

## Superpave Implementation

James Mahoney and Jack E. Stephens

Prepared for  
The New England Transportation Consortium

September 1999

NETCR 18

Project No. 96-1

Prepared by  
University of Connecticut  
Connecticut Transportation Institute  
Connecticut Advanced Pavement Laboratory

This report was sponsored by the New England Transportation Consortium, a cooperative effort of the Department of Transportation and the Land Grant Universities of the six New England States, and the US Department of Transportation's Federal Highway Administration.

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

1. The first part of the report

2. The second part of the report

3. The third part of the report

4. The fourth part of the report

5. The fifth part of the report

6. The sixth part of the report

7. The seventh part of the report

8. The eighth part of the report

9. The ninth part of the report

10. The tenth part of the report

11. The eleventh part of the report

12. The twelfth part of the report

13. The thirteenth part of the report

14. The fourteenth part of the report

15. The fifteenth part of the report

16. The sixteenth part of the report

17. The seventeenth part of the report

18. The eighteenth part of the report

# Technical Report Documentation Page

1. Report No. <b>NETCR 18</b>	2. Government Accession No. <b>N/A</b>	3. Recipient's Catalog No. <b>N/A</b>	
4. Title and Subtitle  <b>SUPERPAVE Implementation</b>		5. Report Date <b>September 1999</b>	
		6. Performing Organization Code <b>N/A</b>	
7. Author(s) <b>James Mahoney, Jack Stephens</b>		8. Performing Organization Report No. <b>CAP Lab 99-2</b>	
9. Performing Organization Name and Address <b>University of Connecticut Connecticut Advanced Pavement Lab Connecticut Transportation Institute Storrs, CT 06269-3037</b>		10. Work Unit No. (TRAIS) <b>N/A</b>	
		11. Contract or Grant No. <b>N/A</b>	
12. Sponsoring Agency Name and Address <b>New England Transportation Consortium 179 Middle Turnpike University of Connecticut, U-202 Storrs, CT 06269-5202</b>		13. Type of Report and Period Covered  <b>Final</b>	
		14. Sponsoring Agency Code <b>NETC 96-1 A study conducted in cooperation with the U. S. DOT</b>	
15. Supplementary Notes  <b>N/A</b>			
16. Abstract  The conversion from viscosity grading asphalt binders to Superpave Performance Graded asphalt binders (PGAB) has required a major change in the testing equipment and protocols. The test results from the PGAB showed large variations between laboratories. These variations could result in disputes between PGAB suppliers and State Highway Agencies. This study looked at possible causes for the test result variations as well as possible remedies. The project also conducted Asphalt Binder Technician Workshops to convey the interim findings of the project as well as to learn where potential problems may exist based upon feedback from the Technicians conducting these tests.			
17. Key Words <b>Binder, Performance Graded Asphalt Binder, Superpave Binder, PGAB</b>		18. Distribution Statement <b>No restrictions. This document is available to the public through the National Technical Information Service, Springfield, VA 22161</b>	
19. Security Classif. (of this report) <b>Unclassified</b>	20. Security Classif. (of this page) <b>Unclassified</b>	21. No. of Pages	21. Price <b>N/A</b>

Form DOT F 1700.7 (8-72)

Reproduction of completed page authorized

## Acknowledgements

The authors would like to acknowledge the State Highway Agencies for the complete cooperation throughout this project. The authors would especially like to thank all the people who participated in this project. Without their complete cooperation and willingness to share their experiences this project would never have been possible.



## Table of Contents

Technical Report Documentation	ii
Acknowledgements	iii
Table of Contents	iv
List of Tables	v
Chapter 1 Introduction	1
Chapter 2 Round Robin 98 Performed in Conjunction With NECEPT	3
Chapter 3 Pre-molded DSR Samples	4
Chapter 4 Round Robin 99	7
Chapter 5 Lab Visits – Observations	8
Chapter 6 NETC Binder Technician Workshops	10
Chapter 7 Lab Visits - Equipment Comparisons	11
Chapter 8 Database of Binder Test Results	15
Chapter 9 Summary of Results	16
Chapter 10 Recommendations	19
References	26
Appendix I – Original Proposal	
Appendix II – Round Robin 98 Report	
Appendix III – Pre-molded DSR Samples Information	
Appendix IV – Round Robin 99 Report	
Appendix V – Lab Visits – Observations	
Appendix VI – NETC Binder Technician Workshop Presentations	
Appendix VII – Lab Visits – Equipment Comparisons Test Data	

## List of Tables

Table 1 – Results from the Premolded DSR Samples	5
Table 2 – Comparison of Round Robin %1S and %1S Values for Lab Visits	14
Table 3 – Average %1S for all Samples Sent Out	17
Table 4 – AASHTO Protocols Referenced in NETC 96-1 Recommendations	19

## CHAPTER 1

### Introduction

The Asphalt industry has undergone dramatic changes over the past several years due to the introduction and implementation of the Superpave system. This new technology has changed both the way an asphalt binder is graded and the way Hot Mix Asphalt is designed. The change from asphalt binder classification by a single viscosity test to performance grading created a number of challenges for the State Highway Agencies (SHAs). Adjustments were needed in material acceptance procedures if the new specifications were to be accommodated. The asphalt binder testing technicians had very limited experience with the test equipment for the determination of the binder properties used in the new performance grading. The early test results showed a large variation in test results between asphalt binder testing laboratories. This variation could cause substantial difficulties between asphalt suppliers and SHAs.

This project was initiated to improve the Performance Graded Asphalt Binder (PGAB) testing which would in turn reduce the between laboratory variation. This study did not address issues relating to the Superpave Mixture but focused only on PGAB issues.

The scope of this study included the following tasks, all pointed toward the goal of improving the reproducibility of the binder test results:

- Development of a round robin testing program to identify the level of variability occurring during testing.
- Observation by the CAP Lab of the actual testing techniques being employed by the SHA technicians.
- Dissemination of information on the techniques observed along with

recommended practices to the technicians through workshops where both SHA technicians as well as private industry technicians could gather to learn about changes to the testing specifications, discuss issues they felt were important and to exchange techniques and ideas between the workshop participants.

- Convey information learned during this project through the use of the CAP Lab website ([www.caplab.uconn.edu](http://www.caplab.uconn.edu)).
- Distribute copies of the reports of each activity to all of the participants of the Round Robins as well as the workshops.
- Providing telephone support for the SHA technicians.

The original research proposal is attached as Appendix 1. The following chapters summarize the activities undertaken by the CAP Lab for this project

## CHAPTER 2

### Round Robin 98 performed in conjunction with NECEPT

Working with the Northeast Center of Excellence for Pavement Technology (NECEPT) provided several benefits to this project. Interaction with the technical personnel at Penn State who developed Performance Grading and were now part of NECEPT would lead to early awareness of equipment and protocol changes. A second benefit was the increase in the size of the pool of participating laboratories. The SHA laboratories of New York, New Jersey, Pennsylvania, Maryland, Delaware and the supplier laboratories of the area joined the New England SHAs forming a pool of 20 labs. The major revelation of the 98 Round Robin was the significant variation in test results between the various labs. A detailed statistical analysis was carried out by NECEPT to estimate the variation of coefficients for the components of variance. See the attached draft report for a detailed presentation and conclusions for the 98 Round Robin (Appendix II).

### CHAPTER 3 Pre-molded DSR Samples

Premolding the DSR samples was intended to remove the effects of differences in handling times and temperatures from the test conditions. Unfortunately, there was no way to ensure that the molds would remain in a horizontal position and because of this some of the samples slumped badly and required some re-handling. The two samples were the same asphalt binder. The raw overall  $G^*$  average was 1.401 with a Standard Deviation (St Dev) of 0.121 and the raw average phase angle was 86.6 with a St Dev of 0.9.  $G^*$  for the second test at lab 105 and both deltas for lab 122 are more than two Standard Deviations from the averages and should be deleted from the computations as outliers. The adjusted  $G^*$  is then 1.420 with a St Dev of 0.085 and the average phase angle 86.9 with St Dev of 0.30.

Late sample delivery forced some labs to test a day late. The mid portion of Table 1 shows the effect of separating the results into two sets of data with the wild ones referred to above still deleted. One day seems insignificant in the life of a binder but as the samples were poured 7/27/98 it did add 50% to the time since major heating which would have destroyed any steric hardening which may have occurred. Statistical computations must be looked at cautiously as the number of data points with similar conditions is small. The  $G^*$  of the late group was 0.096 or 6.9 % greater than that of the on-time group with little change in St Dev. At that rate, a two day delay would have given results more than two St Dev greater than the on-time group. The timing did not affect the average phase angle but did increase its variability.

Table 1 – Results from the Premolded DSR Samples

	Lab #	G* #1, kPa	Delta #1	G* #2, kPa	Delta #2	
	101	1.263	86.87	1.222	86.8	
	103	1.502	86.64	1.58	86.7	
	105	1.372	86.91	1.016	87.46	
	107	1.413	87.6	1.414	87.29	
	108	1.472	86.79	1.423	86.78	
	109	1.361	87.29	1.415	87.08	
	110	1.363	86.61	1.424	86.56	
	113	1.414	87.16	1.389	87.2	
	121	1.448	86.55	1.402	86.68	
	122	1.504	83.6	1.466	84.6	
	123	1.551	86.28			
Overall G* Average		1.401		G* Average without 105 #2	1.420	
Overall G* Std Dev		0.121		G* Std Dev without 105 #2	0.085	
Overall Phase Angle Average		86.6		Phase Angle Average without 122	86.9	
Overall Phase Angle Std Dev		0.9		Phase Angle Std Dev without 122	0.3	
<b>Variations in Day of Testing</b>						
G* Avg Test Performed 7-29-98		1.396		G* Avg Test Performed 7-30-98	1.492	
G* Std D Test Performed 7-29-98		0.075		G* Std D Test Performed 7-30-98	0.077	
<b>Note: Lab 122 Phase Angles not included in calculations</b>						
Phase Angle Avg 7-29-98		86.9		Phase Angle Avg 7-30-98	86.9	
Phase Angle Std Dev 7-29-98		0.3		Phase Angle Std Dev 7-30-98	0.5	
<b>Possible Equipment Variations</b>						
<b>Note: Lab 105 G*#2 not used in the following calculations</b>						
G* Average ATS DSR		1.405		G* Average Other DSR Makers	1.438	
G* Std Dev ATS DSR		0.032		G* Std Dev Other DSR Makers	0.123	
Phase Angle Average ATS DSR		87.1		Phase Angle Avg Other DSRs	86.1	
Phase Angle Std Dev ATS DSR		0.3		Phase Angle Std D Other DSRs	1.2	

The state labs and CAP Lab used ATS Rheologica DSRs while the other labs used DSRs from different manufacturers. The bottom portion of the Table 1 looks at the effect of instrument. Due to the small number of non ATS instruments used, no evaluation of

brands has been attempted. The average  $G^*$  for the non-ATS group was slightly above that of the ATS group and the phase angle slightly below. The major difference was in the magnitude of the standard deviations, which was approximately four times as great. This implies that there was a difference between the units. Due to the small number of tests no conclusions can be made as to whether the difference was between or within a brand. Incidentally the instrument used by the lab whose Deltas were deleted was not an ATS and if retained would not increase the St Dev of that group.

Summary: The results for the ATS group had the least St Dev. Delays after pouring and prior to placing in the test instrument appears to increase  $G^*$  and the St Dev. This would also imply that the higher the temperatures during handling the greater  $G^*$ . The smaller standard deviations obtained by ATS DSRs may be attributable to the frequent maintenance and calibration the machines are receiving from the manufacturer. Additional details of the Pre-Molded Round Robin are attached as Appendix III.



## CHAPTER 4

### Round Robin 99

After the Pre-Molded results were reviewed and distributed to all participants, it was decided that a Round Robin in which explicit instructions regarding sample handling during specimen preparation and testing should be conducted. The report of this effort known as Round Robin 99 is in Appendix IV. During all of the Round Robins, the CAP Lab monitored the test results and was able to notify several labs of problems they were unaware of having with their equipment.

The reduction in the variation between laboratories indicates the need for tighter test protocol specifications and adherence to these specifications regarding sample handling and specimen preparation. The current test protocols do not adequately address the issue of sample handling. For example, the test protocols currently state that the samples should be heated until the material has the consistency of SAE 10W30 motor oil. Unfortunately, this leaves a lot of room for interpretation regarding heating temperatures and length of time to heat samples, thus the samples have a vast difference in thermal histories. This difference in thermal histories will cause variations in the test results.

The sample handling and testing procedure utilized for this round robin may not be appropriate for routine testing. The handling procedures were developed to achieve the greatest uniformity of sample handling. The sample handling instructions are located in Appendix IV. Standardized heating temperatures and times need to be developed.

## CHAPTER 5

### Lab Visits - Observations

The CAP Lab sent a person to five of the six New England State SHA's and to the New York State DOT to observe binder testing. These visits were intended to note departures from the testing protocols. Any techniques which would expedite the testing were also noted provided the technique did not adversely affect the test results. The results from the visits were reported anonymously so as to not embarrass any individuals and encourage everyone to be more open as to the procedures routinely followed. The lab visits indicated that there was very little deviation from lab to lab in regards to the testing techniques. The largest deviation noted from the testing procedure involved the BBR. Several labs were placing the BBR test specimens into the supports and then adjusting the testing loads on the specimens. This caused the beams to deflect before the test had actually begun. The test results did not include any of this deflection and therefore the test results were inaccurate.

The CAP Lab also observed that some of the labs did not do a very good job tracking the amount of time samples were in the Rolling Thin Film Oven (RTFO). This piece of equipment does not have its own timing device on it. Therefore, it requires the operator to track the amount of time the material has been in the oven manually. The AASHTO specification requires the material to be in the oven for 85 +/- 1 minute. The time limits on this test are quite tight because of the rapid rate of aging occurring in the oven. Not adhering to these strict time limits will cause large differences in the material and erroneous test results.

The use of glass cleaning ovens or ignition ovens for cleaning glassware was a major time saver. The glassware is cleaned by burning the asphalt off of the glass. This method of cleaning the glassware is a major improvement from using solvents to accomplish the same task. The glass cleaning oven is faster than solvents and is more environmentally friendly. Detailed visit observations as circulated to the participants can be found in Appendix V.

## CHAPTER 6

### NETC Binder Technician Workshop

A one day binder technician workshop was held in January, 1999. The intent of the workshop was to update the binder technicians as to the progress being made during the project. The workshop also presented an opportunity to inform the technicians as to the upcoming work being done as part of the project. The topics discussed during the workshop include: the Round Robin 98 which was performed in conjunction with NECEPT, the pre-molded DSR samples, the lab visits where observations were made, the upcoming Round Robin 99 and the lab visits which were going to be made during the spring of 1999 to run samples on the state labs equipment. Additional material related to the January 1999 Binder Technician Workshop including slides used is attached as Appendix VI.

## CHAPTER 7

### Lab Visits - Equipment Comparison

The CAP Lab followed up Round Robin 99 by visiting the asphalt binder testing labs and running samples on the lab's equipment. The between laboratory variation found in the round robin results can not be broken down into that caused by equipment differences and that due to technician handling. The first visitations by CAP Lab personnel to the state laboratories monitored the technicians. A second series of visits were undertaken in an attempt to determine what variability was assignable to the equipment.. The purpose of the visits was to determine the degree of variability caused by the testing equipment. If successful, the results could be combined with the Round Robin 99 results to give a better picture of the between laboratory variation. The same person was used to perform the testing at each facility. This was done to eliminate variations which could be caused by multiple operators.

The CAP Lab prepared samples out of the same material used for Round Robin 99. The samples for both the Round Robin 99 and the visitations were all prepared at the same time to ensure the materials were similar. The results obtained from the visits can not be directly compared to the Round Robin 99 results due to differences in sample handling. The CAP Lab samples were prepared to ensure their uniformity. The methods used to prepare the samples did not match the techniques used by the laboratories during the Round Robin testing. Each visitation sample was placed in its own small heat resistant container to avoid repeated re-heating of the samples which would change the properties of the material.

The testing during the visits included Dynamic Shear Rheometer (DSR) testing of original binder and rolling thin film oven (RTFO) residue testing as well as pressure aging vessel (PAV) residue tested in the Bending Beam Rheometer. Both the RTFO and the PAV conditioning of the visitation samples was done at the CAP Lab. The DSR testing was performed at 58° C and 64° C for both the original binder and RTFO residue using the 25 mm parallel plates. The testing performed using the BBR was performed at -18° C. To make the testing of the PAV residue uniform throughout the visits silicone beam molds were used. The same beam molds were used in each lab. The silicone beam molds were chosen because of their ease of use and cleanup. The CAP Lab brought their own tools for the trimming process so that the results would apply to the basic test assemblies present at each lab.

All of the DSR testing performed by the CAP Lab was done in the same six month manufacturer calibration period as Round Robin 99 was performed. The manufacturer calibration could change the settings on the DSR and would make drawing conclusions between Round Robin 99 and the samples run on each lab's equipment very difficult. The laboratories were asked to prepare both the DSR and BBR as they did for the Round Robin samples. This included verifying the temperatures of the equipment in the same manner as was done for the Round Robin. The labs were also asked to calibrate their BBRs as they did for Round Robin 99.

The CAP Lab ran two DSR original binder samples, two DSR RTFO samples and three pairs of beams on the BBR. For the DSR samples, two specimens were mounted in the DSR. One specimen was mounted and tested at both 58° C and 64° C, the second specimen was mounted and tested at 64° C only. This testing regime was similar to the one employed for Round Robin 99. The following pattern was repeated for all of the samples which were to be tested in the DSR. The DSR samples were heated for 20 minutes at 135° C. Each specimen was mounted and tested as soon as possible. The DSR specimens were trimmed using a heated trimming tool. The BBR samples were heated for 45 minutes at 163° C before pouring. The beams were allowed to stand on the countertop and cool for 45-50 minutes. The beams were then placed in an ice bath for 5-7 minutes. An ice bath was used to ensure thermal history uniformity from lab to lab. The beams were then soaked in the BBR bath at -18C for 60 minutes and then tested. Figures 1 through 6 show the averaged values obtained at each of the state binder labs.

Table 2 shows the %1S values for lab visits for the four New England States which participated in both Round Robin 99 and the lab visits and the corresponding %1S values for the four states from Round Robin 99. The %1S value represents one standard deviation as a percentage of the average.

Table 2  
Comparison of Round Robin 99 %1S values for selected states and %1S values for Lab Visits

Sample & Temperature	Four States Round Robin 99	Lab Visits	Total 12 Lab Round Robin
G*, Original Binder, %1S, 58° C	4.51	4.83	4.90
G*, Original Binder, %1S, 64° C after 58° C	6.70	4.74	5.71
G*, Original Binder, %1S, 64° C new specimen	5.39	6.05	5.83
G*, RTFO, %1S, 58° C	6.31	4.43	7.17
G*, RTFO, %1S, 64° C after 58° C	6.91	4.41	8.03
G*, RTFO, %1S, 64° C new specimen	8.12	5.68	8.48
Stiffness, %1S	3.64	4.19	5.65
m-value, %1S	2.08	1.52	2.80

The values obtained for the lab visits are close to the values obtained from Round Robin 99. The smaller the %1S value the closer the test results were to each other. The difference for the G\* of the original material appears random with the visitation %1S greater for one sample and less for two. It would be expected that the RTFO %1S values would be lower for the CAP Lab visits due to homogenization of the samples after the RTFO aging at the CAP Lab. For the Round Robin samples, which were conditioned by each lab, greater variation in the material probably existed when the specimens were poured. This greater variability of the Round Robin samples can not be charged to the equipment as the equipment did not change. The numerical difference in average G\* value for the two series is due to the difference in conditioning but the difference in %1S is probably due to differences in handling. The data from the lab visits can be seen in Appendix VII.



## CHAPTER 8

### Database of Binder Test Results

A portion of this project was intended to build a database where results for binders supplied by the same shipper to different states could be compared. This task could not be completed because most of the states were not recording the supplier's lot numbers with the binder test results from the 1998 construction season. This meant that there was no guarantee that tests were actually on the same binder lot even though the material had come from the same supplier on approximately the same day.

## CHAPTER 9

### Summary of Results

With the implementation of the new equipment, protocols and criteria of the Superpave system, The New England Transportation Consortium initiated this study to identify possible causes for the between-laboratory test variations and recommend solutions to reduce these variations. The major goal was to assist the state agencies with implementation of Superpave Performance Graded Asphalt Binder testing.

After some initial adjustments, the six states were comparably equipped and round robins were conducted as a basis for determining what difficulties existed. Test results by the different labs from Round Robin 98 differed substantially. For one of the tests, the lowest result was approximately half of the highest. Such variation would make use of the results for QC/QA very difficult, so a workshop was held to look for differences in the procedures used by the reporting labs.

When the premolded DSR samples were processed, the variations between reporting labs were greatly reduced though not eliminated. One of the goals of premolded DSR samples was to determine if the variations were due to handling by the technicians or equipment differences. Handling differences for the premolded samples were reduced by preparing all samples for machine mounting by the CAP Lab prior to distribution. The reduction in variation indicated sample handling was a factor.

At the end of the ensuing workshop, which stressed sampling handling, the last round robin (Round Robin 99) was distributed with precise handling, temperature and timing instructions. The detailed instructions included with the Round Robin 99 samples

attempted to eliminate problems with sample handling discovered during the project. The standard deviation for the test results reported by the laboratories was small. Table 3 contains the averages for the %1S (excluding outliers) for each of the testing stages throughout this project. The %1S value is the ratio of the standard deviation for that set of test results to the average for all of the test results expressed as a percentage.

Table 3 – Average %1S for each set of Samples Sent out during this Project

	Round Robin 98	Premolded Samples	Round Robin 99
G*, Original	9.64%	5.99%	5.48%
Phase Ang. Orig	0.86%	0.35%	0.51%
G*, RTFO	9.67%	N/A	7.89%
Phase Ang. Orig.	0.83%	N/A	0.47%
m-value	4.04%	N/A	2.80%
Stiffness	9.62%	N/A	5.65%

The last effort was the identification of equipment differences. Identical samples were packaged in salve cans at the CAP Lab and James Mahoney, manager of the CAP Lab spent a day at each laboratory processing and testing the material. Very little difference was observed from lab to lab. The results of these visits can be seen in chapter 7.

Overall, the variation of test results for each Round Robin has decreased after each workshop and each laboratory visit. The largest decrease in variation was brought about by including explicit instructions regarding heating times, heating temperatures and limiting the amount of time the specimens are allowed to stand prior to testing. In order to realize this reduction in test variation for production testing, explicit heating and timing instructions need to be developed and followed.

It is very apparent that some form of continued effort will be necessary if the between laboratory variation is to be contained when very small changes in test handling or equipment can change results. This continued effort should include Round Robins as well as technician workshops. The Round Robins are a good method to ensure testing uniformity between laboratories as well as identifying equipment problems. The technician workshops are an excellent forum for technicians to discuss issues with changes in the test protocols, equipment changes and exchange information they may have acquired over the previous construction season.

## CHAPTER 10 Recommendations

The following recommendations are drawn from the major findings of this project. The sources for these recommendations were from all aspects of the project. These aspects include the lab observations, workshop discussions, Round Robin results and discussions with testing agencies outside of the New England region.

A number of recommendations are made concerning the techniques of carrying out binder testing. Several other recommendations stress the need for precise timing and temperature control for steps in the testing process. Some of the recommendations provide guidance for steps inadequately described in the current testing specifications.

The last recommendations address somewhat different problems. Superpave binder testing is a new endeavor. As with any new endeavor, many changes can be expected to the testing procedures and these last recommendations attempt to address problems brought forth by these changes. Table 4 provides a quick reference as to the specification(s) each recommendation is referencing.

Table 4, AASHTO Protocols Referenced in NETC 96-1 Recommendations

AASHTO Protocol*	Recommendation Number																	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
MP-1	X		X	X	X	X												
PP-1	X		X		X													
TP-1	X		X		X								X	X	X			
TP-3	X		X		X													
TP-5	X		X		X	X	X	X	X	X						X		
PP-6	X		X	X	X	X												
T-40	X	X	X		X													
T-240	X		X		X							X				X		
TP-48	X		X		X	X					X							

- \* MP-1 *Specification for Performance Graded Asphalt Binder*  
 PP-1 *Practice for Accelerated Aging of Asphalt Binder Using a PAV*  
 TP-1 *Method for Determining the Flexural Creep Stiffness of Asphalt*

- Binder Using the Bending Beam Rheometer*
- TP-3 *Method for Determining the Fracture Properties of Asphalt Binder in Direct Tension*
- TP-5 *Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer*
- PP-6 *Practice for Grading or Verifying the Performance Grade of an Asphalt Binder*
- T-40 *Sampling Bituminous Material*
- T-240 *Effect of Heat and Air on a Moving Film of Asphalt (RTFO Test)*
- PP-26 *Practice for Certifying Suppliers of Performance Graded Asphalt Binders*
- TP-48 *Method for Viscosity Determination of Asphalt Binder Using Rotational Viscometer*

1. Based upon the improvements made in the test deviations in the Round Robins, improved standardized handling (time and temperature) procedures need to be developed. Standardized handling procedures would help to reduce the amount of variation occurring between laboratories.
2. Unlined paint cans should be used for sample containers. The lining used in paint cans was never expected to stand oven temperatures and may melt into the asphalt. Paint cans are easy to fill and most importantly are the easiest to stir for uniformity when re-heating the material for testing. Stirring is important for all samples but will become an increasingly larger issue as modified asphalts become more common. This is also an issue as QC/QA becomes more widely practiced. A state lab may have a way to stir samples in containers other than paint cans but another lab which becomes involved in testing dispute resolution may not have this capability.
3. Limit the amount of time the original sample is heated. As any heating causes binder to age thus affecting test results, any heating beyond that necessary for easy stirring and pouring must be avoided. The length of heating time required

to completely melt the binder depends on the PG grade as well as any modifiers that may be present.

4. When testing polymer modified binders, obtain sample heating instructions from the manufacturer of the material. Binders which have been heavily modified with polymers can be expected to require handling at higher temperatures than binders which have not been modified. The manufacturer should be able to provide temperatures to which the modified binder can be heated without damaging the polymer modifier. Over heating can damage some polymer additives. Destruction of the polymer additives may cause the binder to appear much softer than it is when handled properly.
5. All binder samples should be thoroughly stirred prior to testing to ensure their homogeneity. Particular care should be given to homogenizing polymer modified binders as some polymers tend to settle in the sample container.
6. Original binder, as it is in the rapid aging portion of the aging curve, is affected by heat aging more than PAV samples and should be heated only once. For minimum variation in test results, the original material sample should only be heated until the material is able to be stirred and poured.
7. The amount of time DSR samples stand in the silicon rubber molds should be as short as possible limited to one hour. By allowing these samples to remain in the molds, steric hardening can occur. The rate at which steric hardening occurs varies greatly from binder to binder and variations of steric hardening are difficult to quantify. Avoid steric hardening by limiting time before mounting.

8. Warm the testing plates to testing temperature before setting the zero gap on the DSR. Once the zero gap is set, changing temperature greatly alters the dimensions causing a false gap reading.
9. Trim DSR samples with a heated trimming tool. The use of room temperature trimming tools may cause some of the binder material to be pulled out from between the test plates. Since the material around the perimeter of the sample has the largest moment arm, it is the most sensitive portion of the sample. Therefore, a deficient quantity of material on the outside perimeter of the plates can cause a substantial error in the measured complex shear modulus.
10. Most DSR instrumentation has an offset between the temperature sensor and the true sample temperature. Each lab should be equipped to measure the temperature between the DSR plates. This can be performed with either a thermistor or a thermocouple. With either temperature measuring system, the measuring device needs periodic calibration against a certified temperature measuring device.
11. When calibrating the temperature of the Rotational Viscometer, the spindle should be in the sample and the spindle should be rotating. This will help to reduce the temperature gradients present in the sample. Caution should be used to prevent the thermocouple from becoming entangled with the spindle.
12. Care should be taken observing the length of time binder is left in the Rolling Thin Film Oven. At this stage in aging, the binder ages easily and a few minutes



can cause significant aging. This is the only piece of equipment which does not have an automatic timing system. This requires the operator to pay particular attention to the length of time the sample material is left in the oven. This can be difficult as the operator is usually busy performing some of the many other tests. A simple kitchen timer may be used as a reminder.

13. BBR beams must be cooled sufficiently for ease in de-molding without deforming the beam. Ice baths should be used whenever possible. The ice bath provides a uniform between laboratory temperature for de-molding specimens. Some binders may not become stiff enough in an ice bath for demolding and will need to be placed in a freezer.
14. The aluminum BBR molds should be lined with plastic strips. The use of the glycerin-talc mixture as a bond breaker over all of the beam mold parts will cause the BBR bath fluid to need frequent changes as the glycerin-talc mixture will cloud the fluid. Even more important, uneven application of this mixture to the beam molds will cause uneven beam cross-sections and affect test results.
15. The use of straight alcohol in the BBR bath if allowed by safety codes is the most desirable fluid to use in the bath. The ethylene glycol, methanol and water mixture while not flammable is dense causing the beams to float in the BBR bath. This makes temperature conditioning difficult and increases the chances the beams will be damaged while placing them in testing supports.
16. The use of glass cleaning ovens or ignition ovens to clean glassware is a huge improvement over cleaning the glassware with solvents. Cleaning glassware

with this technique is not nearly as labor intensive as using solvents and there is no chance of contaminating the next binder sample with traces of solvents. Also, with the ovens there are no solvents which need to be disposed.

17. Over the course of the year, several test samples should be sent out to laboratories for testing. The intention of these samples is to allow the laboratory the opportunity to test with as little pressure as possible. The goal is to allow the labs to test the samples as closely as possible to their standard testing technique. Most labs prepare quite differently for testing "pedestal samples" such as the AMRL Proficiency samples than they do for routine testing. By having samples which are not held as "pedestal samples", each lab would have an opportunity to discover potential problems which they otherwise may not be able to detect. This can best be done by having samples where the labs identity is held in confidence and the lab's results are compared to a large pool of data. This also allows the technicians to readily compare their results with those of others. With this concept, there is no penalty for poor performance by labs and yet it would be a very powerful tool to diagnose problems which could exist.

18. As the specifications continue to change and the equipment evolves to meet these changes, a forum for the binder technicians to learn about these changes needs to be maintained. The workshop participants have noted the importance and need for the workshops. The workshops allowed the participants to learn about upcoming changes to the specifications and equipment. The workshops also allowed the participants to voice their concerns about problems they have with the specifications. The feedback from the technicians performing the testing is very important. This provides information as to which areas of the specification may require additional scrutiny.

### References

1. AASHTO, *June 1998 – AASHTO Provisional Standards*, 1998.
2. AASHTO Materials Reference Laboratory, Performance-Graded Binder Samples, [www.nist.gov/aashto/amrl/services/sect3b.html](http://www.nist.gov/aashto/amrl/services/sect3b.html), June 1, 1999.
3. Anderson, David and Marasteanu, Mihai, *Manual of Practice*, NECEPT, 1999.
4. McDaniel, Rebecca S., *Results of a Superpave Binder Testing Program of the North Central Asphalt User Producer Group*, September 1996.
5. Wisconsin Department of Transportation, *Combined State Binder Group Round Robin 99-1*, March 17, 1999.

Appendix I  
Original Proposal

Proposal to; The New England Consortium

Project: SUPERPAVE Implementation

Project Number: 96 - 1

Submitted by: C A P Lab, University of Connecticut

## **Response to N E Consortium Project Number 96-1**

### **SUPERPAVE Implementation**

New England, like the rest of the United States, has used viscosity at 60 degrees C or penetration at 25 degrees C as the prime factor in binder classification. This did not permit comparisons of the performance in thin films as found on aggregate or at low winter temperatures. The SHRP binder tests provides data for such comparisons. As SHRP test data is not available for pavements more than a few years old, selection of design values for the new tests have been based on accelerated testing or extrapolation of short term performance. Each using agency feels the need for the immediate collection of actual field performance data to verify the predicted performance. Every state can be expected to collect field data but several years will pass before any one state has a statistically sound quantity of data. As conditions tend to be similar through out New England, pooling data will make sound statistical analysis possible much sooner.

Area wide pooling of data must be carried out with great care to ensure that the results collected by different agencies have the same significance. Past experience with the application of AASHTO and ASTM test methods has shown that different interpretations of a test method can occur for years after the method has been published. This results both from differences in laboratory equipment and personnel background. As the SHRP program implements both new techniques and equipment, careful control of laboratory work is essential. Round robin testing can be used to both identify problem points in procedures and to determine the degree of conformity of individual laboratories.

### **Organization for "SUPERPAVE" Implementation:**

This project will be under the supervision of the CAP Lab staff. Jack E. Stephens, the director and James Mahoney, the laboratory manager will assign time to the project. Dr. Norman Garrick, the Civil Engineering Department professor responsible for bituminous studies will assist on computer applications. A coordinating committee with a representative from each New England state will advise the project. Research assistants at the laboratory supplemented by student labor will provide additional support time as needed. The Lab has the available SHRP binder and mix equipment in duplicate so that equipment down time should not interfere with testing schedules.

### **Task 1: Establish a WEB site on the Internet**

The University of Connecticut has a well established computer center with long experience in networking. Students at the five branches have networked with the main campus computer center for some 40 years. The Computer Center conducts short courses on WEB site establishment. Should any difficulties be experienced in setting up a web site, the staff of the computer center and of the Engineering Computer Science Program would be available for counsel. The WEB site will be set up through the Transportation Institute. All cooperating laboratories will be expected to forward their SHRP testing results to the CAP Lab. Material for the WEB site will be prepared by a research assistant and reviewed by the CAP Lab director or manager before placement at the WEB

site. Relative linking to other pertinent WEB sites will be included. An information sheet will be supplied all potential users.

## **Task 2: Develop Round Robin Testing Program**

As literally all asphalt testing is related to state usage, the advisors from the states will be able to supply a list of the laboratories equipped for SHRP binder testing. The Director will approach all laboratories and request support through cooperation in a Round Robin testing program. When a sufficient number have joined, the CAP Lab director as a member of the Northeast Users Producers Group will contact the northeast suppliers of P G grade asphalts to secure binders for Round Robin testing. It is anticipated that some suppliers will also be Round Robin testers. AMRL methodology and practices for collecting and distributing samples will be followed as applicable and to the extent practical. The range of grades will be determined by the application of SuperPave methodology to the New England area.

Once testing commences, each laboratory will be asked to bi-monthly fax reports to the CAP Lab. Data at the Web site concerning the Round Robin testing will be presented in statistical format and updated bi-monthly. As SHRP testing is new to most laboratories, the first Round Robin can be expected to show substantial deviations. Using analyse of the data to aid in correcting short comings, successive Round Robins should show progressively better uniformity between laboratories

## **Task 3: Observe Operation of SHRP Binder Equipment at State Highway Agency Laboratories.**

The CAP Lab Director or Manager will visit each SHA laboratory and observe calibration, sample preparation and test procedures for the Rotational Viscosimeter, Dynamic Shear Rheometer, Rolling Thin Film Oven, Pressure Aging Vessel and Bending Beam Rheometer. Any non-specification procedure observed will be noted as an aid in weighting test results and possibly reducing data scatter. Procedure deviations that appear to improve the efficiency or accuracy of the test method will be noted and distributed to the other states by posting at the WEB site. Each laboratory visited will receive a report of the observations at that laboratory. The labortory observations will compliment the Round Robin testing.

## **Task 4: Other Activities that Will Aid the State Highway Agencies (SHA) Laboratories.**

As The New England States adopt PG Grading, test results of locally available binders should become available. The Stastes will be encouraged to send in routine test results for posting at the WEB-Site. . Contact will be made with SHAs, binder producers, University laboratories and User Producer Groups across the country and the Federal Materials group requesting reports or other information as to improvements found in techniques for carrying out SHRP testing that would make for more efficient use of equipment, methods or personnel. Any procedures found worthy would be posted at the WEB site.



## **Task 5. Final Report**

A final report will be prepared including sections of statistical results of the round robin testing, effectiveness of the current SHRP test methods and a summary of non-specification procedures that would improve efficiency or accuracy of testing. Any shortcomings found in procedures that affect interlab comparisons of data will be noted and corrections suggested.

### **Schedule of Activities**

First priority will be given to the establishment of the advisory committee. The committee members will be asked to bring to the first meeting lists of laboratories and suppliers.

A WEB site for New England will be established on the Internet where results of P G Binder routine testing can be posted.

The CAP Lab will immediately contact area laboratories for support of a Round Robin Testing Program using AASHTO provisional methods with any variations agreed to by the advisory panel.

The Director will then approach the suppliers for commitments of materials.

Samples will be distributed to laboratories.

As testing progresses, visits will be made to the laboratories for observation of testing.

As available, testing results will be posted at the WEB site.

Throughout the project, contacts with other agencies will be made and any material concerning SHRP testing improvements or changes posted at the WEB site.

A final statistical analyses will be made and results distributed

Progress reports will be submitted to the Consortium quarterly. A final report will summarize the work.

# Work Schedule

Month of First Year

	1	2	3	4	5	6	7	8	9	10	11	12
Establish Project Committee	X											
Committee Meetings	X			X			X			X		
Task #1 Establish & Update WEB-Site PG Binder Test Results	X	X	X	X	X	X	X	X	X	X	X	X
Task #2 Contact AMRL for Round Robin Procedures	X	X										
Contact Binder Suppliers to Collect Round Robin Samples	X	X				X				X		
Arrange with Labs for Round Robin Participation and Distribute Samples		X				X				X		
Receive and Analyse data	X	X	X	X	X	X	X	X	X	X	X	X
Update Round Robin at WEB-Site	X	X	X	X	X	X	X	X	X	X	X	X
Task #3 Observe Testing at Labs		X	X	X		X	X			X	X	
Post at WEB-Site promising Procedure/technique variations		X	X	X	X	X	X	X	X	X	X	X
Task #4 Contact non-New England Labs and others for Developements			X	X			X	X			X	X
Post Developements at WEB-Site			X	X	X	X	X	X	X	X	X	X
Task #5 Quarterly Reports			X			X			X			X
Final Report												

# Work Schedule

Month of Second Year

	1	2	3	4	5	6	7	8	9	10	11	12
Establish Project Committee												
Committee Meetings	X			X			X			X		
Task #1 Establish & Update WEB-Site PG Binder Test Results	X	X	X	X	X	X	X	X	X	X	X	X
Task #2 Contact AMRL for Round Robin Procedures												
Contact Binder Suppliers to Collect Round Robin Samples		X				X				X		
Arrange with Labs for Round Robin Participation and Distribute Samples		X				X				X		
Receive and Analyse data	X	X	X	X	X	X	X	X	X	X	X	X
Update Round Robin at WEB-Site	X	X	X	X	X	X	X	X	X	X	X	X
Task #3 Observe Testing at Labs		X	X			X	X			X	X	
Post at WEB promising Procedure/technique variations	X	X	X	X	X	X	X	X	X	X	X	X
Task #4 Contact non-New England Labs and others for Developements	X			X	X			X	X			
Post Developements at WEB-Site	X	X	X	X	X	X	X	X	X	X	X	X
Task #5 Quarterly Reports			X			X			X			X
Final Report											X	X

Updated 10, 1996

Date of First Appointment: 1946

Birthdate: 8/17/23 Birthplace: Eaton, Ohio

Education: B.S. 1947 University of Connecticut  
M.S. 1955 Purdue University  
Ph.D. 1959 Purdue University

Experience: 1946- University of Connecticut: Instructor 1946-47;  
Assistant Professor, 1950; Associate Professor,  
1959; Professor, 1962-88; Emeritus Professor,  
1989-; Department Head, 1965-72  
Director Conn Advanced Pavement Lab, 1996 —  
1948-50 Engineer, Connecticut Highway Department  
1958- Owner, Jack E. Stephens Soils Testing Laboratory  
1958-65 Soils and Foundation Consultant, A. J. Macchi  
Engineers  
1962-63 Pavement Consultant, Commissioner of Connecticut  
Highway Department  
1993-95 Consultant to Vermont Transportatio Agency,  
Implementation of SHRP

Professional Societies: North East Asphalt User Producers Group;  
American Society of Photogrammetry; American  
Society of Civil Engineers; American Society of  
Engineering Education; American Congress of  
Surveying and Mapping; American Road Builders  
Association; Association of Asphalt Paving  
Technologists; Connecticut Academy of Science  
and Engineering; Highway Research Board;  
American Society of Testing Materials; Phi Kappa  
Phi; Tau Beta Pi; Chi Epsilon; Sigma Xi

Honors or Distinctions: Automobile Safety Foundation Fellowship, 1958-59;  
Citation, Teaching Excellence, Western Electric  
Fund, 1974;  
University of Connecticut Alumni Association  
Distinguished Public Service Award, 1982;  
University of Conn. Engineering Alumni Award, 1986:

University of Conn. Advisory Boards: Center for Real Estate and Urban  
Economics Research 1966--;  
Joint Highway Research Advisory Council 1962-;  
Transportation Institute 1974-88;  
Technology Transfer Center 1984-;  
CAP Lab, 1996--

Other Tecnical Activities: SHRP Asphalt Advisory Panel, TRB 1986-98  
ConnDOT Pavement Task Force, Chrm 1995--  
NETTCP, Chr Committe for Standardizing Forms, 1995-  
Conn Soc Prof Eng, Laboratory Committee, 1995--

Professional Societies: Conn Society of Civil Engineers, Director 1964,  
Second Vice-President, 1967-68, First  
Vice-President, 1968-69, President, 1969-70,  
Honorary Member, 1969;  
Conn Section, American Society of Civil Engineers,  
Vice-President, 1965-66, President, 1966-67,  
Director, 1967-69

American Road and Transportation Builders,  
Education Division, Director, 1962-65, Vice  
President, 1976, President, 1977;  
Director Transportation Institute, Univ. of Conn.  
1974-77; Committees A2DO4 and A2HO2, Transportation  
Research Board, Chrm. Transp. Comm. and Director,  
Connecticut Academy of Science and Engineering

Field of Specialization: Highway Materials

Research Interest: Problems Associated with Asphalt Paving  
(Including Mix Design Problems, Asphalt Rheology  
and Composition, Pavement Durability, Physical  
Specifications with Chemical Composition  
Specifications)

Publications:

Journal Articles:

Stephens, J. E. and H. A. Sawyer, Jr. 1957. Under-Reinforced Concrete Beams  
Under Long Term Loads. J. Amer. Concrete Inst., Proc. V. 54. 29(1):21-29.

