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| 16. Abstract 16. Abstract A research project, "Evaluation of Shear Strength Property of HMAC for Predicting Performance," was conducted the Texas Transportation Institute (TTI) with an objective to find the "best" Superpave Shear Test (SST) protocol. Researchers performed a comparative analysis of results from SST and other laboratory-scale rutting tests and recommended frequency sweep at constant height (FSCH) as the "best" SST protocol for predicting rutting of hot r asphalt (HMA) mixtures. In order to prepare a precision statement for the FSCH, researchers developed and administered an interlaboratory study conducted at the four regional Superpave Centers, Asphalt Institute, and Fede Highway Administration. Three different hot mix asphalt mixtures were tested using the FSCH test at two different temperatures and 10 different frequencies following standard test procedure AASHTO TP7-01 at each of the six participating laboratories. Before testing, all of the specimens were prepared at TTI. The results were analyzed us both the graphical technique suggested by W. J. Youden and ASTM E 691-99 "Standard Practice for Conducting a Interlaboratory Study." The precision values obtained for the different test parameters were poor due to the high variability of this test procedure. | | | | | | | | |
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PRECISION STATISTICS FOR FREQUENCY SWEEP AT CONSTANT HEIGHT TEST

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Report 0-1819-2 Project Number 0-1819 Project Title: Evaluation of Shear Strength Property of HMAC for Predicting Performance

> Performed in Cooperation with the Texas Department of Transportation and the Federal Highway Administration

> > November 2003

TEXAS TRANSPORTATION INSTITUTE The Texas A&M University System College Station, Texas 77843-3135

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The contents of this report reflect the views of the authors, who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official view or policies of the Federal Highway Administration (FHWA) or the Texas Department of Transportation (TxDOT). This report does not constitute a standard, specification, or regulation. The engineer in charge was Joe W. Button, P.E. (Texas, # 40874).

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Special thanks are extended to the participating laboratories for conducting tests at no cost to this project.

This project was conducted in cooperation with TxDOT and FHWA.

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CHAPTER 1: INTRODUCTION

GENERAL

The Strategic Highway Research Program (SHRP) study developed the Superpave mixture design and analysis process and provided a series of test protocols using the Superpave shear tester (SST) to predict the performance of hot mix asphalt (HMA) mixtures. Excessive time is required to conduct all of these test procedures on each HMA mixture designed, which can significantly increase the cost of mixture design. Conducting all of the tests can be confusing and even conflicting. The objective of this project was to evaluate four selected Superpave shear test protocols and determine which of the test protocols is most suitable for predicting asphalt pavement performance. The predominant pavement performance issue of interest herein is rutting. So, the ultimate goal is to identify the "best" SST test protocol that can evaluate the shearing resistance of HMA.

Researchers selected or developed four different mixtures from very poor to excellent quality using materials from Texas and Georgia. The HMA mixtures selected for this project were: Type C limestone, Type D rounded river gravel, granite stone mastic asphalt (SMA), and granite Superpave. Rutting performance of these mixtures was evaluated using four SST protocols: Frequency Sweep at Constant Height (FSCH), Simple Shear at Constant Height (SSCH), Repeated Shear at Constant Height (RSCH), and Repeated Shear at Constant Stress Ratio (RSCSR). Three laboratory-scale accelerated loaded wheel tests were performed on these four mixtures to compare the results with those from the SST. The loaded wheel tests used in this project were: Asphalt Pavement Analyzer, 1/3-Scale Model Mobile Load Simulator, and Hamburg Wheel Tracking Device (*1*).

Researchers recommended the FSCH test as the "best" SST protocol (*1*). To determine the precision of the FSCH test, compacted specimens from three HMA mixtures were sent to six different laboratories across the United States to conduct an interlaboratory test program. Acceptance criteria for the FSCH test were recommended.

One of the tasks of this research project was to develop acceptance criteria for the "best" SST test protocol. The objective of this task was to develop acceptance values for the test property(s) of the best test. A part of this task was to conduct an interlaboratory study (ILS) to

determine the precision of the engineering test/property identified most suitable for predicting pavement performance. The main idea was to recommend to TxDOT a test protocol using the SST along with acceptance criteria for HMA mixtures that are suitable for use in a guide or specification. In the American Association of State Highway and Transportation Officials (AASHTO) Provisional Standard for Materials Testing, Interim Edition, April 2001, AASHTO suggested some major changes in the SST test protocols. However, they did not provide any precision or bias requirements for any of the test values. Precision and bias are commonly obtained from an interlaboratory testing program, a major task.

The FSCH test findings are consistent with the findings from other agencies. The most important HMA property provided by the FSCH test is the complex shear modulus. The FSCH test also provides the shear phase angle, which assists in accounting for the viscoelastic properties of the HMA mixtures. Soon after deciding the FSCH was the most suitable test, the researchers initiated an interlaboratory testing program among several laboratories throughout the nation.

CHAPTER 2: INTERLABORATORY TESTING

GENERAL

Interlaboratory studies are the most commonly used procedures to evaluate the performance of a test method. In general, results from a test procedure performed on similar materials under similar conditions are not always equivalent. Observations show slight variations in the test results obtained. The variations depend upon many factors such as the inability to reproduce identical test specimens, operator, equipment used, and laboratory temperature control (2). The main idea behind an ILS is to check whether the discrepancies among test results from different laboratories are due to random variation or if there exists some systematic bias affecting the results.

Two methods are commonly used to perform an ILS, one is a graphical method developed by W. J. Youden, and the other is based on American Society for Testing and Materials (ASTM) standard E 691-99. A description of both procedures is provided below.

GRAPHICAL DIAGNOSIS OF INTERLABORATORY TEST RESULTS

W. J. Youden developed a graphical method to evaluate laboratory test procedures (*3*). According to his method, samples from two different materials are sent to various laboratories for testing. The selected materials should be similar to each other and must have a close resemblance regarding the magnitude of the property evaluated. The number of samples to be distributed depends upon the number of laboratories considered. If the number of laboratories considered is small, additional samples are distributed to each laboratory.

Based on the results obtained from the different laboratories, graphs are prepared according to Youden's procedure. The results obtained for one sample are plotted on the x-axis, and the results obtained for the second sample are plotted on the y-axis. The number of points on the graph depends upon the number of laboratories considered. Then a horizontal median line is drawn on the graph such that there are equal points on either side of the line. Similarly, a vertical median line is drawn such that there are equal points on either side. If there are some points very far apart from the median lines, they are not considered for determining the position of the median lines.

The two median lines separate the graph into four quadrants. In an ideal situation, there will be an equal number of points in all four quadrants. But in actual practice, this is rarely the case. The points either tend to concentrate more in the upper quadrants or in the lower quadrants. This phenomenon indicates that the laboratories are producing either high results or low results. In some cases, the points tend to follow a line pattern, scattering around a line that runs from the upper right to lower left quadrant at approximately 45° to the x-axis. When the points lie very close to the 45° line, this implies that each laboratory is very carefully following their own version of test procedure, which may stray from the proper procedure (*3*).

If the data are heterogeneous, the results for some specimens will be high and others will be low and the points will be equally distributed in the four quadrants. On the other hand, if the points follow a circular distribution, then the cause for such a pattern cannot be clearly determined from the graph. It may be due to sampling difficulties or due to poor accuracy of the test results. To overcome the sampling problem, researchers must prepare and assign specimens carefully.

It is common to see one or more points far away from the median line. These points are not considered when determining the position of the median line. The points appear either far from both axes or far from one and close to the other axis. If a point lies close to one axis and far from the other, this indicates that one measurement was better or more accurate than the others. When more points are close to the 45° line, this usually implies that the results from the different laboratories are more consistent (*3*).

The precision of the test method being evaluated is obtained by measuring the perpendicular distance from each point to the 45° line. The standard deviation of a single result is then estimated by multiplying the average of the distances by $\sqrt{\pi/2}$ or 0.886 (3). Based on the calculated standard deviation, a circle is drawn with the median intersection point as the center. The points that lie within the circle are free from bias. A circle with a radius of 2.5 to 3 times the standard deviation will likely contain all of the points that are free from any bias or error.

