar extent, had another source been examined it would not be surprising to determine a coefficient of variation somewhat greater than 7.3%, the point being that a usable estimate for the standard deviation associated with the Abson recovery of non-aged asphalts could reasonably be 10% of the recovered viscosity. Granted this estimate is based upon recovered viscosities in this study which averaged 2,999 poises and no data is herein available to fine tune the viscosity limit above which this estimate would be inappropriate. Without first attempting to define this limit, it is felt that a presentation of this study's results with aged asphalts is in order. The computer ANOVA analysis in Table 21 gives a standard deviation of 4,390 poises when pooled from data inclusive of five asphalt sources in their aged viscosity condition (one source being eliminated as previously discussed). This deviation, expressed as a percentage of the mean recovered viscosity, is also reported in the table as a coefficient of variation of 7.1%. It is of interest to note that the coefficient of variation reported in Table 19, when one particular source of aged viscosity asphalt was tested, yielded a result of 11.4%. To a similar extent, had another source been examined it would not be surprising to determine a coefficient of variation somewhat less

## TABLE 24

## MEAN RESULTS OF MINERAL FINES

MEANS

| FILLER | N | VISC* |
|---|---|---|
| 1 | 8 | 4.56437500 |
| 2 | 7 | 4.35400000 |

| QUANT | N | VISC* |
|---|---|---|
| 1 | 8 | 3.97762500 |
| 2 | 7 | 5.02457143 |

| TEST | N | VISC* |
|---|---|---|
| C | 8 | 4.45912500 |
| R | 7 | 4.47428571 |

| FILLER | QUANT | N | VISC* |
|---|---|---|---|
| 1 | 1 | 4 | 4.00250000 |
| 1 | 2 | 4 | 5.12625000 |
| 2 | 1 | 4 | 3.95275000 |
| 2 | 2 | 3 | 4.88900000 |

| FILLER | TEST | N | VISC* |
|---|---|---|---|
| 1 | C | 4 | 4.41525000 |
| 1 | R | 4 | 4.71350000 |
| 2 | C | 4 | 4.50300000 |
| 2 | R | 3 | 4.15533333 |

| QUANT | TEST | N | VISC* |
|---|---|---|---|
| 1 | C | 4 | 4.33125000 |
| 1 | R | 4 | 3.62400000 |
| 2 | C | 4 | 4.58700000 |
| 2 | R | 3 | 5.60800000 |

| FILLER | QUANT | TEST | N | VISC* |
|---|---|---|---|---|
| 1 | 1 | C | 2 | 4.32100000 |
| 1 | 1 | R | 2 | 3.67700000 |
| 1 | 2 | C | 2 | 4.50250000 |
| 1 | 2 | R | 2 | 5.73000000 |
| 2 | 1 | C | 2 | 4.33450000 |
| 2 | 1 | R | 2 | 3.57100000 |
| 2 | 2 | C | 2 | 4.67150000 |
| 2 | 2 | R | 1 | 5.32400000 |

*Units = poises x $10^{-3}$

than 7.1%, the point being that a usable estimate for the standard deviation associated with the Abson recovery of aged asphalts could reasonably be 10% of the recovered viscosity.

Thus, giving credence to the fact that the absolute value suggested above for either the standard deviation of unaged or aged recovered viscosities could be slightly inaccurate, it is believed that a generalized standard deviation when recovering an unknown source of Louisiana asphalt, whether it be aged or not, is approximately 10% of the recovered viscosity value.

## CONCLUSIONS

The following specific conclusions are deemed validly drawn from the data of this study. It is pointed out, however, that these conclusions are necessarily constrained by the scope of the data acquisition, i.e., Louisiana asphalt sources, trained operators, ranges of time exposure in extraction solvent, ranges of quantity of extraction solvent, and type and ranges of quantity of mineral fines in the extracted mixes.

1. The time an asphalt cement remains dissolved in reagent-grade trichloroethylene during the extraction and recovery process can significantly affect the viscosity of the recovered asphalt. Higher viscosities can be expected with longer dilution times.

2. The quantity of asphalt cement recovered can significantly affect the viscosity of the recovered asphalt. Higher viscosities can be expected when recovering 50 grams rather than 75 grams.

3. Within the same laboratory, and between operators familiar with the Abson recovery, there is no significant difference in recovered asphalt viscosity attributable to the operator.

4. The normal range in quantity of extraction solvent used with a centrifuge type extraction does not significantly affect the viscosity of a recovered asphalt.

5. The quantity of mineral fines in an extracted mix can significantly affect the viscosity of the recovered asphalt when a reflux type extraction is used. Higher viscosities can be expected when the quantity of fines causes excessive extraction and recovery times.

6. The precision of absolute viscosity measurements associated with residues recovered by the Abson procedure can be defined with a standard deviation equal to 10% of the recovered viscosity value.

7. A "softening" of the asphalt cement recovered by the Abson procedure can occur. This occurrence is dependent upon the source of the asphalt.

## RECOMMENDATION

The recovery of asphalt from solution by the Abson method (AASHTO T 170-73) should be strictly adhered to, except that the extraction from the aggregate-asphalt mixture should be in accordance with only Method A of AASHTO T 164-76 (inclusive of 1980 Interim Revisions). Conformance to the above will assure a minimum of 75 grams of asphalt being recovered by a cold extraction procedure within a total time period (extraction through recovery) of eight hours.