Gant, E. V., J. E. Stephens and L. K. Moulton. 1958. Measurement of Forces  
Produced in Piles by Settlement of Adjacent Soil. Highway Res. Board Bull.  
173:20-37.

Stephens, J. E. and W. H. Goetz. 1960. Designing a Fine Bituminous Mixture  
for High Skid Resistance. Highway Res. Board Proc. 39:173-90.

Stephens, J. E. and W. H. Goetz. 1961. A Study of the Effects of  
Aggregate Factors on Pavement Friction. Highway Res. Board Bull.  
302:1-17.

Stephens, J. E. 1963. Discussion: Field Testing of a Nuclear Density  
Device on Bituminous Concrete. Proc. Assoc. Asphalt Paving Technol.  
32:145.

Stephens, J. E. 1963. Discussion: Control of Asphalt Concrete  
Construction by the Air Permeability Test. Proc. Assoc. Asphalt Paving  
Technol. 32:167-69.

Stephens, J. E. 1963. Discussion: Methods to Eliminate Reflection  
Cracking in Asphalt Concrete Resurfacing over Portland Cement Concrete  
Pavements. Proc. Assoc. Asphalt Paving Technol. 32:226-27.

Stephens, J. E. 1963. Discussion: Asphalt Viscosity as Related to  
Pavement Performance. Proc. Assoc. Asphalt Paving Technol. 32:347-51.

Stephens, J. E. 1963. Discussion: Factors That Control Asphalt  
Requirements of Bituminous Paving Mixtures as a Method for Determining  
the Proper Asphalt Content. Proc. Assoc. Asphalt Paving Technol.  
32:549-51.

Stephens, J. E. 1964. Discussion: The Compaction of Asphaltic Concrete on  
the Road. Proc. Assoc. Asphalt Paving Technol. 33:277-83.

Breen, J. J. and J. E. Stephens. 1966. Split Cylinder Test Applied to  
Bituminous Mixtures at Low Temperatures. ASTM J. Mater. 1(1):66-76.  
Appendix I

- Stephens, J. E. and J. J. Breen. 1966. A Preliminary Report on the Fatigue of a Bituminous Mix at Low Temperatures. Proc. Can. Technol. Asphalt Assoc. 11.
- Stephens, J. E. 1957. Discussion: Statistical Evaluation of Equipment and Operator Effects on Results of Asphalt Extraction Tests. Proc. Assoc. Asphalt Paving Technol. 36:253-56.
- Stephens, J. E. 1967. Discussion: Symposium on Compaction of Asphalt Concrete. Proc. Assoc. Asphalt Paving Technol. 36:357-67.
- Stephens, J. E. 1967. Discussion: Thermal Expansion and Contraction Characteristics of Utah Asphaltic Concretes. Proc. Assoc. Asphalt Paving Technol. 36:700-02.
- Stephens, J. E. and J. J. Breen. 1967. The Glass Transition Temperature and the Mechanical Properties of Asphalt. Proc. Can. Technol. Asphalt Assoc. 12.
- Stephens, J. E. 1968. Application of Statistical Principles to Quality Control and Acceptance of Aggregates. Proc. Assoc. Asphalt Paving Technol. 37:265-73.
- Stephens, J. E. and J. J. Breen. 1969. The Interrelationship Between the Glass Transition Temperature and Molecular Characteristics of Asphalt. Proc. Assoc. Asphalt Paving Technol. 39:706-12.
- Stephens, J. E. and K. Sinha. 1978. Effect of Aggregate Shape on Bituminous Mix Character. Proc. Asso. Asphalt Paving Techn. 47.
- Stephens, J. E. 1982. Field Evaluation of Rubber-Modified Bituminous Concrete. Transportation Research Record 843:11-21.
- Stephens, J. E. and G. E. Hoag. 1984. Volatiles in MC-3000 Bituminous Paving Mixtures. Asphalt Paving Technology, Vol. 53, pp. 186.

#### Technical Reports

- Stephens, J. E. 1966. Reduction of Apparent Aggregate Variation Through Improved Sampling. Rep. No. JHR 66-1. Civil Eng. Dept., University of Connecticut.
- Breen, J. J. and J. E. Stephens. 1966. Fatigue and Tensile Characteristics of Bituminous Pavements at Low Temperatures. Rep. No. JHR 66-3. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E., J. J. Breen, R. F. Dawson and K. C. Sinha. 1966. A Study of the Incorporation of Protective Construction and Slanting Techniques in an Urban Planning Project. Rep. No. CE 66-9. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E. and G. J. Gromko. 1967. An Investigation of the Brittle-Plastic Behavior of Asphalt Mixtures by Use of an Impact Device. Rep. No. CE 67-12. Civil Eng. Dept., University of Connecticut. (Also published in: Highway Res. Record No. 256. pp. 22-36.)
- Stephens, J. E. 1973. Final Report. Bituminous Mix Density by Coated Specimen. Project 67-5. Civil Eng. Dept., University of Connecticut.
- Appendix I

- Stephens, J. E. 1973. Final Report. Rainfall and Runoff Study of Small Drainage Areas. Project 51-2. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E. and S. Mokrzewski. 1974. The Effect of Reclaimed Rubber on Bituminous Concrete Pavement Mixtures. Rep. No. JHR 74-75. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E., R. Fitch and L. B. Shih. 1974. Hot White Traffic Paint—Laboratory and Summer Condition Study. Rep. No. JHR 74-76. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E. 1974. Effect of Aggregate Shape on Bituminous Mix Character. Project 63-9 Final Report. Report No. JHR 74-87. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E., T. Pastor and J. Pelleccione. 1976. Split Cylinder Test for Tension Strength of Concrete. Project 75-2 Final Report. Report No. JHR 76-97. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E., T. Pastor and J. F. Pelliccione. 1977. Statistical Analysis Applied to Management Decisions. Project 75-4 Final Report. Report No. JHR 77-104. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E. 1979. Measurement of Bridge Deck Status by Dynamic Modulus. JHRAC Report No. JHR 79-129. Civil Eng. Dept., University of Connecticut.
- Stephens, J. E. 1981. Recycled Rubber in Roads, Final Report. Report No. CE 81-138 Civil Eng. Dept., University of Connecticut.
- Stephens, J. E. 1982. Cost of Deferring Highway Maintenance. Report to Transportation Committee, Connecticut General Assembly, Connecticut Academy of Science and Engineering, 410 Asylum Street, Hartford, CT.
- Stephens, J. E. and Hoag, G. E. Volatiles in MC-3000 Bituminous Paving Mixtures, Final Report Project 81-1, JHRAC Report No. JHR 82-143, Civil Eng. Dept., University of Connecticut.
- Stephens, J. E., D. W. Sundstrom and H. E. Klei. 1983. The Addition of Lignin from Gasohol Plants to Asphalts, Final Report. Project 80-3, JHRAC Report JHR 83-149.
- Stephens, J. E., C. F. Davis and M. F. Makuch. 1984. An Analysis of Highway Pavement Deterioration and a General Technique for Optimization of Maintenance/Deconstruction, Final Report. Project 82-4, JHRAC Report JHR 84-156.
- Stephens, J. E., Rivenburgh, J. An Evaluation of Aging of Recycled Asphalt, Final Report Project 83-3, JHRAC Report No. JHR 86-166, Civil Engineering Dept., University of Connecticut.
- Stephens, J. E. 1987. Waste Conversion for Public Construction, Report to Connecticut General Assembly, Connecticut Academy of Science and Engineering, 410 Asylum Street, Hartford, CT.
- Stephens, J. E. 1989. Nine Year Evaluation of Recycled Rubber in Roads, Final Report. Project 86-8, JHRAC Report JHR 89-183.

Stephens, J. E., C. F. Davis and H. H. Ridgeway. Further Developments of Optimization Procedures in Pavement Management, Final Report. Project 83-1, JHRAC Report 89-186.

Stephens, J. E. 1990. The Effects of Temperature Cycles on the Aging of Bituminous Concrete, Final Report. Project 86-9, JHRAC Report 90-190.

Stephens, J. E. and W. Santosa. 1992. Aging of Bituminous Concrete, Final Report. Project 87-5, JHRAC Rep No. JHR 92-211. Civil Eng. Dept.



**James Mahoney**  
200 West Road, Apt 115  
Ellington, CT 06029  
(H) 860-870-5959 (W) 860-486-5956

**PROFESSIONAL  
EXPERIENCE**

**Laboratory Manager**, Connecticut Advanced Pavement Laboratory, University of Connecticut, March 1996 - Present. Manage daily lab operations, including: lab construction; Strategic Highway Research Program (SHRP) testing equipment setup; training; supervision of student labor. Attended SHRP training short courses sponsored by the Federal Highway Administration.

**Adjunct Professor**, University of Connecticut - Torrington Branch, Fall 1995 - Present. Classes taught include Statics and Dynamics.

**Computer Consultant**, Cornwall Planning Group, 1993 - Present. Research recommend and secure new equipment, maintain computer operations, and development of database format and specialized business applications.

**Vice President**, James J. Torrant, Inc., 1987 - 1996. Performed wide range of management responsibilities including: work schedules; extensive public interface; supervising field work, equipment repairs, computerization of operations, and accounting functions.

**EDUCATION**

**Master of Science; Geotechnical Engineering**, University of Connecticut, August 1995. Thesis research included the development and implementation of a laboratory testing program.

**Bachelor of Science; Civil Engineering**, University of Connecticut, May 1993.

**AWARDS -  
RECOGNITION**

E. Russell Johnston, Jr. Award, 1992. Excellence in Civil Engineering Award; Top ranked Junior Civil Engineering student.

Tau Beta Pi, National Engineering Honor Society, Inducted in 1992.

Chi Epsilon, National Civil Engineering Honor Society, Inducted in 1992.

**PROFESSIONAL  
ORGANIZATION**

Member, American Society of Civil Engineers, 1991 - Present.

## Appendix II

### Round Robin 98 Report Performed in Conjunction with NECEPT

# 1998 NORTHEAST DSR-BBR ROUND ROBIN

## A JOINT EFFORT BETWEEN:

North East Center for Excellence for Pavement Technology  
Regional Pooled Fund Study Work Order 18, Task R3

New England Transportation Consortium  
Superpave Implementation Project 96-1  
Connecticut Advanced Pavement Laboratory

Workshops at both Penn State and UConn.  
Preliminary statistics and slides by CAP Lab

Round Robin distribution carried out jointly

Statistical Analysis by NECEPT

Draft Report prepared by NECEPT, assisted by CAP Lab

Personnel: D. A. Anderson  
J. Mahoney  
M. O. Marasteanu  
J. Stephens

Final Report expected by late summer.

# DRAFT

## TABLE OF CONTENTS

		Page
1.	INTRODUCTION.....	1
	Background .....	1
	Scope of this Report .....	2
2.	MATERIALS AND TESTING PROGRAM .....	3
	Materials.....	3
	Testing Program .....	3
	Test Results .....	4
3.	STATISTICAL ANALYSIS .....	5
4.	CONCLUSIONS AND RECOMMENDATIONS .....	19
5.	REFERENCES .....	21
7.	APPENDIX A Round Robin Announcement.....	23
8.	APPENDIX B Testing Program.....	25
9.	APPENDIX C Round Robin Test Results.....	31
10.	APPENDIX D Plots of Round Robin Test Results .....	55
11.	APPENDIX E Slides Used in Dr. Jack Stephens' Presentation .....	91
12.	APPENDIX F Statistical Analysis Details .....	101

## LIST OF FIGURES

	Page
Figure D1. Rotational viscosity data for unaged PSU1 and PSU4 at 135°C.....	56
Figure D2. Rotational viscosity data for unaged PSU1 and PSU4 at 165°C.....	57
Figure D3. Rotational viscosity data for unaged PSU2 and PSU3 at 135°C.....	58
Figure D4. Rotational viscosity data for unaged PSU2 and PSU3 at 165°C.....	59
Figure D5. G* data for unaged PSU1 and PSU4 at 70°C.....	60
Figure D6. G* data for unaged PSU1 and PSU4 at 76°C.....	61
Figure D7. G* data for unaged PSU2 and PSU3 at 64°C.....	62
Figure D8. G* data for unaged PSU2 and PSU3 at 70°C.....	63
Figure D9. G* data for RTFOT PSU1 and PSU4 at 70°C .....	64
Figure D10. G* data for RTFOT PSU1 and PSU4 at 76°C .....	65
Figure D11. G* data for RTFOT PSU2 and PSU3 at 64°C .....	66
Figure D12. G* data for RTFOT PSU2 and PSU3 at 70°C .....	67
Figure D13. Stiffness data for PAV PSU1 and PSU4 at -12°C.....	68
Figure D14. m-value data for PAV PSU1 and PSU4 at -12°C.....	69
Figure D15. Stiffness data for PAV PSU2 and PSU3 at -12°C.....	70
Figure D16. m-value data for PAV PSU2 and PSU3 at -12°C.....	71
Figure D17. Stiffness data for PAV PSU1 and PSU4 at -18°C.....	72
Figure D18. m-value data for PAV PSU1 and PSU4 at -18°C.....	73
Figure D19. Stiffness data for PAV PSU2 and PSU3 at -18°C.....	74
Figure D20. m-value data for PAV PSU2 and PSU3 at -18°C.....	75
Figure D21. Stiffness data for pre-PAV 1 and 4 at -18°C.....	76
Figure D22. m-value data for pre-PAV 1 and 4 at -18°C.....	77
Figure D23. Stiffness data for pre-PAV 2 and 3 at -18°C.....	78
Figure D24. m-value data for pre-PAV 2 and 3 at -18°C.....	79

Figure D25. Strain sweeps $G^*$ data for unaged PSU1 .....	80
Figure D26. Strain sweeps phase angle data for unaged PSU1 .....	81
Figure D27. Strain sweeps $G^*$ data for unaged PSU2.....	82
Figure D28. Strain sweeps phase angle data for unaged PSU2.....	83
Figure D29. Strain sweeps $G^*$ data for unaged PSU3.....	84
Figure D30. Strain sweeps phase angle data for unaged PSU3 .....	85
Figure D31. Strain sweeps $G^*$ data for unaged PSU4.....	86
Figure D32. Strain sweeps phase angle data for unaged PSU4.....	87
Figure D33. Complex viscosity data for the reference fluid at 64°C .....	88
Figure D34. Complex viscosity data for the reference fluid at 76°C .....	89
Figure D35. Complex viscosity versus capillary viscosity for the reference fluid at 64°C.....	90

## LIST OF TABLES

	Page
Table 3.1. Variance components and corresponding coefficients of variation obtained in the round robin.....	9
Table 3.2. Summary of D1S and D2S values obtained in the round robin.....	12
Table 3.3. Outliers omitted in the statistical analysis performed in the round robin.....	15
Table C.1. Rotational viscosity data.....	32
Table C.2. G* test results for the unaged asphalt binder samples. ....	33
Table C.3. Phase angle test results for the unaged asphalt binder samples. ....	34
Table C.4. G* test results for the RTFOT asphalt binder samples. ....	35
Table C.5. Phase angle test results for the RTFOT asphalt binder samples. ....	36
Table C.6. Stiffness test results at -12°C for the PAV asphalt binder samples. ....	37
Table C.7. m-value test results at -12°C for the PAV asphalt binder samples. ....	39
Table C.8. Stiffness test results at -18°C for the PAV asphalt binder samples. ....	41
Table C.9. m-value test results at -18°C for the PAV asphalt binder samples. ....	43
Table C.10. Stiffness test results at -18°C for the pre-PAV asphalt binder samples.....	45
Table C.11. m-value test results at -18°C for the pre-PAV asphalt binder samples.....	47
Table C.12. Complex viscosity test results for the reference fluid.....	49
Table C.13. G* strain sweeps test results for the unaged asphalt binder samples.....	50
Table C.14. Phase angle strain sweeps test results for the unaged asphalt binder samples.....	52





## 1. INTRODUCTION

### Background

In 1996 a round robin was held in the Northeast to evaluate the repeatability and reproducibility of dynamic shear rheometer (DSR) measurements. This round robin is described in detail elsewhere.<sup>(1)</sup> From this round robin it was concluded that both the repeatability and reproducibility for the DSR were unsatisfactory for specification purposes, regardless of the manufacturer, and that improvements in both the repeatability and reproducibility of the DSR measurements were needed.<sup>(1)</sup>

Rather than simply conduct a second round robin as a follow-up to the first round robin, it was decided that an effort should be made to identify current testing practice and to improve, if possible, that practice. Therefore, prior to a second round robin, two Binder Technician Workshops were held in January of 1998, one at the CAP Lab at the University of Connecticut and one at NECEPT at Penn State University. At these binder technician workshops the techniques and procedures used by the laboratories in the northeast were reviewed. A detailed description of the two two Binder Technician Workshops is given elsewhere.<sup>(2)</sup>

Following the two workshops samples were sent to the workshop participants as part of the second round robin (see Appendix A for round robin announcement). The main objective of this round robin was to provide support for NECEPT work plan R-6 that addresses binder QC/QA issues. As a consequence, the round robin included specification testing for both DSR and BBR, RTFOT aging followed by PAV aging, as well as tests on the DSR reference fluid, DSR strain sweeps, and rotational viscometer testing.

Twenty laboratories, including state agencies, asphalt binder suppliers, and private laboratories participated in this round robin. A number of the participants were not able to send a complete set of information due to equipment limitations and to busy schedules. Nevertheless, the statistical procedure used with the test data allowed for a robust analysis of the results.

David Anderson, Mihai Marasteanu, and Charles Antle developed the testing schedule and the instructions sent to the round robin participants. Mihai Marasteanu was responsible for creating the Excel files sent to the participants for data input. Mihai Marasteanu and Jim Mahoney were responsible for organizing the round robin data for further analysis. Charles Antle, Mihai Marasteanu, and David Anderson were responsible for the statistical analysis. Jack Stephens and Mihai Marasteanu made presentations on the round robin results at the workshops held at NECEPT and CAP Lab in January 1999.

### **Scope of This Report**

This report documents the development and the results of the second round robin held in the northeast in spring-summer 1998. Specifically, this report includes:

1. Instructions and testing schedule sent to participants.
2. Test results.
3. A commentary of the test results and the statistical analysis of the round robin results.
4. Conclusions and recommendations.

The chapters that follow present these topics in detail. Supporting information is contained in the appendices to this report.

## **2. MATERIALS AND TESTING PROGRAM**

As mentioned in the previous chapter, the main objective of this round robin was to provide support for NECEPT work plan R-6 that addresses binder QC/QA issues. As a consequence, the round robin included specification testing for both DSR and BBR, RTFOT aging followed by PAV aging, as well as tests on the DSR reference fluid, DSR strain sweeps, and rotational viscometer testing.

### **Materials**

Two sets of asphalt binders were sent to the participants. The first set contained four asphalt binder samples in the unaged condition. The four samples were labeled PSU1, PSU2, PSU3, and PSU4. However, PSU1 was identical with PSU4 and PSU2 was identical with PSU3. The asphalt binder in the PSU2 and PSU3 samples was a plain PG 64 – 22, while the asphalt binder in the PSU1 and PSU4 samples was a modified PG 70 – 22. Each participant received enough material to be able to perform a complete MP1 set of tests.

The second set contained four asphalt binders that have been previously aged in the PAV at the CAPLab facility. The four samples were labeled 1, 2, 3, 4, but similar to the previous set, samples 1 and 4 were identical and samples 2 and 3 were identical. These samples were used only for BBR testing at – 18°C.

### **Testing Program**

The testing program included specification testing for DSR including strain sweeps, BBR testing, tests on the DSR reference fluid, and rotational viscometer testing. The 8-mm parallel plate testing was not included in the round robin. They will be soon replaced with the 12-mm plates and therefore there was no benefit to include additional testing in the round robin. A detailed description of the testing program is given in Appendix B. No other special testing instructions were given; it was assumed that each laboratory was familiar with and used the current AASHTO test methods.

## Test Results

Each of the participating laboratories received, in addition to the samples to be tested and the testing program, a floppy disk with specially formatted Excel files to input the test results. This approach simplified considerably the handling of the large volume of data received from the participants. The test results from the participating laboratories are tabulated in Appendix C. The identification of the participants was coded and each participant received only their own individual code number. Plots of the test results are given in Appendix D. Additional interpretation of the test results is given in Dr. Jack Stephens' presentation displayed in Appendix E. The tabulated data was further used to calculate the variability within and between laboratories. A detailed description of the statistical analysis performed in this study is provided in the next chapter.

### 3. STATISTICAL ANALYSIS

The testing program described in chapter 2 consisted of several separate experiments performed on each of the materials sent to the participants. Each of these experiments were by design nested experiments. The analyses of the resulting data from these experiments were performed using the Nested Procedure in the SAS statistical package. The SAS procedure provided estimates for the appropriate variance components that were the basis for the evaluation of the sources of variation which occur with each of the measurement processes. This approach is detailed in the subsequent paragraphs.

Two models were used for the analysis of variance. The first model was used with the data obtained on the asphalt binder samples for which aging was not required:

$$Y(i,j) = \mu + \text{LAB}(i) + \text{ERR}(i,j) \quad (1)$$

where

$Y(i,j)$  = the Y value measured by laboratory i using sample j

$\mu$  =  $\mu$  is the overall mean for  $Y(i,j)$

$\text{LAB}(i)$  = the laboratory effect for laboratory i in the study. This is the laboratory bias at laboratory i under the conditions that were present when these measurements were made. This bias may well depend upon conditions as well as materials. Thus, a separate analysis was done for each material. The  $\text{LAB}(i)$  effects (bias) are assumed to be normal random variables from some population with mean of zero and variance of  $\text{VAR}(\text{LAB})$ . The  $\text{VAR}(\text{LAB})$  or the coefficient of variation due to the Lab,  $\text{CV}(\text{Lab})$ , is needed to establish a bias statement.

$\text{ERR}(i,j)$  = the final error component in the measured values and it is often called the experimental error. It is better described as the within laboratory measurement error. It is assumed to be from a normal population with mean of zero and variance of  $\text{VAR}(\text{ERR})$  with corresponding coefficient of variation of  $\text{CV}(\text{ERR})$ . The  $\text{CV}(\text{ERR})$  is for a single measured value of a given item. The standard deviation, which is equal to the square root of the  $\text{VAR}(\text{ERR})$ , is the within laboratory D1S, an estimate of the "repeatability" used in the precision and bias

statement.

The second model was used with the data obtained on the asphalt binder samples for which aging was required:

$$G^*(i,j,k) = \mu + LAB(i) + AGING(i,j) + ERR(i,j,k) \quad (2)$$

where

- $Y(i,j,k)$  = the Y value at LAB i using material from AGING run j (at LAB i) with sample k (from AGING run j in LAB
- $\mu$  =  $\mu$  is the overall mean for  $Y(i,j, k)$
- $LAB(i)$  = same as in the first model
- $AGING(i,j)$  = is the effect of run j of AGING at laboratory i and is assumed to also be a normal random variable with mean of zero and variance of  $VAR(AGING)$  with corresponding coefficient of variation of  $CV(AGING)$ . The  $CV(AGING)$  or coefficient of variation due to replicate aging runs is needed in order to establish a precision and bias statement.
- $ERR(i,j,k)$  = same as in the first model.

A summary of the estimated variance components obtained by applying the two models described above to the test data is given in table 3.1. Detailed descriptions of each of the analysis of variance performed in this study are given in Appendix F. Table 3.1 also includes the corresponding coefficients of variation for the variance components, which are needed to make judgements and recommendations. The coefficients of variation were calculated as a percentage of the overall mean for the measurement of interest. Some important observations can be made based on the results summarized in table 3.1. For the DSR based specification,  $G^*$  dictates the amount of variation observed in  $G^*/\sin\delta$ . The phase angle  $\delta$  has a very small coefficient of variation, typically less than 0.5%, and has almost no influence on the variability of the specification values. An interesting trend is noticed in the DSR results on the aged materials. For asphalt binders PSU1 and PSU4 (identical) the coefficients of variation due to laboratory effects, at both test temperatures, were lower than the coefficients of variation due to aging. The opposite situation is noticed for PSU2 and PSU3 (identical) for which the aging effects are lower than the laboratory effects. This trend is not noticed in the BBR data. However, the BBR stiffness data on PAV-aged materials revealed a different trend. At the higher test temperatures, characterized by smaller stiffness values, the laboratory effects are significantly lower than the laboratory effects at the lower temperatures characterized

by higher stiffness values. This observation is valid for both PSU1&PSU4 and PSU2&PSU3. For the pre-PAV materials the laboratory coefficients of variation were similar with the coefficients of variation for the PAV materials at the lower temperature. This suggests that the aging process, including both RTFOT and PAV, is very repeatable from one laboratory to another.

The coefficients of variation that correspond to the appropriate sources of variation provide the basis for the estimation of the D1S and D2S for both the within laboratory (repeatability) and between laboratories (reproducibility) sources of variability. In all cases the within laboratory D1S is simply the within laboratory standard deviation, or if a percentage D1S is desired, it is the error coefficient of variation CV(ERR). This represents the best estimate of the standard deviation for the repeated measurements on a given material at a given laboratory. If we have two such measured values for the same material at a given laboratory, then the difference of the two measured values will be a random variable with mean zero and a standard deviation of 1.414 D1S. It follows that this difference in absolute value should be less than 2.8 D1S, with a probability of 95%. This value of 2.8D1S represents the within laboratory D2S value.

The measures for the reproducibility, i.e., the between laboratories D1S and D2S are not so simply defined. The D1S in this case is the standard deviation of a random measurement of a given material if we choose a laboratory at random, then choose an AGING run at random (if any aging is included), then perform the measurement on the sample. This entire process is to be repeated so that the set of such values will have different laboratory effects, different AGING effects (if aging is required), and different measurement effects. This standard deviation is a good indicator of the variation to be expected when measured values are obtained from various laboratories. If two identical materials are sent to two different laboratories, and each of these then age the samples and then measure a property of interest, the difference in these two measurements should be less than 2.8D1S, with a probability of 95%. This value of 2.8D1S represents the between laboratories D2S value. A summary of the D1S and D2S values obtained in this study are given in table 3.2.

It should be mentioned here that the data from some laboratories were deleted from the analyses when it became clear that their inclusion would have distorted the results in an unreasonable manner. Engineering judgement and consideration of means and standard deviations were used to select the outliers that were omitted in the statistical calculations. All such omissions were noted in the extensive statistical analyses presented in Appendix F. A summary of the laboratories that have data removed as outliers is given in table 3.3. Please note that in the case of the DSR test data if one laboratory had its G\* results

removed as outliers its  $G^*/\sin\delta$  results would be also removed as outliers. Discussions with the affected laboratories made it clear that the data in these cases were not at all representative of their ability to carry out these tests in the future. These extremes do, however, add to the evidence that there must be some means for each laboratory to evaluate their own performance carefully in the future, with or without the aid of outside help.

One of the means available for monitoring performance on the DSR 25-mm parallel plate testing is Cannon Instrument Company viscosity standard (reference fluid) N2700000SP. The fluid, a polybutene, is considered to be Newtonian at 52°C and above, and as a consequence the viscosity written on the bottle is approximately equal to the complex modulus  $G^*$  divided by the measurement frequency in radians per second,  $\eta = |G^*|/\omega$ . The fluid is linear over a wide range of strains and is significantly less temperature-dependent than asphalt binders. This makes the fluid a good tool for verifying the calibration of a rheometer, provided that the temperature measuring system of the rheometer is first properly calibrated.

In this round robin, the reference fluid was tested at two temperatures, 64°C and 76°C, using a combination of two strain levels, 1% and 10%, and two frequencies, 1rad/s and 10rad/s. Each test was replicated once. The results are summarized in figures 3.1 and 3.2 in Appendix D. The statistical analysis performed on the reference fluid data indicated that at both test temperatures laboratory, replicate, frequency, and strain level had significant effects on the reference fluid data. The effects were stronger for laboratory, replicate and frequency, which were characterized by p-values of 0.0001, than the strain effect characterized by a p-value of 0.05. However, this conclusion should be interpreted with caution as only 5 laboratories sent complete information for the reference fluid testing. In addition, due to the limited amount of test data available no outliers were eliminated from the set of data. Figure 3.3 in Appendix D shows the percent differences of the results from the viscosity value of 264.5 Pa.s at 64°C. No reference fluid viscosity value is given on the bottle for 76°C.

The strain sweeps performed in this round robin did not indicate any significant strain dependency for the complex modulus and the phase angle. The results did indicate however increased levels of testing noise, especially in the phase angle data. The noise is associated with the performance of the equipment rather than tested material properties. The strain sweeps test results are displayed in graphic format in Appendix D.



Table 3.1. Variance components and corresponding coefficients of variation obtained in the round robin.

Table 3.1. Variance components and corresponding coefficients of variation obtained in the aging tests										
Condition	Test	Parameter	Sample	Temperature	Mean	STD		CV%		
						Lab	Aging	Error	Lab	Aging
Tank	RV	Viscosity	PSU1&PSU4	135°C	1014	71.7		26.3	7.1%	2.6%
				165°C	285	38.0		9.6	13.4%	3.4%
			PSU2&PSU3	135°C	450	38.7		9.7	8.6%	2.2%
				165°C	126	13.6		5.0	10.8%	4.0%
	G*	PSU1&PSU4	70°C	1395	82.2		51.1	5.9%	3.7%	
			76°C	765	43.2		28.4	5.6%	3.7%	
		PSU2&PSU3	64°C	1458	111.8		55.4	7.7%	3.8%	
			70°C	702	44.3		29.1	6.3%	4.1%	
	DSR	PSU1&PSU4	70°C	75.9	0.336		0.798	0.4%	1.1%	
			76°C	78.0	0.478		0.343	0.6%	0.4%	
		PSU2&PSU3	64°C	86.4	0.410		0.281	0.5%	0.3%	
			70°C	87.8	0.735		0.274	0.8%	0.3%	
	G* /sin δ	PSU1&PSU4	70°C	1439	86.8		54.5	6.0%	3.8%	
			76°C	782	44.1		29.4	5.6%	3.8%	
		PSU2&PSU3	64°C	1462	101.2		55.1	6.9%	3.8%	
			70°C	702	44.4		29.2	6.3%	4.2%	

Table 3.1. Variance components and corresponding coefficients of variation obtained in the round robin, cont'd.

Condition	Test	Parameter	Sample	Temperature	Mean	STD		CV%	
						Lab	Aging	Lab	Aging
RTFOT	DSR	G*	PSU1&PSU4	70°C	3299	95.7	248	2.9%	7.5%
				76°C	1784	68.9	150.1	3.9%	8.4%
			PSU2&PSU3	64°C	3313	184.9	136.5	5.6%	4.1%
				70°C	1555	105.2	55.9	6.8%	3.6%
		$\delta$	PSU1&PSU4	70°C	71.0	0.329	0.423	0.5%	0.6%
				76°C	73.2	0.341	0.456	0.5%	0.6%
			PSU2&PSU3	64°C	82.8	0.526	0.159	0.6%	0.2%
				70°C	84.8	0.608	0.129	0.7%	0.2%
		G* /sin $\delta$	PSU1&PSU4	70°C	3490	106.1	270.8	3.0%	7.8%
				76°C	1863	73.5	160.3	3.9%	8.6%
			PSU2&PSU3	64°C	3339	188.7	138.7	5.7%	4.2%
				70°C	1561	106.4	56.53	6.8%	3.6%

Table 3.1. Variance components and corresponding coefficients of variation obtained in the round robin, cont'd.

Condition	Test	Parameter	Sample	Temperature	Mean	STD			CV%		
						Lab	Aging	Error	Lab	Aging	Error
PAV	BBR	S	PSU1&PSU4	-12°C	134	8.3	6.2	4.6	6.2%	4.6%	3.4%
				-18°C	279	28.2	7.1	10.7	10.1%	2.5%	3.8%
			PSU2&PSU3	-12°C	149	6.3	5.9	6.2	4.2%	4.0%	4.2%
				-18°C	323	31.3	8.6	11.1	9.7%	2.7%	3.4%
		m	PSU1&PSU4	-12°C	0.327	0.0081	0.0052	0.0057	2.5%	1.6%	1.7%
				-18°C	0.281	0.0094	0.0032	0.0041	3.3%	1.1%	1.5%
			PSU2&PSU3	-12°C	0.351	0.0104	0.0037	0.0070	3.0%	1.0%	2.0%
				-18°C	0.293	0.0148	0.0039	0.0066	5.1%	1.3%	2.2%
PRE-PAV	BBR	S	1&4	-18°C	164	16.15		7.348	9.8%		4.5%
			2&3		297	32.26		9.889	10.9%		3.3%
		m	1&4	-18°C	0.352	0.0131		0.0058	3.7%		1.7%
			2&3		0.310	0.0173		0.0071	5.6%		2.3%

Table 3.2. Summary of D1S and D2S values obtained in the round robin.

Condition	Test	Parameter	Sample	Temperature	Mean	Within		Between	
						D1S	D2S	D1S	D2S
Appendix II	RV	Viscosity	PSU1&PSU4	135°C	1013.6	2.6%	7.3%	7.5%	21.3%
				165°C	284.5	3.4%	9.5%	13.8%	39.0%
			PSU2&PSU3	135°C	450.2	2.2%	6.1%	8.9%	25.1%
				165°C	126.2	4.0%	11.2%	11.5%	32.5%
	[G*]		PSU1&PSU4	70°C	1394.8	3.7%	10.4%	6.9%	19.6%
				76°C	765.1	3.7%	10.5%	6.8%	19.1%
			PSU2&PSU3	64°C	1457.6	3.8%	10.7%	8.6%	24.2%
				70°C	702.1	4.1%	11.7%	7.5%	21.3%
	DSR	$\delta$	PSU1&PSU4	70°C	75.9	1.1%	3.0%	1.1%	3.2%
				76°C	78.0	0.4%	1.2%	0.8%	2.1%
			PSU2&PSU3	64°C	86.4	0.3%	0.9%	0.6%	1.6%
				70°C	87.8	0.3%	0.9%	0.9%	2.5%
Tank			PSU1&PSU4	70°C	1438.6	3.8%	10.7%	7.1%	20.1%
				76°C	782.1	3.8%	10.6%	6.8%	19.2%
			PSU2&PSU3	64°C	1461.6	3.8%	10.7%	7.9%	22.3%
				70°C	701.8	4.2%	11.8%	7.6%	21.4%

Table 3.2. Summary of D1S and D2S values obtained in the round robin, cont'd.

Condition	Test	Parameter	Sample	Temperature	Mean	Within		Between	
						D1S	D2S	D1S	D2S
RTFOT	DSR	G*	PSU1&PSU4	70°C	3298.5	4.4%	12.5%	9.2%	26.0%
				76°C	1783.6	3.7%	10.3%	10.0%	28.2%
			PSU2&PSU3	64°C	3312.8	5.1%	14.4%	8.6%	24.3%
				70°C	1554.5	4.4%	12.4%	8.8%	25.0%
		$\delta$	PSU1&PSU4	70°C	70.98	0.6%	1.6%	0.9%	2.7%
				76°C	73.24	0.5%	1.5%	1.0%	2.7%
			PSU2&PSU3	64°C	82.79	0.2%	0.6%	0.7%	2.0%
				70°C	84.8	0.2%	0.7%	0.8%	2.2%
		G* /sin $\delta$	PSU1&PSU4	70°C	3490.2	4.6%	13.0%	9.5%	26.9%
				76°C	1863.1	3.8%	10.7%	10.2%	28.8%
			PSU2&PSU3	64°C	3338.8	5.1%	14.4%	8.7%	24.5%
				70°C	1560.9	4.4%	12.4%	8.9%	25.1%

Table 3.2. Summary of D1S and D2S values obtained in the round robin, cont'd.

Condition	Test	Parameter	Sample	Temperature	Mean	Within		Between	
						D1S	D2S	D1S	D2S
PAV	BBR	S	PSU1&PSU4	-12°C	134.4	3.4%	9.7%	8.4%	23.9%
				-18°C	279.3	3.8%	10.8%	11.1%	31.4%
			PSU2&PSU3	-12°C	149.0	4.2%	11.8%	7.1%	20.2%
				-18°C	322.6	3.4%	9.7%	10.6%	30.1%
		m	PSU1&PSU4	-12°C	0.327	1.7%	4.9%	3.4%	9.7%
				-18°C	0.281	1.5%	4.1%	3.8%	10.8%
			PSU2&PSU3	-12°C	0.351	2.0%	5.6%	3.7%	10.5%
				-18°C	0.293	2.2%	6.3%	5.7%	16.1%
PRE-PAV	BBR	S	1&4	-18°C	164.26	4.5%	12.7%	10.8%	30.5%
			2&3		296.540	3.3%	9.4%	11.4%	32.2%
		m	1&4	-18°C	0.352	1.7%	4.7%	4.1%	11.5%
			2&3		0.310	2.3%	6.5%	6.0%	17.1%

Table 3.3. Outliers omitted in the statistical analysis performed in the round robin.

Condition	Test	Parameter	Sample	Temperature	Laboratory identification number																					
					1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20		
	RV	Viscosity	PSU1&PSU4	135°C																X						
				165°C																						
			PSU2&PSU3	135°C																		X				
				165°C																				X	X	
		$ G^* $	PSU1&PSU4	70°C														X								
				76°C															X			X				
			PSU2&PSU3	64°C																X	X					
				70°C																X	X					
Tank	DSR	$\delta$	PSU1&PSU4	70°C																X						
				76°C																X	X					
			PSU2&PSU3	64°C																	X					
				70°C																X						
		$ G^* /\sin \delta$	PSU1&PSU4	70°C															X							
				76°C																X	X					
			PSU2&PSU3	64°C																	X	X				
				70°C																	X	X				







#### 4. CONCLUSIONS AND RECOMMENDATIONS

The round robin held in the northeast in 1998 provided an extensive set of data that was statistically analyzed to identify and quantify the sources of variability in the testing of asphalt binders. The experimental design of the round robin made possible the analysis of the contributions of both RTFOT and PAV aging process in addition to the laboratory effects and the effects of the final measuring process.

The magnitude of the various sources of variability and of the resulting D1S and D2S were summarized in chapter 3. A detailed description of the statistical analysis performed is given in Appendix F. A few important comments should be made here. All the components of variance have been estimated with a very large degree of accuracy as evidenced by their large degrees of freedom. This is due to the pooling of this information over the laboratories. That is why particular care was taken to exclude all the unreasonable outliers in the data set so that this pooling would not be affected by the inclusion of bad data points. The laboratory component was estimated with the least accuracy. This is a characteristic of round robin studies in general.

In this study, different asphalt binders were tested at different temperatures. It was very fortunate that when the components of variance were evaluated for different temperatures or different materials it was found that the corresponding coefficients of variance remained quite stable. This is important because the data can be further pooled to provide increased accuracy in the estimation of the components needed in the development and evaluation of QA/QC plans.