The points that lie outside the circle need investigation. The laboratories producing such results should be examined to determine whether they are following the method correctly, and, if possible, another pair of samples of different materials should be sent to these laboratories for testing to obtain additional results. With the additional results, new graphs are plotted and the

median lines are drawn for each material. Once the charts are ready, all the graphs are superimposed so that the medians coincide with one another. Keeping this as reference, all of the points are transferred to a single sheet having one pair of median lines common for all. Based on this chart, it is possible to compare the points plotted earlier and points obtained from testing the new pair of materials.

Based on the Youden method, unit plots for this experiment demonstrated the ILS test results obtained from the six laboratories on three different mixes at two different test temperatures and 10 different frequencies. In those plots, some laboratories were consistent while the other laboratories had either high values or low values. Figures A1 and A2 show typical Youden plots.

ASTM STANDARD E 691-99

This section describes ASTM E 691-99, "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method," which can be used to determine the precision of a test method. The standard specifies a procedure to develop a precision statement based on within-laboratory repeatability and between-laboratory reproducibility. The procedure follows three main steps as described below (2):

- planning the interlaboratory study,
- guiding the test phase of the study, and
- analyzing the test results.

Planning the Interlaboratory Study

Important decisions regarding the design of the ILS are made at this stage: the number of laboratories to include in the study, the type of materials to test, and decisions regarding the test results, for example. According to the ASTM standard, an ILS should include not more than 30 and not less than 6 different laboratories.

The reason behind considering a maximum number of laboratories is to reduce the detrimental effects of the laboratories that produce bad results. Only laboratories having proper facilities, good testing equipment, and trained technicians should participate in the study. Proper training should be given to the laboratory technicians regarding the test method to be analyzed.

The decision regarding the number of laboratory results must be based on the accuracy in estimating the measure of repeatability. The number of results should be kept to a minimum because of time and cost considerations.

Guiding the Test Phase of the Study

This phase of the ILS involves preparing the test specimens, distributing them to the various laboratories, keeping track of the testing progress, and checking the data collected from all the laboratories. Evaluators should prepare 50 percent more material than needed. The specimens should be randomly selected and sent to each laboratory. At regular intervals, the progress should be checked and the final data sheets from all the laboratories should be collected and reviewed for any irregularities.

Analyzing the Test Results

The main tasks of this phase are to:

- determine whether the collected data are consistent enough to form the basis for a test method precision statement,
- investigate and act on any data considered inconsistent, and
- obtain the precision statistics on which the precision statement can be based.

Consistency verification of the test results is important because the presence of outliers may lead to invalidation of the analysis. A simple one-way analysis of variance can check data consistency. For ease of analyzing the data, the results are represented in the form of a table where each row contains data from one laboratory for all frequencies and the column contains the data obtained from all laboratories for one frequency.

The data are then divided into cell statistics, intermediate statistics, precision statistics, and consistency statistics, as described in the following paragraphs.

Cell Statistics

The first step involves calculation of the cell average for each frequency. The equation used is:

$$\overline{x} = \sum_{1}^{n} \frac{x}{n}$$

Where,

x = individual test result

n = number of test results per cell

Cell standard deviation is calculated by using the equation given below:

$$s = \sqrt{\sum_{1}^{n} \left(x - \overline{x}\right)^2 / (n - 1)}$$

Where,

s = cell standard deviation

Intermediate Statistics

Average of cell averages is calculated using the equation below:

$$\overline{\mathcal{X}} = \sum_{1}^{p} \overline{x} / p$$

Where,

 \overline{x} = cell average

p = number of laboratories

• Cell deviation, *d*, is calculated by subtracting the average of cell averages from cell average:

$$d = \overline{x} - \overline{X}$$

• Standard deviation of cell averages is calculated as follows:

$$S_{\bar{x}} = \sqrt{\sum_{1}^{p} d^2 / (p-1)}$$

Precision Statistics

The fundamental precision statistics are the repeatability standard deviation and the reproducibility standard deviation.

• Repeatability standard deviation:

$$S_r = \sqrt{\sum_{1}^{p} s^2 / p}$$

• Reproducibility standard deviation (S_R) : In this case, the larger of the values obtained from the equation below or the value of S_r is considered as reproducibility standard deviation:

Larger of
$$S_r$$
 and ${S_R}^* = \sqrt{(S_{\bar{x}})^2 + (S_r)^2 (n-1)/n}$

Consistency Statistics

• For each cell the value of *h* is calculated using the formula given below:

$$h = d / \left(S_{\overline{x}} \right)$$

Where,

h = between-laboratory consistency statistic

• For each cell the value of k is calculated using the formula given below:

 $k = s / S_r$

Where,

k = within-laboratory consistency statistic

To facilitate easy representation, bar charts are prepared from the calculated values of h

and *k*. The bar charts are prepared in two ways (2):

- frequencies grouped by laboratory and
- laboratories grouped by frequencies.

The critical values for 0.5 percent significance level for both h and k are recommended based on experience. When 1 percent significance level was used, most of the cells were flagged and when 0.1 percent was used very few cells were flagged. The values that exceed the critical values are marked in the appendix tables.

In the plots by laboratory, there are usually three general patterns for the h plot (2):

- All laboratories have both positive and negative *h* values.
- The *h* values for individual laboratories are either positive or negative, and the number of negative laboratories equals the number of positive laboratories.
- One laboratory shows all positive *h* values while the other laboratories show all negative *h* values.

The first two patterns indicate that there is no variation in the test procedure and there is no need of any investigation, while the last type suggests the need for an investigation.

In the case of the k plots by laboratory, a k value from one of the laboratories either too small or too large when compared to those from the other laboratories, suggests the need for an investigation. Small k values indicate some error in measurement, and high k values indicate a large variation in data (2).

The plots by material are necessary to compare the plots by laboratory type when the values of h and k for the plot by laboratory are close to the critical values. If the values of h and k for one laboratory are considerably different from the values for other laboratories then an investigation of the offset laboratory is suggested.

Once the data are analyzed and the flawed cells are identified, a detailed investigation must be conducted to determine the reason for the variation in the results. Retesting of the materials is an option, and h and k values will be determined again. With the corrected data, the 95 percent repeatability (r) and reproducibility (R) limits can be determined using the following equations (2):

 $r = 2.8S_r$

 $R = 2.8S_{R}$

CHAPTER 3: LABORATORY TESTING AND DATA ANALYSIS

GENERAL

Other than Texas Transportation Institute (TTI), there was no institution in Texas capable of conducting SST tests. Researchers contacted most agencies in the U.S. known to operate SST, including the other four Superpave centers, the Asphalt Institute (AI), and Federal Highway Administration (FHWA). Among them, five organizations agreed to perform the FSCH test without cost to the project. These organizations are:

- FHWA,
- Asphalt Institute,
- North Central Superpave Center at Purdue University,
- Western Superpave Center at University of Nevada at Reno (UNR),
- Southeastern Superpave Center at Auburn University (NCAT), and
- TTI South Central Superpave Center.

A few other qualified organizations were contacted but they did not commit due to their time constraints. The target was to involve at least six different laboratories, as recommended by ASTM E691-99, "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method." AI and UNR used SST machines manufactured by Cox Co, Inc., and rest of the laboratories used SST machines manufactured by Interlaken, Inc. Tests on the main project at TTI were conducted using a Cox SST machine.

Due to the time limitation of the participating laboratories, researchers decided to send three specimens from each of the three mixtures. The mixtures selected for this part of the project were Type C limestone, Type D river gravel, and granite SMA. Researchers further decided to conduct the FSCH test at only two temperatures: 68°F (20°C) and 104°F (40°C). There were two reasons for eliminating the tests at 39°F (4°C). Test results show that the FSCH test at that low temperature is not sensitive enough to discriminate between shearing strengths of different HMA mixtures. Moreover, researchers from the other participating laboratories complained about the time required to stabilize the temperature at 39°F (4°C).

Three specimens from each of the three mixtures were prepared at TTI according to AASHTO TP4 and AASHTO TP7-01. Participating laboratories were instructed to conduct the FSCH test following the most recent AASHTO TP7-01 (4). The specimens were mailed to participating laboratories in October 2001. The laboratories were requested to complete the testing within a short time period, but due to different reasons, some laboratories could not conduct the test in a timely manner. Not all of the laboratories performed the test during the same time period. The interlaboratory test was completed in a 9-month time period. The time difference for testing between the laboratories may have significantly contributed to the variability of the test results.

The FSCH test method has no bias because the values determined are defined only in terms of the test method. In other words, there are no standard values to compare test results with, so it was not possible to establish bias.

DATA ANALYSIS

The test results at each frequency for each mix type and test temperature were analyzed following the ASTM E 691-99 standard procedure as described in the background section. In this analysis, the names of the participating laboratories are reassigned numerically as Lab 1 through Lab 6. The statistics for between-laboratory consistency (*h*) and within-laboratory consistency (*k*) were determined for the complex shear modulus (G*) and the shear phase angle (δ) parameters. These results are presented in the Appendix. Tables A1 to A8 are for the limestone mix; Tables A9 to A16 are for the river gravel mix; and Tables A17 to A24 are for the granite mix.

Results obtained by one of the laboratories (Lab 6) were not considered in the analysis due to the insufficient number of replicates available. They tested only two specimens instead of three. Another laboratory (Lab 5) did not report the data for tests conducted at 0.01 Hz, 0.02 Hz, and 0.05 Hz.

The critical values that appear at the bottom of each of the tables in the Appendix (Tables A1 through A24) are from ASTM E 691-99 and they correspond to a 0.5 percent significance level (2). The critical value of h depends on the number of laboratories, and the critical value of

k depends on both the number of laboratories and the number of replicates per frequency. The numbers that exceeded the critical values appear highlighted in the tables of the Appendix.

Next, the values of *h* and *k* were plotted by laboratory and by material to observe the variability of the test method. Typical plots by laboratory for G^* and δ for each test temperature appear in Figures A3 and A4.

Observing the graphs by laboratory, the *h* plots follow a pattern of both positive and negative values among the materials. In some cases, the individual laboratories tend to be either positive or negative for all materials, and in the majority of the cases, the number of negative laboratories balances with the number of laboratories having positive values. According to ASTM E 691-99, these patterns do not demand further investigation and the values of *h* can be used to determine the test method variability (2).

The k plots from Lab 5 data show the smallest k values, meaning that Lab 5 might have a protocol or test measurement problem. On the other hand, in some plots, Lab 2 shows very high k values, probably representing within-laboratory test measurement imprecision.

The plots by material are useful to observe which laboratories have h or k values near the critical thresholds. The h and k values for Lab 2, Lab 5, and, in some cases, Lab 4 are near or even exceeding the critical limit of h and k. This is consistent with the pattern observed in the k plots by laboratory, where Lab 2 had very high values and Lab 5 had very low values.

The 95 percent repeatability (*r*) and reproducibility (*R*) limits were computed for G* and δ according to the ASTM E 691-99. All results are presented in the Appendix. Tables A25 through A30 show the results for G* of three mixtures at two different temperatures. The repeatability and reproducibility values for δ are displayed in Tables A31 through A36. The format of the tables follows the requirement of Section A21 of the Form and Style of ASTM Standards. Repeatability and reproducibility limit terms are used as specified in ASTM E 177-90a (*5*).

Tables 1 and 2 demonstrate the precision statistics of shear modulus and shear phase angle, respectively, measured at 10 Hz and 40°C. With respect to rutting, the FSCH test conducted at a higher temperature yielded more discriminatory results, which identify good and bad mixtures, and 10 Hz frequency corresponds to highway traffic. The Appendix contains the precision statistics determined at two temperatures and 10 frequencies for three different mixtures.

| Material | Average of Cell Average | Repeatability Standard Deviation, S _r | Reproducibility Standard Deviation, S _R | 95% Repeatability Limit, r | 95% Reproducibility Limit, R |
|--------------|----------------------------|--|--|----------------------------------|------------------------------------|
| Limestone | 138,374 | 16,728 | 42,235 | 46,838 | 118,259 |
| River Gravel | 37,477 | 3,817 | 7,806 | 10,688 | 21,857 |
| Granite | 68,228 | 3,480 | 16,362 | 9,743 | 45,813 |

Table 1. Precision Statement for Shear Modulus (G* in psi) Measured at 10 Hz and 40 °C.

Table 2. Precision Statement for Phase Angle (* in degree) Measured at 10 Hz and 40 °C.

| Material | Average of Cell AverageRepeatability Standard Deviation, Sr | | Reproducibility Standard Deviation, S _R | 95% Repeatability Limit, r | 95% Reproducibility Limit, R |
|--------------|--|------|--|----------------------------------|------------------------------------|
| Limestone | 35.02 | 2.67 | 4.25 | 7.48 | 11.91 |
| River Gravel | 46.57 | 0.91 | 3.40 | 2.54 | 9.51 |
| Granite | 47.56 | 1.13 | 3.31 | 3.17 | 9.27 |

CHAPTER 4: SUMMARY AND RECOMMENDATIONS

A preliminary analysis of an ILS using the Youden method created unit plots using the replicate test results of six different laboratories. Several unit plots showed a very strong linear relationship among the replicate values. Following Youden's method, a 95 percent standard deviation circle was computed for those plots, and, in some cases, the majority of the points were inside the circle, while in other cases, all of the points were outside. Some laboratories had very high values in some plots and very low values in others. Because of this observed behavior, researchers decided to use the ASTM E 691-99 standard to analyze the data and obtain the precision statement for the test procedure.

ASTM E 691-99 was used to obtain the between-laboratory (h) and within-laboratory (k) consistency statistics. Some of the results exceeded the critical values at the 0.5 percent significance level. Since there was no indication of typographical errors or reported deviations from the standard procedure, all test results were used to compute the repeatability and reproducibility limits. The only exception was Lab 6, whose results were not considered in the statistical analysis due to the insufficient number of replicates reported.

The precision statement for the FSCH test at several test conditions is described in the Appendix. The repeatability and reproducibility limits presented in the Appendix represent the absolute maximum value difference expected in about 95 percent of all pairs of test results from laboratories similar to those in the study. Because of the reduced number of laboratories involved in this ILS, the precision statistics presented in the Appendix should not be considered as exact mathematical quantities applicable to all cases and circumstances. The data serve as general guidelines only. There will certainly be times when the results from the same or different laboratories will have greater differences than the ones predicted by this ILS. If more precise statistics are required, additional studies involving a greater number of laboratories and designed according to the specific mix type and testing conditions should be performed.

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APPENDIX: INTERLABORATORY TESTING



Figure A1. Replicate 1 versus Replicate 2 for G* of Granite SMA at 20 °C and 10 Hz.



Figure A2. Replicate 1 versus Replicate 2 for G* of Granite SMA at 40 °C and 10 Hz.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | -1.09 | -1.15 | -1.25 | -0.55 | -0.57 | -0.55 | -0.50 | -0.52 | -0.47 | -0.31 |
| Lab 2 | 0.45 | 0.37 | 0.45 | 0.76 | 1.06 | 1.09 | 1.76 | 1.65 | 1.76 | 1.05 |
| Lab 3 | -0.52 | -0.40 | -0.27 | 0.23 | 0.18 | 0.29 | -0.30 | -0.08 | -0.28 | 0.45 |
| Lab 4 | 1.16 | 1.17 | 1.07 | 1.00 | 0.74 | 0.60 | -0.25 | -0.06 | -0.30 | 0.38 |
| Lab 5 | 0.00 | 0.00 | 0.00 | -1.44 | -1.41 | -1.44 | -0.71 | -0.99 | -0.71 | -1.57 |

 Table A1. Between-Laboratory Consistency Statistic (h) for G* of Limestone at 20 °C.