The study also indicated that more research is needed to further investigate some of the issues addressed in this study. One issue is the increased laboratory variance component observed in BBR tests where higher stiffness values are measured. Another issue is the effect of frequency and strain level on the reference fluid viscosity measured with the DSR equipment. The statistical analysis indicated that both were significant, but the set of data was very limited due to the reduced number of laboratories that sent data. The testing program proposed in this study should be repeated in order to obtain a complete set of data that would validate if strain level and frequency have a significant effect on the measured reference fluid viscosity.



## REFERENCES

1. *Precision of Dynamic Shear Rheometer in the Northeast Round Robin Study*, NECEPT Regional Pooled Fund Study, PTI Report 9932, May 1999.
2. *Northeast DSR-BBR Workshop*, NECEPT Regional Pooled Fund Study, PTI Report 9930, September 1998.

**APPENDIX A**  
**ROUND ROBIN ANNOUNCEMENT**



# CAPLab

Connecticut Advanced Pavements laboratory

# NEAU/PG

Northeast Asphalt User/Producer Group

## ROUND ROBIN ANNOUNCEMENT

---

April 6, 1998

You are invited to participate in a round robin organized by NECEPT and the Connecticut Advanced Pavements Laboratory (CAPLab). The round robin is a follow up of the two DSR/BBR Test Procedure Workshops held in January. You will find enclosed the asphalt binders to be tested in the round robin, the testing instructions and a floppy disk containing an Excel 5.0 file to input the test results. If you have any questions please contact

Mihai Marasteanu  
NECEPT  
201 Research Office Building  
The Pennsylvania Transportation Institute  
University Park, PA 16802  
Tel. No. (814) 863-8010  
FAX No. (814) 865-3039  
e-mail: mom1@psu.edu

After you finish all your testing, enter the test results in the Excel file contained in the provided floppy disk and return the disk to the above address. We expect to receive the floppy disk with your results between May 15<sup>th</sup> and May 31<sup>st</sup>, 1998.

Thank you for your participation.

**APPENDIX B**  
**ROUND ROBIN TESTING PROGRAM SENT TO PARTICIPANTS**

## 1. MATERIALS AND TESTING SCHEDULE

### 1.1. Reference Fluid

The reference fluid (viscosity fluid) is not supplied with the samples shipped to you. It must be purchased separately from Canon Instrument. The part number for the viscosity standard is: N2700000SP. Canon can be reached at Canon Instrument Company, PO Box 16, State College, PA 16804, Tel: (814) 353-8000, Fax.: (814) 353-8007. When ordering the material, ask for a 55ml container. The fluid should come with a calibration at 64°C (other temperatures, such as 58 and 70°C may be included but they are not needed in this work.) A small amount of viscosity fluid will last for a considerable amount of time and there is a definite expiration date for the fluid. It should not be used beyond the expiration date.

The objective in using the reference fluid is to test at two temperatures, two frequencies, and two strain levels. Because different models and brands of rheometers vary in their sensitivity it is difficult to recommend a common set of test conditions. Try to use the following schedule:

- Mount the reference fluid between the 25-mm plates with the usual sample bulge and start the testing at 64°C and 1 rad/s (if you cannot set your machine for 1.0 rad/s skip to 10 rad/s.)
- Increase the stress or strain until you are in the lower limit of the range of stress or strain that can be reliably measured by the instrument. At this point the strain will likely be 0.5 to 2 percent. This will be your "low strain" measurement.
- Increase the stress or strain by a factor of 10 - 20 and obtain a second reading. This will be your "high strain" measurement.
- With the same sample, increase the frequency to 10 rad/s and repeat the above.
- With the same sample, increase the temperature to 76°C and repeat the steps above.

The fluid is very linear in the 64 to 82°C range. You can vary your strain values from 0.5 to 60 percent when making measurements but also stay within the capabilities of your instrument. Not all instruments can cover this range of strain values when testing the fluid.

If you want to validate the calibration of your rheometer, your measured  $G^*$  value in Pa (at 10 rad/s and 64°C) should be equal to ten times the 64°C viscosity value in Pa-s (printed on the bottle). If the two values disagree by more than 3 to 4 percent the calibration of your DSR may be suspect.



### 1.2. Asphalt Binders for BBR Testing – No Aging Required

You will receive four, three ounce cans of material for BBR testing. They are labeled as 1, 2, 3, and 4. The testing required is shown in Figure 1. Each can contains sufficient material for preparing a set of two or three test beams. From each can, prepare one set of beams for testing at  $-18^{\circ}\text{C}$ . The beams should be poured simultaneously, not at different points in time. If you prepare a set of three beams, report the values from the first two beams tested. Do not test three beams and selectively report the data from two of the beams. Use the third beam in the set only in the event that one of the first two beams break or is otherwise destroyed during the testing operation. If possible, measure the thickness of the two mold end inserts used to cast each beam and enter the average of the two measurements as the thickness of the sample in the BBR software.

### 1.3. Asphalt Binders for Aging – RTFOT and PAV Aging Required

The asphalt binders in the one-quart cans labeled PSU 1 through PSU 4 are for RTFOT aging followed by PAV aging. A flow diagram describing the aging and testing sequence for the asphalt binders in the cans labeled PSU 1 through PSU 4 is shown in the attached figure. The material is to be aged in the RTFOT (use all 8 bottles and mix them together before pouring the material for the PAV and the DSR testing; mass loss is not required in this experiment) followed by aging in the PAV. Use a separate PAV runs for the material in each of the four cans. This will require four separate PAV runs. The schedule of testing for samples PSU1 through PSU4 is as follows:

- Tank material - one viscosity test. Test the tank material in the rotational viscometer at  $135^{\circ}\text{C}$  and  $165^{\circ}\text{C}$  by increasing the temperature from  $135^{\circ}\text{C}$  to  $165^{\circ}\text{C}$ .
  - Tank material - two DSR tests using the 25-mm plate. This means mounting two separate samples in the DSR. The two samples may be tested immediately one after the other.
  - Tank material - one strain sweep. Once the grading temperature has been determined, if your equipment permits, conduct strain sweeps on a sample from each can, PSU1 through PSU4. See Section 1.3.1 for additional directions.
  - RTFOT residue - two DSR tests using the 25-mm plate. Again, this means mounting two separate samples in the DSR. The two samples may be tested immediately one after the other.
  - PAV residue - two BBR beams at  $-12^{\circ}\text{C}$  and two BBR beams at  $-18^{\circ}\text{C}$ .
- \* Note: This round robin does not include any testing with the 8mm DSR plate.

The above testing schedule must be repeated for each of the four cans, PSU1 through PS4. Use 70°C as the starting temperature for PSU1 and PSU4. Use 64°C as the starting temperature for PSU2 and PSU3.

### 1.3.1. Strain Sweeps

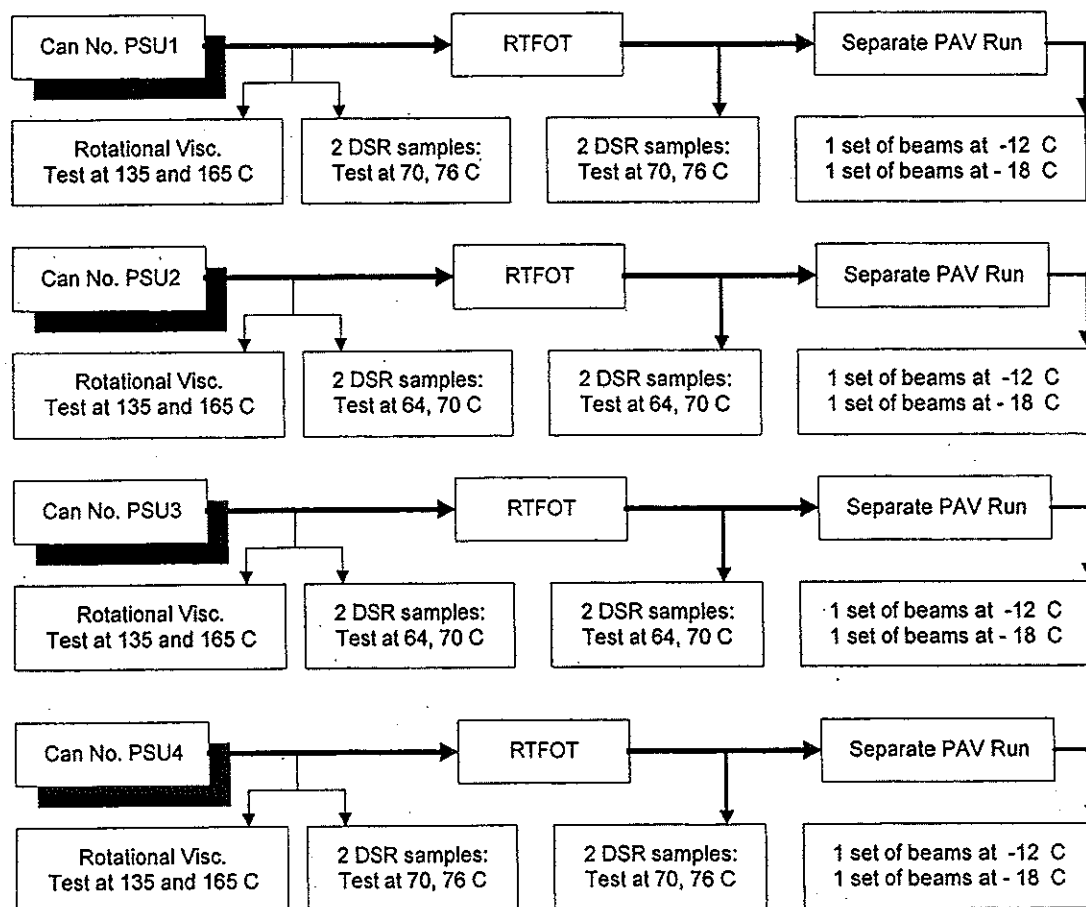
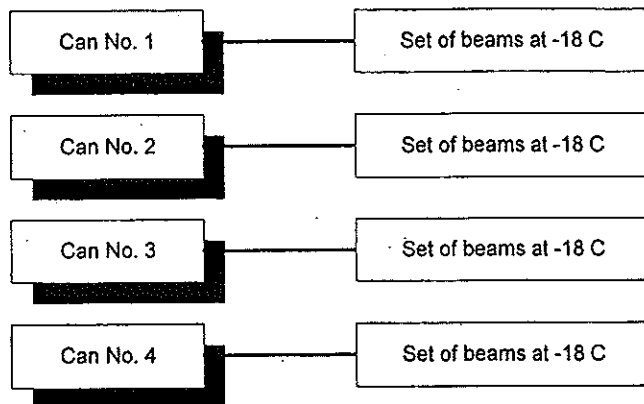
The current version of the AASHTO specification requires strain sweeps (AASHTO Designation: TP5-93, Edition 1C, June 1997). Specific instructions for determining strain sweeps are given in the specification. The strain sweep must be conducted on a newly mounted sample at the grading temperature.

For the purposes of this round robin, conduct one strain sweep (four sweeps in total) on the unaged material from cans PSU1 through PSU 4. The strain sweep is conducted at the grading temperature. A copy of the Annex to the specification that describes how strain sweeps are to be measured is attached. If you do not know how to do a strain sweep please contact your rheometer manufacturer. You may need new software in order to conduct the strain sweep. If you are unable to conduct strain sweeps, proceed with the other testing and omit the strain sweeps.

## 2. REPORTING OF DATA

A 3.5 in floppy disk is included with this instruction sheet or with the test samples. The floppy disk includes an Excel spreadsheet file for entering the data. The worksheets within the file have been protected so that you do not have to worry about changing the format. Please enter the data as directed on the spreadsheet. The floppy is to be returned to the address printed on the floppy. Do not try to reformat the file. If the file format does not fit the data, call Jim at 860-486-5956 (UConn) or Mihai at 814-863-8010 (NECEPT).

In general, all data should be reported to three significant digits, e.g. 64.1°C, 981, 0.000675, etc. The only exception is the  $R^2$  for the BBR, which should be entered as per the data sheet for your BBR.



Note: In the above, "2 DSR samples" means to mount two separate samples in DSR and test one after other. A "set of beams" means two BBR beams molded at same time and tested one after the other.



**APPENDIX C**  
**ROUND ROBIN TEST RESULTS**

Table C1. Rotational viscosity data.

Can no.	Test Temp °C	1	2	3	4	5	6	7	9	8	10	11	13	14	16	17	18	19	20
		21		27	27	21	504	21			93	23	21	29		27	21		21
PSU1	135	1010	1054	1050	1013	1055	1000	1005	947	1200	1440	995	1050	1127	968	1050	990	875	935
		1010		1050	1013	1055	1000	1005		1188		995	1038	1127	961	1050		866	935
		1010		1050	1013	1055	1000	1002		1188		995	1050	1127	957	1050		866	935
				1050		1000				1188									
PSU2	165	273	290	263	275	288	295	280	382	325	312	274	288	304	256	295	280	193	253
		273		263	263	288	295	280		325		274	288	304	256	295	268	193	253
		273		263	275	288	295	278		325		274	288	304	256	295		193	253
				265		295	295			325									
PSU3	135	425	536	438	438	450	475	435	519	448	732	440	438	445	417	488	425	313	378
		425		438	438	450	463	433		445		440	438	445	416	488	422	312	378
		425		435	425	450	463	433		445		440	438	445	415	488		313	378
				437		463				445									
PSU4	165	117	157	135	113	126	135	118	123	123	216	126	113	120	113	140	120	75	108
		117		130	113	126	135	115.0		120		126	113	120	112	140	117	73	108
		117		133	113	126	135	118		120		126	113	120	112	140		74	108
				133		135				118									
PSU3	135	425	535	433	425	450	500	448	545	443	655	445	438	448	442	563	425	390	400
		425		433	425	450	500	450		443		440	450	448	438	563	423	385	400
		425		433	425	450	500	445		443		440	438	448	436	563		385	400
				433		500				443									
PSU4	165	116	158	140	113	128	145	125.0	346	123	205	124	125	121	115	160	120	60	108
		116		138	113	128	140	123		123		124	125	121	114	155	118	61	108
		117		138	113	128	140	125.0		125		124	125	121	115	160		62	108
				138		140				123									
PSU4	135	995	1054	1010	1000	1030	1025	1083	951	1125	1320	990	1025	1130	961	1187	1035	808	940
		995		1010	1000	1030	1025	1080		1113		995	1025	1130	962	1150	1006	807	940
		995		1005	1000	1030	1025	1083		1113		995	1025	1130	962	1150		808	940
				1010		1025				1113									
PSU4	165	268	293	285	275	285	290	295	388	300	420	276	275	307	269	325	295	179	255
		268		285	263	285	290	298		300		274	275	307	270	325	276	173	255
		268		283	263	285	290	298		300		274	275	307	271	325		173	255
				283		295				300									



Table C3. Phase angle test results for the unaged asphalt binder samples.

Sample ID	Rep. no.	Target test temp, °C	1	2	3	4	5	6	7	8	9	10	11	13	14	15	16	17	18	19	20
PSU1	1	64	75.5	76.5	76.4	76.4	76.2	74.2	76.3	76.0	76.4	76.8	75.8	75.5	76.0	76.1	75.9	73.9	75.6	76.0	
		70	77.4	78.2	78.6		77.8	75.8	78.5	79.2		79.0	77.4	77.3	77.9	77.9		76.0			75.6
		76																77.9			77.5
	2	64	75.7	76.4	76.3		76.2	74.2	76.3		78.5	76.8	76.0	75.5	74.6	76.0		74.0			76.0
		70	77.8	78.2	78.6	78.4	78.1	75.9	78.5		78.5	79.0	77.8	77.5	76.9	78.2	79.3	76.1	78.0	78.7	77.9
		76																78.2			
PSU2	1	64	86.2	86.5	86.7	86.3	86.3	86.4	86.5	88.2	86.6	86.8	86.4	86.4	86.3	84.3	87.0	85.7	86.5	87.0	85.9
		70	87.6	87.3	88.4		86.9	87.9	87.6	91.1		88.2	87.5	86.4	88.2	87.7		86.6			87.2
		76																87.3			
	2	64	86.3	86.2	86.7		86.1	86.1	86.4			86.8	86.4	87.4	85.6	82.7		85.7			86.3
		70	87.6	87.4	88.3	88.1	87.4	87.9	87.7		88.0	88.2	87.6	87.4	86.8	86.9	88.7	86.6	87.7	88.6	87.5
		76							89.1									87.7			
PSU3	1	64	86.3	86.6	86.8	86.7	86.1	86.4	86.3	87.5	86.6	86.8	86.3	86.2	86.4	85.9	86.8	85.8	86.8		86.3
		70	87.8	87.3	88.3		87.6	87.5	87.5	90.3		88.3	87.9	87.0	87.6	87.3		87.3	87.7		87.5
		76							88.5									88.3			
	2	64	86.0	86.1	86.6		86.4	86.4	86.3			86.7	86.5	86.3	85.3	86.1		84.9	86.4		86.5
		70	87.3	87.2	88.3	88.1	87.6	87.5	87.5		87.9	88.2	87.9	87.4	86.6	87.9	88.6	86.2	88.5		87.7
		76							88.6									86.6			
PSU4	1	64	75.7	75.6	76.2	76.6	75.6	74.1	75.7	75.9	76.6	76.6	75.7	75.6	75.9	76.0	76.1	73.1	75.4	76.8	75.8
		70	77.8	77.9	78.4		77.8	75.7	77.8	79.1		78.8	77.9	77.5	78.2	78.0		75.0			77.7
		76																76.9			
	2	64	75.6	76.0	76.1		75.8	74.1	75.5		78.6	76.7	76.1	75.6	74.9	75.8		73.3			75.8
		70	77.5	77.8	78.3	78.7	77.5	75.8	77.6			78.8	77.7	77.6	77.0	77.4	78.2	70.0	77.7	78.9	77.7
		76																76.7			



Table C4. G\* test results for the RTFOT asphalt binder samples.

Sample ID	Rep. no.	Target test temp, °C	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU1	1	64						5978	2679	3543	3040	3092	3370	3509	3280	3184	5534	3138		
		70	3390	3093	3277	3400	3565	3250	1426	1985	1700	1617	1747	1923	1790	1761	2833	1656	3442	3506
		76	1857	1663	1790	1846	1914										1482			1900
	2	64	3403	2832	3296		3588	5949	2784		3590	3237	3315	3747	3240		5610			3512
		70	1858	1597	1870		1896	3216	1480		2000	1671	1786	2045	178		2717			1941
PSU2	1	64						3173	3254	2776	3290	3230	3383	3516	4890	3500	2923	3370	3115	3411
		70	3381	3172	3044	2815	3244	1538	1526	1338	1580	1477	1612	1710	1740	1634	1317	1538		1608
		76	1592	1546	1391	1347	1498										617			
	2	64	3380	3340	3127		3178	3188	3305		3450	3308	3397	4071	3850		3187		3484	
		70	1571	1513	1409		1483	1572	1549		1660	1474	1615	1997	1700		1435		1481	1643
PSU3	1	64						3185	3031	2771	3360	3275	3252	3958	2920	3332	3264	3082	3794	3529
		70	3364	3195	3990	2766	3299	1548	1405	1358	1600	1480	1576	1900	2090	1564	1488	1462		1668
		76	1555	1559	1813	1290	1574										721			
	2	64	3335	3307	3360		3301	3167	3095		3280	3106	3388	3956	3150		3924			3464
		70	1544	1572	1506		1567	1533	1434		1550	1501	1597	1906	1280		1593		1761	1603
PSU4	1	64						5723	3008	3127	3080	3263	3332	3687	4150	4828	7374	3441	3259	3625
		70	3287	3239	3449	2666	3142	3033	1605	1673	1690	1763	1825	2026	2160	2743	3639	1835		1969
		76	1788	1690	1908	1493	1701										7427			
	2	64	3280	3277	3528		3094	5558	3080		2900	3251	3281	3819	3470		3688			3924
		76	1790	1701	1950		1695	3008	1644		1610	1779	1793	2113	2000		2027		1745	2145

Table C5. Phase angle test results for the RTFOT asphalt binder samples.

Sample ID	Rep. no.	Target test temp, °C	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU1	1	64						69.4									69.7			
		70	70.5	71.5	71.3	71.2	70.6	71.3	71.8	70.3	72.0	71.4	70.5	70.9	70.9	72.1	71.4	71.0	71.2	70.7
		76	72.7	73.5	73.7	73.3	73.2		74.1	73.1	74.3	74.0	72.8	73.1	73.3	74.6	74.0	73.3		73.0
	2	64	70.4	71.6	71.2		70.6	69.3	71.5		71.0	71.4	70.7	69.8	70.8		69.8			70.7
		70	72.6	73.4	73.4		73.2	71.2	73.4		73.2	73.5	72.9	72.3	73.3		71.9			73.0
PSU2	1	64	82.4	82.9	83.4	83.8	82.9	82.9	82.5	84.0	83.0	83.2	82.8	82.7	81.4	83.3	82.9	82.7	83.3	82.7
		70	84.4	84.4	85.5	85.7	84.8	84.8	84.5	87.0	85.0	85.1	84.7	84.3	84.2	85.4	84.0	84.8		84.7
		76																		
	2	64	82.4	83.0	83.4		83.0	82.9	82.6		82.8	82.9	82.6	81.9	81.9		82.9			82.7
		70	84.4	84.8	85.5		84.9	84.7	84.6		84.9	84.9	84.6	84.0	84.3		84.8	85.4		84.7
PSU3	1	64	82.6	82.8	82.9	83.9	83.0	82.8	82.9	84.3	83.3	82.7	82.9	81.9	81.4	83.5	82.3	82.8	82.8	82.5
		70	84.5	84.6	85.1	85.8	84.8	84.8	84.7	87.1	85.3	85.1	84.7	84.0	83.2	85.7	84.1	84.8		84.5
		76															85.8			
	2	64	82.6	82.4	83.0		82.7	82.8	83.0		83.3	83.2	82.7	81.8	81.2		82.7			82.6
		70	84.7	84.8	85.2		84.5	84.7	84.9		85.3	84.9	84.7	83.9	83.7		84.8	84.4		84.6
PSU4	1	64						69.3									69.7			
		70	70.7	71.1	70.9	73.1	71.4	71.4	70.6	71.7	72.1	71.4	70.9	70.2	69.8	69.4	71.4	70.5	71.3	70.1
		76	72.9	73.0	73.2	75.3	73.7		72.8	74.7	74.4	73.6	73.2	72.4	71.9	71.6	73.9	73.0		72.1
	2	64	70.5	70.4	70.9		70.8	69.4	70.8		72.5	71.2	70.8	70.1	70.7		67.8			69.6
		70	72.7	72.8	73.1		73.1	71.2	73.0		74.8	73.5	73.1	72.3	73.0		72.2	73.3		71.8



Table C6. Stiffness test results at -12°C for the PAV asphalt binder samples, cont'd.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU3	1	8	312	295	259	270	265	264	278	275	286	314	284	299		297	284	302	270	304
		15	261	241	240	226	219	220	230	228	236	261	237	248		245	234	250	224	255
		30	212	191	193	184	174	177	184	182	190	210	191	198		196	186	200	180	207
		60	163	150	153	147	136	139	146	144	149	166	151	156	183	156	146	156	141	166
		120	133	116	120	115	105	107	113	111	115	129	116	121		121	112	121	109	130
		240	102	88	91	89	79	82	87	85	88	98	89	93		92	85	91	82	100
2		8	296	294	271	267	266	243	274	289	295	313	278	306		273	289	282	301	259
		15	249	242	228	223	219	201	227	240	245	261	233	253		227	239	235	250	218
		30	201	192	186	181	175	162	181	191	196	209	188	203		185	190	189	200	178
		60	160	151	150	144	137	128	144	151	154	165	148	160	187	143	149	150	157	144
		120	125	116	119	113	106	99	112	117	118	130	116	124		117	114	116	120	115
		240	96	88	93	87	80	75	85	89	91	100	89	94		91	86	88	92	90
PSU4	1	8	238	255	221	205	256	233	239	253	245	259	235	270		260	224	261	268	231
		15	202	214	185	172	215	196	199	213	206	218	198	227		219	187	219	225	197
		30	166	174	151	140	176	159	163	174	167	178	162	184		178	151	177	183	162
		60	134	141	121	113	141	128	131	137	133	144	130	148	182	143	120	141	146	132
		120	107	112	95	89	112	100	104	111	106	113	102	117		114	93	111	115	106
		240	83	88	74	70	87	78	81	87	82	89	79	91		89	71	86	88	83
2		8	252	274	232	199	253	228	233	241	241	266	236	273		246	241	253	265	254
		15	212	222	196	167	212	192	193	202	202	224	198	229		209	200	212	222	215
		30	174	179	159	136	173	156	157	164	164	182	160	186		173	161	171	181	176
		60	140	143	127	109	139	125	124	131	131	147	128	149	184	142	127	136	145	142
		120	111	113	101	86	110	99	99	103	103	117	100	118		114	99	107	114	112
		240	86	88	79	67	86	78	77	80	80	92	77	93		91	76	83	89	87

Table C7. m-value test results at -12°C for the PAV asphalt binder samples.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU1	1	8	0.259	0.294	0.249	0.261	0.271	0.265	0.278	0.252	0.253	0.268	0.265	0.266		0.267	0.297	0.282	0.235	0.270
		15	0.278	0.309	0.270	0.280	0.292	0.286	0.294	0.274	0.272	0.288	0.287	0.285		0.277	0.299	0.300	0.258	0.283
		30	0.299	0.326	0.294	0.301	0.314	0.310	0.311	0.299	0.294	0.309	0.312	0.306		0.288	0.321	0.320	0.284	0.298
		60	0.319	0.343	0.318	0.322	0.337	0.333	0.328	0.323	0.315	0.331	0.336	0.327	0.322	0.298	0.343	0.340	0.309	0.312
		120	0.340	0.360	0.341	0.342	0.359	0.357	0.345	0.347	0.336	0.353	0.361	0.348		0.309	0.366	0.360	0.335	0.327
		240	0.360	0.376	0.365	0.363	0.382	0.381	0.362	0.371	0.358	0.374	0.384	0.369		0.319	0.388	0.381	0.360	0.342
2		8	0.258	0.285	0.264	0.256	0.273	0.266		0.270	0.255	0.269	0.254	0.270		0.257	0.258	0.272	0.255	0.253
		15	0.279	0.303	0.286	0.277	0.290	0.289		0.288	0.274	0.287	0.278	0.289		0.272	0.282	0.294	0.277	0.269
		30	0.301	0.322	0.310	0.300	0.309	0.314		0.308	0.295	0.306	0.304	0.310		0.288	0.308	0.318	0.300	0.287
		60	0.324	0.342	0.335	0.332	0.328	0.339		0.328	0.315	0.326	0.330	0.332	0.322	0.305	0.334	0.343	0.323	0.304
		120	0.347	0.361	0.359	0.345	0.347	0.364		0.347	0.336	0.345	0.356	0.353		0.321	0.361	0.367	0.346	0.322
		240	0.369	0.381	0.384	0.368	0.366	0.389		0.367	0.356	0.365	0.382	0.375		0.337	0.387	0.391	0.370	0.339
PSU2	1	8	0.286	0.310	0.272	0.266	0.283	0.286	0.286	0.289	0.283	0.284	0.281	0.285		0.271	0.292	0.291	0.270	0.273
		15	0.309	0.327	0.296	0.290	0.305	0.311	0.308	0.307	0.306	0.305	0.309	0.305		0.290	0.316	0.316	0.294	0.289
		30	0.334	0.345	0.322	0.315	0.329	0.338	0.333	0.327	0.331	0.329	0.340	0.327		0.312	0.343	0.342	0.320	0.306
		60	0.359	0.364	0.348	0.341	0.353	0.365	0.358	0.347	0.358	0.353	0.371	0.349	0.346	0.333	0.370	0.369	0.346	0.324
		120	0.384	0.382	0.374	0.367	0.377	0.395	0.383	0.367	0.382	0.377	0.402	0.371		0.354	0.396	0.396	0.372	0.341
		240	0.409	0.400	0.400	0.392	0.401	0.419	0.408	0.387	0.407	0.400	0.432	0.393		0.376	0.423	0.423	0.398	0.358
2		8	0.257	0.304	0.266	0.270	0.279	0.287	0.293	0.296	0.282	0.283	0.299	0.287		0.277	0.293	0.297	0.280	0.257
		15	0.281	0.323	0.284	0.291	0.302	0.310	0.309	0.315	0.306	0.304	0.323	0.309		0.292	0.313	0.319	0.300	0.280
		30	0.307	0.343	0.303	0.315	0.327	0.336	0.328	0.335	0.332	0.328	0.350	0.333		0.309	0.338	0.343	0.322	0.306
		60	0.333	0.364	0.323	0.339	0.352	0.361	0.347	0.355	0.358	0.351	0.376	0.357		0.326	0.362	0.368	0.344	0.332
		120	0.359	0.384	0.343	0.362	0.378	0.387	0.365	0.376	0.385	0.374	0.408	0.381		0.343	0.386	0.392	0.367	0.358
		240	0.385	0.405	0.362	0.386	0.403	0.413	0.384	0.396	0.411	0.397	0.429	0.405		0.359	0.410	0.416	0.389	0.384

Table C7. m-value test results at -12°C for the PAV asphalt binder samples, cont'd.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU3	1	8	0.269	0.306	0.272	0.269	0.295	0.282	0.286	0.289	0.288	0.280	0.278	0.289		0.290	0.294	0.287	0.280	0.266
		15	0.291	0.325	0.293	0.289	0.317	0.305	0.307	0.309	0.310	0.303	0.301	0.310		0.309	0.317	0.311	0.305	0.288
		30	0.316	0.345	0.322	0.312	0.342	0.331	0.330	0.332	0.335	0.328	0.327	0.333		0.330	0.343	0.338	0.333	0.313
		60	0.340	0.365	0.345	0.335	0.367	0.357	0.353	0.354	0.359	0.353	0.353	0.355	0.343	0.352	0.365	0.364	0.361	0.338
		120	0.365	0.385	0.374	0.358	0.392	0.383	0.376	0.377	0.383	0.378	0.379	0.378		0.373	0.394	0.391	0.389	0.363
		240	0.390	0.405	0.400	0.381	0.416	0.408	0.399	0.399	0.407	0.404	0.404	0.401		0.394	0.420	0.418	0.417	0.388
2		8	0.267	0.298	0.266	0.270	0.293	0.280	0.287	0.288	0.290	0.285	0.271	0.286		0.274	0.292	0.273	0.289	0.262
		15	0.292	0.319	0.284	0.292	0.315	0.304	0.308	0.309	0.312	0.304	0.295	0.308		0.293	0.315	0.298	0.312	0.280
		30	0.319	0.341	0.304	0.316	0.339	0.330	0.331	0.332	0.335	0.325	0.321	0.333		0.313	0.341	0.326	0.337	0.301
		60	0.345	0.364	0.323	0.341	0.364	0.356	0.354	0.356	0.359	0.346	0.345	0.358	0.334	0.353	0.367	0.354	0.361	0.321
		120	0.372	0.387	0.343	0.365	0.389	0.383	0.378	0.379	0.382	0.367	0.374	0.383		0.353	0.393	0.381	0.386	0.341
		240	0.399	0.409	0.362	0.390	0.413	0.409	0.401	0.402	0.406	0.388	0.401	0.408		0.372	0.419	0.409	0.411	0.361
PSU4	1	8	0.248	0.268	0.264	0.266	0.263	0.262	0.271	0.284	0.272	0.263	0.254	0.267		0.269	0.278	0.272	0.264	0.246
		15	0.270	0.285	0.285	0.285	0.283	0.284	0.288	0.295	0.291	0.282	0.279	0.286		0.286	0.300	0.292	0.287	0.266
		30	0.294	0.303	0.309	0.305	0.305	0.308	0.306	0.308	0.311	0.304	0.306	0.307		0.304	0.324	0.315	0.313	0.288
		60	0.316	0.327	0.313	0.326	0.328	0.332	0.324	0.320	0.331	0.326	0.333	0.328	0.309	0.322	0.343	0.337	0.339	0.310
		120	0.343	0.340	0.356	0.347	0.350	0.357	0.342	0.333	0.351	0.348	0.360	0.349		0.341	0.372	0.360	0.365	0.332
		240	0.367	0.358	0.380	0.368	0.373	0.381	0.360	0.345	0.371	0.370	0.388	0.370		0.359	0.396	0.382	0.390	0.354
2		8	0.257	0.306	0.267	0.266	0.268	0.268	0.287	0.270	0.268	0.266	0.271	0.271		0.246	0.284	0.275	0.266	0.254
		15	0.278	0.316	0.286	0.285	0.287	0.286	0.301	0.290	0.288	0.283	0.292	0.288		0.263	0.305	0.295	0.286	0.276
		30	0.302	0.326	0.307	0.306	0.308	0.306	0.316	0.312	0.311	0.303	0.316	0.307		0.282	0.329	0.317	0.309	0.301
		60	0.325	0.336	0.328	0.327	0.329	0.326	0.330	0.334	0.333	0.322	0.339	0.326	0.305	0.301	0.352	0.339	0.331	0.325
		120	0.349	0.346	0.349	0.348	0.350	0.346	0.345	0.356	0.356	0.341	0.363	0.345		0.319	0.376	0.362	0.354	0.350
		240	0.372	0.356	0.371	0.369	0.371	0.365	0.360	0.378	0.378	0.361	0.386	0.364		0.338	0.400	0.384	0.376	0.374

Table C8. Stiffness test results at -18°C for the PAV asphalt binder samples.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU1	1	8	457	502	431	412	451	425	342	463	436	498	444	506		425	443	508	533	463
		15	397	431	378	355	397	369	291	398	387	432	382	437		368	382	439	462	403
		30	337	355	323	301	336	310	242	336	328	368	321	368		314	319	369	389	341
		60	280	294	261	249	278	257	198	280	274	306	265	305	283	266	264	304	324	285
		120	229	238	213	205	228	209	160	229	227	252	214	250		223	214	248	267	236
		240	186	190	171	166	183	166	126	182	184	202	170	202		184	170	197	216	192
PSU2	1	8	454	498	445	409	466	426	335	436	488	514	480	526		397	453	592	531	385
		15	394	431	386	353	404	368	286	378	421	446	411	456		347	391	514	460	333
		30	334	353	322	299	342	309	237	318	355	375	346	384		294	329	431	390	279
		60	278	286	268	247	285	256	196	263	293	311	285	318	283	246	271	356	339	233
		120	228	235	218	202	232	207	159	215	239	255	231	259		209	220	290	269	192
		240	185	188	175	162	185	165	127	173	192	206	184	207		174	176	232	217	157
PSU2	2	8	532	525	521	497	576	511		504	613	611	500	619		494	519	596	581	519
		15	456	448	450	427	501	438		434	521	528	426	534		415	450	515	500	453
		30	384	373	374	359	423	366		365	436	446	356	446		349	378	432	418	382
		60	318	304	306	297	350	298		299	356	370	292	366	353	286	314	365	341	316
		120	259	243	247	241	281	240		242	288	299	232	294		250	254	287	272	257
		240	206	191	197	193	227	188		192	229	237	182	233		209	203	228	216	204
PSU2	2	8	540	564	515	509	564	500	409	520		615	525	579		527	543	599	559	400
		15	468	478	437	441	491	431	344	450		536	454	501		459	469	518	488	347
		30	394	404	359	372	416	358	283	375		447	382	419		386	394	432	407	292
		60	327	323	288	308	345	293	229	310		370	313	345		324	325	356	356	244
		120	265	257	226	248	279	235	181	249		301	250	279		273	264	287	271	205
		240	210	203	175	197	223	185	142	199		241	197	222		226	210	227	215	169

Table C8. Stiffness test results at -18°C for the PAV asphalt binder samples, cont'd.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU3	1	8	541	565	513	483	558	500	404	489	575	599	561	616		518	528	609	585	534
		15	471	487	445	421	478	434	343	419	494	516	491	530		448	457	523	508	463
		30	404	406	371	350	398	360	280	352	409	436	416	442		382	383	438	420	388
		60	340	324	305	289	325	295	225	289	335	360	343	363	413	321	316	358	344	322
		120	282	260	250	236	263	237	176	235	270	293	277	293		269	255	288	273	263
		240	229	205	200	189	206	188	133	187	213	233	219	233		221	203	227	210	211
2		8	571	570	541	541	549	513	375	519	563	623	582	610		493	559	623	531	450
		15	498	482	470	468	470	441	315	445	480	540	507	525		426	480	537	462	389
		30	421	404	392	395	398	367	258	372	401	455	424	438		360	403	447	385	328
		60	350	326	321	328	326	301	208	305	329	377	346	359	363	300	332	366	315	274
		120	286	262	259	265	260	241	164	247	264	308	284	290		249	268	294	252	226
		240	229	206	206	211	204	188	129	196	207	247	225	230		204	212	231	200	185
PSU4	1	8	483	480	444	384	464	452	334	450	476	517	487	532		457	450	540	482	326
		15	421	408	388	333	410	392	281	390	411	448	425	460		398	288	466	416	285
		30	356	341	328	281	349	329	231	328	345	380	361	386		335	325	391	350	244
		60	296	279	272	234	287	272	167	272	286	315	246	320	386	260	263	324	288	205
		120	242	228	222	191	233	220	149	222	234	258	242	260		230	218	264	234	173
		240	195	183	178	154	187	176	119	179	187	207	194	209		187	175	210	187	144
2		8	489	482	466	416	475	418		453	480	507	470	516		455	441	520	478	380
		15	425	414	405	360	412	360		392	414	441	407	445		400	380	449	411	322
		30	360	353	340	303	347	305		330	348	371	343	373		341	319	376	344	264
		60	299	284	283	252	288	251		276	289	309	282	308	341	285	265	311	286	211
		120	245	232	230	206	234	203		226	236	254	230	251		240	21	254	237	173
		240	197	186	184	166	188	161		182	189	203	184	201		198	173	203	191	141



Table C9. m-value test results at -18°C for the PAV asphalt binder samples.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU1	1	8	0.211	0.240	0.204	0.221	0.198	0.211	0.240	0.218	0.191	0.205	0.222	0.224		0.216	0.223	0.218	0.219	0.212
		15	0.231	0.257	0.230	0.238	0.223	0.235	0.259	0.238	0.215	0.227	0.243	0.241		0.227	0.245	0.240	0.236	0.229
		30	0.253	0.275	0.258	0.257	0.251	0.261	0.280	0.260	0.241	0.251	0.268	0.260		0.239	0.268	0.265	0.255	0.249
		60	0.275	0.294	0.286	0.276	0.276	0.285	0.301	0.282	0.267	0.275	0.293	0.275	0.270	0.250	0.291	0.289	0.274	0.263
		120	0.297	0.313	0.314	0.295	0.306	0.312	0.322	0.304	0.293	0.299	0.318	0.297		0.262	0.314	0.314	0.292	0.287
		240	0.319	0.331	0.343	0.314	0.333	0.338	0.343	0.326	0.319	0.324	0.342	0.315		0.274	0.338	0.339	0.311	0.307
2		8	0.213	0.244	0.221	0.216	0.206	0.215	0.239	0.218	0.222	0.222	0.223	0.215		0.217	0.219	0.219	0.205	0.229
		15	0.232	0.260	0.241	0.237	0.230	0.238	0.256	0.238	0.241	0.239	0.245	0.237		0.227	0.240	0.240	0.225	0.242
		30	0.253	0.278	0.263	0.259	0.256	0.263	0.275	0.260	0.262	0.259	0.268	0.261		0.237	0.264	0.263	0.248	0.256
		60	0.274	0.296	0.285	0.281	0.282	0.289	0.294	0.283	0.263	0.278	0.291	0.285	0.270	0.248	0.288	0.288	0.271	0.271
		120	0.296	0.314	0.307	0.303	0.308	0.314	0.313	0.305	0.305	0.297	0.315	0.309		0.258	0.312	0.309	0.294	0.285
		240	0.317	0.332	0.330	0.325	0.334	0.339	0.332	0.327	0.326	0.317	0.338	0.333		0.269	0.336	0.332	0.317	0.299
PSU2	1	8	0.220	0.233	0.232	0.224	0.212	0.228		0.221	0.240	0.209	0.225	0.223		0.234	0.212	0.221	0.227	0.205
		15	0.241	0.257	0.253	0.244	0.236	0.252		0.245	0.258	0.234	0.251	0.247		0.238	0.236	0.243	0.251	0.230
		30	0.264	0.283	0.275	0.265	0.262	0.278		0.270	0.278	0.262	0.280	0.273		0.242	0.262	0.269	0.277	0.258
		60	0.288	0.309	0.298	0.287	0.288	0.304		0.296	0.298	0.299	0.309	0.299	0.281	0.246	0.288	0.294	0.304	0.288
		120	0.311	0.336	0.321	0.309	0.313	0.330		0.321	0.317	0.318	0.337	0.325		0.250	0.314	0.319	0.330	0.315
		240	0.334	0.362	0.344	0.331	0.339	0.356		0.347	0.337	0.346	0.366	0.351		0.255	0.340	0.344	0.357	0.343
2		8	0.209	0.235	0.246	0.209	0.204	0.227	0.258	0.225		0.220	0.210	0.220		0.227	0.218	0.221	0.214	0.230
		15	0.234	0.259	0.272	0.235	0.229	0.251	0.277	0.246		0.241	0.239	0.243		0.236	0.241	0.245	0.239	0.239
		30	0.262	0.286	0.301	0.263	0.257	0.277	0.298	0.270		0.264	0.271	0.269		0.245	0.265	0.272	0.267	0.248
		60	0.289	0.313	0.331	0.292	0.285	0.304	0.319	0.293		0.287	0.303	0.294		0.254	0.290	0.298	0.294	0.257
		120	0.317	0.340	0.360	0.320	0.312	0.331	0.340	0.317		0.310	0.335	0.319		0.263	0.315	0.325	0.322	0.267
		240	0.345	0.367	0.389	0.348	0.340	0.357	0.362	0.341		0.332	0.367	0.344		0.272	0.339	0.351	0.349	0.276