 Table A2. Between-Laboratory Consistency Statistic (h) for G* of Limestone at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | -0.06 | -0.26 | -0.46 | -0.19 | -0.31 | -0.42 | -0.46 | -0.52 | -0.44 | -0.42 |
| Lab 2 | -0.64 | -0.50 | -0.41 | 0.01 | 0.15 | 0.41 | 0.48 | 0.60 | 0.65 | 0.73 |
| Lab 3 | -0.73 | -0.71 | -0.63 | -0.23 | -0.20 | -0.14 | -0.17 | -0.20 | -0.09 | -0.03 |
| Lab 4 | 1.43 | 1.47 | 1.49 | 1.59 | 1.56 | 1.42 | 1.40 | 1.35 | 1.24 | 1.13 |
| Lab 5 | 0.00 | 0.00 | 0.00 | -1.18 | -1.19 | -1.27 | -1.25 | -1.23 | -1.36 | -1.41 |

Critical value at the 0.5% significance level = 1.74

Table A3. Within-Laboratory Consistency Statistic (k) for G* of Limestone at 20 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.42 | 0.32 | 0.26 | 0.25 | 0.14 | 0.25 | 0.04 | 0.12 | 0.07 | 0.71 |
| Lab 2 | 1.11 | 1.31 | 1.17 | 1.35 | 1.95 | 1.73 | 2.23 | 2.20 | 2.23 | 1.74 |
| Lab 3 | 1.54 | 1.40 | 1.52 | 1.68 | 1.04 | 1.33 | 0.15 | 0.37 | 0.17 | 1.19 |
| Lab 4 | 0.47 | 0.49 | 0.50 | 0.49 | 0.30 | 0.39 | 0.04 | 0.08 | 0.03 | 0.21 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.21 | 0.11 | 0.11 | 0.01 | 0.02 | 0.01 | 0.07 |

Critical value at the 0.5\% significance level = 1.92

Table A4. Within-Laboratory Consistency Statistic (*k*) for G* of Limestone at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.74 | 0.81 | 0.85 | 1.07 | 1.16 | 1.10 | 0.79 | 0.35 | 0.65 | 0.32 |
| Lab 2 | 0.47 | 0.26 | 0.15 | 0.34 | 0.41 | 1.26 | 1.61 | 2.06 | 0.40 | 1.61 |
| Lab 3 | 0.51 | 0.70 | 0.79 | 1.06 | 1.39 | 1.45 | 1.24 | 0.74 | 2.02 | 1.50 |
| Lab 4 | 1.73 | 1.67 | 1.62 | 1.61 | 1.24 | 0.28 | 0.50 | 0.31 | 0.56 | 0.25 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.09 | 0.08 | 0.06 | 0.04 | 0.03 | 0.10 | 0.08 |

Critical value at the 0.5% *significance level* = 1.92

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.82 | 0.81 | 0.82 | 0.18 | -0.07 | 0.73 | 0.69 | 0.01 | -0.02 | 0.90 |
| Lab 2 | -0.27 | 0.03 | 0.14 | 0.42 | 1.41 | -1.47 | 0.48 | 1.66 | 1.72 | 1.27 |
| Lab 3 | 0.75 | 0.57 | 0.47 | -0.24 | -0.48 | 0.12 | -0.76 | -0.79 | -0.73 | -0.82 |
| Lab 4 | -1.30 | -1.42 | -1.44 | -1.54 | -1.27 | -0.44 | -1.35 | -0.78 | -0.62 | -0.67 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 1.17 | 0.42 | 1.05 | 0.94 | -0.11 | -0.35 | -0.68 |

Table A5. Between-Laboratory Consistency Statistic (*h*) for δ of Limestone at 20 °C.

Table A6. Between-Laboratory Consistency Statistic (*h*) for δ of Limestone at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | -0.53 | -0.65 | -0.53 | -0.78 | -0.67 | -0.14 | -0.06 | 0.65 | 0.69 | 0.99 |
| Lab 2 | -1.10 | -0.18 | -0.98 | -0.90 | -0.83 | -0.87 | -1.02 | -1.69 | 0.07 | -1.11 |
| Lab 3 | 0.52 | -0.63 | 0.20 | -0.01 | -0.23 | -0.16 | 0.25 | 0.43 | 0.28 | 0.65 |
| Lab 4 | 1.11 | 1.46 | 1.31 | 0.08 | 0.06 | -0.53 | -0.71 | -0.12 | -1.72 | -1.05 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 1.61 | 1.67 | 1.71 | 1.54 | 0.73 | 0.68 | 0.51 |

Critical value at the 0.5% significance level = 1.74

Table A7. Within-Laboratory Consistency Statistic (k) for δ of Limestone at 20 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.38 | 0.63 | 0.98 | 0.85 | 0.36 | 0.82 | 0.34 | 0.24 | 0.24 | 0.37 |
| Lab 2 | 1.23 | 0.48 | 0.13 | 1.66 | 2.17 | 1.93 | 2.18 | 2.20 | 2.18 | 2.15 |
| Lab 3 | 1.31 | 1.78 | 1.67 | 1.10 | 0.30 | 0.32 | 0.13 | 0.09 | 0.11 | 0.27 |
| Lab 4 | 0.78 | 0.43 | 0.49 | 0.40 | 0.19 | 0.66 | 0.33 | 0.33 | 0.39 | 0.36 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.41 | 0.13 | 0.25 | 0.09 | 0.07 | 0.09 | 0.18 |

Critical value at the 0.5% significance level = 1.92

| Table A8. Within-Laborator | y Consistency Sta | tistic (k) for δ of Lin | nestone at 40 °C. |
|----------------------------|-------------------|-------------------------|-------------------|
|----------------------------|-------------------|-------------------------|-------------------|

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.58 | 0.05 | 0.51 | 0.68 | 1.39 | 0.70 | 0.41 | 0.47 | 0.81 | 0.89 |
| Lab 2 | 1.62 | 1.75 | 1.52 | 1.64 | 0.48 | 1.97 | 2.12 | 2.11 | 1.80 | 1.49 |
| Lab 3 | 0.37 | 0.91 | 0.43 | 1.00 | 1.08 | 0.67 | 0.51 | 0.49 | 1.00 | 1.36 |
| Lab 4 | 0.95 | 0.30 | 1.12 | 0.74 | 1.26 | 0.41 | 0.25 | 0.32 | 0.33 | 0.35 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.55 | 0.27 | 0.09 | 0.02 | 0.01 | 0.05 | 0.12 |

Critical value at the 0.5% significance level = 1.92

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | -0.20 | -0.43 | -0.40 | 0.18 | 0.29 | 0.55 | 0.47 | 0.43 | 0.57 | 0.69 |
| Lab 2 | -1.24 | -1.09 | -1.15 | -0.02 | 0.10 | -0.03 | 0.58 | 0.86 | 0.84 | 0.81 |
| Lab 3 | 1.16 | 1.25 | 1.18 | 0.94 | 0.81 | 0.62 | 0.21 | 0.15 | -0.06 | -0.11 |
| Lab 4 | 0.27 | 0.27 | 0.37 | 0.56 | 0.53 | 0.59 | 0.52 | 0.27 | 0.34 | 0.29 |
| Lab 5 | 0.00 | 0.00 | 0.00 | -1.67 | -1.72 | -1.72 | -1.77 | -1.72 | -1.69 | -1.67 |

Table A9. Between-Laboratory Consistency Statistic (*h*) for G* of River Gravel at 20 °C.