Table C9. m-value test results at -18°C for the PAV asphalt binder samples, cont'd.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	13	14	15	16	17	18	19	20
PSU3	1	8	0.197	0.234	0.222	0.222	0.229	0.221	0.241	0.229	0.233	0.216	0.198	0.229		0.218	0.217	0.224	0.212	0.219
		15	0.218	0.258	0.243	0.242	0.252	0.246	0.272	0.249	0.255	0.239	0.227	0.250		0.230	0.241	0.248	0.245	0.239
		30	0.240	0.285	0.265	0.264	0.278	0.274	0.306	0.270	0.278	0.263	0.259	0.273		0.242	0.266	0.275	0.281	0.261
		60	0.262	0.312	0.283	0.287	0.304	0.301	0.340	0.292	0.302	0.288	0.292	0.296	0.289	0.255	0.292	0.301	0.317	0.283
		120	0.285	0.339	0.311	0.309	0.329	0.328	0.374	0.313	0.326	0.313	0.323	0.319		0.268	0.318	0.327	0.353	0.305
2		240	0.307	0.366	0.334	0.331	0.355	0.356	0.408	0.335	0.350	0.338	0.356	0.341		0.280	0.343	0.354	0.389	0.327
		8	0.205	0.245	0.222	0.210	0.213	0.220	0.268	0.231	0.229	0.213	0.219	0.228		0.227	0.220	0.223	0.220	0.224
		15	0.228	0.264	0.245	0.235	0.241	0.247	0.282	0.251	0.253	0.235	0.242	0.249		0.239	0.244	0.248	0.245	0.238
		30	0.254	0.286	0.271	0.262	0.272	0.278	0.302	0.274	0.279	0.258	0.267	0.273		0.252	0.270	0.276	0.274	0.253
		60	0.280	0.368	0.296	0.289	0.303	0.305	0.322	0.296	0.304	0.282	0.292	0.297	0.283	0.266	0.295	0.303	0.302	0.268
PSU4	1	120	0.306	0.330	0.322	0.316	0.334	0.338	0.343	0.319	0.330	0.305	0.317	0.321		0.279	0.321	0.331	0.330	0.284
		240	0.331	0.351	0.348	0.343	0.365	0.368	0.363	0.341	0.356	0.328	0.342	0.344		0.293	0.347	0.358	0.359	0.299
		8	0.210	0.245	0.204	0.217	0.194	0.217	0.263	0.219	0.221	0.211	0.206	0.222		0.217	0.226	0.220	0.220	0.209
		15	0.231	0.259	0.228	0.236	0.222	0.239	0.278	0.238	0.241	0.232	0.230	0.241		0.234	0.245	0.241	0.241	0.220
		30	0.254	0.275	0.255	0.257	0.253	0.264	0.295	0.259	0.262	0.255	0.257	0.263		0.253	0.266	0.264	0.265	0.233
2		60	0.277	0.290	0.261	0.273	0.283	0.289	0.311	0.280	0.284	0.279	0.284	0.285	0.266	0.242	0.287	0.287	0.289	0.246
		120	0.300	0.305	0.308	0.299	0.314	0.314	0.328	0.301	0.305	0.302	0.311	0.306		0.291	0.309	0.310	0.313	0.259
		240	0.324	0.321	0.334	0.320	0.345	0.339	0.345	0.322	0.327	0.326	0.337	0.328		0.230	0.330	0.333	0.337	0.271
		8	0.210	0.225	0.212	0.218	0.213	0.213		0.218	0.220	0.211	0.218	0.224		0.201	0.221	0.225	0.232	0.285
		15	0.231	0.246	0.235	0.237	0.235	0.238		0.236	0.239	0.232	0.239	0.244		0.217	0.241	0.244	0.245	0.289
2		30	0.255	0.268	0.260	0.259	0.259	0.264		0.256	0.261	0.255	0.263	0.265		0.235	0.262	0.265	0.260	0.292
		60	0.276	0.290	0.265	0.280	0.284	0.291		0.276	0.283	0.278	0.286	0.286	0.266	0.252	0.284	0.285	0.275	0.296
		120	0.302	0.313	0.310	0.301	0.309	0.318		0.296	0.304	0.301	0.309	0.308		0.270	0.306	0.306	0.290	0.300
		240	0.326	0.335	0.335	0.323	0.333	0.344		0.315	0.326	0.324	0.332	0.329		0.288	0.327	0.327	0.305	0.304

Table C10. Stiffness test results at -18°C for the pre-PAV asphalt binder samples.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	12	13	14	15	16	17	18	19	20
1	1	8	341	311	284	309	312	317	222	314	313	336	316	312	340		267	300	336	341	327
		15	284	256	237	257	260	217	180	261	260	280	265	259	283		222	253	277	287	273
		30	228	203	190	207	210	208	141	210	209	224	214	210	227		181	206	221	233	219
		60	129	159	150	163	166	163	108	166	163	178	171	165	173	163	145	164	173	186	173
		120	138	122	116	126	128	125	82	129	126	139	134	128	139		117	129	134	146	135
		240	105	93	89	97	98	94	62	98	96	107	102	97	106		93	100	102	112	103
2	2	8	328	322	301	314	309	259	228	297	304	347		297	342		271	316	349	364	282
		15	274	263	257	264	262	215	184	247	252	287		248	286		225	264	289	306	236
		30	221	208	209	212	212	172	144	195	202	229		200	230		183	214	230	246	192
		60	175	163	168	167	167	135	111	152	160	181		157	182	162	148	169	181	196	154
		120	136	126	131	130	129	103	83.8	119	125	141		121	141		118	132	139	153	122
		240	104	95	101	99	99	79	62	90	96	109		92	107		94	101	106	117	95
2	1	8	550	576	528	510	544	527	395	478	545	585		516	545		358	516	515	579	434
		15	472	507	447	434	473	451	329	409	467	506		443	464		307	443	443	497	372
		30	393	402	372	366	395	370	266	338	392	421		364	382		257	371	369	409	311
		60	321	330	298	304	322	298	212	274	318	343		300	308	305	215	305	301	334	255
		120	257	260	235	248	259	233	165	218	255	277		239	244		182	247	240	264	205
		240	202	203	183	199	203	179	127	171	200	217		187	189		153	195	188	206	163
2	2	8	543	542	526	512	534	494	356	490	537	563		531	546		413	563	539	562	434
		15	468	460	449	449	465	423	297	420	458	483		459	462		354	484	459	474	374
		30	388	377	368	376	388	346	239	346	378	402		382	376		298	403	379	396	311
		60	319	305	302	312	313	279	191	282	307	330		314	301	303	249	330	307	318	256
		120	255	244	238	253	252	219	148	227	245	266		249	235		207	265	243	254	206
		240	201	190	186	202	197	169	114	179	192	211		194	181		172	208	191	197	163

Table C10. Stiffness test results at -18°C for the pre-PAV asphalt binder samples, cont'd.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	12	13	14	15	16	17	18	19	20
3	1	8	507	545	503	498	544	540	359	472	539	592	514	526	538		457	511	515	580	373
		15	443	454	430	424	466	456	300	403	459	508	445	450	455		392	442	438	488	319
		30	373	376	358	354	388	377	243	332	374	423	370	377	371		328	372	381	412	267
		60	307	303	289	288	314	304	192	269	301	346	302	308	296	351	273	307	292	333	224
		120	248	241	232	232	251	238	152	216	239	277	239	246	232		224	249	233	265	185
		240	197	188	181	182	196	183	118	170	185	216	188	192	178		180	198	181	204	150
4	2	8	535	549	484	485	541	516	373	482	518	585	492	537	553		453	529	526	559	431
		15	459	466	412	417	465	439	313	409	443	502	420	463	471		389	458	447	478	372
		30	383	377	343	347	387	363	252	340	365	416	347	388	386		327	383	369	396	315
		60	313	308	278	280	316	291	202	277	296	341	283	317	308	327	271	312	299	322	263
		120	251	246	223	225	255	230	158	221	235	276	229	254	241		227	252	237	256	215
		240	197	190	174	175	200	177	122	174	182	218	176	199	184		187	199	183	200	171
4	1	8	333	340	328	290	288	305	220	304	330	343	323	302	341		301	329	317	353	287
		15	277	276	273	242	243	253	178	251	274	285	270	256	283		252	276	264	292	241
		30	223	219	219	194	197	204	139	201	221	230	218	206	227		205	223	211	233	196
		60	176	170	174	152	157	160	107	157	174	181	173	163	179	186	164	177	165	184	155
		120	137	130	137	118	123	123	82	123	134	140	134	126	139		129	138	128	142	121
		240	105	98	104	91	94	93	62	93	102	107	101	97	105		101	106	97	106	93
4	2	8	333	327	325	321	306	314	218		318	351	312	307	344		300	304	317	326	292
		15	277	264	274	269	257	260	177		264	291	260	257	285		252	255	262	272	244
		30	223	210	222	219	207	209	136		212	233	210	208	229		204	207	209	218	197
		60	177	164	175	175	163	164	106		167	184	166	164	181	195	165	165	165	172	158
		120	138	126	136	137	127	127	81		130	143	129	127	140		131	129	127	133	125
		240	105	95	103	106	97	95	61		98	110	98	97	107		103	99	96	101	97

Table C11. m-value test results at -18°C for the pre-PAV asphalt binder samples.

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	12	13	14	15	16	17	18	19	20
1	1	8	0.279	0.303	0.279	0.280	0.274	0.289	0.323	0.281	0.286	0.284	0.263	0.273	0.280		0.283	0.261	0.296		
		15	0.304	0.322	0.303	0.303	0.299	0.314	0.343	0.304	0.309	0.304	0.288	0.299	0.303		0.293	0.284	0.316		
		30	0.331	0.344	0.329	0.328	0.326	0.342	0.365	0.329	0.334	0.326	0.315	0.328	0.329		0.304	0.309	0.338		
		60	0.358	0.366	0.355	0.353	0.354	0.369	0.387	0.355	0.360	0.348	0.343	0.356	0.355	0.347	0.315	0.335	0.360	0.341	0.350
		120	0.385	0.387	0.381	0.378	0.381	0.396	0.408	0.380	0.385	0.370	0.370	0.384	0.380		0.325	0.360	0.382	0.368	0.374
		240	0.412	0.409	0.407	0.403	0.408	0.424	0.430	0.406	0.411	0.392	0.397	0.412	0.406		0.336	0.386	0.404	0.395	0.398
2	2	8	0.273	0.307	0.248	0.275	0.262	0.290	0.323	0.302	0.284	0.299		0.276	0.274		0.286	0.273	0.290	0.268	0.269
		15	0.298	0.326	0.275	0.299	0.289	0.312	0.344	0.320	0.304	0.315		0.301	0.298		0.295	0.295	0.313	0.292	0.288
		30	0.324	0.346	0.306	0.326	0.319	0.337	0.368	0.340	0.326	0.333		0.329	0.326		0.306	0.320	0.338	0.319	0.309
		60	0.351	0.366	0.337	0.353	0.349	0.361	0.391	0.360	0.348	0.351		0.357	0.353	0.347	0.316	0.345	0.363	0.345	0.329
		120	0.378	0.387	0.367	0.380	0.379	0.386	0.415	0.380	0.370	0.368		0.384	0.381		0.327	0.371	0.387	0.372	0.350
		240	0.404	0.407	0.398	0.406	0.409	0.410	0.438	0.400	0.392	0.386		0.412	0.408		0.338	0.396	0.412	0.399	0.371
1	1	8	0.229	0.243	0.241	0.230	0.213	0.235	0.277	0.236	0.225	0.223		0.232	0.242		0.257	0.222	0.224	0.233	0.230
		15	0.253	0.268	0.267	0.246	0.242	0.265	0.297	0.261	0.251	0.248		0.256	0.267		0.255	0.245	0.251	0.259	0.252
		30	0.280	0.296	0.296	0.265	0.273	0.299	0.320	0.288	0.279	0.276		0.283	0.296		0.252	0.271	0.280	0.289	0.275
		60	0.306	0.323	0.324	0.284	0.304	0.332	0.342	0.316	0.307	0.304		0.309	0.324	0.300	0.250	0.267	0.310	0.316	0.289
		120	0.333	0.351	0.353	0.302	0.336	0.365	0.365	0.343	0.335	0.332		0.336	0.353		0.247	0.322	0.339	0.347	0.322
		240	0.360	0.379	0.381	0.321	0.367	0.398	0.387	0.370	0.364	0.360		0.362	0.381		0.245	0.348	0.369	0.376	0.346
2	2	8	0.224	0.250	0.240	0.207	0.218	0.239	0.277	0.240	0.241	0.233		0.212	0.253		0.243	0.227	0.242	0.239	0.228
		15	0.249	0.272	0.264	0.232	0.246	0.267	0.298	0.261	0.264	0.254		0.243	0.280		0.249	0.251	0.266	0.264	0.250
		30	0.277	0.295	0.291	0.260	0.278	0.299	0.321	0.284	0.288	0.277		0.277	0.309		0.255	0.278	0.292	0.291	0.274
		60	0.305	0.319	0.318	0.287	0.308	0.330	0.344	0.308	0.313	0.300		0.311	0.336	0.297	0.261	0.305	0.318	0.319	0.299
		120	0.332	0.343	0.345	0.315	0.339	0.362	0.368	0.331	0.337	0.323		0.344	0.368		0.267	0.331	0.344	0.346	0.323
		240	0.360	0.366	0.372	0.342	0.370	0.393	0.391	0.354	0.362	0.346		0.378	0.397		0.273	0.358	0.370	0.373	0.348

Table C11. m-value test results at -18°C for the pre-PAV asphalt binder samples, cont'd.

Appendix II

Sample ID	Rep. no.	Loading time	1	2	3	4	5	6	7	8	10	11	12	13	14	15	16	17	18	19	20
3	1	8	0.205	0.261	0.233	0.234	0.229	0.238	0.281	0.247	0.252	0.222	0.218	0.223	0.254		0.230	0.215	0.242	0.225	0.235
		15	0.233	0.280	0.258	0.257	0.255	0.267	0.298	0.267	0.275	0.249	0.247	0.250	0.281		0.246	0.238	0.266	0.255	0.246
		30	0.263	0.300	0.285	0.281	0.284	0.299	0.318	0.289	0.300	0.279	0.279	0.279	0.310		0.263	0.264	0.292	0.287	0.259
		60	0.293	0.327	0.312	0.305	0.313	0.333	0.337	0.311	0.326	0.309	0.312	0.308	0.339	0.384	0.280	0.298	0.319	0.319	0.271
		120	0.323	0.341	0.340	0.330	0.341	0.363	0.357	0.333	0.352	0.339	0.344	0.337	0.368		0.298	0.316	0.345	0.351	0.284
		240	0.353	0.362	0.367	0.354	0.370	0.395	0.376	0.355	0.377	0.369	0.376	0.336	0.397		0.315	0.341	0.372	0.383	0.296
	2	8	0.225	0.255	0.235	0.228	0.226	0.238	0.271	0.239	0.237	0.234	0.234	0.218	0.241		0.242	0.218	0.238	0.235	0.209
		15	0.250	0.275	0.259	0.255	0.250	0.266	0.292	0.261	0.263	0.255	0.259	0.245	0.271		0.249	0.244	0.264	0.260	0.231
		30	0.278	0.298	0.285	0.284	0.277	0.297	0.316	0.286	0.291	0.278	0.286	0.276	0.306		0.256	0.272	0.293	0.287	0.256
		60	0.306	0.327	0.317	0.314	0.304	0.327	0.339	0.310	0.319	0.300	0.314	0.306	0.340	0.290	0.264	0.301	0.322	0.315	0.280
		120	0.334	0.344	0.338	0.343	0.331	0.358	0.362	0.334	0.348	0.323	0.341	0.336	0.374		0.272	0.329	0.352	0.342	0.301
		240	0.362	0.366	0.364	0.373	0.358	0.389	0.386	0.359	0.376	0.346	0.369	0.360	0.408		0.279	0.357	0.381	0.370	0.328
4	1	8	0.277	0.312	0.276	0.260	0.263	0.280	0.327	0.293	0.278	0.280	0.267	0.267	0.282		0.277	0.269	0.285	0.284	0.267
		15	0.301	0.333	0.298	0.307	0.287	0.306	0.345	0.313	0.303	0.303	0.294	0.293	0.305		0.294	0.293	0.309	0.309	0.291
		30	0.326	0.353	0.323	0.331	0.313	0.334	0.364	0.335	0.331	0.329	0.324	0.322	0.331		0.312	0.319	0.335	0.337	0.316
		60	0.352	0.374	0.348	0.354	0.340	0.362	0.384	0.357	0.353	0.354	0.354	0.350	0.350	0.334	0.330	0.345	0.362	0.364	0.342
		120	0.377	0.396	0.373	0.378	0.367	0.390	0.404	0.379	0.386	0.380	0.383	0.376	0.382		0.348	0.370	0.388	0.392	0.368
		240	0.403	0.417	0.398	0.401	0.393	0.418	0.424	0.400	0.414	0.405	0.413	0.407	0.407		0.367	0.396	0.415	0.419	0.393
	2	8	0.278	0.316	0.259	0.264	0.274	0.278	0.327		0.282	0.291	0.271	0.270	0.285		0.273	0.264	0.291	0.281	0.281
		15	0.300	0.333	0.289	0.287	0.298	0.304	0.344		0.305	0.310	0.297	0.296	0.307		0.289	0.288	0.313	0.304	0.296
		30	0.325	0.351	0.322	0.313	0.324	0.333	0.364		0.331	0.331	0.325	0.323	0.330		0.306	0.315	0.338	0.330	0.314
		60	0.350	0.369	0.354	0.338	0.350	0.362	0.383		0.356	0.352	0.354	0.352	0.354	0.335	0.323	0.342	0.362	0.355	0.334
		120	0.374	0.388	0.387	0.363	0.377	0.390	0.402		0.382	0.372	0.382	0.379	0.378		0.340	0.369	0.387	0.381	0.348
		240	0.399	0.406	0.420	0.388	0.403	0.419	0.422		0.408	0.393	0.411	0.407	0.402		0.357	0.396	0.411	0.406	0.365

Table C12. Complex viscosity test results for the reference fluid.

Replicate	Target test temperature °C	Strain %	Frequency rad/s	1	4	5	6	7	9	10	11	13	14	17	18	19	20
1	64	1	1	262	276		269		262	272		272	276			261	290
	64	10	1	262	270		268		255	271		271	272		262	270	278
	64	1	10	259	261	270	269	261		270	267	264	272	278		249	276
	64	10	10	259	259	265	266			267	267	271	266	267		262	271
	76	1	1	128	129		135		129	128		131	139			128	146
	76	10	1		129		132		128	128		133	138			125	138
	76	1	10	127	128	132	132	126	127	129	127	131	139	134		126	134
	76	10	10	127	127	131	133		125	130	127	132	138	126		120	131
2	64	1	1	256			276			267			292		278		298
	64	10	1	255			271			268			292				280
	64	1	10	254		251		258		266	270		289	271			286
	64	10	10	252		250				264	265		288	285	272		273
	76	1	1	124			140			128			146				144
	76	10	1	124			136			128			146				138
	76	1	10	124		123		124.0		128	124		144	125	134		134
	76	10	10	123		124				128	125		145	125	131		132

Table C13. G\* strain sweeps test results for the unaged asphalt binder samples.

Sample ID	Loading time	1	3	4	5	6	7	9	8	10	13	14	16	17	19	20
PSU1	2	1354	1330	1290	1427	1401	1308	1540	1498	1297	1740	1672	1760	1455	1410	1471
	4	1358	1330	1300	1410	1362	1290	1520	1507	1293	1746	1679	1760	1384	1400	1449
	6	1362	1330	1300	1418	1353	1279	1510	1513	1288	1753	1669	1760	1444	1410	1440
	8	1359	1330	1290	1420	1354	1274	1510	1512	1288	1759	1670	1750	1491	1410	1433
	10	1360	1330	1280	1435	1346	1274	1500	1514	1284	1761	1668	1740	1439	1410	1434
	12	1362	1340	1290	1415	1344	1270	1490	1514	1282	1761	1670	1740	1432	1410	1431
	14	1362	1340	1280	1413	1338	1267	1480	1507	1282	1764	1665	1740	1452	1410	1428
	16	1358	1330	1270	1413	1329	1270	1470	1504	1287	1766	1666	1730	1441	1410	1427
	18	1357	1330	1270	1398	1326	1274	1460	1504	1290	1769	1665	1720	1433	1410	1429
	20	1356	1330	1270	1393	1320	1272	1460	1502	1292	1768	1663	1720	1432	1420	1428
	22	1357	1330	1260	1399	1317	1266	1460	1501	1289	1769	1661	1710	1436	1420	1426
	24	1353	1330	1260	1414	1314	1262	1460	1497	1283	1778	1661	1710	1428	1420	1422
	26	1354	1330	1260	1400	1312	1261	1470	1494	1277	1778	1662	1700	1432	1420	1421
	28	1352	1330	1260	1402	1310	1257	1480	1492	1267	1774	1661	1690	1431	1420	1421
	30	1350	1330	1250	1388	1309	1265	1480	1489	1260	1772	1662	1690	1431	1420	1419
PSU2	2	1464	1390	1140	1306	1423	1370	1610	1375	1378	1756	1792	1640	1942	1400	1525
	4	1465	1390	1130	1321	1448	1344	1590	1382	1384	1762	1813	1640	1891	1400	1504
	6	1465	1390	1120	1323	1449	1333	1580	1384	1388	1765	1815	1630	1899	1390	1488
	8	1463	1400	1110	1314	1448	1330	1580	1383	1389	1770	1808	1630	1842	1390	1482
	10	1464	1390	1110	1310	1444	1325	1570	1377	1393	1774	1811	1630	1890	1400	1482
	12	1466	1400	1120	1317	1447	1326	1570	1380	1391	1778	1811	1620	1902	1410	1480
	14	1465	1400	1120	1327	1440	1330	1590	1376	1397	1777	1815	1620	1895	1400	1480
	16	1465	1400	1110	1321	1442	1331	1620	1375	1395	1778	1814	1610	1897	1400	1484
	18	1464	1390	1110	1352	1442	1333	1630	1374	1408	1779	1817	1620	1880	1400	1491
	20	1464	1390	1110	1324	1441	1334	1620	1377	1414	1781	1817	1610	1874	1410	1483
	22	1464	1390	1100	1309	1442	1329	1600	1378	1409	1784	1816	1600	1882	1410	1481
	24	1464	1390	1110	1307	1441	1319	1570	1379	1395	1785	1816	1600	1861	1410	1482
	26	1463	1390	1100	1347	1445	1319	1560	1379	1382	1785	1818	1600	1877	1410	1477
	28	1463	1390	1100	1319	1445	1329	1580	1374	1373	1789	1818	1610	1865	1410	1478
	30	1462	1390	1090	1340	1447	1322	1590	1373	1371	1786	1818	1610	1860	1410	1474



Table C13. G\* strain sweeps test results for the unaged asphalt binder samples, cont'd.

Sample ID	Loading time	1	3	4	5	6	7	9	8	10	13	14	16	17	19	20
PSU3	2	1406	1410	1550	1404	1459	1475	1690	1331	1402	2177	1999	1570	2233	1410	1484
	4	1401	1430	1530	1395	1438	1453	1670	1329	1399	2188	2007	1570	2338	1410	1467
	6	1403	1420	1520	1401	1430	1435	1660	1331	1407	2201	2010	1570	2286	1400	1456
	8	1407	1420	1520	1385	1420	1436	1650	1336	1410	2208	2017	1560	2264	1410	1453
	10	1411	1420	1530	1391	1412	1439	1630	1331	1416	2210	2012	1560	2283	1410	1450
	12	1409	1420	1520	1383	1407	1435	1610	1331	1416	2213	2007	1550	2260	1410	1445
	14	1409	1420	1530	1389	1401	1433	1610	1327	1422	2216	2016	1550	2261	1410	1452
	16	1409	1410	1520	1366	1401	1432	1600	1326	1423	2215	2014	1540	2268	1420	1458
	18	1409	1410	1520	1396	1397	1431	1600	1329	1435	2222	2011	1540	2259	1420	1453
	20	1407	1410	1510	1400	1393	1430	1620	1329	1438	2220	2011	1540	2264	1420	1443
	22	1410	1400	1520	1366	1393	1429	1640	1329	1432	2223	2009	1540	2255	1420	1443
	24	1409	1400	1520	1396	1394	1429	1660	1331	1424	2226	2006	1540	2229	1430	1446
	26	1408	1400	1520	1379	1393	1429	1640	1327	1408	2228	2007	1540	2227	1430	1449
	28	1408	1390	1510	1379	1393	1428	1610	1330	1405	2230	2005	1550	2231	1430	1451
	30	1407	1390	1520	1361	1396	1428	1600	1332	1404	2225	2003	1550	2227	1430	1446
PSU4	2	1389		1120	1330	1366	1456	818	1294	1306	1509	1839	1980	1603	1330	1523
	4	1385		1120	1328	1353	1436	809	1304	1297	1511	1822	1970	1650	1330	1498
	6	1388		1120	1326	1349	1427	804	1299	1296	1516	1829	1970	1620	1330	1484
	8	1385		1130	1323	1343	1419	801	1303	1299	1518	1833	1960	1594	1340	1476
	10	1383		1120	1333	1350	1419	800	1302	1300	1520	1839	1960	1629	1340	1482
	12	1382		1110	1333	1345	1419	799	1300	1305	1523	1829	1950	1595	1340	1482
	14	1382		1120	1333	1338	1419	799	1300	1309	1524	1829	1940	1587	1340	1476
	16	1380		1120	1324	1342	1415	805	1300	1313	1524	1826	1930	1597	1340	1479
	18	1380		1120	1322	1338	1411	814	1297	1309	1525	1824	1930	1583	1340	1477
	20	1379		1110	1321	1338	1412	821	1294	1303	1525	1829	1920	1581	1340	1473
	22	1378		1110	1317	1336	1409	821	1290	1293	1527	1823	1910	1567	1340	1467
	24	1377		1110	1331	1335	1408	816	1289	1285	1525	1823	1900	1561	1350	1467
	26	1377		1110	1317	1333	1409	815	1287	1278	1525	1820	1900	1555	1350	1470
	28	1375		1110	1321	1334	1407	811	1284	1272	1521	1821	1890	1566	1350	1472
	30	1373		1110	1314	1336	1408	802	1281	1266	1521	1820	1890	1561	1350	1467

Table C14. Phase angle strain sweeps test results for the unaged asphalt binder samples.

Sample ID	Loading time	1	3	4	5	6	8	9	10	13	14	16	17	19	20
PSU1	2	75.7	76.7	77.0	76.1	76.4	77.3		77.1	74.7	74.1	76.5	76.0	75.9	76.0
	4	75.8	76.5	76.7	75.8	76.3	76.3		76.8	74.6	74.6	76.3	76.0	75.9	76.0
	6	75.7	76.4	76.6	75.9	76.3	76.0		76.8	74.6	74.8	76.2	76.1	75.9	76.0
	8	75.7	76.3	76.6	76.0	75.9	75.8		76.7	74.6	74.6	76.2	75.7	75.9	76.0
	10	75.6	76.3	76.6	76.0	76.0	75.8		76.7	74.6	74.6	76.3	75.7	75.6	76.0
	12	75.7	76.3	76.6	75.8	76.2	75.7		76.7	74.6	74.7	76.3	76.1	76.0	76.0
	14	75.8	76.3	76.6	76.1	76.4	75.6		76.7	74.6	74.5	76.3	75.7	76.0	76.0
	16	75.7	76.4	76.6	76.2	76.2	75.6		76.8	74.6	74.8	76.3	75.7	76.0	76.0
	18	75.7	76.4	76.7	76.1	76.2	75.6		76.8	74.6	74.7	76.4	75.9	76.1	76.0
	20	75.7	76.4	76.7	76.2	76.2	75.7		76.8	74.6	74.7	76.4	76.1	76.1	76.0
	22	75.8	76.4	76.7	76.1	76.2	75.7		76.9	74.7	74.8	76.4	75.8	76.1	76.0
	24	75.8	76.5	76.8	76.3	76.2	75.7		76.9	74.7	74.8	76.5	76.1	76.2	76.1
	26	75.7	76.5	76.8	76.3	76.3	75.7		76.9	74.7	74.8	76.5	76.2	76.2	76.1
	28	75.8	76.5	76.8	75.9	76.2	75.8		77.0	74.7	74.7	76.6	75.8	76.3	76.1
	30	75.8	76.6	76.9	76.3	76.3	75.8		77.0	74.7	74.8	76.6	75.8	76.3	76.1
PSU2	2	86.2	87.1	86.9	86.6	85.3	87.1	88.0	85.9	85.1	87.2	85.3		86.7	86.5
	4	86.4	86.8	86.7	86.4	86.1	86.9	87.0	85.8	85.6	87.0	85.3		86.7	86.4
	6	86.1	86.7	86.7	86.4	86.7	86.8	86.7	85.9	85.6	86.9	85.8		86.7	86.4
	8	86.0	86.7	86.6	86.3	86.3	86.8	86.5	85.9	85.5	86.9	85.5		86.7	86.4
	10	86.1	86.7	86.7	86.1	86.4	86.7	86.4	85.8	85.6	86.9	85.4		86.7	86.4
	12	86.1	86.7	86.7	86.4	86.5	86.7	86.3	85.8	85.5	86.9	85.3		86.7	86.4
	14	86.0	86.7	86.7	86.1	86.3	86.7	86.2	85.8	85.5	86.8	85.5		86.7	86.4
	16	86.0	86.7	86.7	86.5	86.4	86.7	86.3	85.8	85.6	86.8	85.6		86.8	86.4
	18	86.1	86.7	86.7	86.5	86.3	86.7	86.3	85.8	85.5	86.8	85.5		86.8	86.4
	20	86.0	86.7	86.7	82.6	86.3	86.8	86.2	85.8	85.5	86.8	85.6		86.8	86.4
	22	86.1	86.7	86.7	87.4	86.3	86.8	86.2	85.8	85.6	86.8	85.6		86.8	86.4
	24	86.1	86.7	86.7	87.3	86.3	86.8	86.2	85.8	85.5	86.8	85.5		86.8	86.4
	26	86.1	86.7	86.7	83.9	86.3	86.7	86.2	85.8	85.6	86.8	85.7		86.9	86.4
	28	86.1	86.7	86.7	86.1	86.3	86.7	86.2	85.8	85.6	86.9	85.3		86.9	86.4
	30	86.1	86.7	86.7	86.3	86.3	86.7	86.2	85.9	85.6	86.9	85.6		86.9	86.4

Table C14 Phase angle strain sweeps test results for the unaged asphalt binder samples, cont'd.

Sample ID	Loading time	1	3	4	5	6	8	9	10	13	14	16	17	19	20
PSU3	2	86.3	87.1	86.8	86.2	85.9	87.1	88.0	85.4	86.0	87.4	83.8		86.8	86.6
	4	86.2	86.8	86.6	86.1	86.1	86.8	87.0	85.3	86.0	87.2	84.1		86.8	86.4
	6	86.2	86.8	86.5	86.5	86.2	86.8	86.6	85.3	85.8	87.1	84.0		86.8	86.4
	8	86.2	86.7	86.5	86.0	86.3	86.7	86.5	85.3	85.9	87.1	84.0		86.8	86.4
	10	86.2	86.7	86.5	86.5	86.4	86.7	86.4	85.3	85.9	87.0	84.0		86.8	86.4
	12	86.2	86.7	86.5	86.3	86.3	86.7	86.3	85.3	85.8	87.0	84.2		86.8	86.4
	14	86.2	86.7	86.5	86.3	86.3	86.7	86.2	85.3	85.7	87.0	84.3		86.8	86.4
	16	86.1	86.7	86.5	86.4	86.3	86.7	86.2	85.3	85.6	87.0	84.3		86.8	86.4
	18	86.2	86.7	86.5	86.5	86.3	86.7	86.2	85.3	85.6	87.0	83.9		86.8	86.4
	20	86.2	86.7	86.5	86.4	86.4	86.7	86.2	85.3	85.7	87.0	83.9		86.8	86.4
	22	86.2	86.7	86.5	86.4	86.4	86.7	86.2	85.3	85.6	87.0	84.4		86.8	86.4
	24	86.2	86.7	86.5	86.4	86.3	86.7	86.2	85.3	85.7	87.0	84.2		86.9	86.4
	26	86.2	86.7	86.5	86.1	86.4	86.7	86.2	85.3	85.7	87.0	84.3		86.9	86.4
	28	86.2	86.7	86.5	86.3	86.4	86.7	86.2	85.3	85.7	87.0	84.0		86.9	86.4
	30	86.2	86.7	86.5	86.0	86.3	86.7	86.2	85.3	85.7	87.1	84.3		86.9	86.4
PSU4	2	75.9	77.3	77.3	75.6	76.0	76.9	79.4	75.6	74.8	75.5	75.0		76.5	75.7
	4	75.6	77.1	77.1	75.5	75.5	76.7	78.6	75.4	75.0	75.3	74.9		76.5	75.6
	6	75.8	77.0	77.0	75.9	76.4	76.6	78.2	75.4	74.8	75.3	74.9		76.5	75.6
	8	75.6	77.0	77.0	76.0	76.3	76.6	78.0	75.4	75.0	75.3	75.2		76.6	75.6
	10	75.6	77.0	77.0	75.8	75.8	76.6	77.9	75.4	75.1	75.3	74.7		76.6	75.6
	12	75.7	77.0	77.0	75.5	76.0	76.6	77.8	75.4	74.9	75.3	74.7		76.6	75.6
	14	75.6	77.0	77.0	75.6	75.9	76.6	77.8	75.4	75.1	75.4	75.0		76.6	75.6
	16	75.6	77.0	77.0	75.9	76.0	76.7	77.8	75.5	75.1	75.4	74.8		76.7	75.6
	18	75.6	77.1	77.1	75.5	76.1	76.7	77.8	75.5	75.1	75.4	74.9		76.7	75.6
	20	75.6	77.1	77.1	75.8	76.1	76.7	77.9	75.5	75.1	75.5	75.1		76.7	75.6
	22	75.7	77.1	77.1	75.9	76.2	76.7	77.9	75.5	75.1	75.5	75.2		76.8	75.6
	24	75.6	77.1	77.1	75.9	76.1	76.7	77.9	75.5	75.2	75.6	75.1		76.8	75.6
	26	75.7	77.2	77.2	75.8	76.1	76.8	77.9	75.5	75.2	75.6	74.8		76.9	75.7
	28	75.7	77.2	77.2	75.7	76.2	76.8	77.9	75.6	75.2	75.6	75.3		76.9	75.7
	30	75.7	77.2	77.2	75.5	76.2	76.8	77.9	75.6	75.2	75.7	75.1		76.9	75.7

**APPENDIX D**  
**PLOTS OF ROUND ROBIN TEST RESULTS**

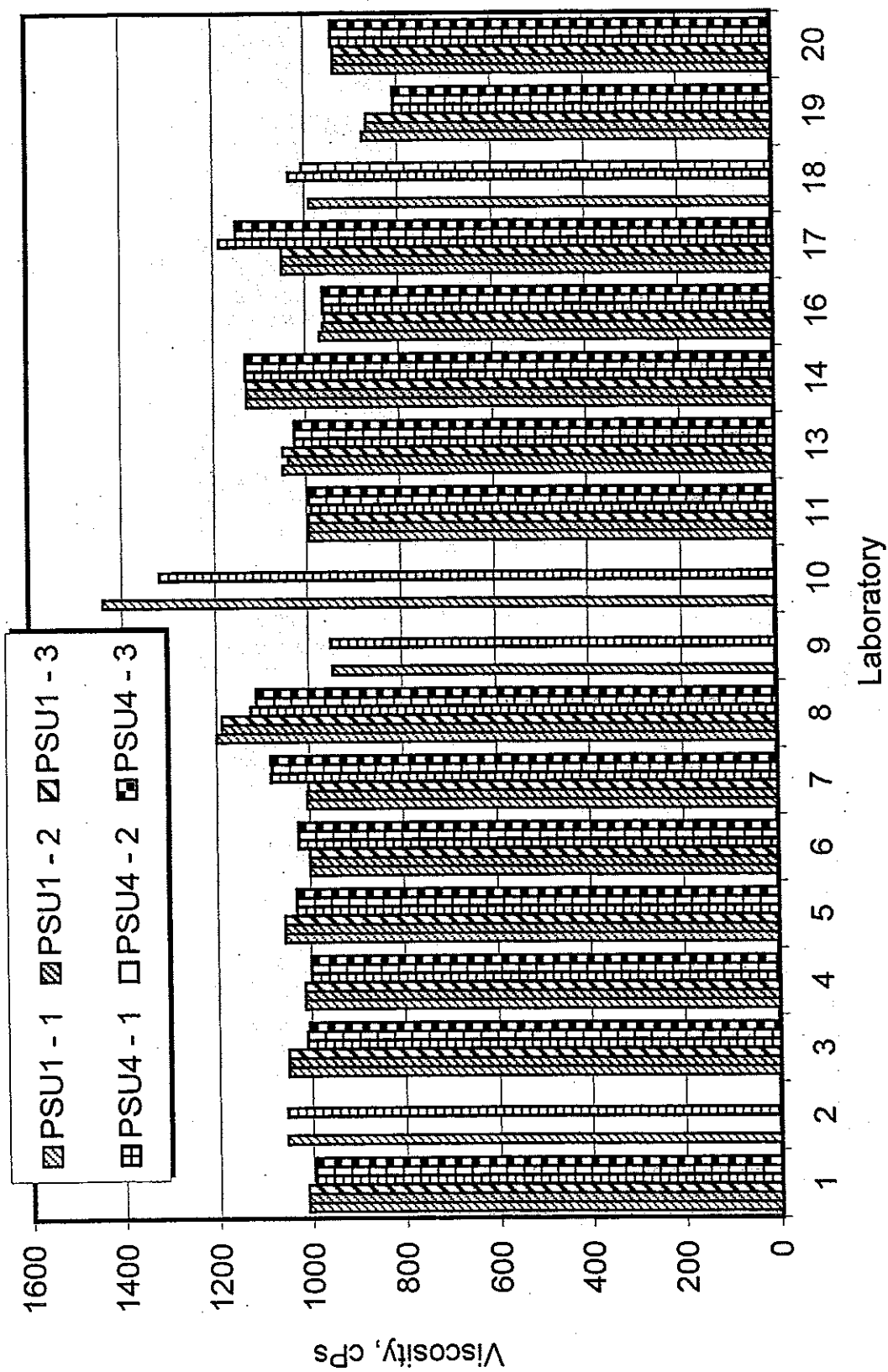


Figure D1. Rotational viscosity data for unaged PSU1 and PSU4 at 135°C.

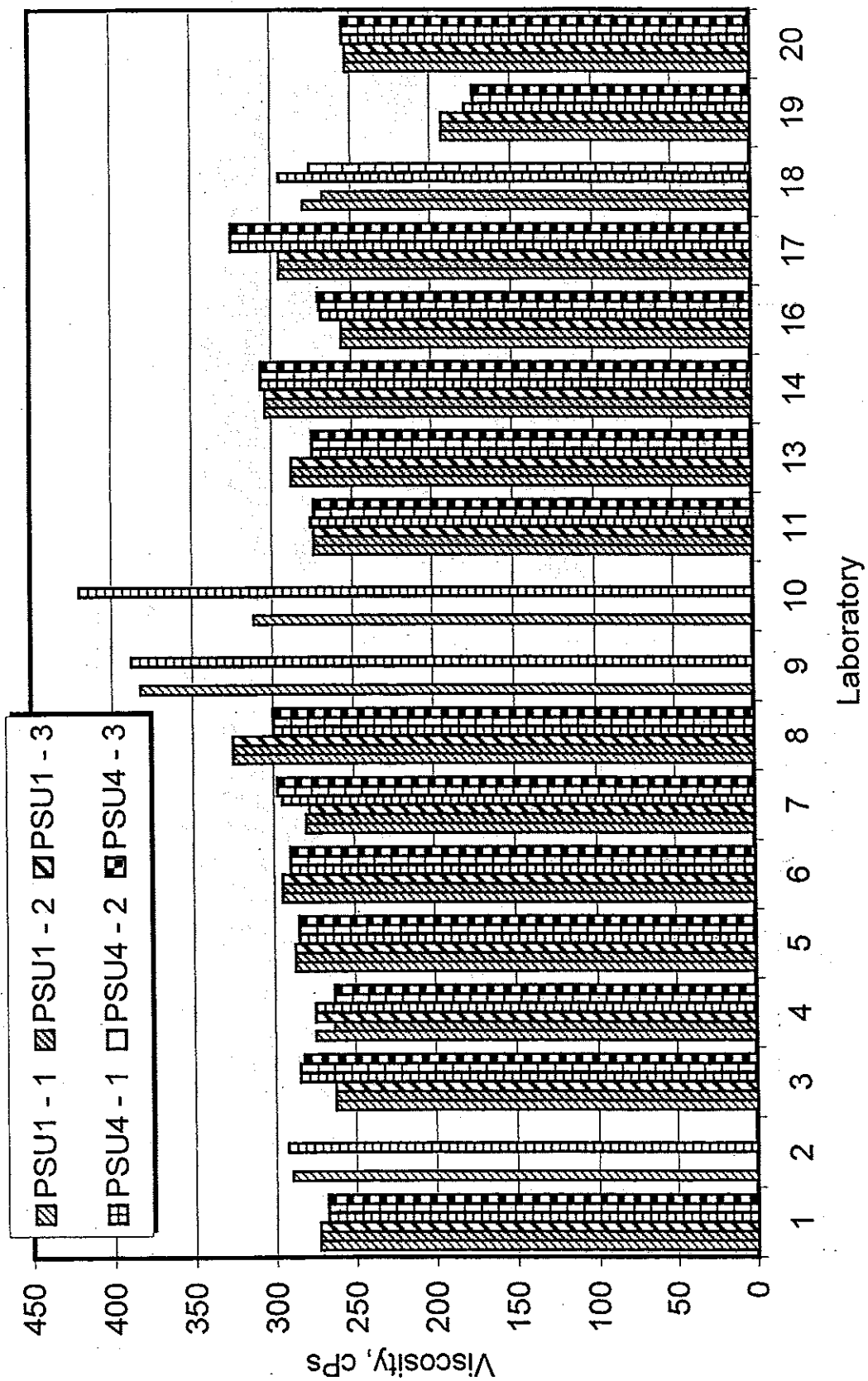


Figure D2. Rotational viscosity data for unaged PSU1 and PSU4 at 165°C.

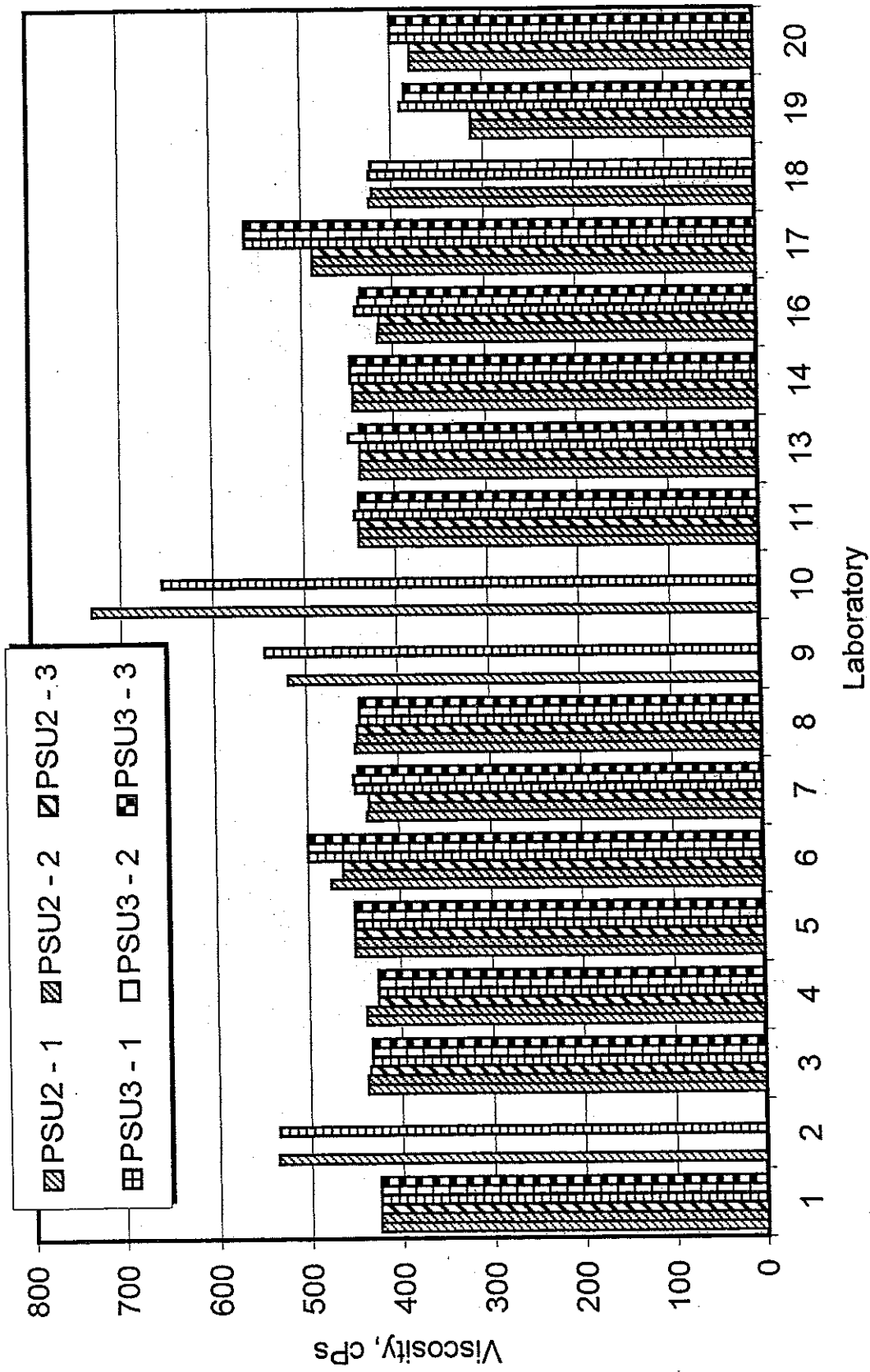


Figure D3. Rotational viscosity data for unaged PSU2 and PSU3 at 135°C.

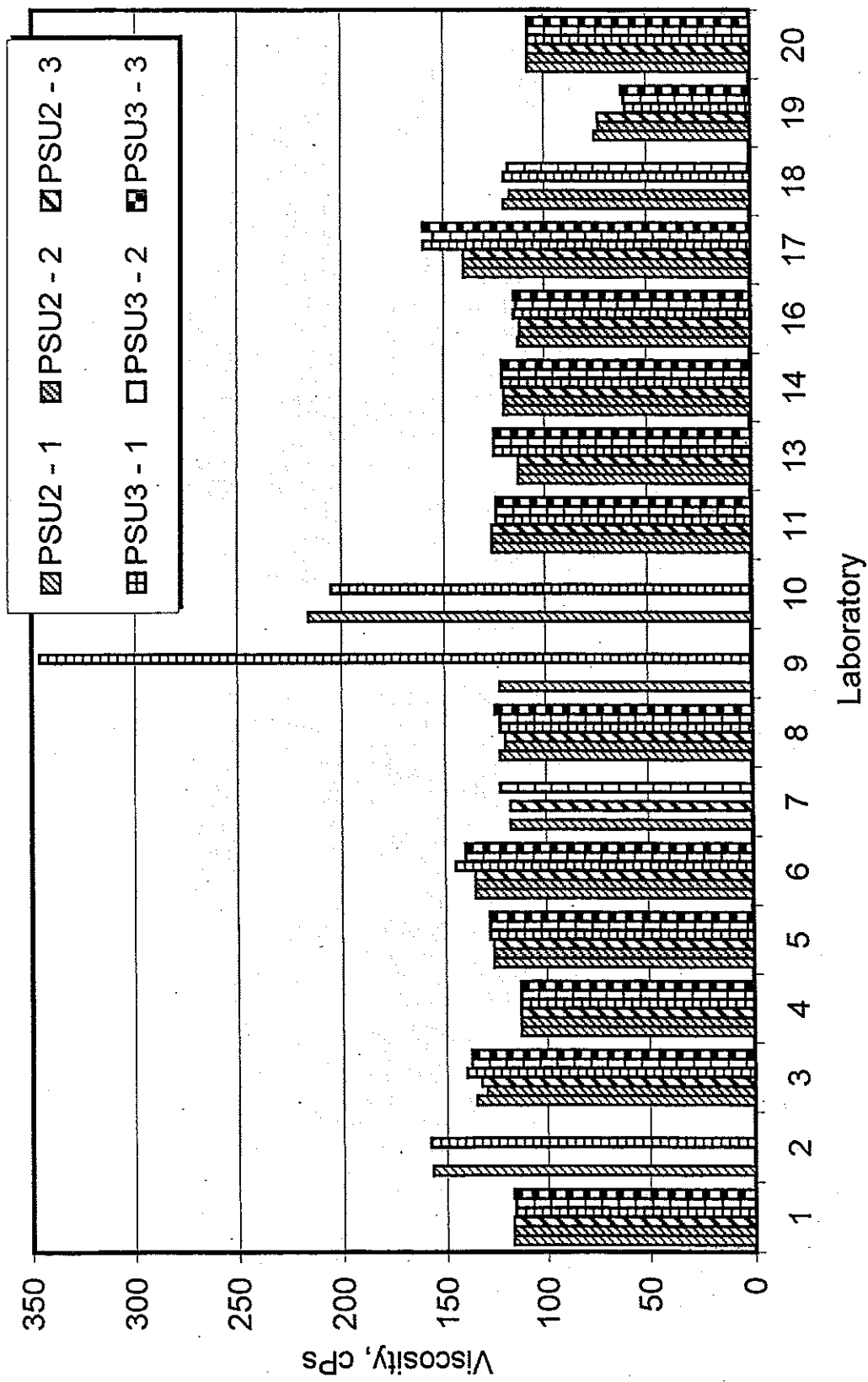


Figure D4. Rotational viscosity data for unaged PSU2 and PSU3 at 165°C.



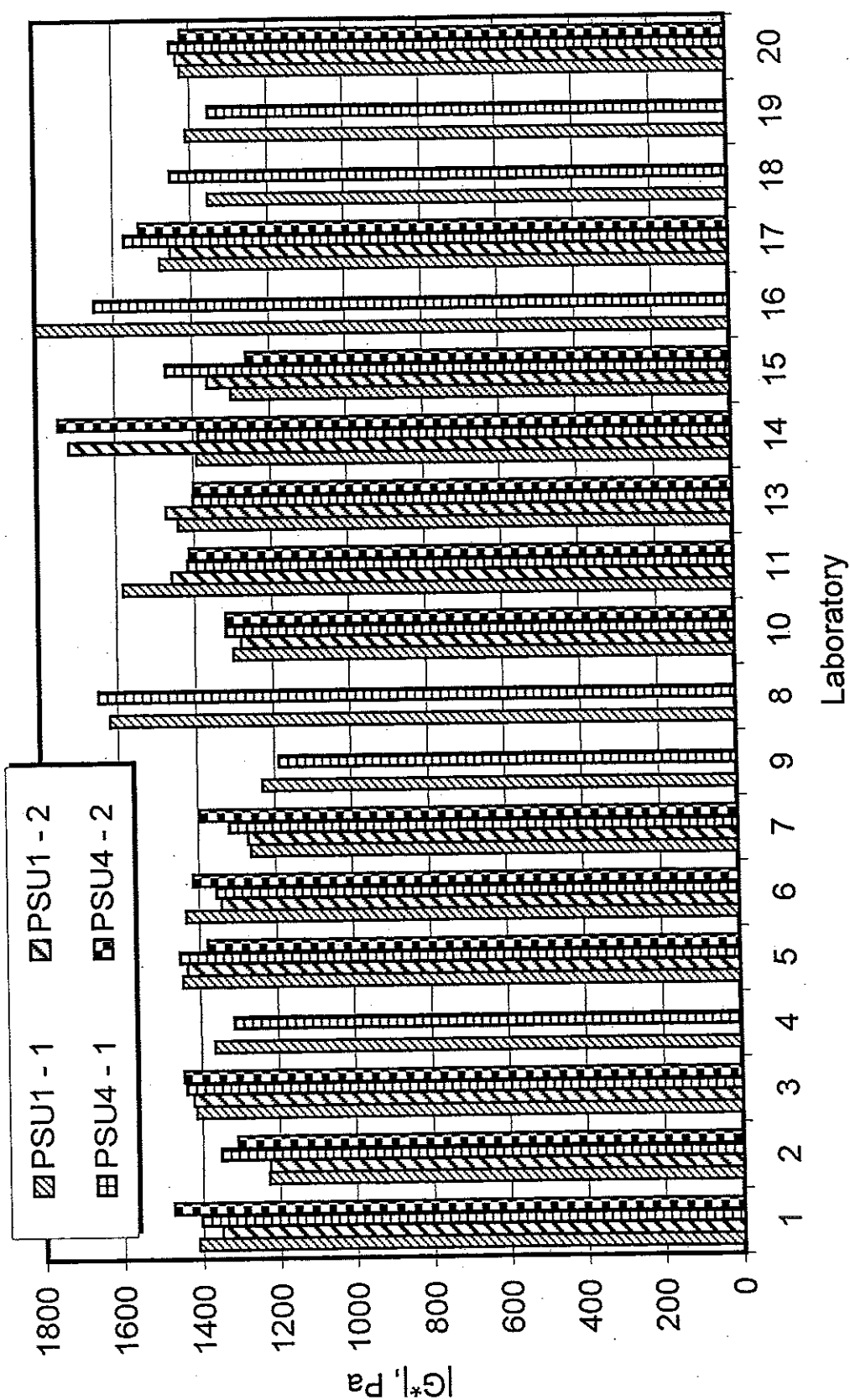


Figure D5.  $G^*$  data for unaged PSU1 and PSU4 at 70°C.

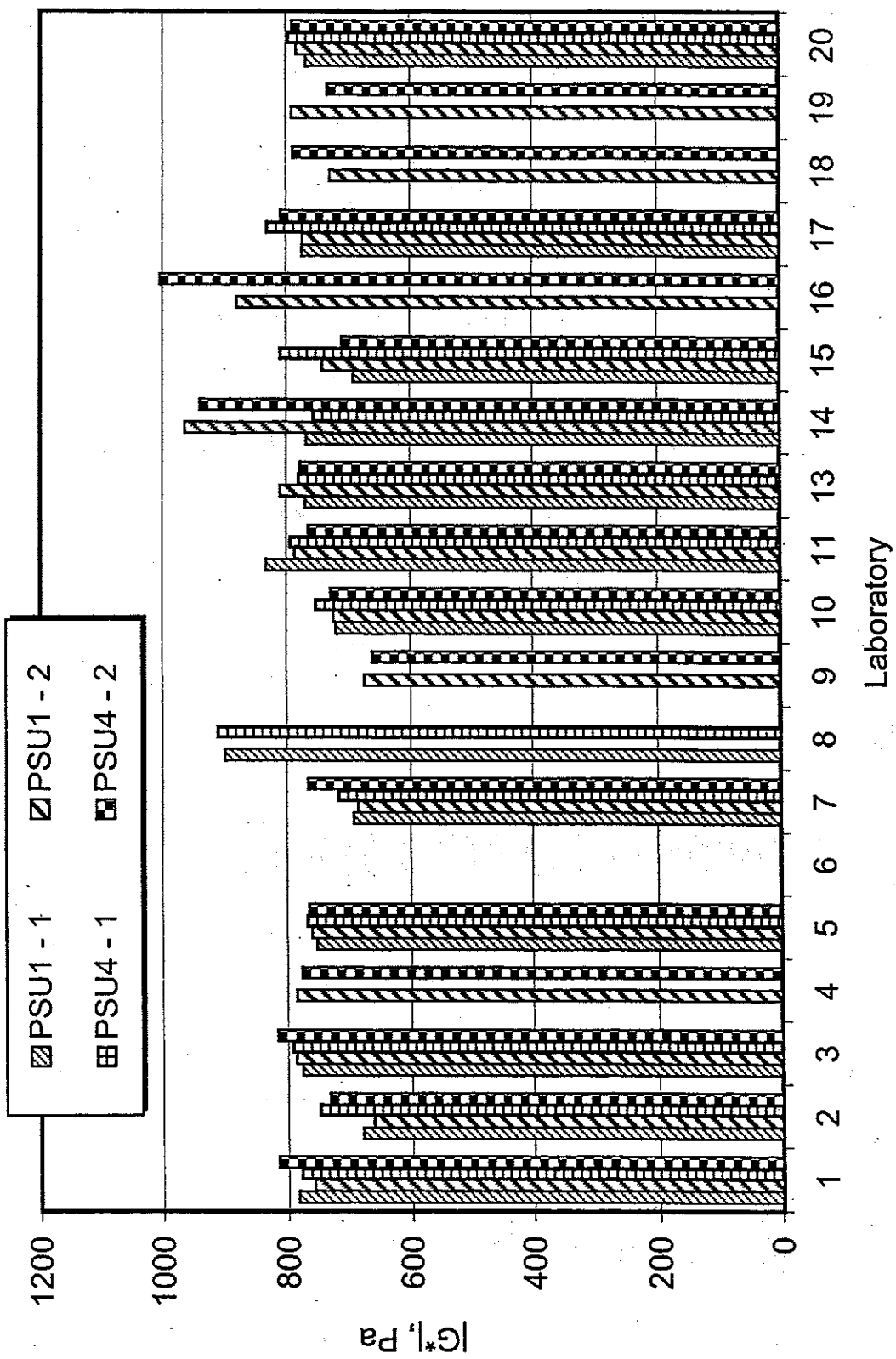


Figure D6. G\* data for unaged PSU1 and PSU4 at 76°C.

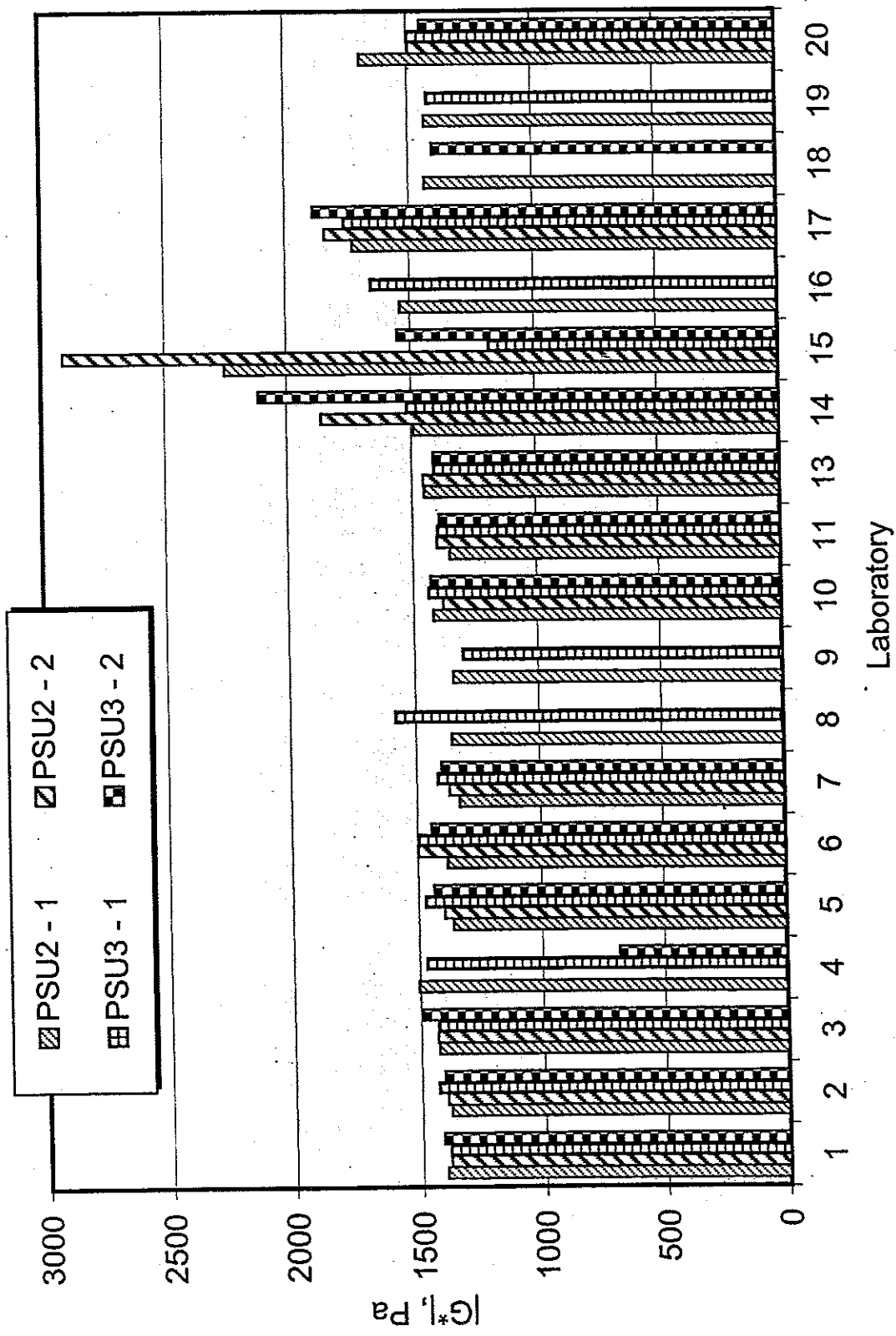
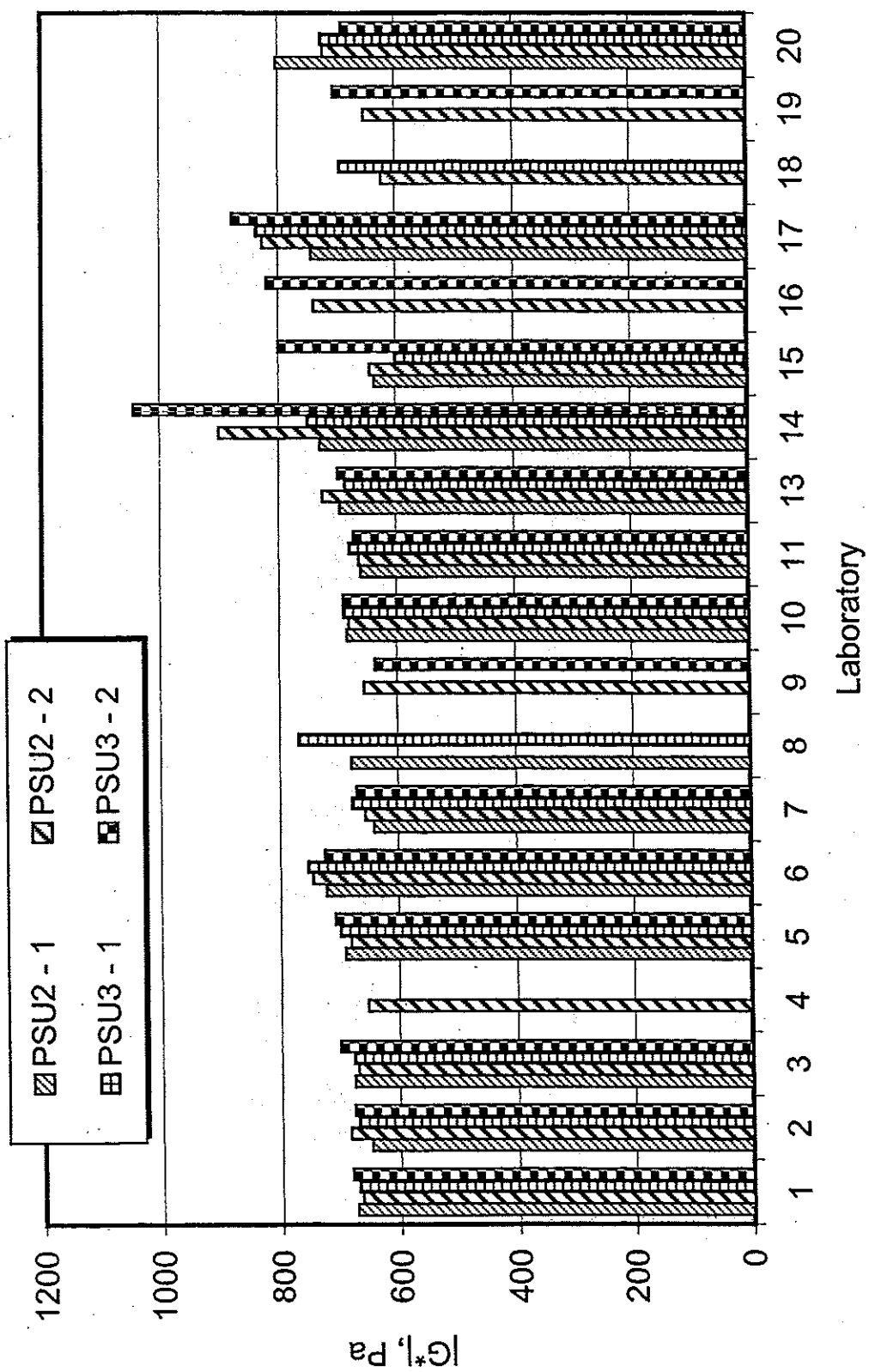


Figure D7.  $G^*$  data for unaged PSU2 and PSU3 at 64°C.

Figure D8.  $G^*$  data for unaged PSU2 and PSU3 at 70°C.

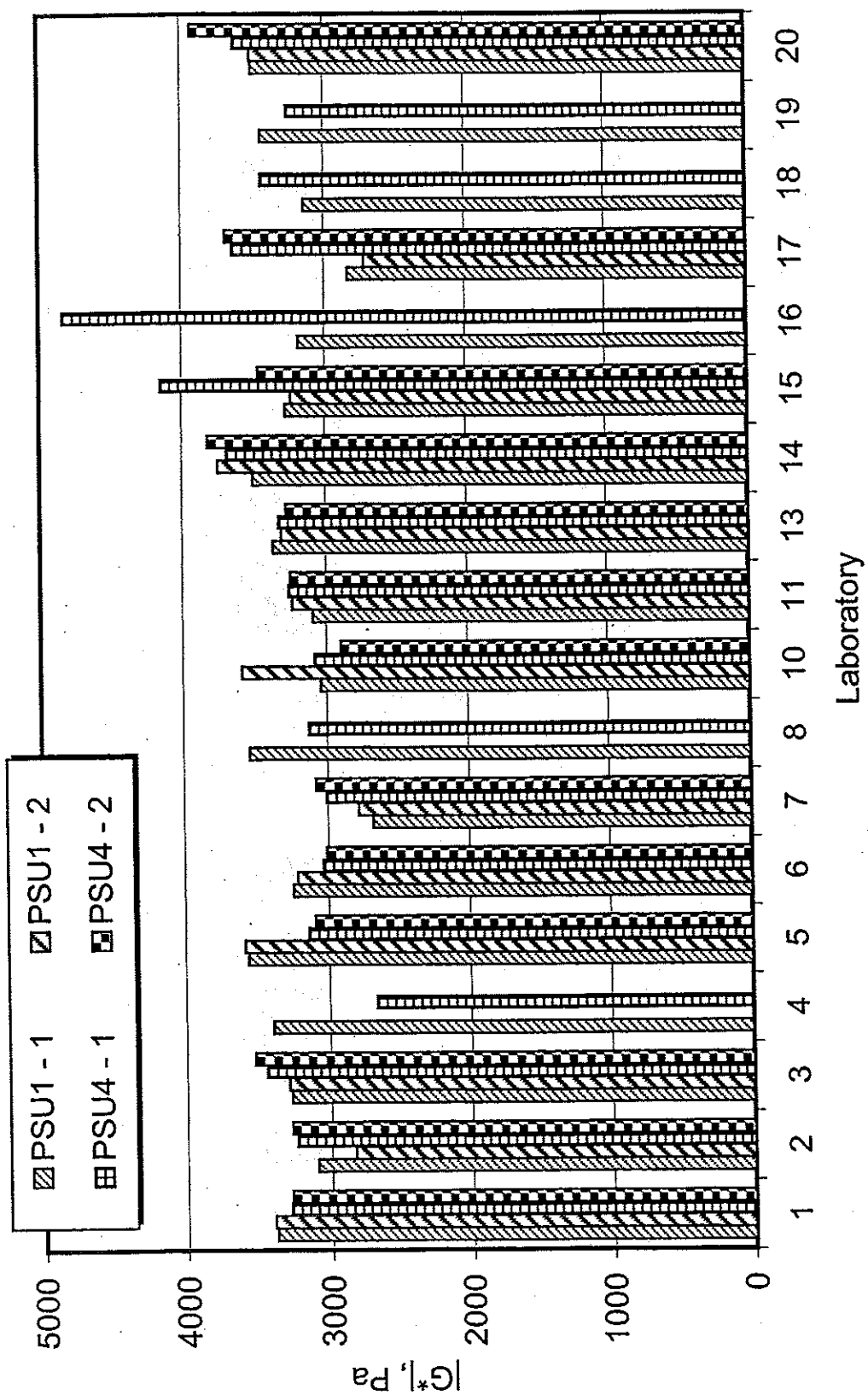


Figure D9.  $G^*$  data for RTFOT PSU1 and PSU4 at 70°C.

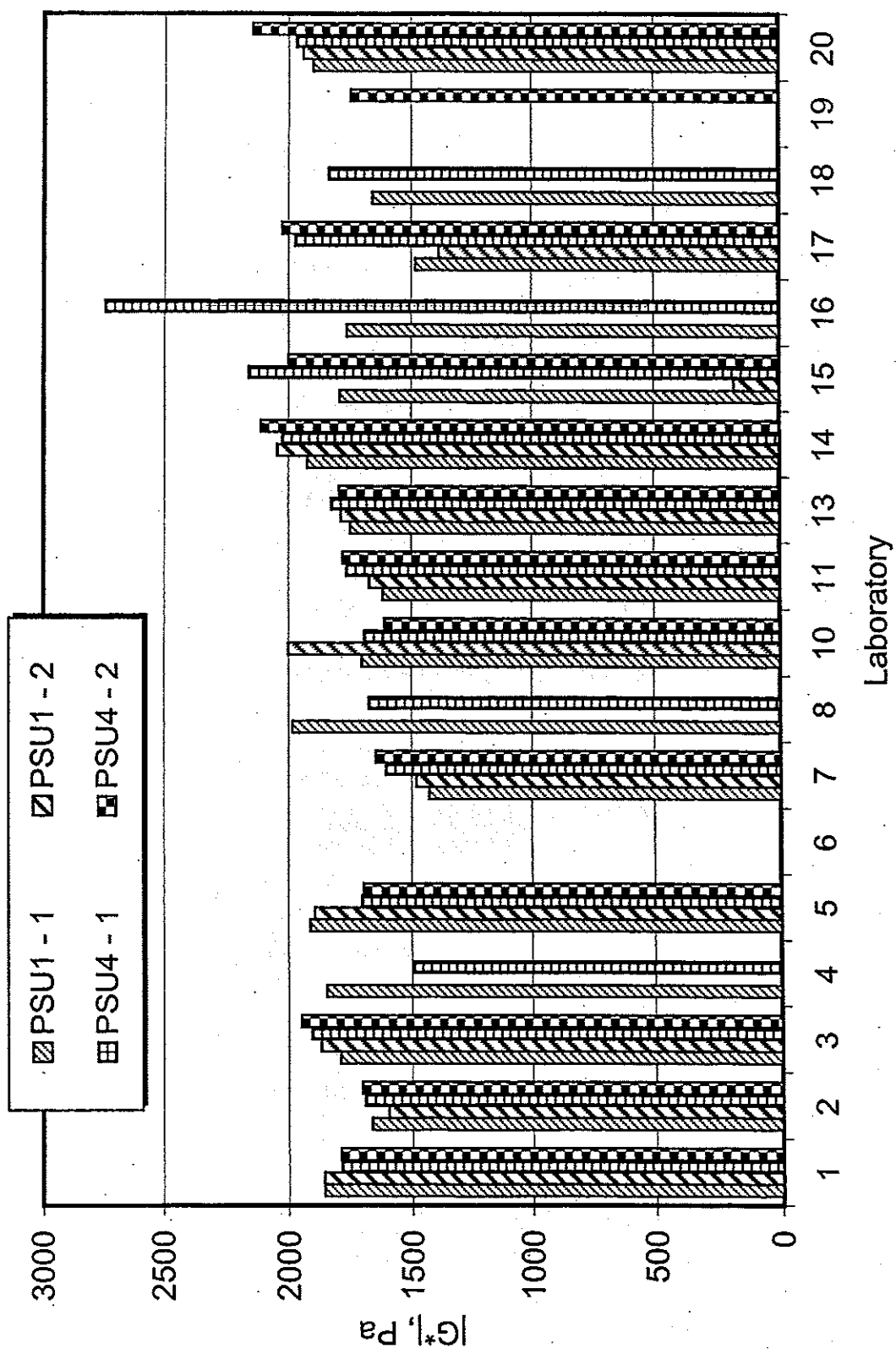


Figure D10.  $G^*$  data for RTFOT PSU1 and PSU4 at 76°C.

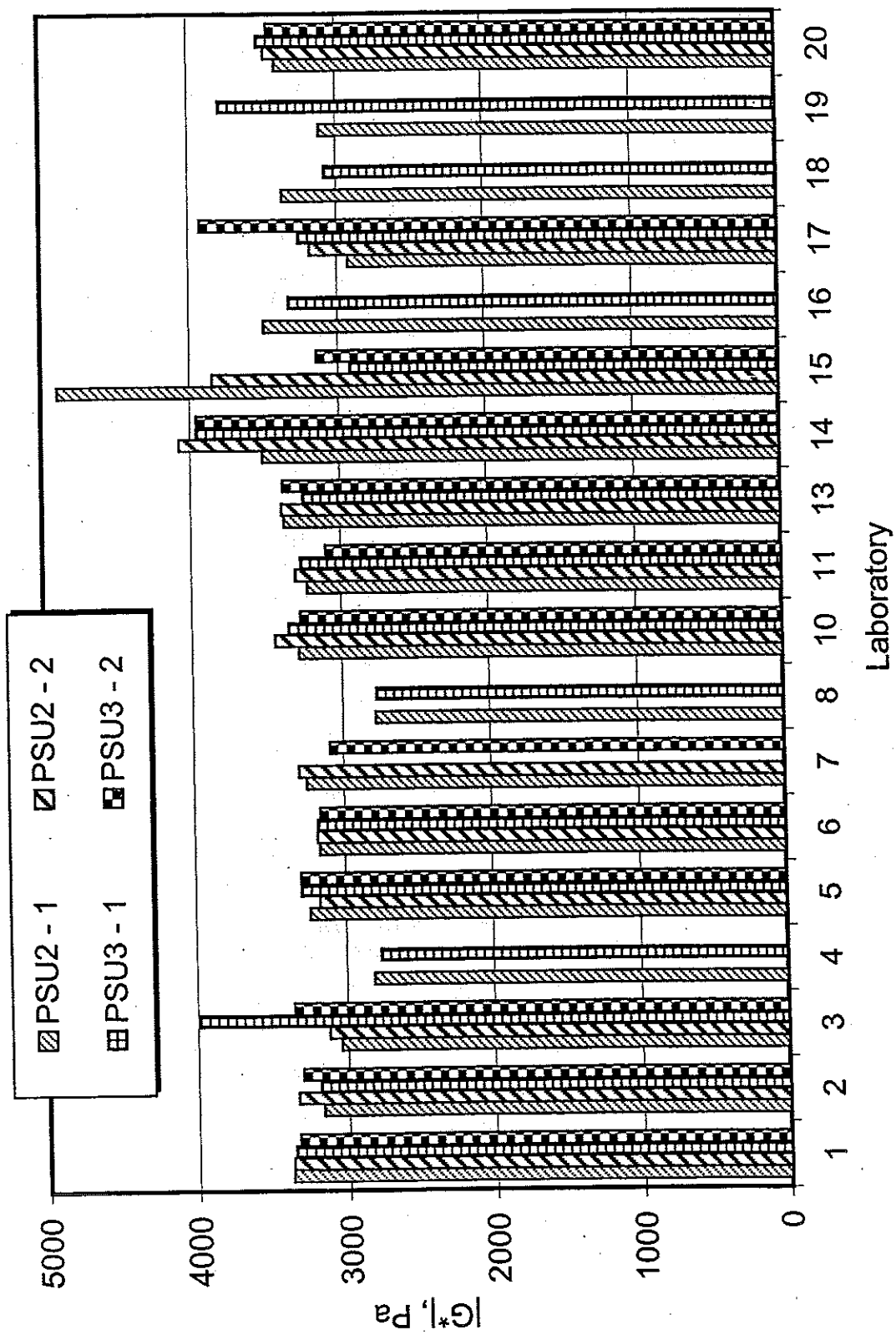
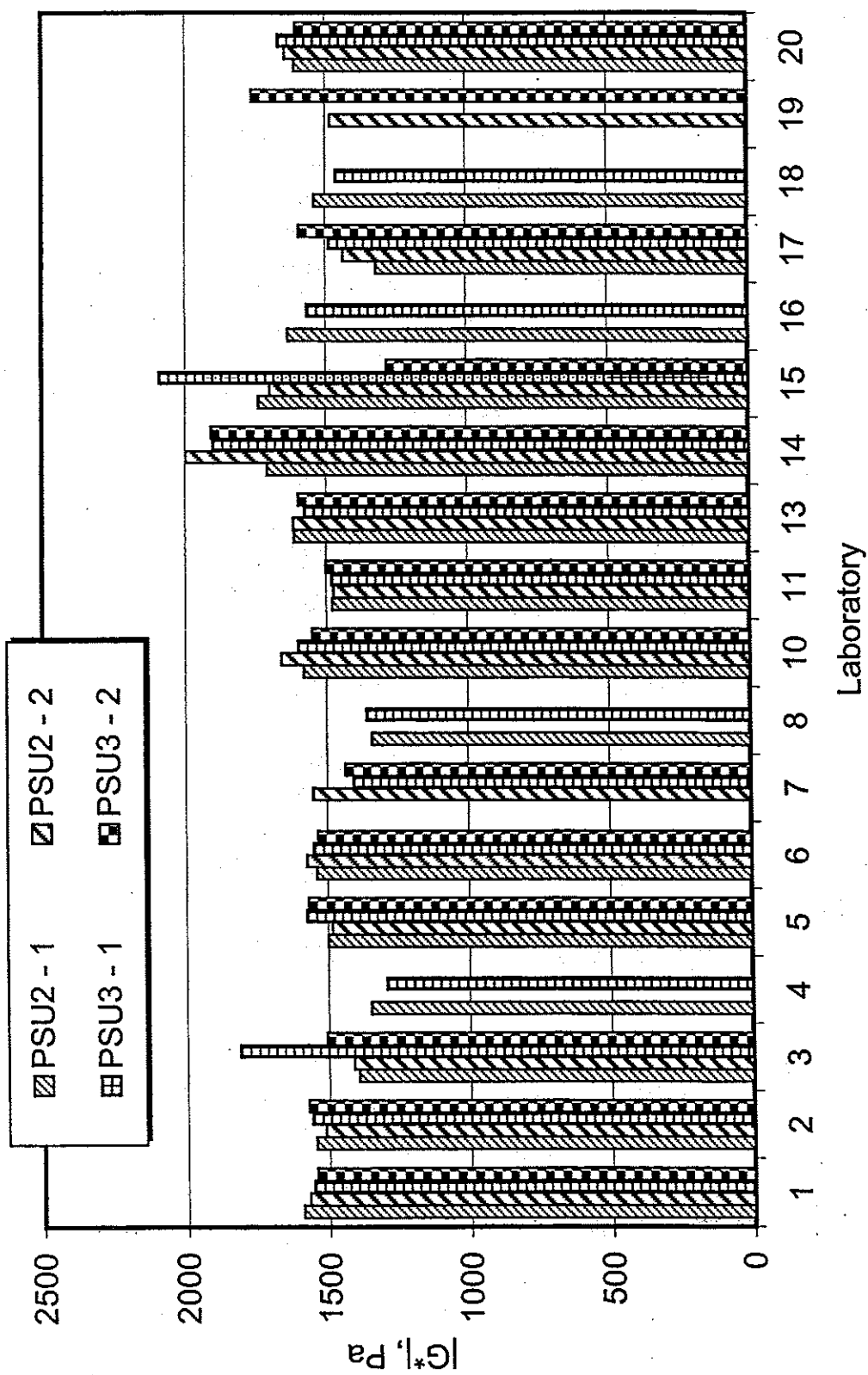


Figure D11.  $G^*$  data for RTFOT PSU2 and PSU3 at 64°C.