 Table A10.
 Between-Laboratory Consistency Statistic (h) for G* of River Gravel at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 1.37 | 1.20 | 1.22 | 1.43 | 1.44 | 1.29 | 1.22 | 1.20 | 1.19 | 1.13 |
| Lab 2 | -1.00 | -1.17 | -1.16 | -1.18 | -1.10 | -0.79 | -0.43 | -0.15 | -0.25 | 0.16 |
| Lab 3 | -0.35 | -0.34 | -0.33 | 0.17 | 0.34 | 0.80 | 0.89 | 0.84 | 0.87 | 0.77 |
| Lab 4 | -0.01 | 0.31 | 0.27 | 0.26 | 0.09 | -0.32 | -0.60 | -0.79 | -0.67 | -1.00 |
| Lab 5 | 0.00 | 0.00 | 0.00 | -0.69 | -0.78 | -0.98 | -1.08 | -1.10 | -1.14 | -1.05 |

Critical value at the 0.5% significance level = 1.74

| Table A11. Within-Labora | atory Consistency | y Statistic (k) for | r G* of River | Gravel at 20 °C. |
|--------------------------|-------------------|---------------------|---------------|------------------|
| | | | | |

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.72 | 0.84 | 0.89 | 1.02 | 1.02 | 1.09 | 0.77 | 0.89 | 0.82 | 0.76 |
| Lab 2 | 0.38 | 0.08 | 0.25 | 0.38 | 0.72 | 0.28 | 1.53 | 1.14 | 1.21 | 1.09 |
| Lab 3 | 1.35 | 1.36 | 1.26 | 1.28 | 1.13 | 1.04 | 0.68 | 0.83 | 0.51 | 0.42 |
| Lab 4 | 1.23 | 1.20 | 1.24 | 1.41 | 1.41 | 1.57 | 1.23 | 1.44 | 1.58 | 1.73 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.41 | 0.42 | 0.44 | 0.29 | 0.36 | 0.31 | 0.29 |

Critical value at the 0.5\% significance level = 1.92

| Table A12. Within-Laborato | y Consistency | Statistic (k) for | G* of River | Gravel at 40 °C. |
|----------------------------|---------------|-------------------|-------------|------------------|
|----------------------------|---------------|-------------------|-------------|------------------|

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.91 | 1.53 | 0.77 | 0.80 | 0.64 | 0.17 | 0.08 | 0.17 | 0.37 | 0.54 |
| Lab 2 | 0.96 | 0.72 | 0.93 | 1.21 | 0.76 | 1.16 | 1.16 | 1.29 | 1.31 | 1.49 |
| Lab 3 | 0.78 | 0.47 | 0.46 | 0.27 | 0.53 | 0.46 | 0.47 | 0.51 | 0.54 | 0.55 |
| Lab 4 | 1.28 | 0.96 | 1.53 | 1.62 | 1.87 | 1.78 | 1.73 | 1.59 | 1.50 | 1.26 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.46 | 0.50 | 0.51 | 0.65 | 0.72 | 0.78 | 0.76 |

Critical value at the 0.5% significance level = 1.92

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.95 | 1.00 | 0.98 | 1.05 | 0.98 | 0.80 | 0.14 | 0.94 | 1.03 | 1.08 |
| Lab 2 | 0.49 | 0.32 | 0.48 | 0.75 | 0.87 | 1.17 | 1.67 | 1.17 | 1.14 | 1.09 |
| Lab 3 | -1.37 | -1.37 | -1.34 | -1.51 | -1.48 | -1.30 | -0.76 | -1.07 | -0.91 | -0.68 |
| Lab 4 | -0.07 | 0.05 | -0.11 | -0.24 | -0.27 | -0.51 | -0.67 | -0.70 | -0.74 | -0.57 |
| Lab 5 | 0.00 | 0.00 | 0.00 | -0.05 | -0.11 | -0.17 | -0.38 | -0.34 | -0.52 | -0.92 |

Table A13. Between-Laboratory Consistency Statistic (*h*) for δ of River Gravel at 20 °C.

Table A14. Between-Laboratory Consistency Statistic (*h*) for δ of River Gravel at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | -1.32 | -0.81 | -1.34 | -0.96 | -0.78 | -0.54 | -0.72 | -0.27 | -0.07 | -0.31 |
| Lab 2 | -0.16 | -0.20 | 0.69 | 0.63 | 0.70 | 0.35 | 0.13 | 0.16 | -0.63 | 0.85 |
| Lab 3 | 1.00 | -0.45 | -0.18 | -0.34 | -0.78 | -0.87 | -0.66 | -0.82 | -0.46 | -0.67 |
| Lab 4 | 0.48 | 1.45 | 0.83 | 1.42 | 1.41 | 1.59 | 1.69 | 1.65 | 1.74 | 1.23 |
| Lab 5 | 0.00 | 0.00 | 0.00 | -0.75 | -0.55 | -0.53 | -0.44 | -0.72 | -0.58 | -1.10 |

Critical value at the 0.5% significance level = 1.74

| Table A15. W | ithin-Laboratory | Consistency | Statistic (k | k) for δ o | f River (| Gravel at 20 ° | ΥĊ. |
|--------------|------------------|-------------|---------------------------------------|------------|-----------|----------------|-----|
| | | | · · · · · · · · · · · · · · · · · · · | / | | | |

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.19 | 0.03 | 0.27 | 0.09 | 0.04 | 0.22 | 0.26 | 0.18 | 0.46 | 0.72 |
| Lab 2 | 1.66 | 1.40 | 1.77 | 1.91 | 2.07 | 1.96 | 2.03 | 2.09 | 1.63 | 1.88 |
| Lab 3 | 0.62 | 0.71 | 0.52 | 0.72 | 0.48 | 0.59 | 0.26 | 0.30 | 0.46 | 0.30 |
| Lab 4 | 0.90 | 1.23 | 0.71 | 0.88 | 0.69 | 0.87 | 0.86 | 0.70 | 1.38 | 0.92 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.25 | 0.04 | 0.05 | 0.05 | 0.06 | 0.12 | 0.09 |

Critical value at the 0.5% significance level = 1.92

Table A16. Within-Laboratory Consistency Statistic (*k*) for δ of River Gravel at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.69 | 0.72 | 0.26 | 0.59 | 1.08 | 0.79 | 1.32 | 0.76 | 0.79 | 1.57 |
| Lab 2 | 1.68 | 1.60 | 1.76 | 2.03 | 1.42 | 1.87 | 1.50 | 0.24 | 1.90 | 0.83 |
| Lab 3 | 0.78 | 0.15 | 0.80 | 0.44 | 0.75 | 0.29 | 0.51 | 0.26 | 0.32 | 0.64 |
| Lab 4 | 0.29 | 0.94 | 0.45 | 0.51 | 1.10 | 0.88 | 0.83 | 2.06 | 0.81 | 1.04 |
| Lab 5 | | 0.00 | 0.00 | 0.25 | 0.25 | 0.20 | 0.22 | 0.24 | 0.10 | 0.60 |

Critical value at the 0.5% significance level = 1.92

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 1.21 | 1.33 | 1.35 | 1.10 | 0.96 | 0.48 | 0.81 | 0.51 | 0.53 | 0.68 |
| Lab 2 | -1.23 | 0.13 | -0.18 | -0.43 | -0.11 | 0.91 | -0.30 | 0.54 | 0.38 | 0.13 |
| Lab 3 | -0.08 | -1.00 | -1.07 | 0.09 | -0.01 | -0.34 | -0.14 | -0.31 | -0.34 | -0.28 |
| Lab 4 | 0.09 | -0.47 | -0.10 | 0.69 | 0.75 | 0.54 | 1.07 | 0.87 | 1.00 | 1.02 |
| Lab 5 | 0.00 | 0.00 | 0.00 | -1.46 | -1.58 | -1.59 | -1.45 | -1.61 | -1.57 | -1.55 |

 Table A17. Between-Laboratory Consistency Statistic (h) for G* of Granite at 20 °C.