Figure D12.  $G^*$  data for RTFOT PSU2 and PSU3 at 70°C.



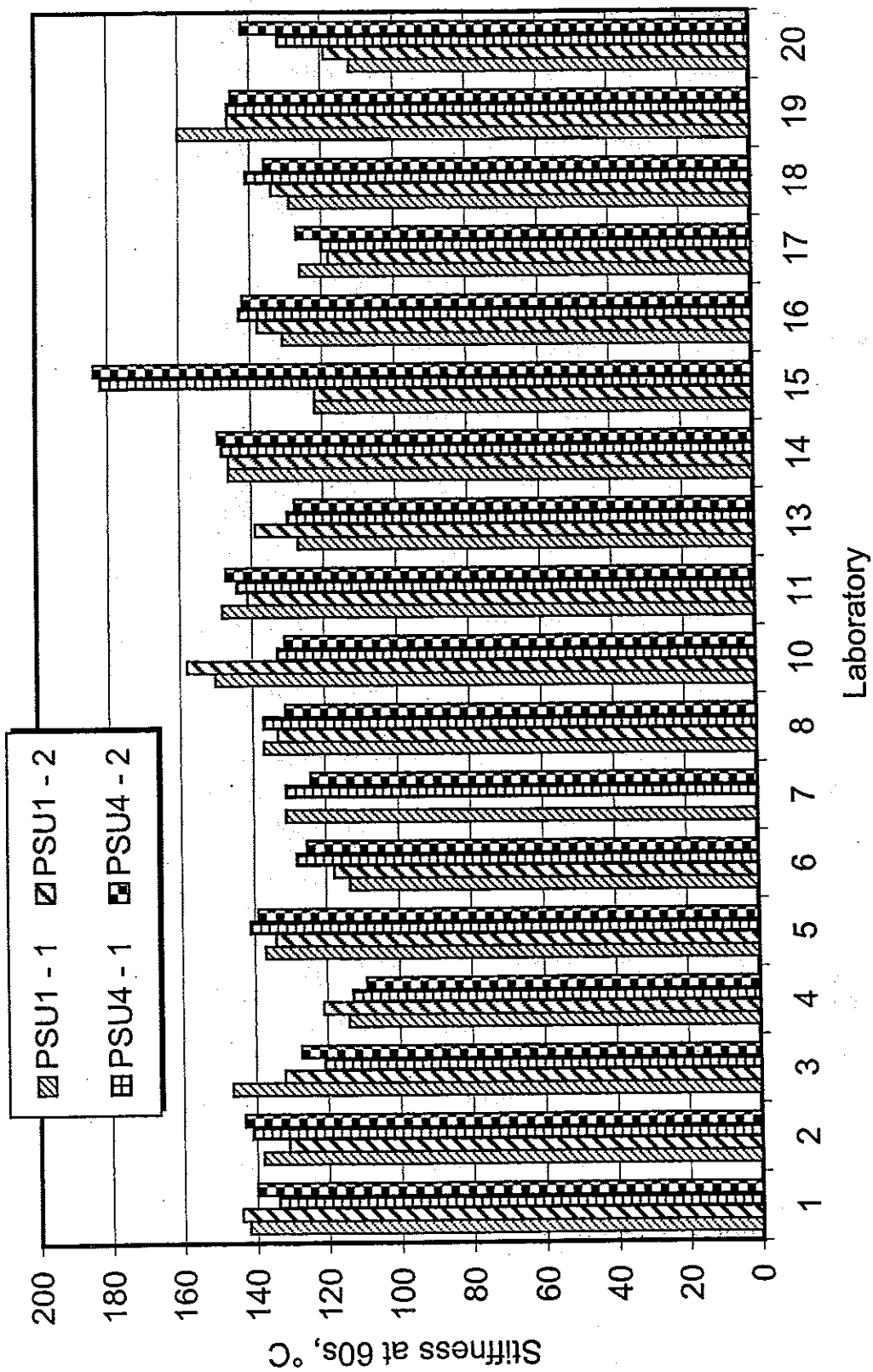


Figure D13. Stiffness data for PAV PSU1 and PSU4 at -12°C.

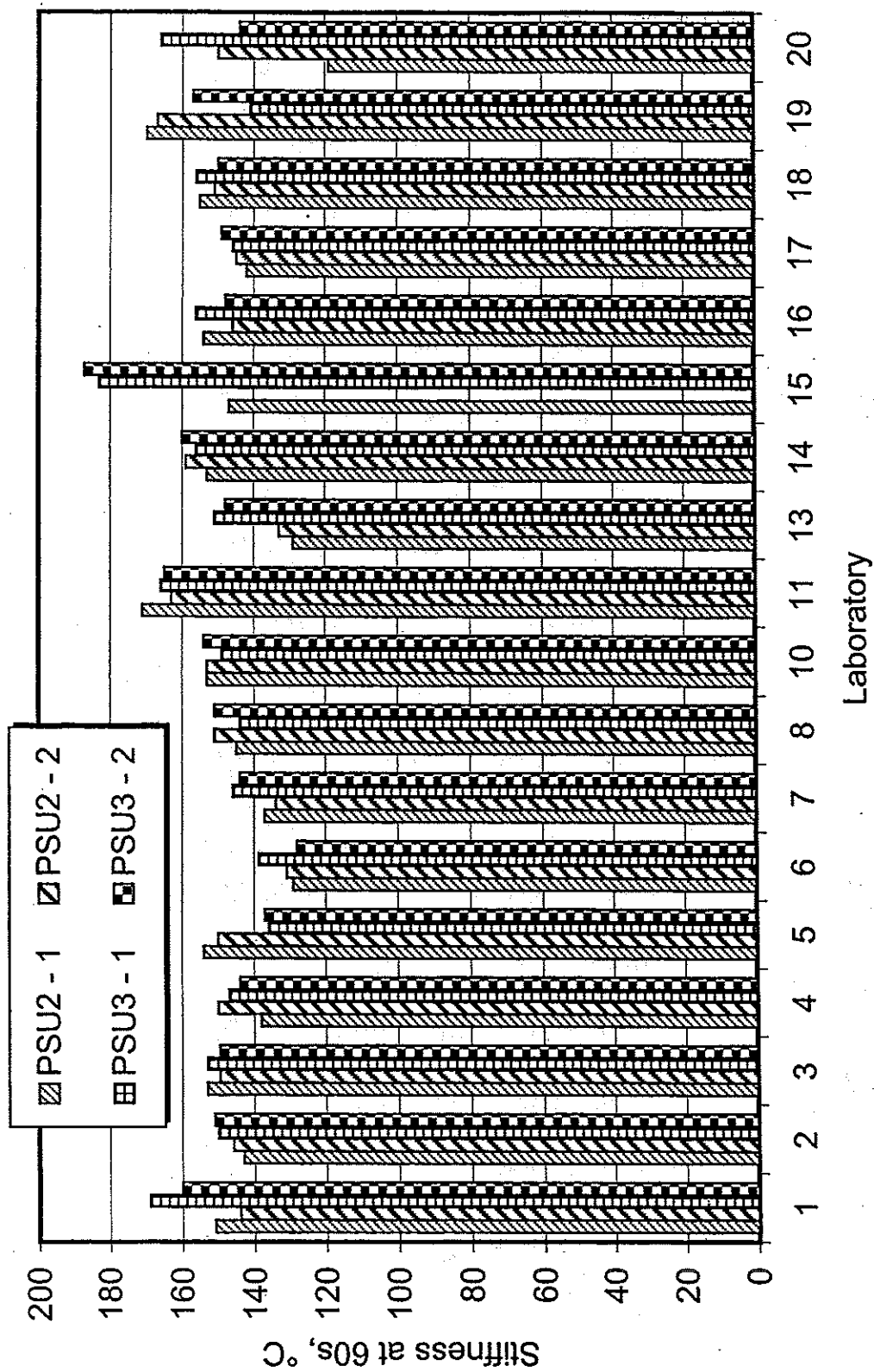


Figure D14. Stiffness data for PAV PSU2 and PSU3 at -12°C.

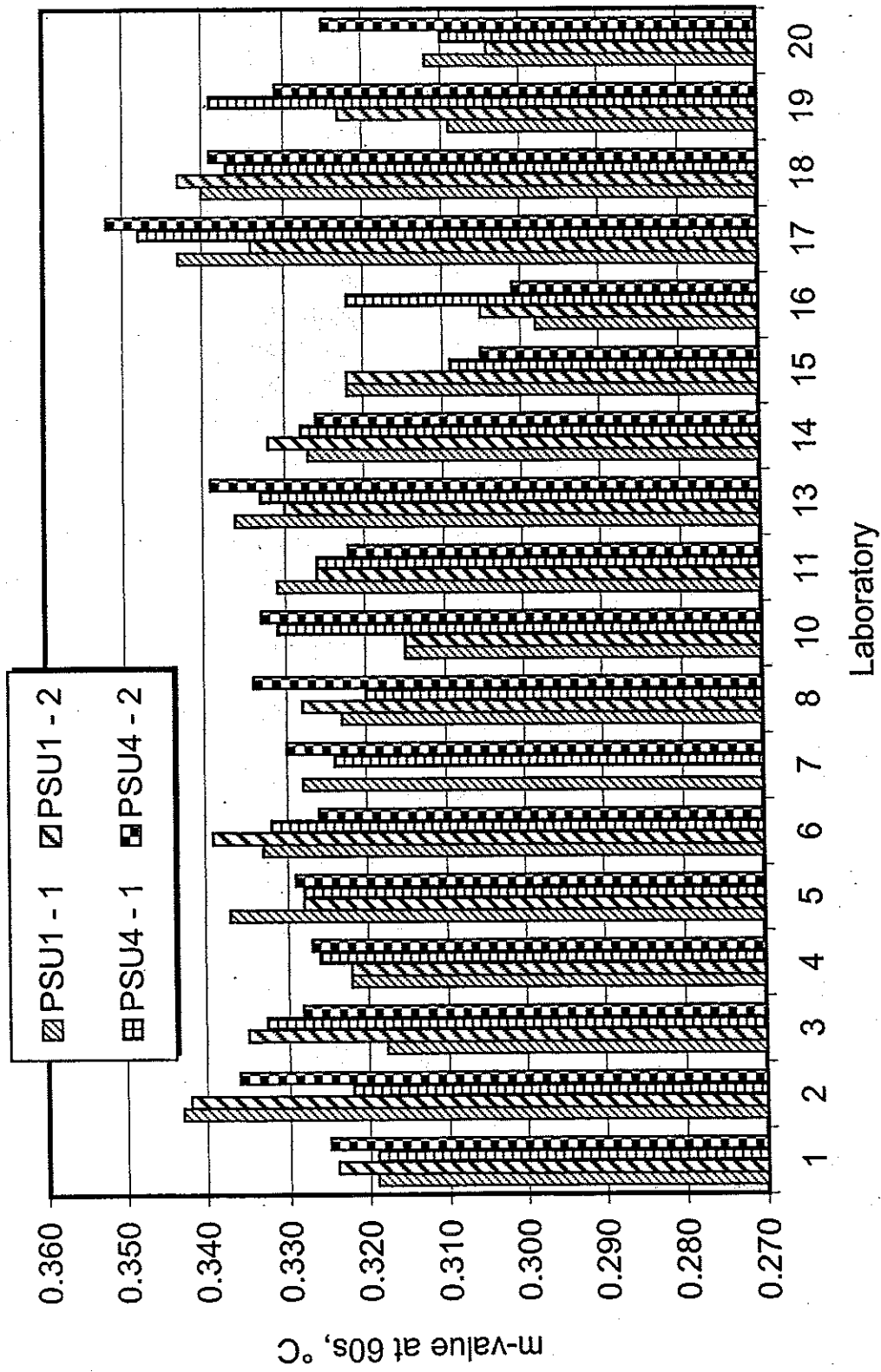


Figure D15. m-value data for PAV PSU1 and PSU4 at -12°C.

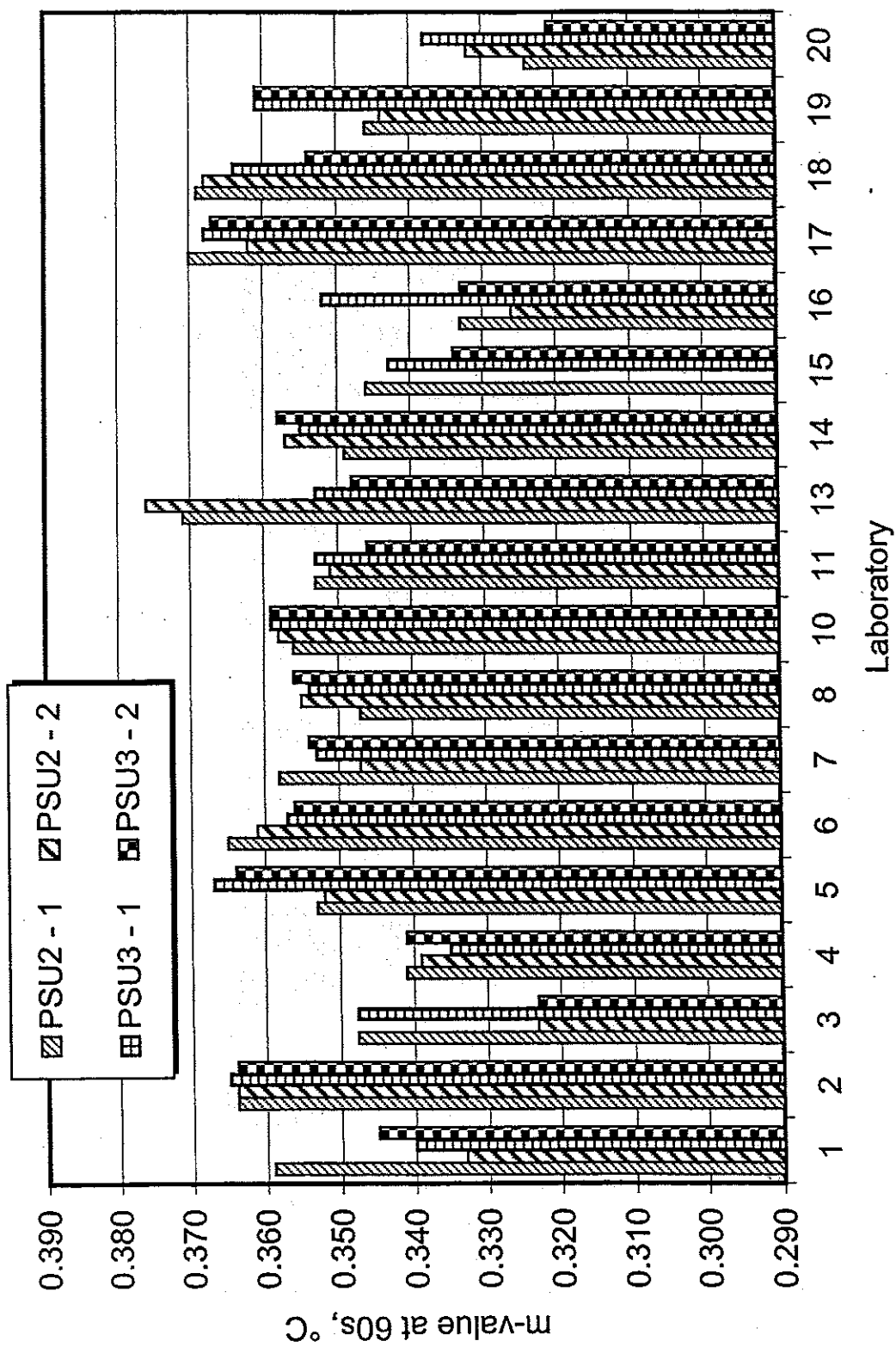


Figure D16. m-value data for PAV PSU2 and PSU3 at -12°C.

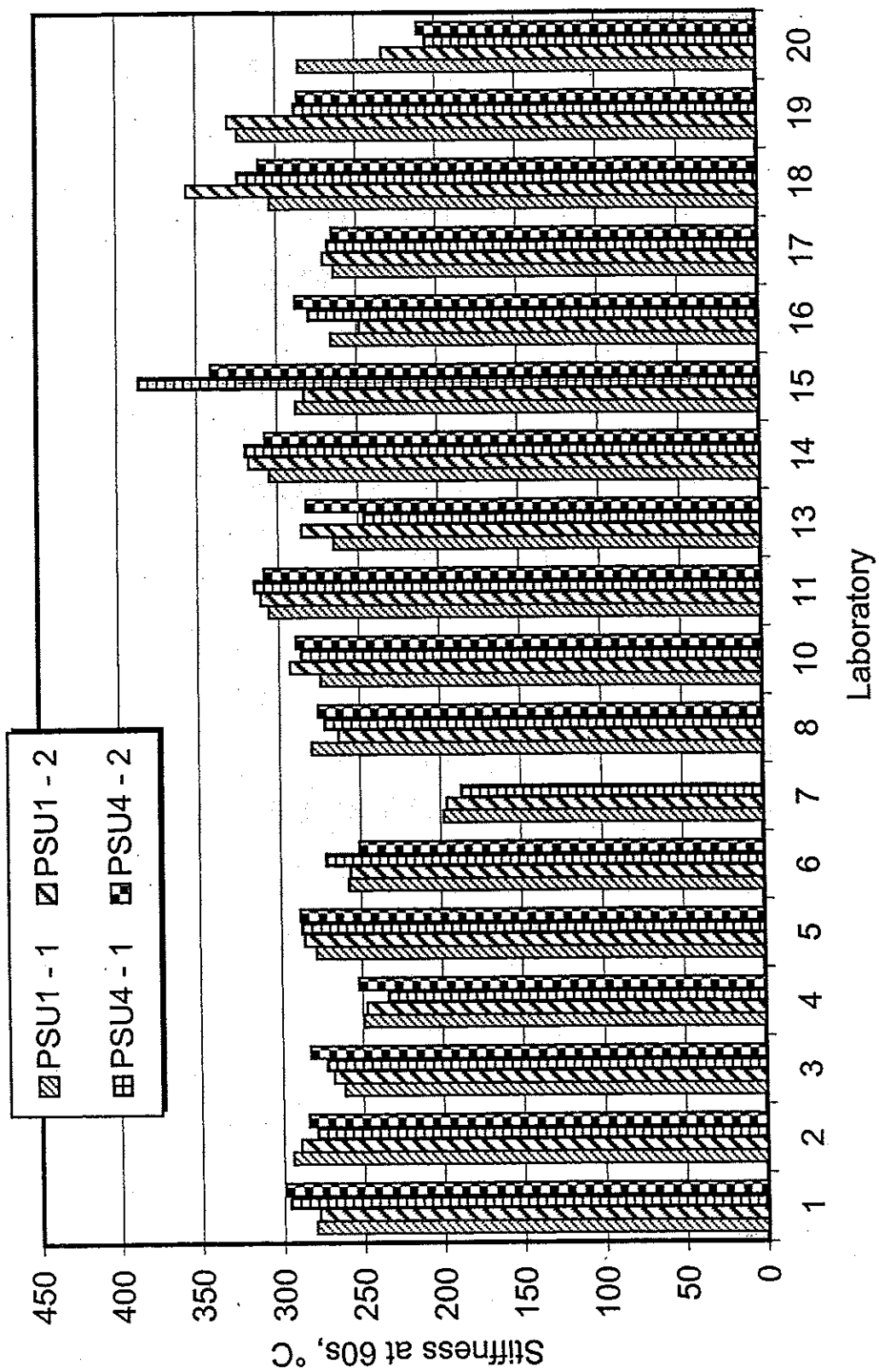


Figure D17. Stiffness data for PAV PSU1 and PSU4 at -18°C.

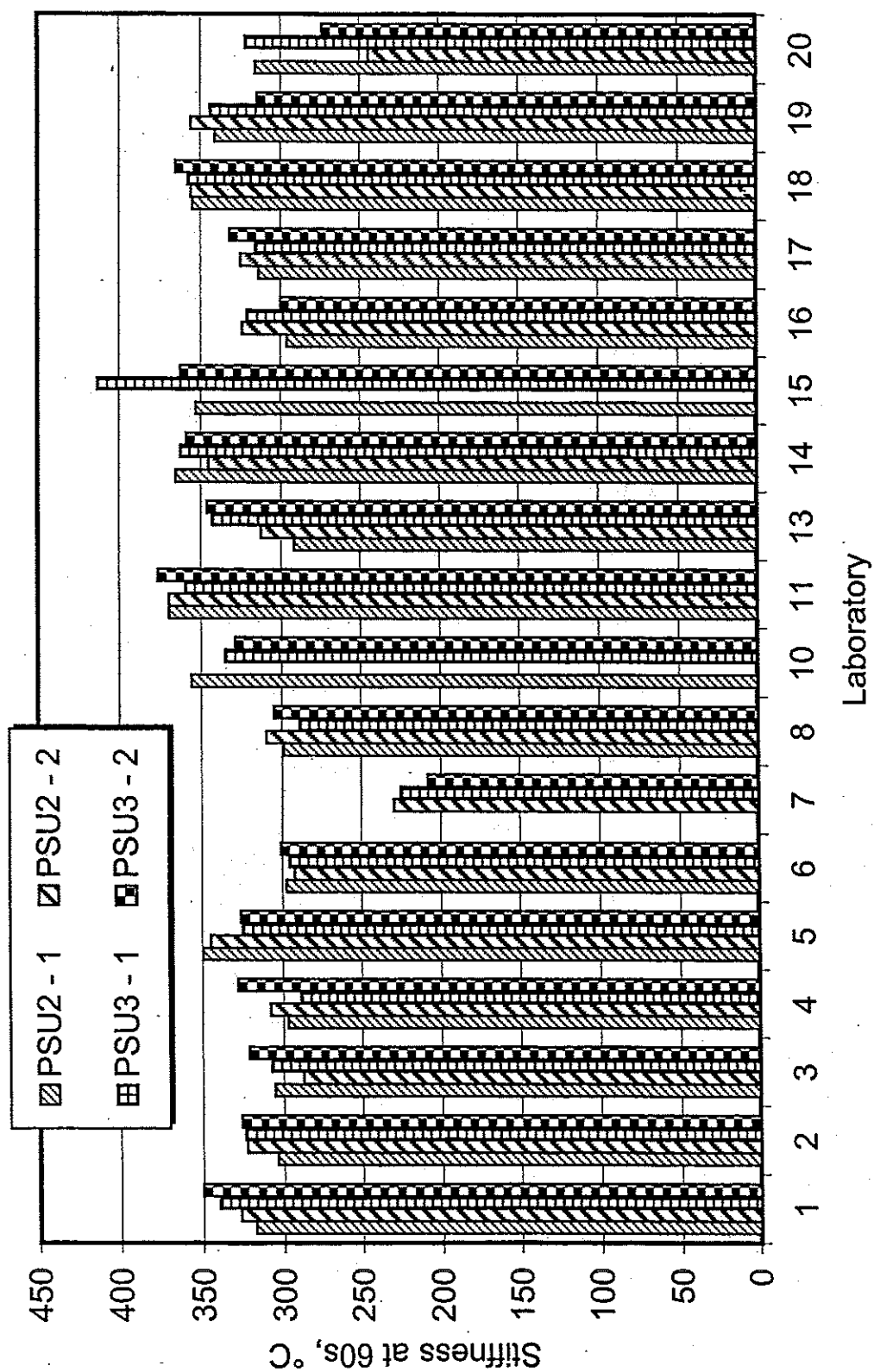


Figure D18. Stiffness data for PAV PSU2 and PSU3 at -18°C.

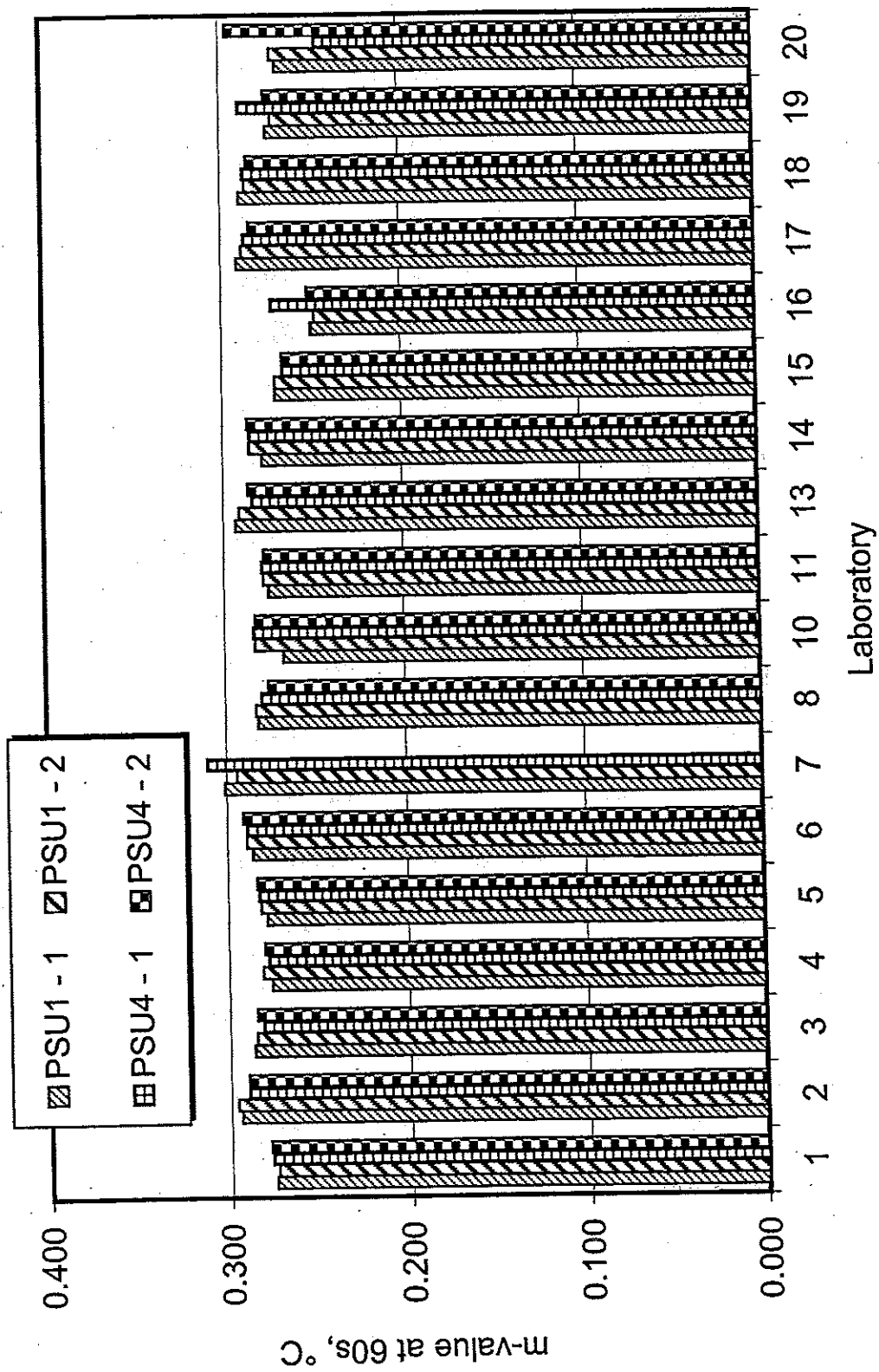


Figure D19. m-value data for PAV PSU1 and PSU4 at -12°C.

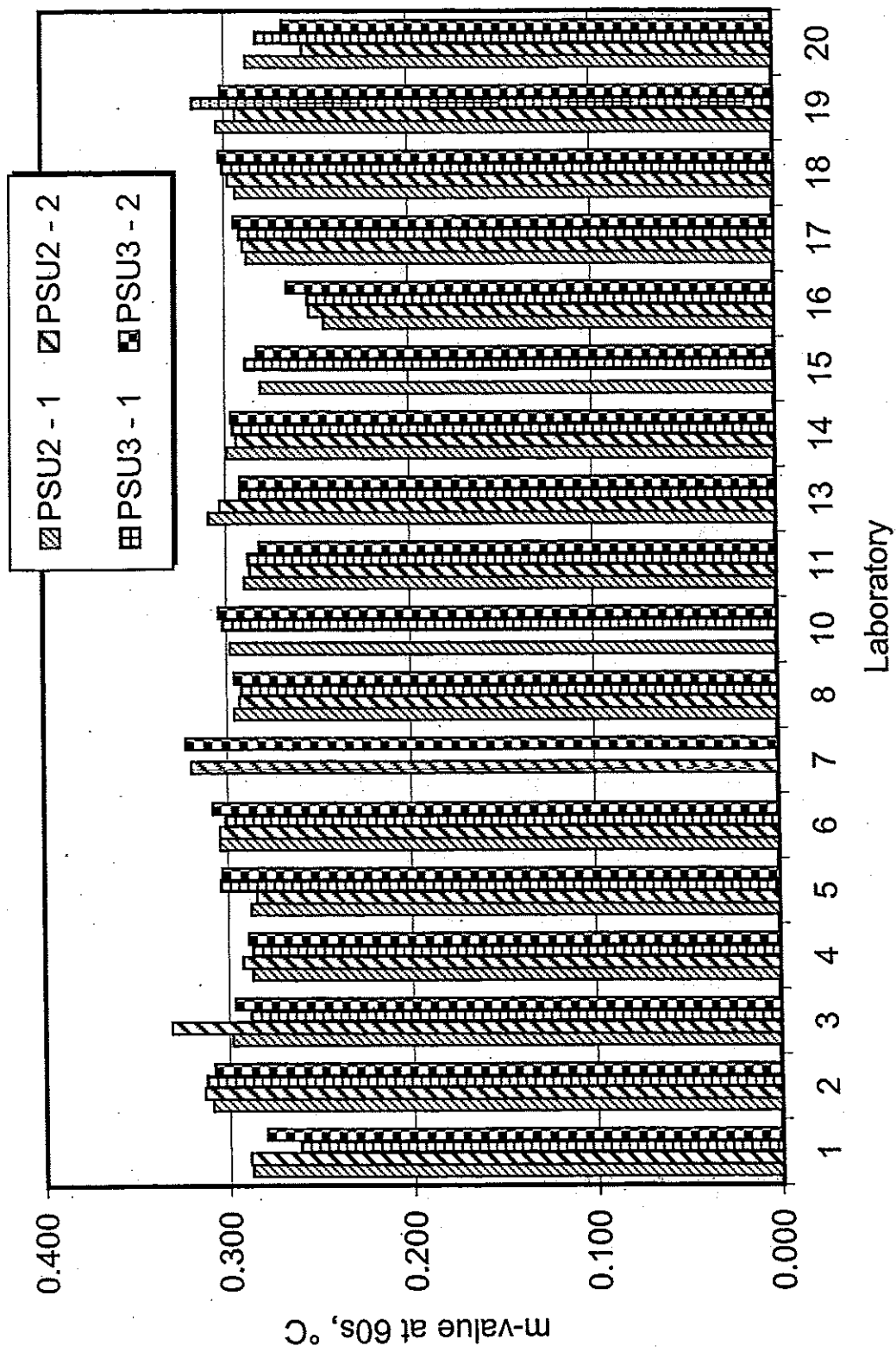


Figure D20. m-value data for PAV PSU2 and PSU3 at -18°C.



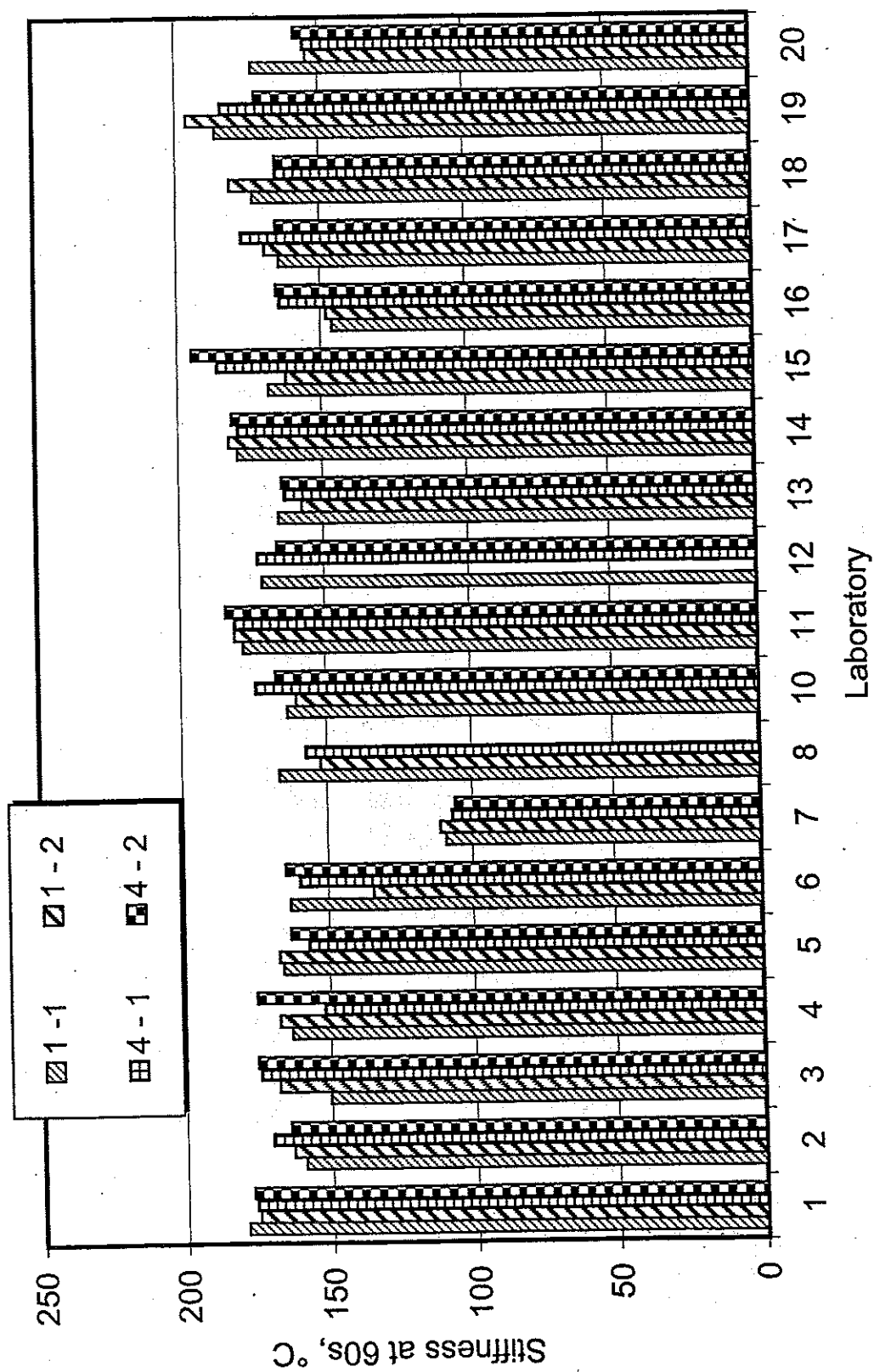


Figure D21. Stiffness data for pre-PAV 1 and 4 at -18°C.

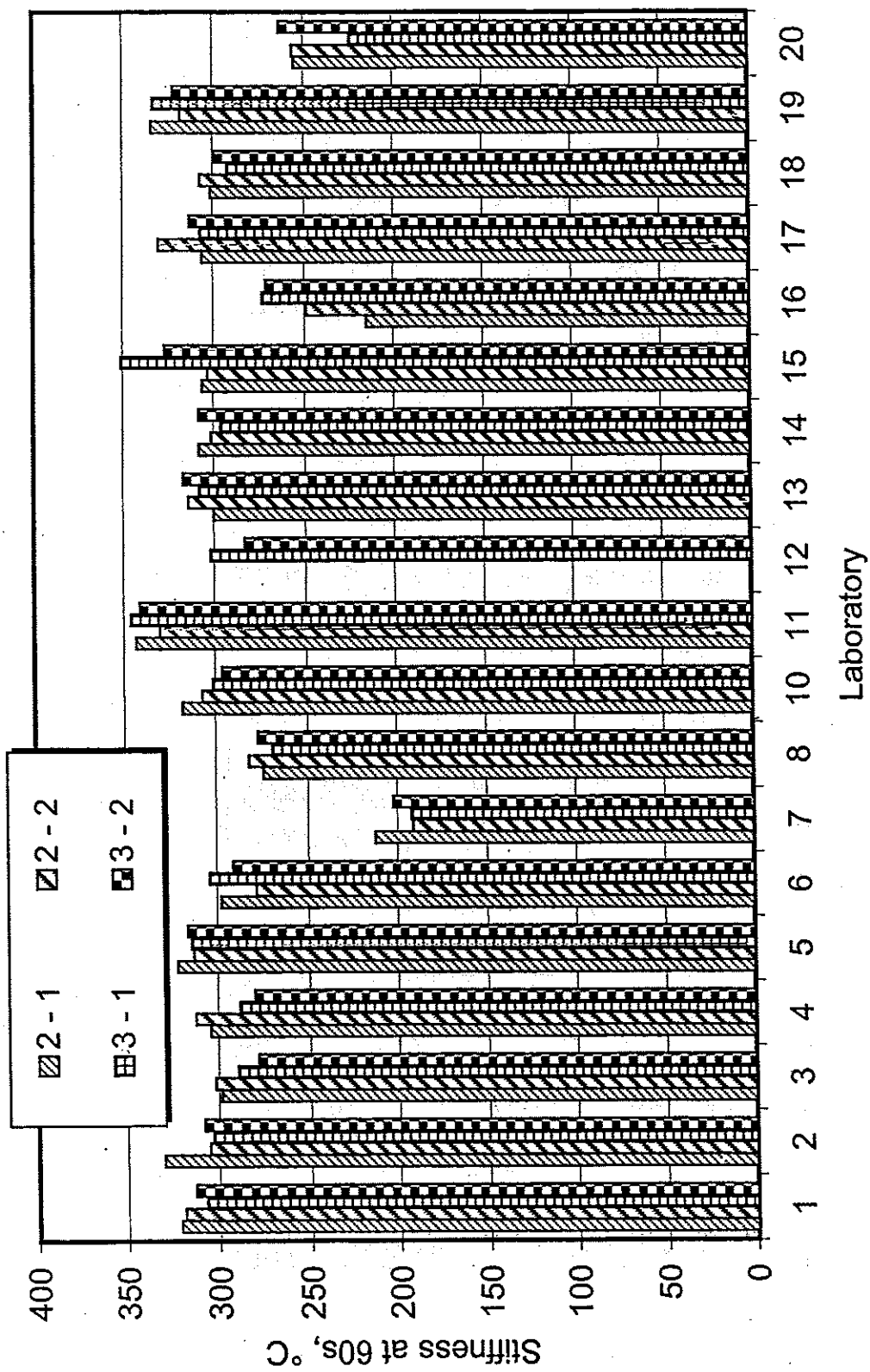


Figure D22. Stiffness data for pre-PAV 2 and 3 at -18°C.