 Table A18. Between-Laboratory Consistency Statistic (h) for G* of Granite at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 1.13 | 1.12 | 1.05 | 1.18 | 1.12 | 1.03 | 1.11 | 1.17 | 1.29 | 1.40 |
| Lab 2 | -1.28 | -1.16 | -1.24 | -0.99 | -0.93 | -0.90 | -0.81 | -0.67 | -0.65 | -0.58 |
| Lab 3 | -0.11 | -0.43 | -0.32 | -0.16 | -0.06 | 0.08 | 0.07 | 0.10 | 0.21 | 0.14 |
| Lab 4 | 0.25 | 0.48 | 0.50 | 0.88 | 0.90 | 0.92 | 0.83 | 0.70 | 0.45 | 0.31 |
| Lab 5 | 0.00 | 0.00 | 0.00 | -0.91 | -1.03 | -1.13 | -1.19 | -1.30 | -1.29 | -1.26 |

Critical value at the 0.5% significance level = 1.74

Table A19. Within-Laboratory Consistency Statistic (k) for G* of Granite at 20 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 1.06 | 0.60 | 0.80 | 1.66 | 1.91 | 0.62 | 1.62 | 1.20 | 1.40 | 1.66 |
| Lab 2 | 1.65 | 1.90 | 1.80 | 1.17 | 0.47 | 2.12 | 1.18 | 1.73 | 1.48 | 1.08 |
| Lab 3 | 0.36 | 0.17 | 0.29 | 0.55 | 0.53 | 0.14 | 0.21 | 0.14 | 0.22 | 0.15 |
| Lab 4 | 0.09 | 0.10 | 0.18 | 0.39 | 0.43 | 0.16 | 0.45 | 0.32 | 0.45 | 0.49 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.65 | 0.82 | 0.30 | 0.85 | 0.68 | 0.76 | 0.91 |

Critical value at the 0.5\% significance level = 1.92

| Table A20. Within-Laborator | y Consistency | Statistic (k) |) for G* | of Granite at 40 ° | C. |
|-----------------------------|---------------|---------------|----------|--------------------|----|
|-----------------------------|---------------|---------------|----------|--------------------|----|

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 1.29 | 1.13 | 0.93 | 1.30 | 1.22 | 1.26 | 1.58 | 1.64 | 1.53 | 1.10 |
| Lab 2 | 0.37 | 0.47 | 1.18 | 0.44 | 0.77 | 0.65 | 0.98 | 1.09 | 1.32 | 1.30 |
| Lab 3 | 0.56 | 0.82 | 0.35 | 0.70 | 0.58 | 0.51 | 0.51 | 0.46 | 0.44 | 0.63 |
| Lab 4 | 1.38 | 1.35 | 1.27 | 1.59 | 1.55 | 1.58 | 0.91 | 0.72 | 0.50 | 0.80 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.33 | 0.43 | 0.51 | 0.66 | 0.63 | 0.70 | 1.03 |

Critical value at the 0.5% *significance level* = 1.92

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | -0.84 | 0.01 | -0.79 | -0.36 | -0.66 | -0.58 | 0.07 | -0.08 | -0.13 | 0.46 |
| Lab 2 | 1.32 | -1.32 | 0.62 | -1.28 | -0.38 | 1.70 | 0.34 | 1.69 | 1.76 | 1.52 |
| Lab 3 | -0.71 | 0.21 | -0.91 | -0.36 | -1.04 | -0.84 | -1.39 | -0.91 | -0.61 | -0.48 |
| Lab 4 | 0.24 | 1.10 | 1.08 | 0.80 | 0.72 | -0.28 | -0.37 | -0.51 | -0.53 | -0.50 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 1.21 | 1.35 | 0.00 | 1.35 | -0.20 | -0.49 | -1.00 |

Table A21. Between-Laboratory Consistency Statistic (*h*) for δ of Granite at 20 °C.

Table A22. Between-Laboratory Consistency Statistic (*h*) for δ of Granite at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|-------|-------|-------|-------|-------|-------|-------|-------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.15 | -1.32 | -0.72 | -1.42 | -0.97 | -1.30 | -1.53 | -1.18 | -0.92 | -0.97 |
| Lab 2 | -1.46 | 0.19 | -0.88 | 0.44 | -0.66 | -0.25 | 0.21 | 0.99 | 1.65 | 1.60 |
| Lab 3 | 0.72 | 0.03 | 0.33 | -0.40 | -0.40 | -0.34 | -0.37 | -0.97 | -0.63 | -0.44 |
| Lab 4 | 0.58 | 1.10 | 1.26 | 1.27 | 1.45 | 1.38 | 0.97 | 0.61 | 0.03 | 0.30 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.10 | 0.59 | 0.51 | 0.72 | 0.56 | -0.13 | -0.48 |

Critical value at the 0.5% significance level = 1.74

| Table A23. Within-Laborator | y Consistency | y Statistic (<i>k</i> | k) for δ | of Granite at 20 ° | °C. |
|-----------------------------|---------------|------------------------|----------|--------------------|-----|
| | , | / | ., | | |

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.84 | 0.27 | 0.55 | 0.28 | 0.32 | 0.36 | 0.65 | 0.31 | 0.12 | 0.33 |
| Lab 2 | 1.49 | 1.98 | 1.91 | 2.21 | 2.19 | 2.16 | 1.74 | 2.09 | 2.06 | 1.43 |
| Lab 3 | 0.65 | 0.05 | 0.07 | 0.11 | 0.17 | 0.19 | 0.35 | 0.16 | 0.18 | 0.40 |
| Lab 4 | 0.81 | 0.12 | 0.20 | 0.12 | 0.22 | 0.36 | 1.12 | 0.69 | 0.82 | 1.53 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.18 | 0.19 | 0.20 | 0.41 | 0.22 | 0.22 | 0.59 |

Critical value at the 0.5% significance level = 1.92

Table A24. Within-Laboratory Consistency Statistic (k) for δ of Granite at 40 °C.

| | Frequen | cy (Hz) | | | | | | | | |
|------------|---------|---------|------|------|------|------|------|------|------|------|
| Laboratory | 0.01 | 0.02 | 0.05 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 10 |
| Lab 1 | 0.27 | 0.20 | 0.21 | 0.57 | 1.12 | 0.82 | 0.63 | 0.85 | 0.43 | 0.92 |
| Lab 2 | 1.87 | 1.93 | 1.82 | 1.30 | 1.27 | 0.88 | 0.95 | 1.80 | 2.08 | 1.37 |
| Lab 3 | 0.14 | 0.29 | 0.54 | 0.85 | 1.00 | 1.17 | 1.39 | 0.37 | 0.09 | 0.53 |
| Lab 4 | 0.64 | 0.40 | 0.60 | 1.50 | 1.01 | 1.41 | 1.22 | 0.51 | 0.55 | 1.10 |
| Lab 5 | 0.00 | 0.00 | 0.00 | 0.11 | 0.31 | 0.44 | 0.52 | 0.79 | 0.41 | 0.90 |

Critical value at the 0.5% significance level = 1.92



Figure A3. Between-Laboratory Consistency (*h*) for G* of Type C Limestone at 20 °C.



Figure A4. Between-Laboratory Consistency (*h*) for G* of Type C Limestone at 40 °C.

| Frequency | Average of | Repeatability | Reproducibility | 95% Repeatability | 95% Reproducibility |
|-----------|---------------|----------------------|----------------------|-------------------|---------------------|
| (Hz) | cell averages | Std Deviation, S_r | Std Deviation, S_R | Limit, r | Limit, R |
| 0.01 | 119,741 | 12,082 | 27,612 | 33,830 | 77,313 |
| 0.02 | 143,857 | 17,191 | 32,016 | 48,135 | 89,645 |
| 0.05 | 180,746 | 22,353 | 37,242 | 62,588 | 104,278 |
| 0.1 | 194,280 | 24,431 | 56,150 | 68,406 | 157,219 |
| 0.2 | 231,602 | 49,003 | 75,324 | 137,210 | 210,907 |
| 0.5 | 277,016 | 48,145 | 80,608 | 134,806 | 225,702 |
| 1 | 436,487 | 507,403 | 527,687 | 1,420,727 | 1,477,525 |
| 2 | 403,011 | 243,549 | 262,946 | 681,938 | 736,248 |
| 5 | 557,996 | 648,905 | 667,200 | 1,816,934 | 1,868,159 |
| 10 | 434,260 | 85,781 | 111,370 | 240,188 | 311,836 |

Table A25. Precision Statistics of G* for Limestone at 20 °C.