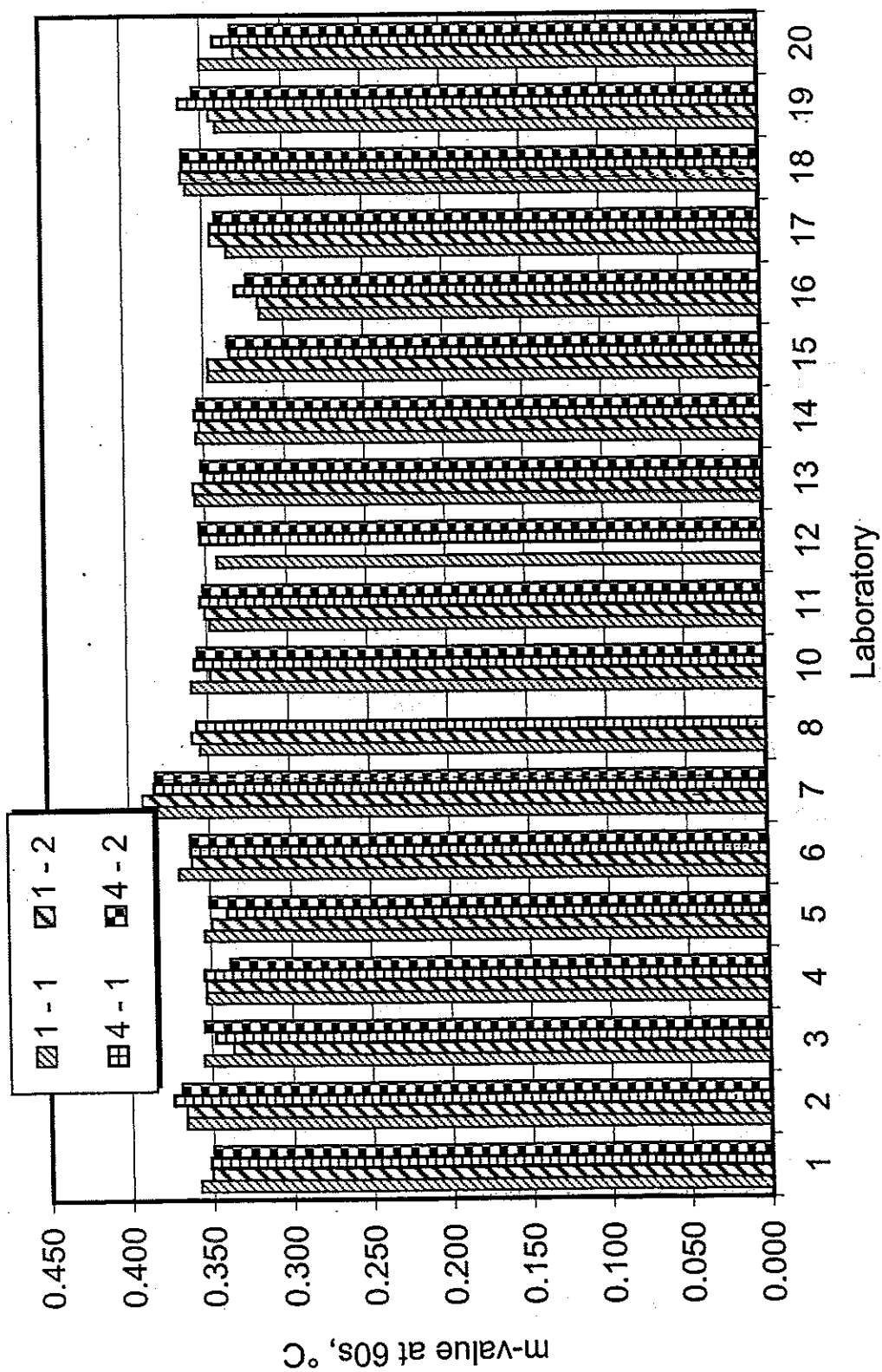


Figure D23. m-value data for pre-PAV 1 and 4 at -18°C.

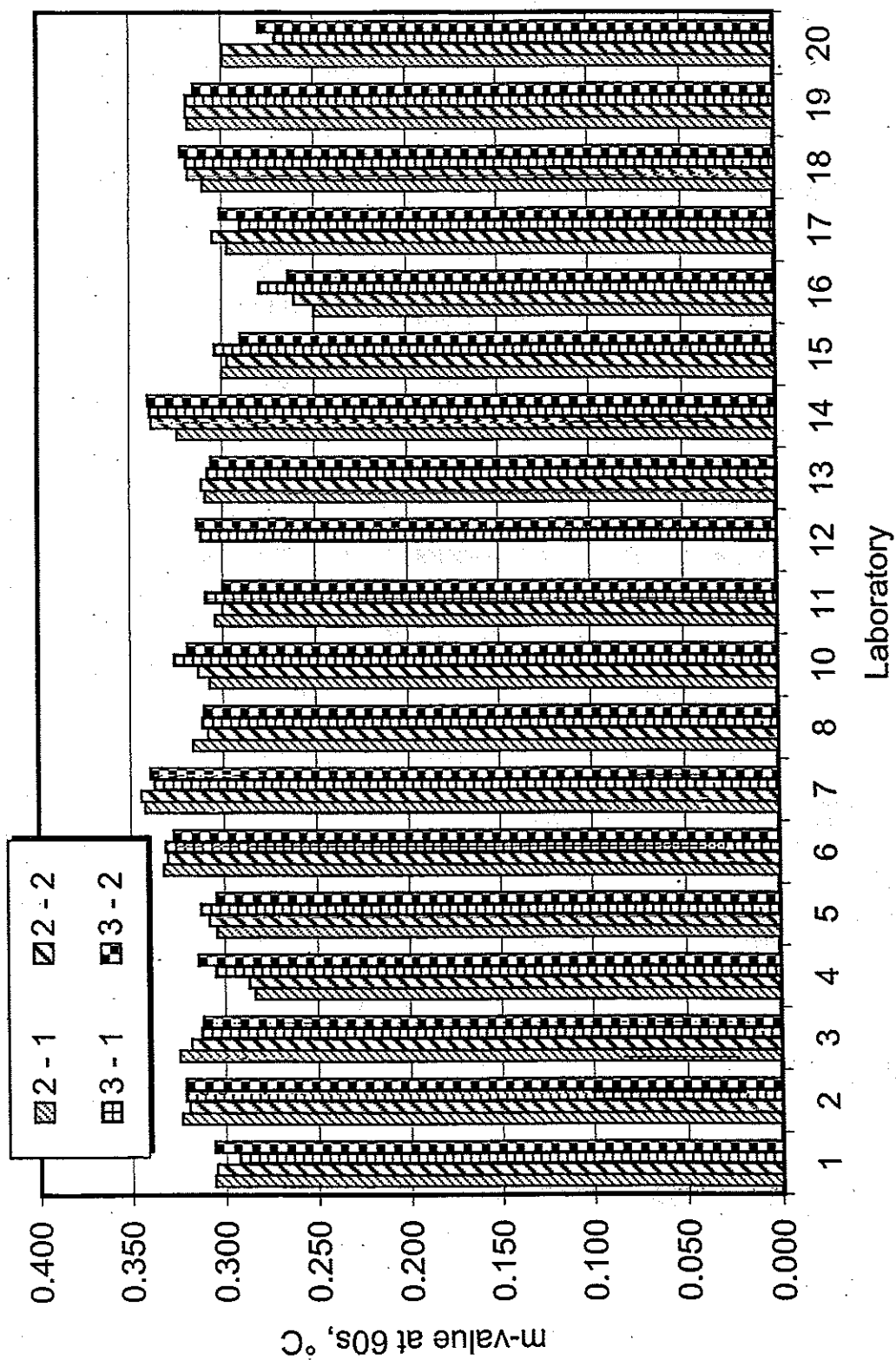


Figure D24. m-value data for pre-PAV 2 and 3 at -18°C.

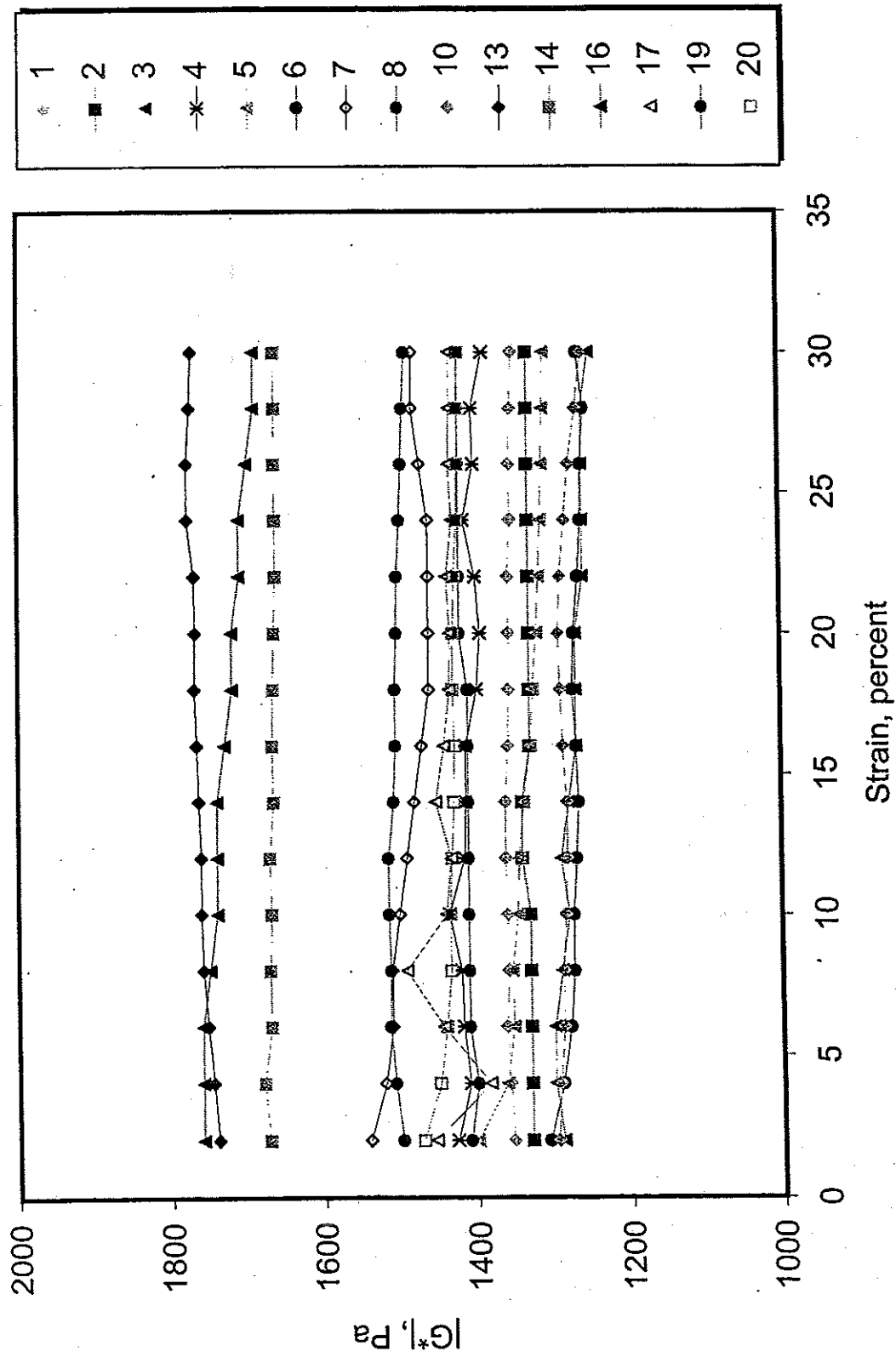


Figure D25. Strain sweeps  $G^*$  data for unaged PSU1.

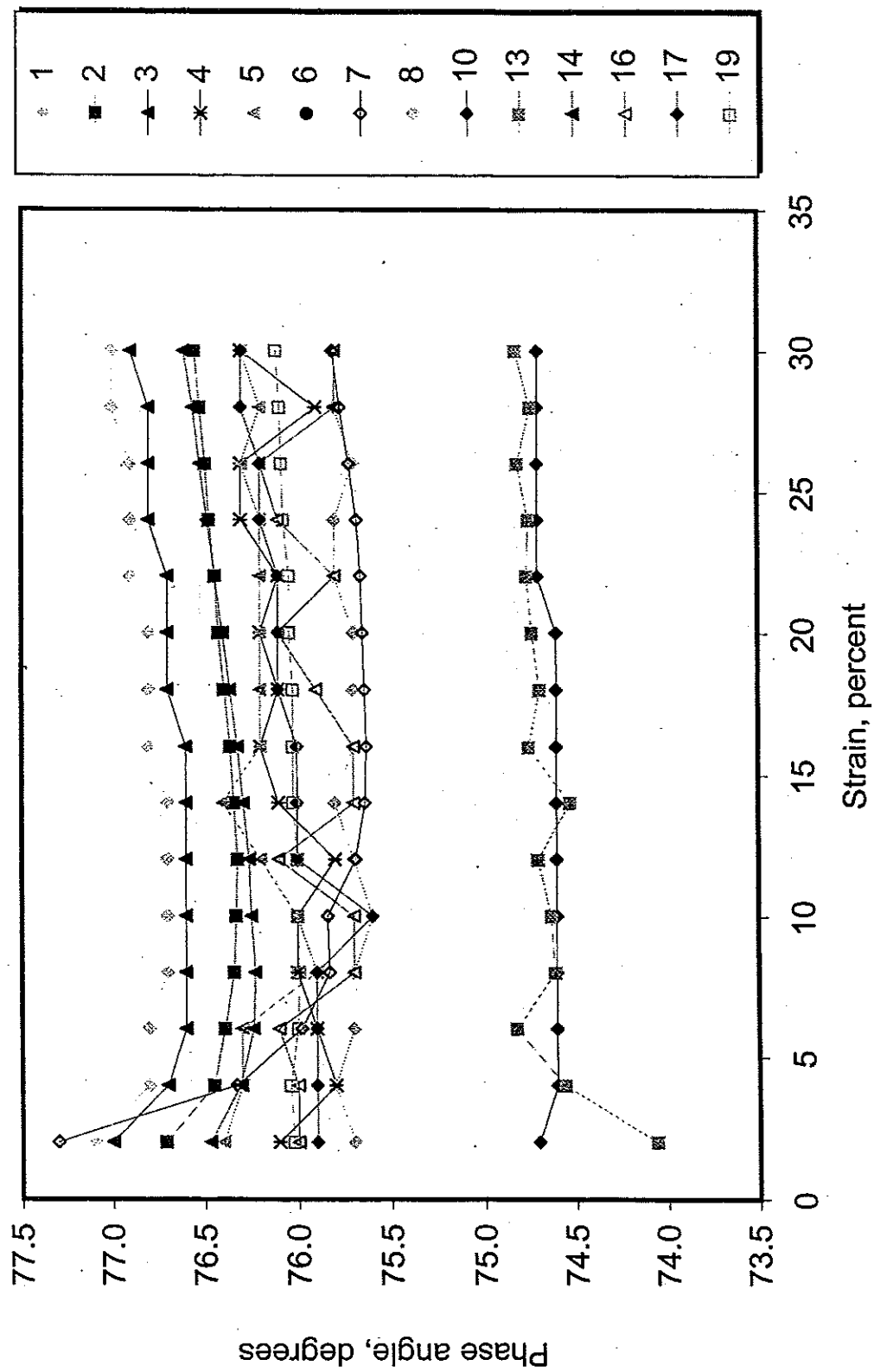


Figure D26. Strain sweeps phase angle data for unaged PSU1.

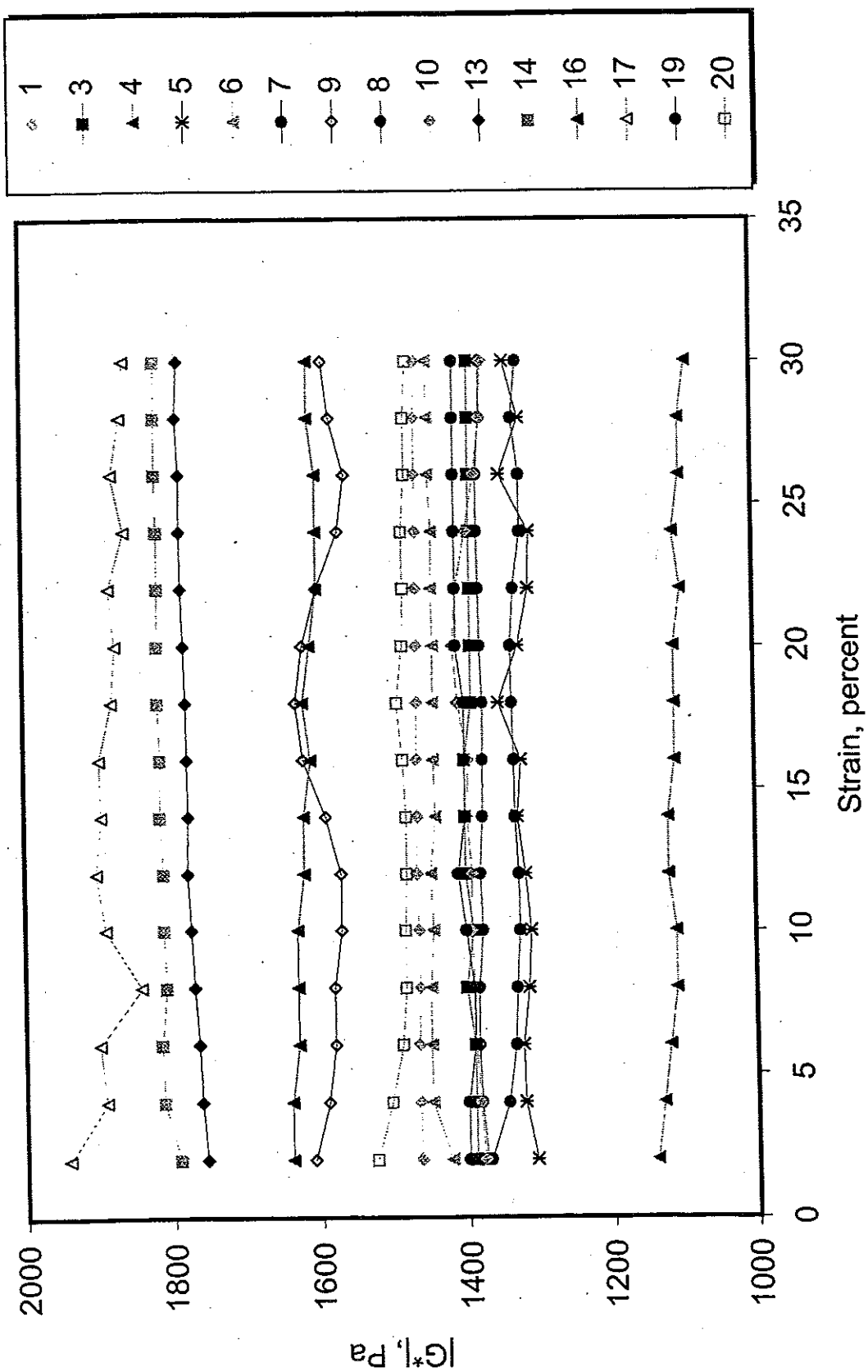


Figure D27. Strain sweeps  $G^*$  data for unaged PSU2.

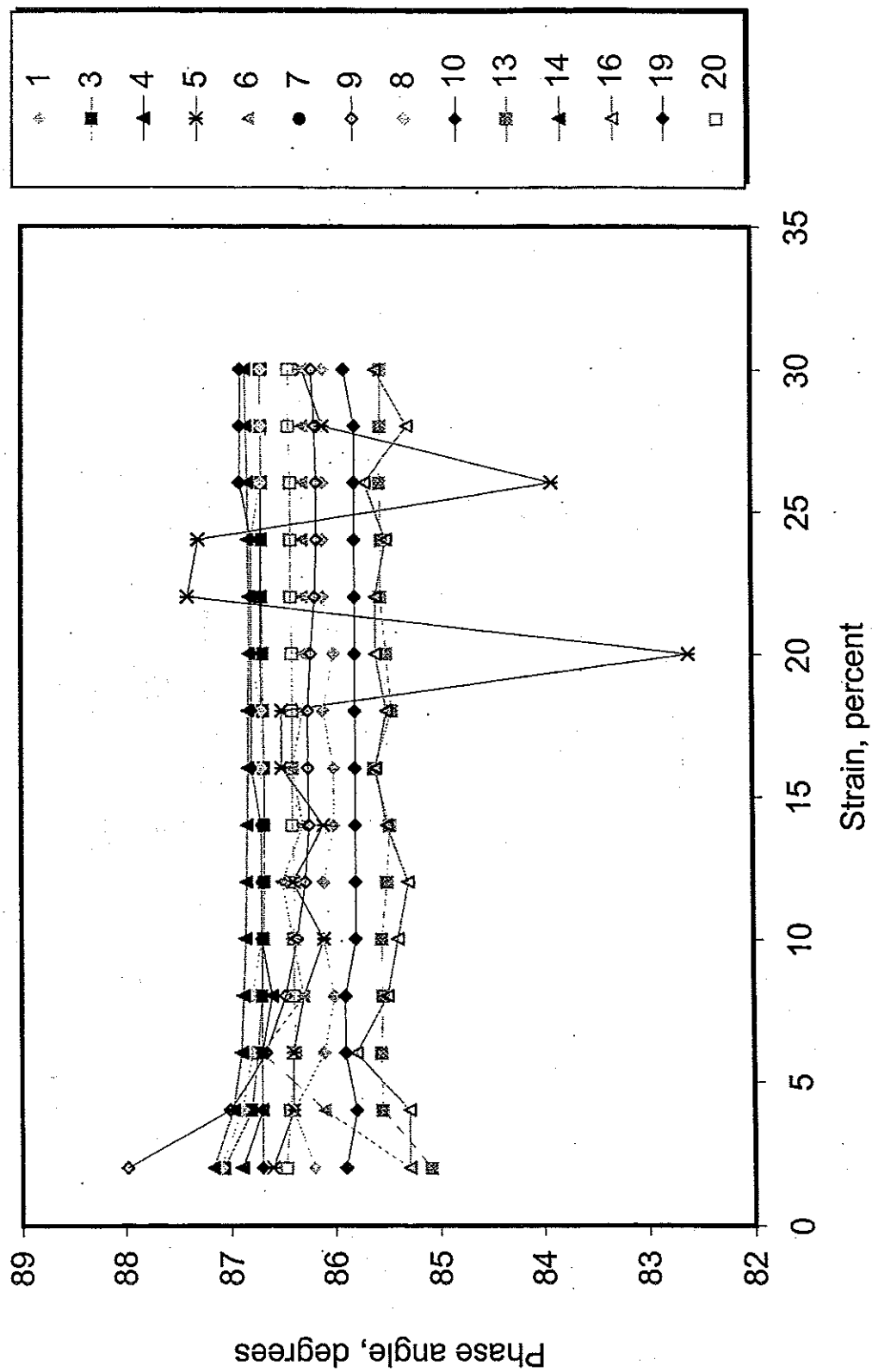


Figure D28. Strain sweeps phase angle data for unaged PSU2.



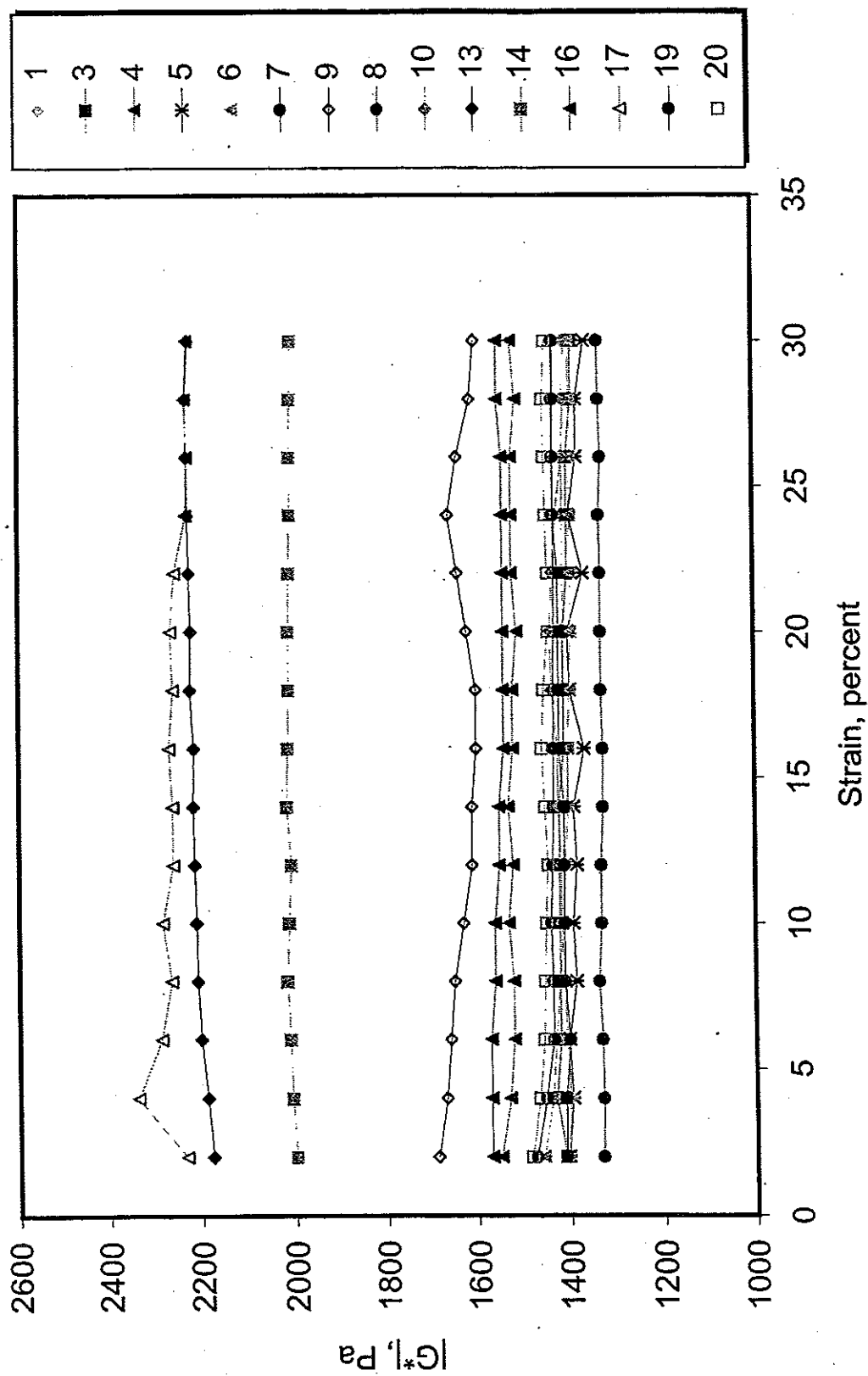


Figure D29. Strain sweeps  $G^*$  data for unaged PSU3.

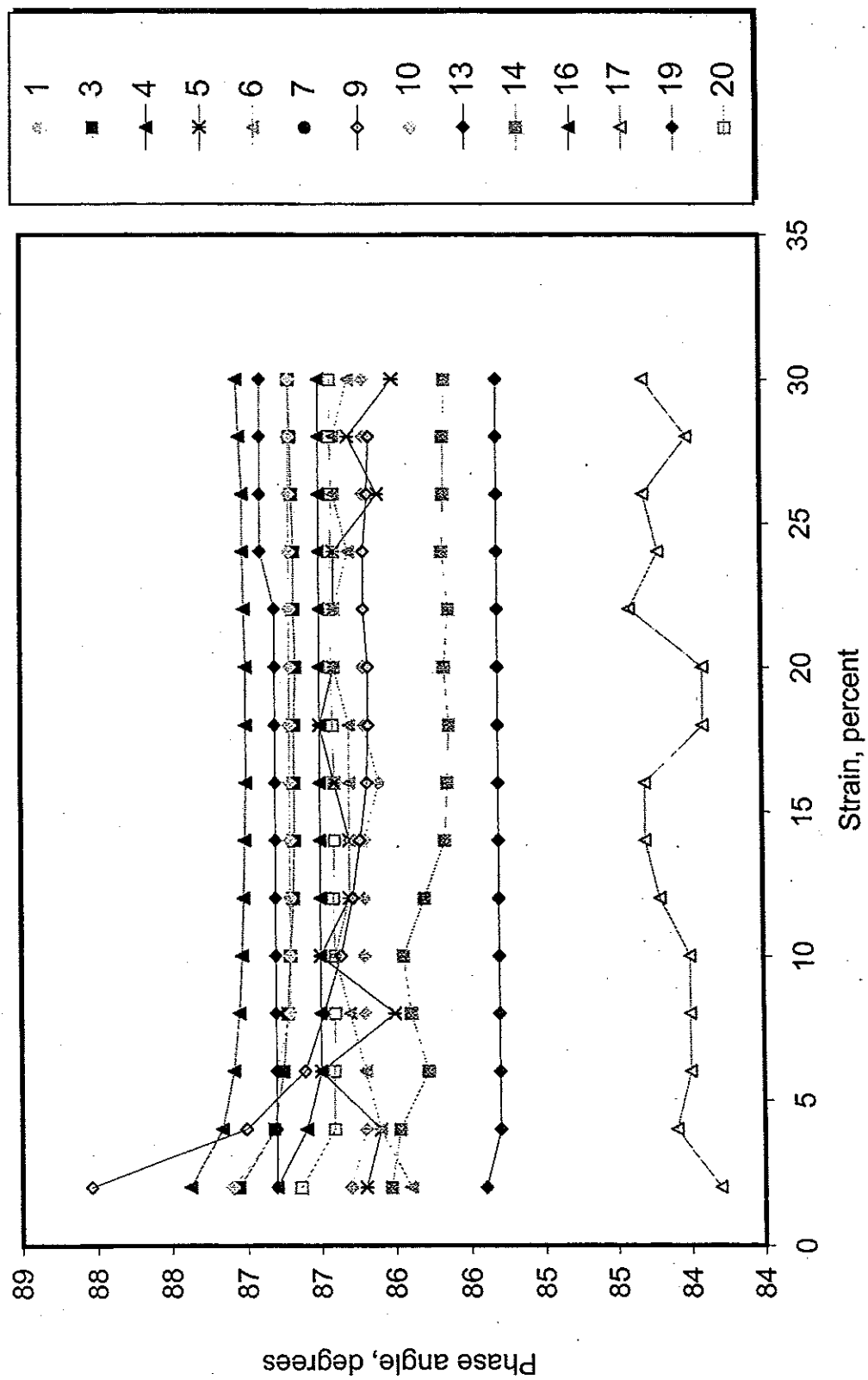


Figure D30. Strain sweeps phase angle data for unaged PSU3.

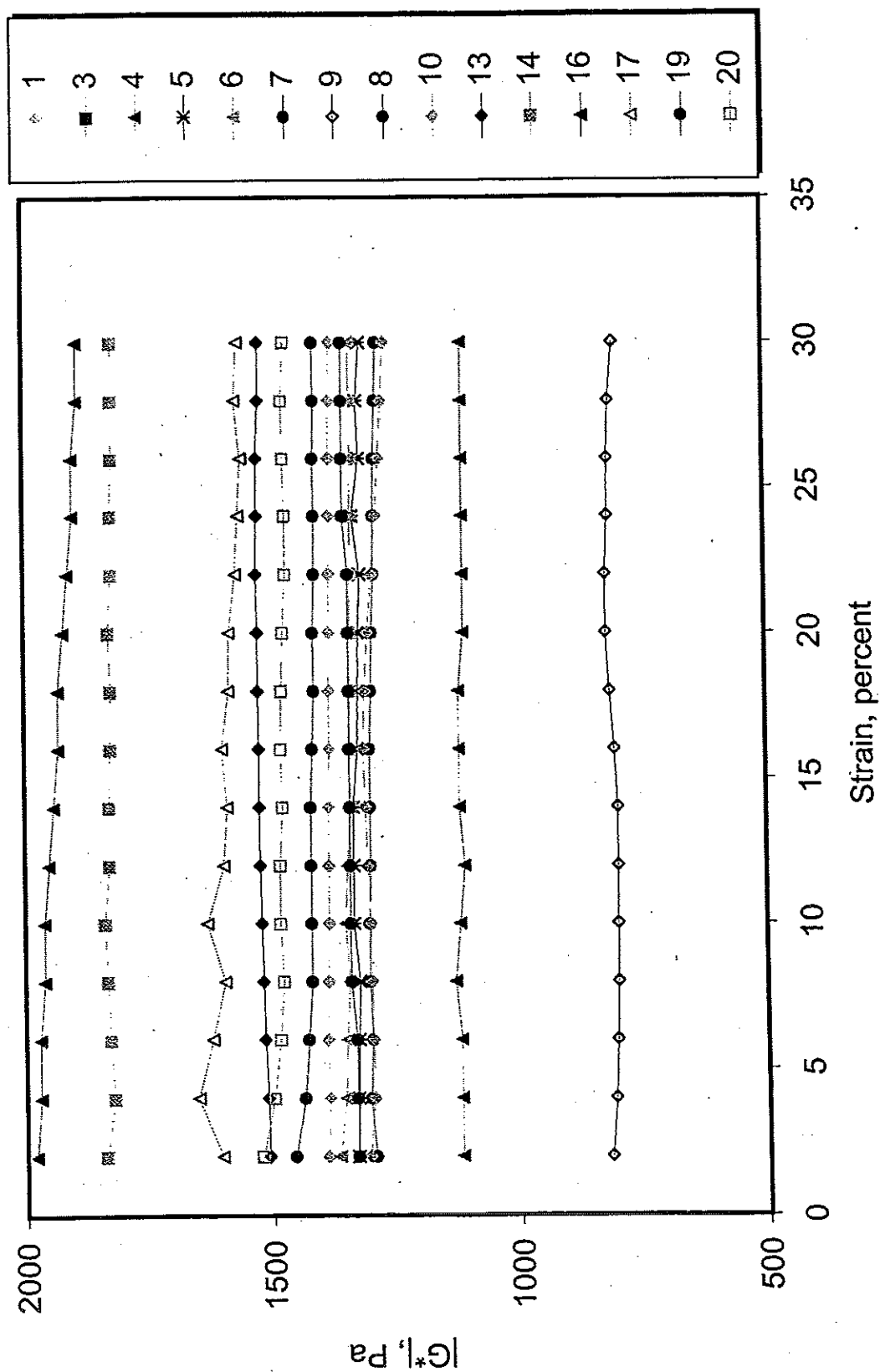


Figure D31. Strain sweeps  $G^*$  data for unaged PSU4.

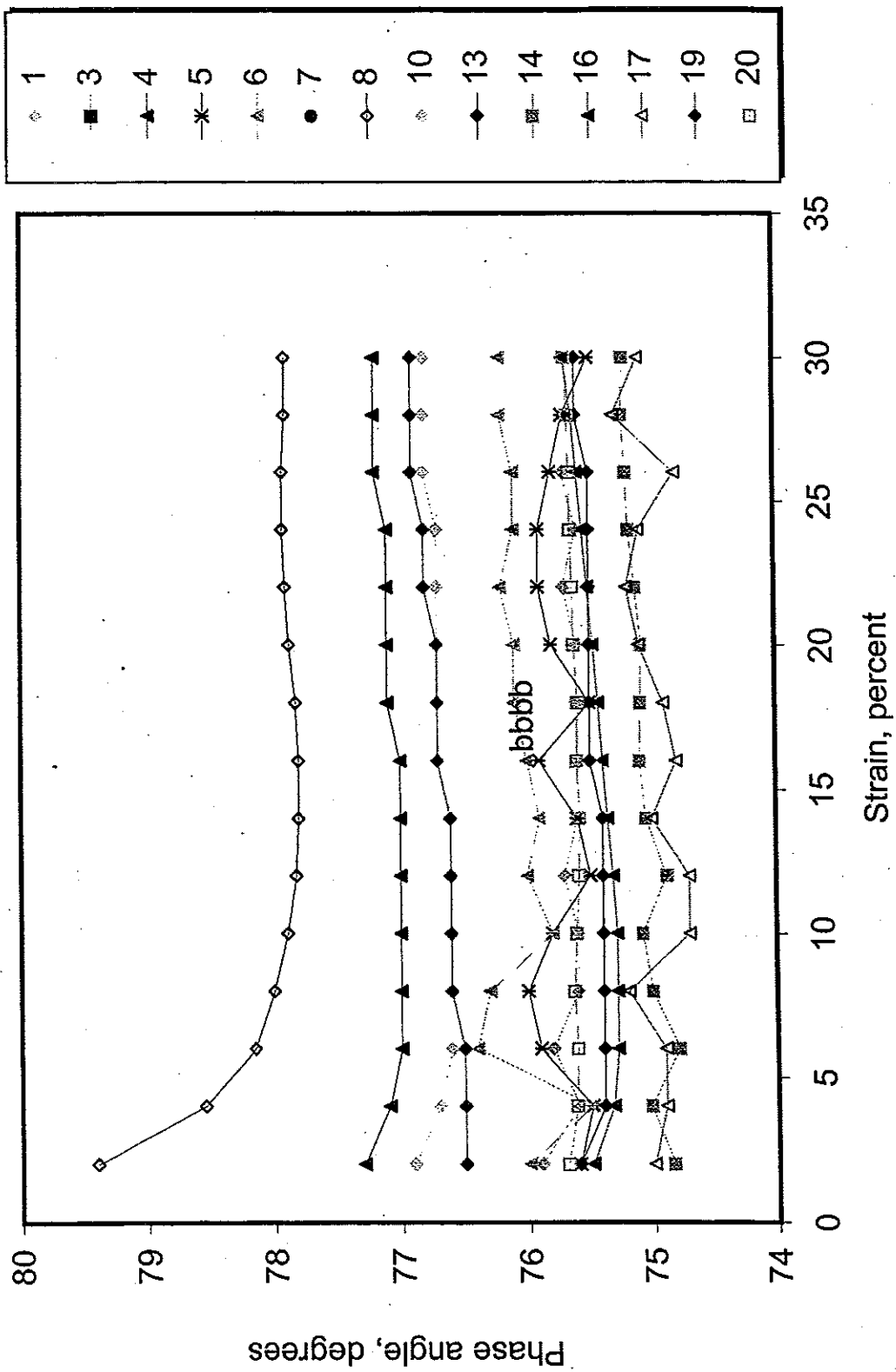


Figure D32. Strain sweeps phase angle data for unaged PSU4.

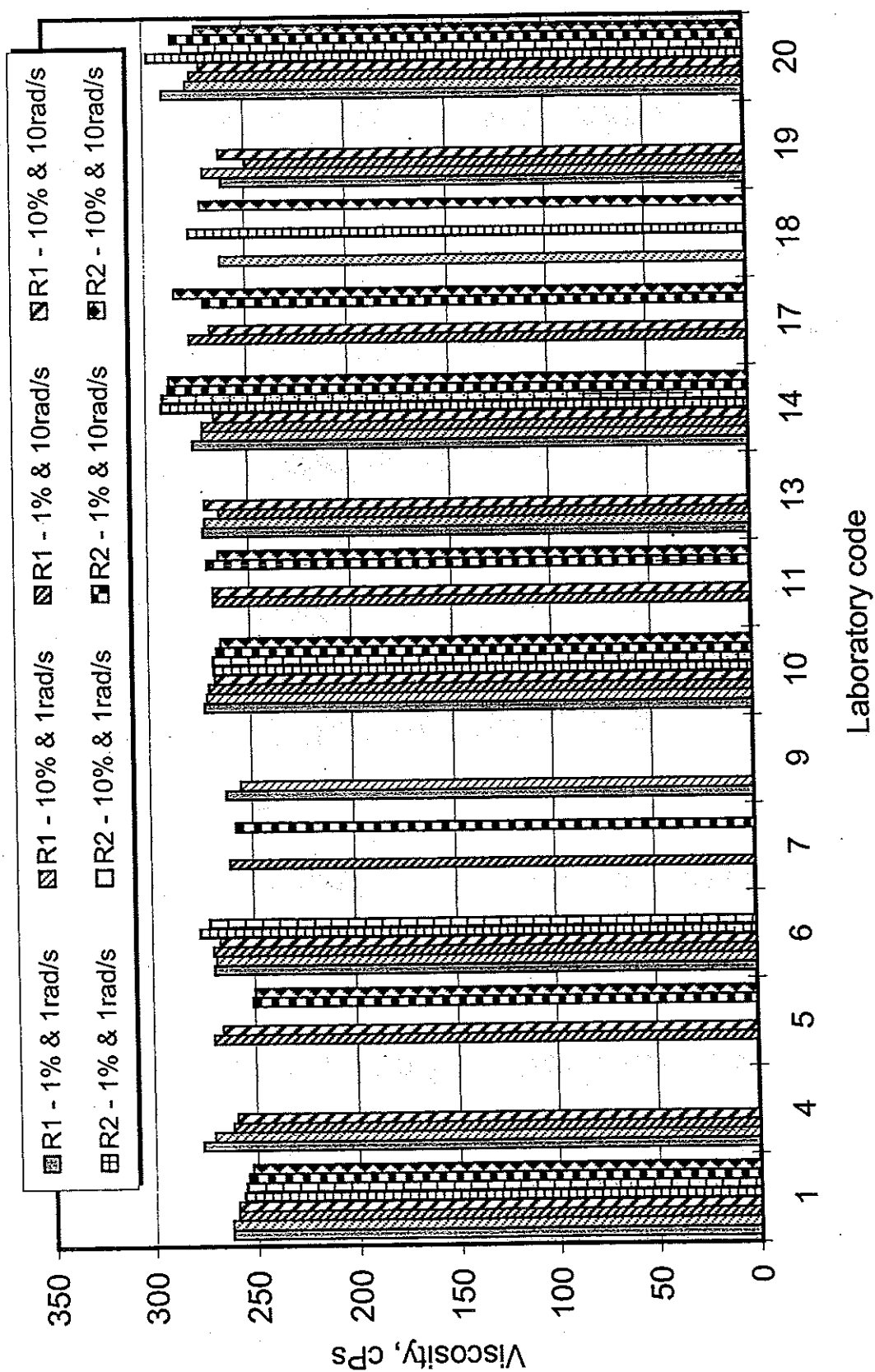


Figure D33. Complex viscosity data for the reference fluid at 64°C.