* Units of repeatability and reproducibility limits are PSI

| TADIC A20, IT CUSION STATISTICS OF G TOT LINESTONE AT 40 V | Ta | ble | A26. | Precision | Statistics | of G* | for | Limestone at | 40 | °C |
|--|----|-----|------|-----------|-------------------|-------|-----|--------------|----|----|
|--|----|-----|------|-----------|-------------------|-------|-----|--------------|----|----|

| Frequency | Average of | Repeatability | Reproducibility | 95% Repeatability | 95% Reproducibility |
|-----------|--------------|----------------------|----------------------|-------------------|---------------------|
| (nz) | cen averages | Stu Deviation, S_r | Stu Devlation, S_R | Lillint, I | Lillit, K |
| 0.01 | 12,672 | 2,279 | 5,228 | 6,382 | 14,637 |
| 0.02 | 16,248 | 2,580 | 6,641 | 7,223 | 18,595 |
| 0.05 | 22,328 | 3,230 | 8,587 | 9,043 | 24,043 |
| 0.1 | 25,169 | 2,827 | 11,415 | 7,914 | 31,961 |
| 0.2 | 32,264 | 2,857 | 13,816 | 7,998 | 38,685 |
| 0.5 | 45,272 | 3,954 | 17,104 | 11,071 | 47,892 |
| 1 | 60,514 | 6,380 | 23,149 | 17,863 | 64,817 |
| 2 | 82,391 | 15,895 | 34,115 | 44,507 | 95,523 |
| 5 | 109,743 | 8,461 | 35,630 | 23,691 | 99,764 |
| 10 | 138,374 | 16,728 | 42,235 | 46,838 | 118,259 |

* Units of repeatability and reproducibility limits are PSI

| Table A27. Precision S | Statistics of G | * for River | Gravel at 20 °C |
|------------------------|-----------------|-------------|-----------------|
|------------------------|-----------------|-------------|-----------------|

| Frequency | Average of | Repeatability | Reproducibility | 95% Repeatability | 95% Reproducibility |
|-----------|---------------|-------------------|-------------------------------|-------------------|---------------------|
| (Hz) | cell averages | Std Deviation, Sr | Std Deviation, S _R | Limit, r | Limit, R |
| 0.01 | 29,332 | 3,976 | 4,858 | 11,131 | 13,602 |
| 0.02 | 37,839 | 4,233 | 4,954 | 11,853 | 13,872 |
| 0.05 | 51,793 | 5,685 | 6,109 | 15,917 | 17,106 |
| 0.1 | 61,821 | 5,957 | 10,043 | 16,679 | 28,120 |
| 0.2 | 77,022 | 7,093 | 12,098 | 19,860 | 33,875 |
| 0.5 | 101,319 | 8,752 | 15,533 | 24,505 | 43,494 |
| 1 | 126,771 | 15,464 | 22,072 | 43,299 | 61,802 |
| 2 | 156,530 | 14,591 | 27,234 | 40,856 | 76,256 |
| 5 | 195,878 | 20,353 | 35,621 | 56,988 | 99,738 |
| 10 | 233,227 | 25,657 | 44,982 | 71,839 | 125,950 |

* Units of repeatability and reproducibility limits are PSI

| | 10010112 | | | | |
|-----------|---------------|-------------------|-------------------------------|-------------------|---------------------|
| Frequency | Average of | Repeatability | Reproducibility | 95% Repeatability | 95% Reproducibility |
| (Hz) | cell averages | Std Deviation, Sr | Std Deviation ,S _R | Limit, r | Limit, R |
| 0.01 | 3,363 | 1,047 | 1,351 | 2,932 | 3,784 |
| 0.02 | 3,728 | 603 | 1,571 | 1,688 | 4,400 |
| 0.05 | 4,886 | 767 | 1,599 | 2,146 | 4,476 |
| 0.1 | 5,846 | 837 | 1,650 | 2,343 | 4,620 |
| 0.2 | 7,475 | 1,012 | 1,963 | 2,834 | 5,497 |
| 0.5 | 10,630 | 1,493 | 2,386 | 4,181 | 6,680 |
| 1 | 14,310 | 1,645 | 2,970 | 4,605 | 8,316 |
| 2 | 19,309 | 2,047 | 4,002 | 5,733 | 11,206 |
| 5 | 28,296 | 2,781 | 5,821 | 7,788 | 16,297 |
| 10 | 37,477 | 3,817 | 7,806 | 10,688 | 21,857 |

Table A28. Precision Statistics of G* for River Gravel at 40 °C.

* Units of repeatability and reproducibility limits are PSI

| Frequency (Hz) | Average of cell averages | Repeatability Std Deviation, S _r | Reproducibility Std Deviation , S_R | 95% Repeatability Limit, r | 95% Reproducibility Limit, R |
|-------------------|--------------------------|--|---------------------------------------|-------------------------------|---------------------------------|
| 0.01 | 49,528 | 9,644 | 15,207 | 27,004 | 42,581 |
| 0.02 | 69,598 | 18,951 | 18,951 | 53,063 | 53,063 |
| 0.05 | 93,610 | 17,033 | 17,033 | 47,691 | 47,691 |
| 0.1 | 102,622 | 9,502 | 25,534 | 26,605 | 71,495 |
| 0.2 | 130,223 | 10,389 | 29,650 | 29,090 | 83,021 |
| 0.5 | 180,706 | 41,422 | 53,915 | 115,982 | 150,962 |
| 1 | 203,320 | 19,595 | 47,565 | 54,867 | 133,182 |
| 2 | 251,269 | 30,893 | 59,705 | 86,500 | 167,173 |
| 5 | 302,486 | 35,904 | 72,203 | 100,530 | 202,167 |
| 10 | 335.254 | 35.831 | 76,774 | 100.328 | 214.967 |

Table A29. Precision Statistics of G* for Granite at 20 °C.

* Units of repeatability and reproducibility limits are PSI

| | Tuble 160, 11 celsion Statistics of G 101 Granite at 10 C. | | | | | | | |
|-----------|--|-------------------------------|----------------------|-------------------|---------------------|--|--|--|
| Frequency | Average of | Repeatability | Reproducibility | 95% Repeatability | 95% Reproducibility | | | |
| (Hz) | cell averages | Std Deviation, S _r | Std Deviation, S_R | Limit, r | Limit, R | | | |
| 0.01 | 5,787 | 878 | 2,488 | 2,459 | 6,966 | | | |
| 0.02 | 6,413 | 896 | 2,968 | 2,507 | 8,310 | | | |
| 0.05 | 8,094 | 1,251 | 3,404 | 3,504 | 9,531 | | | |
| 0.1 | 9,578 | 1,016 | 3,728 | 2,845 | 10,440 | | | |
| 0.2 | 12,256 | 1,011 | 4,294 | 2,830 | 12,023 | | | |
| 0.5 | 17,414 | 1,279 | 5,380 | 3,581 | 15,064 | | | |
| 1 | 23,916 | 1,347 | 6,861 | 3,771 | 19,210 | | | |
| 2 | 33,196 | 2,131 | 8,742 | 5,967 | 24,478 | | | |
| 5 | 49,984 | 3,361 | 12,589 | 9,412 | 35,248 | | | |
| 10 | 68,228 | 3,480 | 16,362 | 9,743 | 45,813 | | | |

Table A30. Precision Statistics of G* for Granite at 40 °C.

* Units of repeatability and reproducibility limits are PSI

| Frequency (Hz) | Average of cell averages | Repeatability Std Deviation, S _r | Reproducibility Std Deviation, S_R | 95% Repeatability Limit, r | 95% Reproducibility Limit, R |
|-------------------|--------------------------|--|--------------------------------------|-------------------------------|---------------------------------|
| 0.01 | 29.04 | 2.41 | 3.58 | 6.74 | 10.04 |
| 0.02 | 27.14 | 1.61 | 2.81 | 4.50 | 7.87 |
| 0.05 | 24.41 | 1.56 | 2.79 | 4.38 | 7.80 |
| 0.1 | 23.71 | 2.17 | 3.35 | 6.07 | 9.39 |
| 0.2 | 23.09 | 5.98 | 6.41 | 16.74 | 17.94 |
| 0.5 | 17.73 | 2.85 | 4.82 | 7.97 | 13.50 |
| 1 | 17.97 | 6.71 | 6.71 | 18.80 | 18.80 |
| 2 | 18.23 | 9.03 | 9.03 | 25.27 | 25.27 |
| 5 | 17.92 | 6.92 | 8.97 | 19.36 | 25.11 |
| 10 | 15.78 | 3.14 | 3.69 | 8.80 | 10.33 |

Table A31. Precision Statistics of δ for Limestone at 20 °C.

* Units of repeatability and reproducibility limits are degrees

| Frequency (Hz) | Average of cell averages | Repeatability Std Deviation, S _r | Reproducibility Std Deviation, S _R | 95% Repeatability Limit, r | 95% Reproducibility Limit, R |
|-------------------|--------------------------|--|--|-------------------------------|---------------------------------|
| 0.01 | 41.57 | 1.86 | 3.68 | 5.20 | 10.31 |
| 0.02 | 40.91 | 4.76 | 4.76 | 13.32 | 13.32 |
| 0.05 | 40.11 | 1.69 | 2.66 | 4.73 | 7.44 |
| 0.1 | 41.86 | 1.89 | 3.34 | 5.30 | 9.36 |
| 0.2 | 41.63 | 1.52 | 3.34 | 4.24 | 9.34 |
| 0.5 | 41.51 | 3.53 | 3.84 | 9.88 | 10.75 |
| 1 | 40.30 | 5.47 | 5.47 | 15.33 | 15.33 |
| 2 | 35.10 | 6.15 | 11.48 | 17.21 | 32.15 |
| 5 | 37.18 | 3.45 | 4.35 | 9.67 | 12.19 |
| 10 | 35.02 | 2.67 | 4 25 | 7 48 | 11 91 |

Table A32. Precision Statistics of δ for Limestone at 40 °C.

* Units of repeatability and reproducibility limits are degrees

| | | | | | ÷. |
|-------------------|--------------------------|--|--|-------------------------------|---------------------------------|
| Frequency (Hz) | Average of cell averages | Repeatability Std Deviation, S _r | Reproducibility Std Deviation, S_{R} | 95% Repeatability Limit, r | 95% Reproducibility Limit, R |
| 0.01 | 38.99 | 2.31 | 3.43 | 6.47 | 9.61 |
| 0.02 | 37.08 | 1.29 | 3.24 | 3.61 | 9.08 |
| 0.05 | 34.99 | 1.56 | 3.16 | 4.37 | 8.86 |
| 0.1 | 33.68 | 1.66 | 2.96 | 4.64 | 8.28 |
| 0.2 | 32.21 | 2.73 | 3.40 | 7.64 | 9.53 |
| 0.5 | 30.57 | 2.26 | 3.62 | 6.33 | 10.13 |
| 1 | 30.79 | 3.05 | 7.28 | 8.55 | 20.40 |
| 2 | 26.98 | 3.64 | 4.57 | 10.21 | 12.80 |
| 5 | 25.24 | 3.00 | 4.65 | 8.39 | 13.02 |
| 10 | 24.95 | 3.27 | 4.78 | 9.16 | 13.38 |

Table A33. Precision Statistics of δ for River Gravel at 20 °C.

* Units of repeatability and reproducibility limits are degrees

| Frequency (Hz) | Average of cell averages | Repeatability Std Deviation, S _r | Reproducibility Std Deviation, S _R | 95% Repeatability Limit, r | 95% Reproducibility Limit, R |
|-------------------|--------------------------|--|--|-------------------------------|---------------------------------|
| 0.01 | 46.22 | 9.38 | 9.38 | 26.25 | 26.25 |
| 0.02 | 44.84 | 7.07 | 7.80 | 19.80 | 21.83 |
| 0.05 | 48.89 | 5.74 | 7.81 | 16.08 | 21.86 |
| 0.1 | 46.60 | 2.72 | 4.73 | 7.60 | 13.23 |
| 0.2 | 47.12 | 1.70 | 4.55 | 4.75 | 12.74 |
| 0.5 | 47.70 | 2.91 | 3.90 | 8.14 | 10.92 |
| 1 | 47.56 | 2.56 | 3.91 | 7.16 | 10.96 |
| 2 | 47.99 | 1.65 | 3.87 | 4.63 | 10.85 |
| 5 | 45.41 | 2.64 | 3.82 | 7.38 | 10.68 |
| 10 | 46.57 | 0.91 | 3.40 | 2.54 | 9.51 |

Table A34. Precision Statistics of δ for River Gravel at 40 °C.

* Units of repeatability and reproducibility limits are degrees

| Frequency (Hz) | Average of cell averages | Repeatability Std Deviation S | Reproducibility Std Deviation Sp | 95% Repeatability | 95% Reproducibility |
|-------------------|--------------------------|----------------------------------|-------------------------------------|-------------------|---------------------|
| 0.01 | 38.49 | 2.77 | 4.61 | 7.75 | 12.90 |
| 0.02 | 33.60 | 8.91 | 8.91 | 24.93 | 24.93 |
| 0.05 | 33.63 | 4.46 | 4.46 | 12.48 | 12.48 |
| 0.1 | 31.27 | 8.78 | 8.78 | 24.58 | 24.58 |
| 0.2 | 30.02 | 6.39 | 6.39 | 17.89 | 17.89 |
| 0.5 | 29.30 | 5.29 | 6.72 | 14.81 | 18.82 |
| 1 | 24.56 | 2.58 | 2.58 | 7.22 | 7.22 |
| 2 | 23.45 | 4.71 | 5.05 | 13.18 | 14.13 |
| 5 | 22.07 | 4.19 | 6.54 | 11.74 | 18.31 |
| 10 | 20 37 | 1 49 | 3 55 | 4 18 | 9 95 |

Table A35. Precision Statistics of δ for Granite at 20 °C.

* Units of repeatability and reproducibility limits are degrees

| Frequency (Hz) | Average of cell averages | Repeatability Std Deviation, S _r | Reproducibility Std Deviation, S _R | 95% Repeatability Limit, r | 95% Reproducibility Limit, R |
|-------------------|--------------------------|--|--|-------------------------------|---------------------------------|
| 0.01 | 34.86 | 9.33 | 15.03 | 26.12 | 42.08 |
| 0.02 | 39.35 | 10.44 | 10.44 | 29.24 | 29.24 |
| 0.05 | 40.04 | 8.62 | 8.62 | 24.13 | 24.13 |
| 0.1 | 42.83 | 2.67 | 3.54 | 7.48 | 9.92 |
| 0.2 | 44.53 | 2.39 | 3.14 | 6.68 | 8.79 |
| 0.5 | 47.74 | 2.00 | 2.94 | 5.60 | 8.24 |
| 1 | 49.02 | 1.36 | 2.28 | 3.82 | 6.39 |
| 2 | 49.51 | 1.14 | 1.47 | 3.20 | 4.11 |
| 5 | 49.01 | 2.17 | 4.22 | 6.07 | 11.82 |
| 10 | 47.56 | 1.13 | 3.31 | 3.17 | 9.27 |

Table A36. Precision Statistics of G* for Granite at 40 °C.

* Units of repeatability and reproducibility limits are degrees