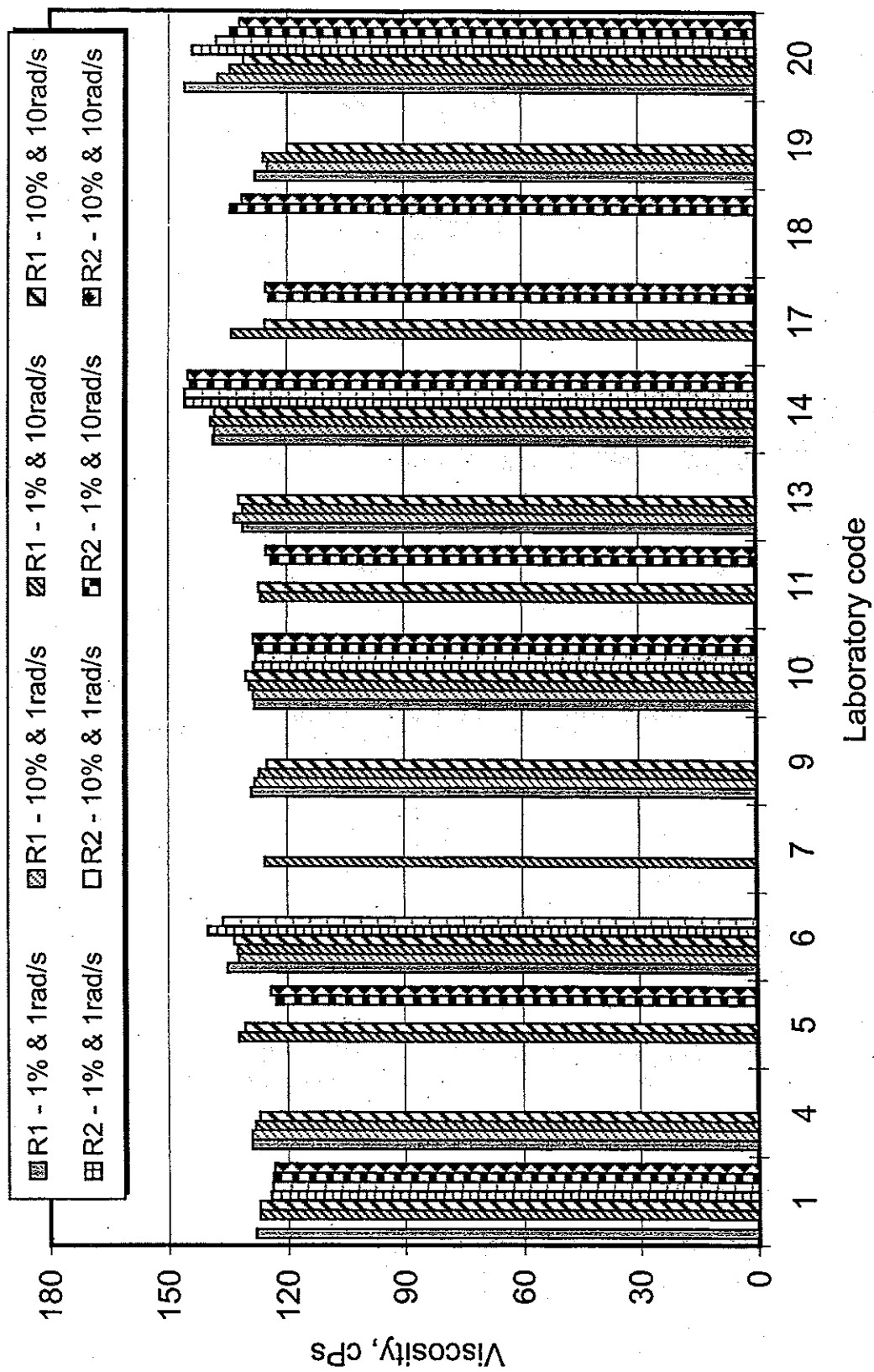


Figure D34. Complex viscosity data for the reference fluid at 76°C.

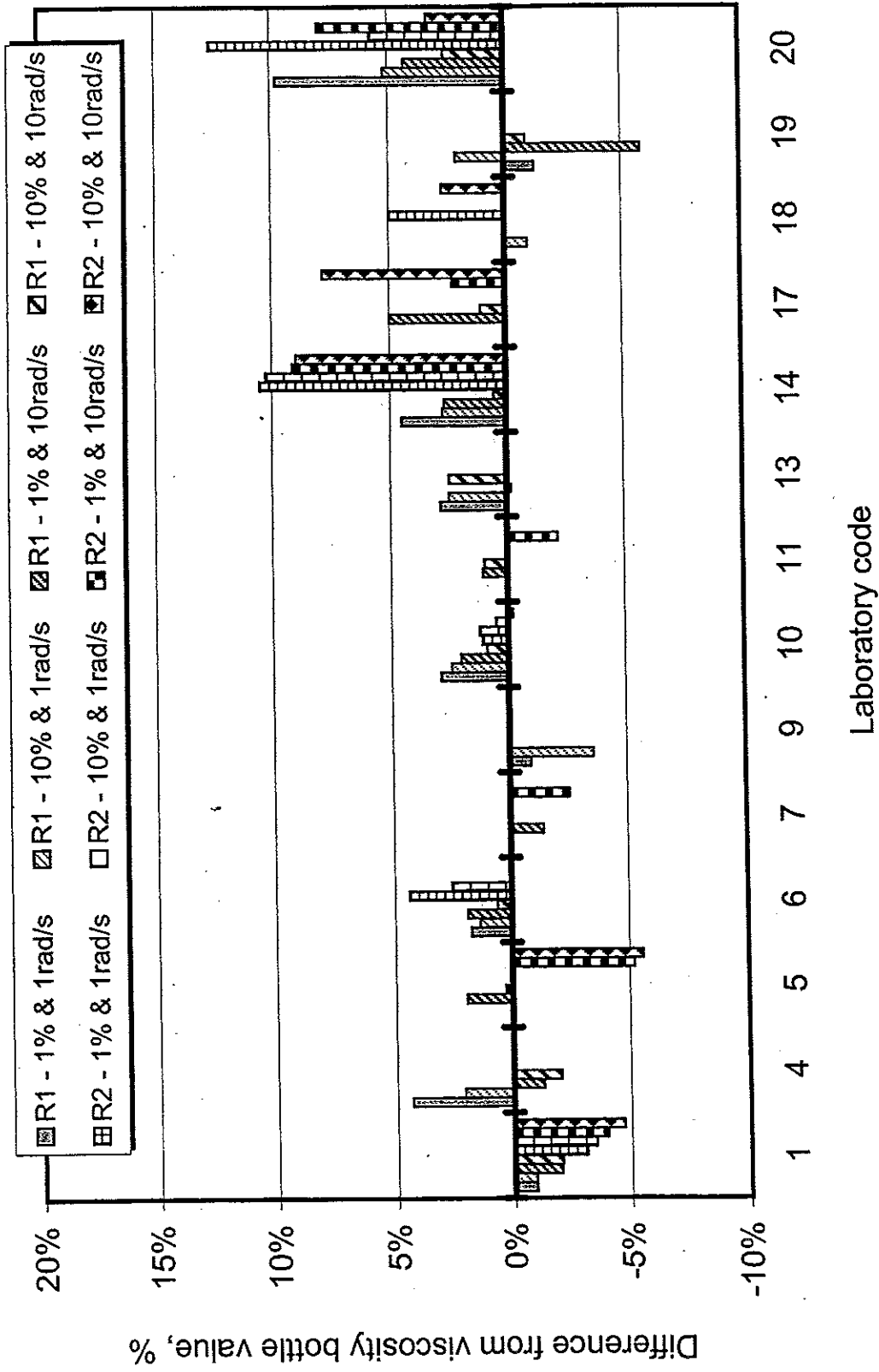


Figure D35. Complex viscosity versus capillary viscosity for the reference fluid at 64°C.

**APPENDIX E**  
**SLIDES USED IN DR. JACK STEPHENS' PRESENTATION**



## Round Robin 2 (March 98)

NETC Project 96-1  
Superpave Implementation

## 4 Binder Samples Were Sent

- Samples 1A & 4A were the same material
- Samples 2A & 3A were the same material
- Pre-aged samples 1 & 4 were the same
- Pre-aged samples 2 & 3 were the same

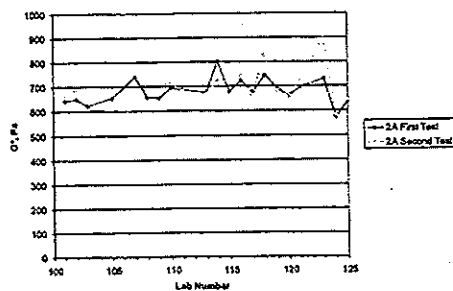
## DSR Testing

- Performed on Original and RTFO Material
- 1A and 4A tested at 70 & 76 C
- 2A and 3A tested at 64 & 70 C
- Duplicate samples were run of each material at each temperature

## Figure 1

- PSU 2A Original @ 70 C
- Most data points are the average of two  $G^*$  values
- More erratic on the high side
- $G^*$  ranges from 625 to 815 Pa,
- The average was 686 Pa
- The range is 28% of the average

Figure 1,  $G^*$  Original 2A 70C



## Figure 1 Continued

- Individual values of  $G^*$  for PSU 2A
- 15 Labs reported 2 values of  $G^*$
- Labs 107, 114, 118 & 123  $G^*$  values appear high
- There were no outstanding low values for  $G^*$

Figure 2

- PSU 3A G\* @ 70 C
- Same material as PSU 2A
- For Figures 1 & 2, 4 of the highs are #2

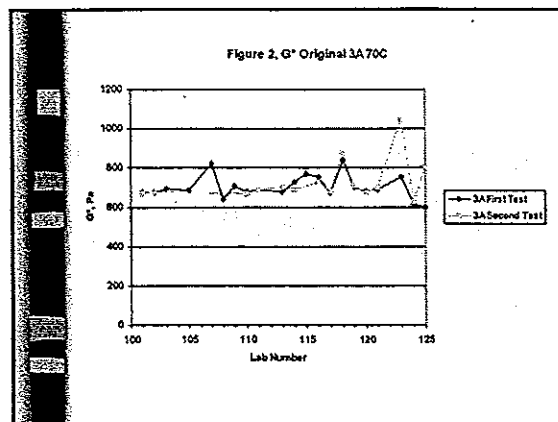


Figure 3

- Original PSU 2A & 3A @ 70 C
- The second G\* value was higher 17 out of the 21 times
- Labs 17, 118 and 123 were high for both the first and second tests for both 2A and 3A

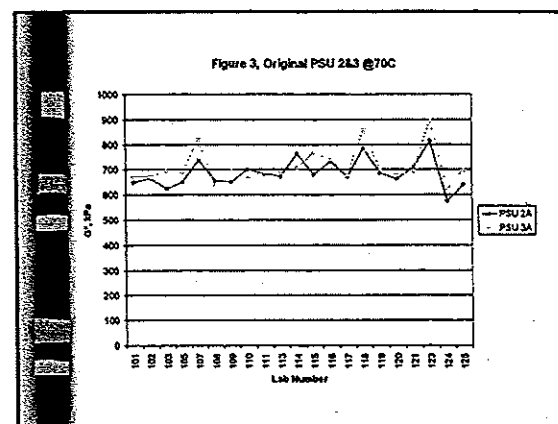


Figure 4

- G\* Original 2A @ 64 C
- 34 out of 42 on line
- Labs 114, 118, 123 and 125 again out
- Range less at 64 C than at 70 C

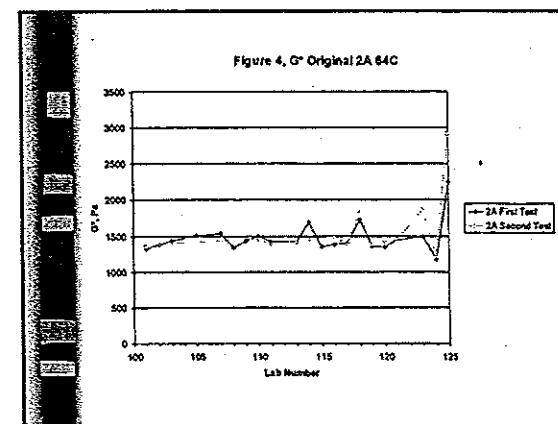


Figure 5

- G\* Original 1A & 4A @ 70 & 76 C
- Each Lab is in good agreement

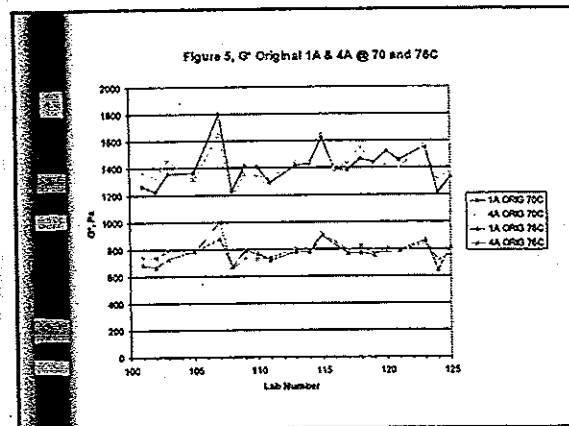


Figure 6

- G\* Original 1A, 2A, 3A and 4A @ 70 C
- Labs 107 & 115 only reported one value for G\*
- Labs 107, 115, 118 and 123 tend to be high

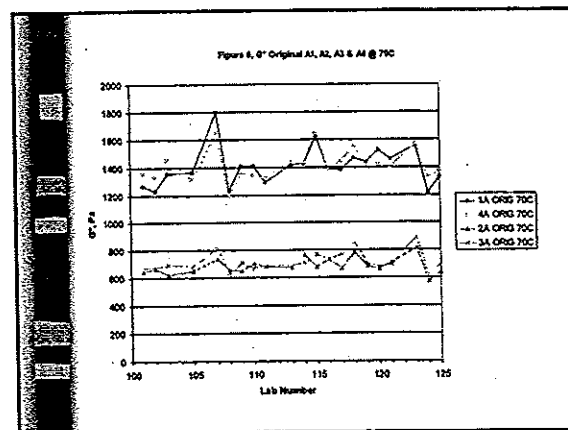


Figure 7

- G\* Original 1A & 4A @ 70 C and 2A & 3A @ 64 C
- Lab Pattern
- Labs 107, 115, 118 and 123 Consistently High
- Labs 108 and 124 are low
- Discard outlier at 125

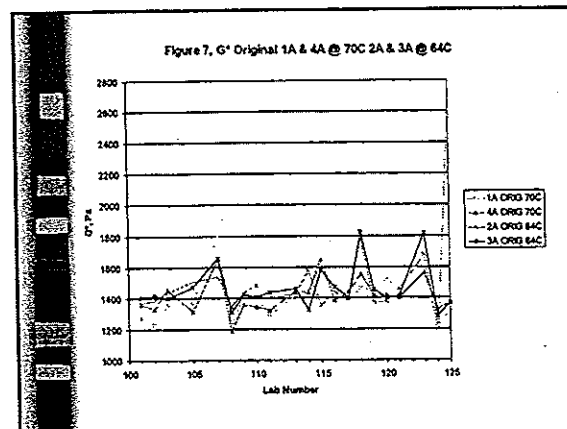


Figure 8

- G\* Original 2A and 3A @ 64 and 70 C
- Look at Lab Differences
- Note 107, 118 and 123 are consistently high
- Outlier at 125

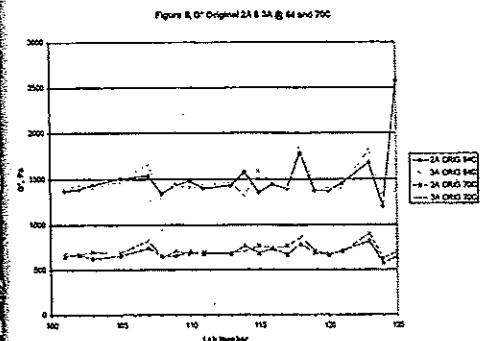


Figure 9

- G\* RTFO 2A & 3A @ 64 & 70C
- Lab 105 and 115 G\* values are low
- Lab 123 G\* continues to be high
- Lab 125 is erratic
- 64 C is more consistent than 70 C
- Compare variation of 1500 Pa to 3300 Pa
- Similar Percentages

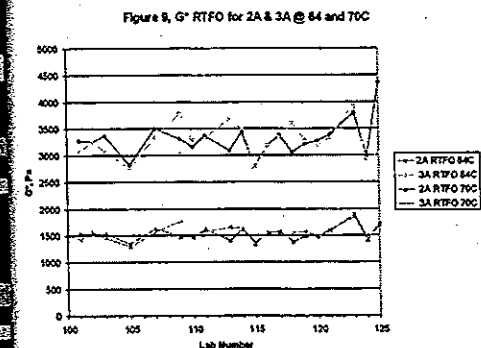


Figure 10

- G\* RTFO 1A & 4A @ 70 C and 2A & 3A @ 64 C
- Average G\* for materials about the same at the two temperatures
- Lab 103, 105, 107, 109 and 115 reported only one value for G\*
- Second test could temper the differences

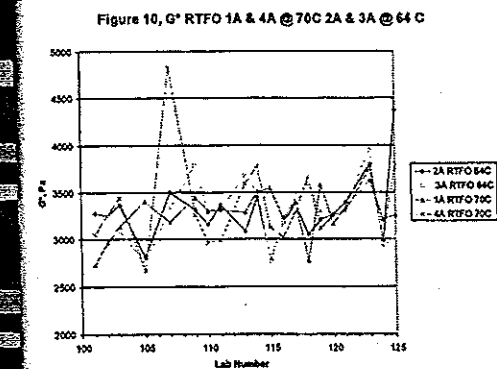


Figure 11

- G\* Original & RTFO 1A & 4A @ 70C
- Why is G\* Original much more consistent than G\* RTFO?
- Labs 107, 115, 118 and 123 still tend to be high

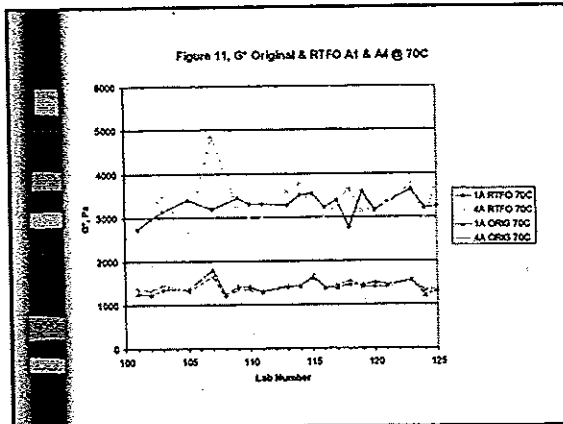


Figure 12

- G\* at Lowest Test Temp. per Sample Stage
- Lab 105, 107, 108, 109 and 115 only reported one value
- For RTFO Material Lab 105 tend to be low and Lab 123 tend to be High
- For Original Material Lab 107, 118 and 123 tend to be high and Lab 108 and 124 tend to be low

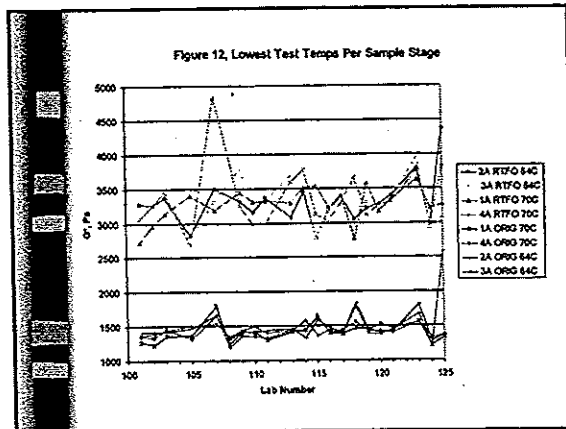


Figure 13

- PSU 2A @ 70 C First and Second Phase Angles
- Twelve Labs have less than 0.5 degree difference
- Lab 115 appears as outlier but is only 3.5 degrees from the average

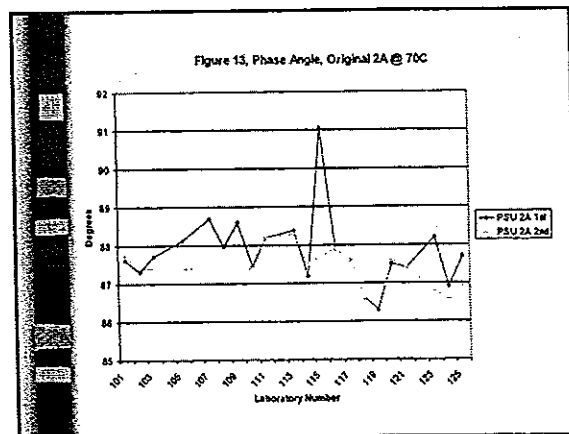


Figure 14

- PSU 3A, First and Second Phase Angles
- Eleven Labs with less than 0.5 Degree Difference
- Lab Number 115 appears as an outlier but is only 2.5 degrees from the average

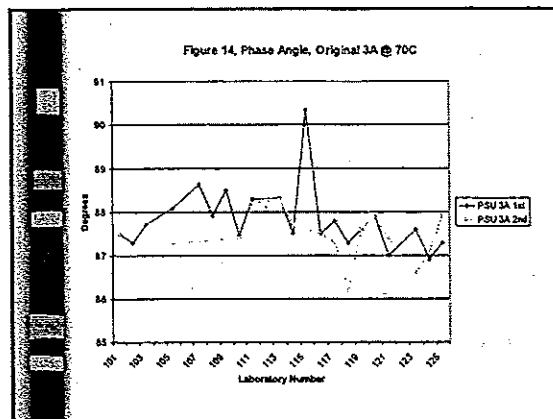


Figure 15

- PSU 2A & 3A @ 70C Average of both reported values - if two were reported
- Nineteen Labs with less than 0.5 degree difference
- Lab 115, only one test for each point

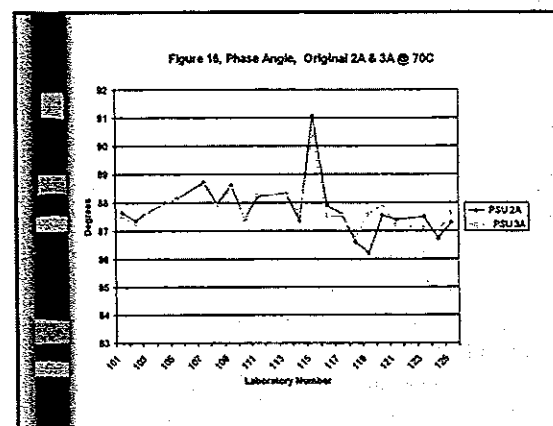


Figure 16

- Very little difference from lab-to-lab

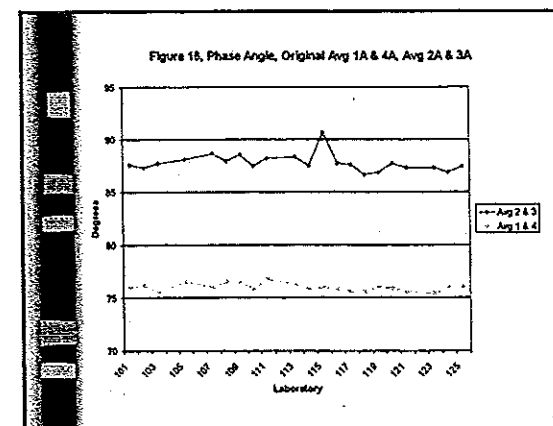


Figure 17

- Pre-molded Sample  $G^*$
- Only 11 Labs Participated
- For Lab 105, which data point is an outlier?
- Number 101 has two similar values but both are low

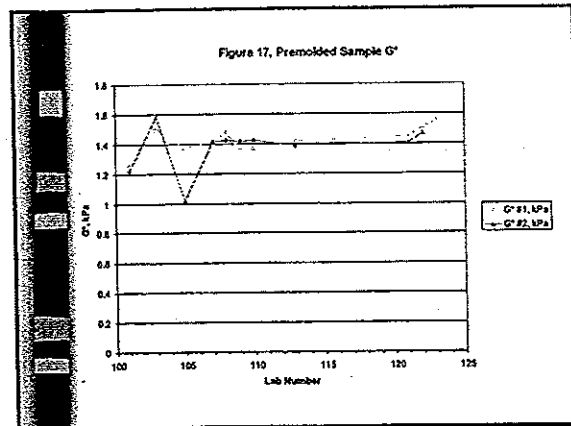
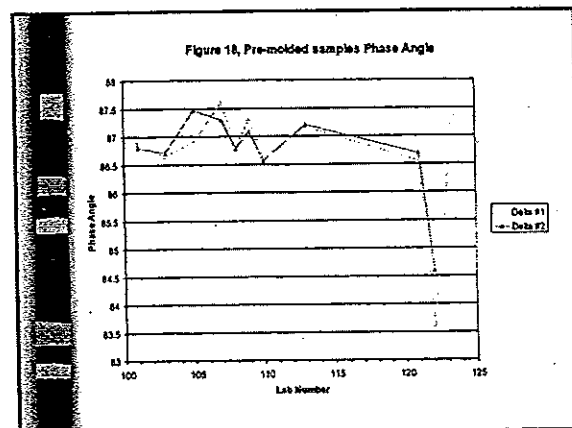


Figure 18

- Pre-molded Samples Phase Angle
- Both results from Lab Number 122 are Quite Low



### Summary Table

- Summation of all  $G^*$  for All Samples including the pre-molded samples
- All of the round robin samples had at least 3 labs with  $G^*$  more than 1 standard deviation away from the average - pre-molded samples had 1
- Every Round Robin sample had at least one lab more than 2 standard deviations away from the average - pre-molded none.

### DSR Results Summary

- Average  $G^*$  of Pre-molded samples approximately the same as original 1A & 4A @ 70 C and RTFO 2A and 3A - standard deviation of pre-molded samples less than any of these
- Repeated similarities in lab versus property curves indicate equipment differences.
- Random Differences Indicate Technique Differences

Figure 19

- BBR Stiffness 1A & 4A -18C @ 60 s
- Data very similar between labs except labs 109, 114 and 125

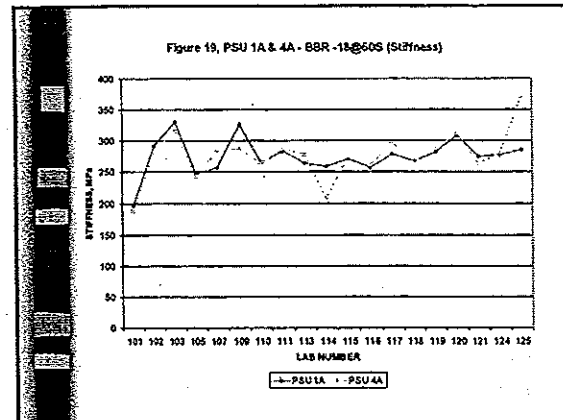


Figure 20

- BBR Average m-value PSU 2A & 3A -12C
- Differences between labs very consistent
- For example lab 113 & 114 are low while lab 118 is high
- At -18C the within lab difference is small as well

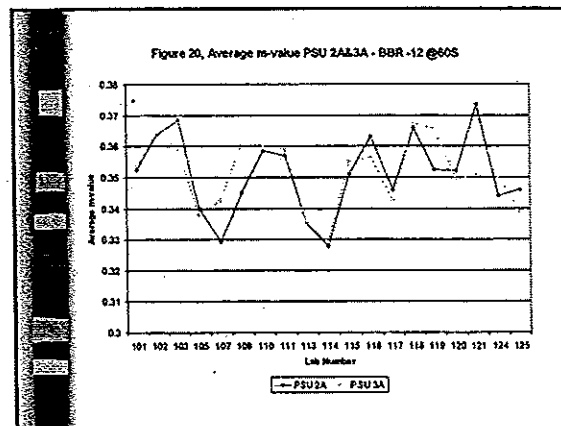


Figure 21

- BBR Stiffness 1A, 2A, 3A & 4A -12C @ 60s
- Note Similarity of curve shape from Lab 101 to Lab 110 & 115 to 124

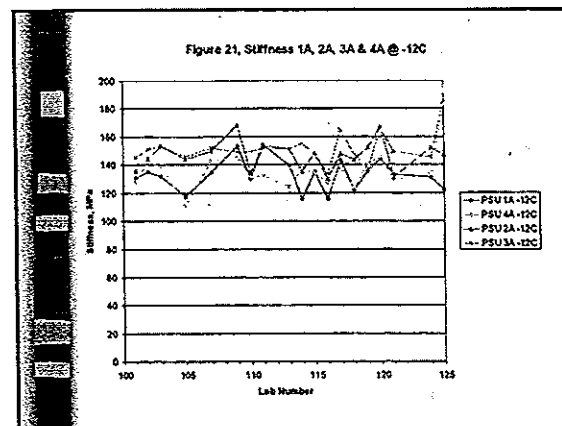




Figure 22

- BBR Stiffness 1A, 2A, 3A and 4A 12C @ 60s
- Differences between labs is similar throughout graph

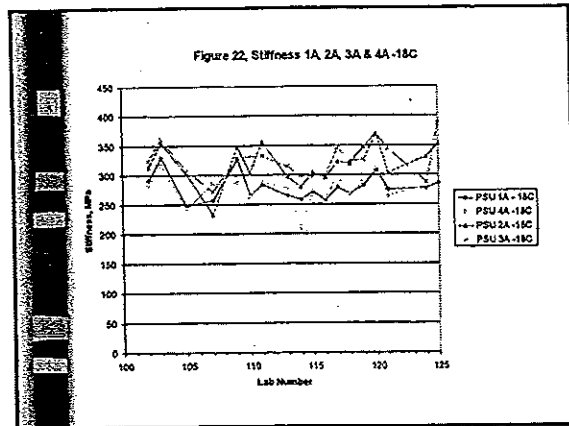


Figure 23

- BBR Stiffness Pre-aged Samples -18C @ 60 s
- Again lab-to-lab differences similar for all

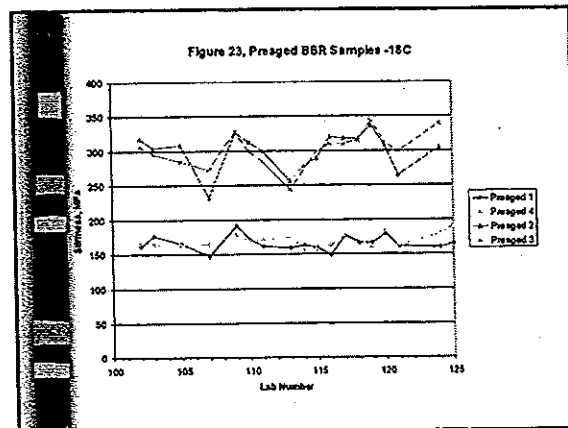
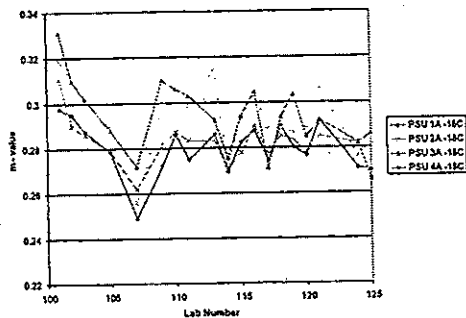


Figure 24, Average m-value 1A, 2A, 3A & 4A @ -18C



**APPENDIX F**  
**STATISTICAL ANALYSIS DETAILS**

## PRELIMINARY STATISTICAL ANALYSIS OF VISCOSITY DATA FOR MATERIALS PSU1 AND PSU4

This experiment provides the data by which the noise in the measured viscosity of the materials PSU1 and PSU4 may be assigned to the laboratory and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j) = \text{Lab}(i) + \text{Error}(i,j)$$

$\text{Lab}(i)$  is a random variable with mean of 0 and standard deviation of  $\text{SIG}(\text{LAB})$

$\text{Error}(i,j)$  is a random variable with mean of 0 and standard deviation of  $\text{SIG}(\text{ERROR})$

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given. Summaries which include the other quantities of interest will be given in a later section.

When the data in this experiment were first analyzed it was clear that two laboratories had excessive within laboratory variation when tested at 135C and one of these also had excessive within laboratory variation at 165C. With these omitted the estimated values for the standard deviations, CV% and mean are given below.

	Temperature					
	135C			165C		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	71.7	7.1%	1013.6	38.0	13.3%	284.5
ERROR	26.3	2.6%		9.6	3.4%	

The computer output is shown in the next page.

THIS IS FOR the viscosity for PSU1 AND PSU4 UNAGED  
 SAMPLES FROM 10 are NOT USED at either temp.  
 SAMPLES FROM 17 are not used at temp 135  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

----- TEMP=135 -----

Nested Random Effects Analysis of Variance for Variable VISC

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	31	175730			
LAB	15	164642	15.839	0.0000	ERROR
ERROR	16	11088			
Variance Source	Mean Square	Variance Component		Percent of Total	
TOTAL	5668.694677	5834.552104		100.0000	
LAB	10976	5141.580229		88.1230	
ERROR	692.971875	692.971875		11.8770	
	Mean		1013.61250000		
	Standard error of mean		18.52037082		

----- TEMP=165 -----

Nested Random Effects Analysis of Variance for Variable VISC

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	33	49223			
LAB	16	47653	32.247	0.0000	ERROR
ERROR	17	1570.110000			
Variance Source	Mean Square	Variance Component		Percent of Total	
TOTAL	1491.612692	1535.339357		100.0000	
LAB	2978.319301	1442.979945		93.9844	
ERROR	92.359412	92.359412		6.0156	
	Mean		284.49411765		
	Standard error of mean		9.35936037		

## PRELIMINARY STATISTICAL ANALYSIS OF VISCOSITY DATA FOR MATERIALS PSU2 AND PSU3

This experiment provides the data by which the noise in the measured viscosity of the materials PSU2 and PSU3 may be assigned to the laboratory and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j) = \text{Lab}(i) + \text{Error}(i,j)$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB)

Error(i,j) is a random variable with mean of 0 and standard deviation of SIG(ERROR)

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given. Summaries which include the other quantities of interest will be given in a later section.

When the data in this experiment were first analyzed it was clear that three laboratories had excessive within laboratory variation when tested at 135C and three (two repeats, one different) had excessive within laboratory variation at 165C. With these omitted the estimated values for the standard deviations, CV% and means are given below.

	Temperature					
	135C			165C		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	38.7	8.6%	450.2	13.6	10.8%	126.2
ERROR	9.7	2.1%		5.0	3.9%	

The computer output is shown in the next page.

THIS IS FOR THE VISCOSITY FOR PSU2 AND PSU3 UNAGED  
 SAMPLES FROM 10, 17, AND 19 ARE NOT USED AT TEMP 135  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

Nested Random Effects Analysis of Variance for Variable VISC

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	29	44738			
LAB	14	43334	33.078	0.0000	ERROR
ERROR	15	1403.625000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	1542.684195	1594.438095	100.0000
LAB	3095.301190	1500.863095	94.1312
ERROR	93.575000	93.575000	5.8688

Mean 450.2166667  
 Standard error of mean 10.15759353

THIS IS FOR THE VISCOSITY FOR PSU2 AND PSU3 UNAGED  
 SAMPLES FROM 9, 10 and 19 ARE NOT USED AT TEMP 165  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

Nested Random Effects Analysis of Variance for Variable VISC

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	29	5921.341667			
LAB	14	5551.216667	16.070	0.0000	ERROR
ERROR	15	370.125000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	204.184195	210.595238	100.0000
LAB	396.515476	185.920238	88.2832
ERROR	24.675000	24.675000	11.7168

Mean 126.1166667  
 Standard error of mean 3.63554433

## PRELIMINARY STATISTICAL ANALYSIS OF G\* EXPERIMENT WITH UNAGED PSU1 & PSU4

1. This experiment provides the data by which the total noise in the measured values of G\* may be assigned to the Laboratory and the measurement error. Accordingly, an appropriate model for the measured values is;

$$Y(i,j,k) = \text{Lab}(i) + \text{Error}(i,j) \text{ where}$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB),  
Error(i,j) is a random variable with mean of 0 and standard deviation of SIG(ERROR).

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual DIS, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that two laboratories had excessive within laboratory variation. Accordingly, the data for these laboratories were omitted for both temperatures. With these omitted the estimated values for the standard deviations, means, and coefficients of variation(%) are given below. A total of 17 laboratories had acceptable data for temperature of 70 and 16 laboratories had acceptable data for temperature of 76.

	Temperature					
	70			76		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	82.2	5.9%	1394.8	43.2	5.6%	765.1
ERROR	51.1	3.7%		28.4	3.7%	

The analyses upon which the above were obtained are given in the next page.

THIS IS FOR THE G STAR FOR UNAGED PSU 1&4  
 WE HAVE THE COMPONENTS DUE TO THE LABS AND THE ERROR  
 LABS 14 AND 16 ARE REMOVED FROM THE DATA SET  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

----- TEMP=70 -----

Nested Random Effects Analysis of Variance for Variable GSTAR

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	57	515639			
LAB	16	408787			
ERROR	41	106851			
Variance Source	Mean Square	Variance Component		Percent of Total	
TOTAL	9046.291591	9360.944251		100.0000	
LAB	25549	6754.816203		72.1596	
ERROR	2606.128049	2606.128049		27.8404	
Mean			1394.75862069		
Standard error of mean			21.69385178		

----- TEMP=76 -----

Nested Random Effects Analysis of Variance for Variable GSTAR

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	53	136643			
LAB	15	106019			
ERROR	38	30623			
Variance Source	Mean Square	Variance Component		Percent of Total	
TOTAL	2578.163005	2670.686984		100.0000	
LAB	7067.965117	1864.814287		69.8253	
ERROR	805.872697	805.872697		30.1747	
Mean			765.09629630		
Standard error of mean			11.84346818		



## PRELIMINARY STATISTICAL ANALYSIS OF G\* EXPERIMENT WITH UNAGED PSU2 & PSU3

2. This experiment provides the data by which the total noise in the measured values of G\* may be assigned to the Laboratory and the measurement error. Accordingly, an appropriate model for the measured values is;

$$Y(i,j) = \text{Lab}(i) + \text{Error}(i,j) \text{ where}$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB),  
Error(i,j) is a random variable with mean of 0 and standard deviation of SIG(ERROR).

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that three laboratories had excessive within laboratory variation. Accordingly, the data for these laboratories were omitted for both temperatures. With these omitted the estimated values for the standard deviations, means, and coefficients of variation (%) are given BELOW. A total of 16 laboratories had acceptable data for both temperatures.

	Temperature					
	64			70		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	111.8	7.7%	1457.6	44.3	6.3%	702.1
ERROR	55.4	3.8%		29.1	4.2%	

The analyses upon which the above were obtained are given in the next page.

THIS IS FOR THE G STAR FOR UNAGED PSU 2&3  
 WE HAVE THE COMPONENTS DUE TO THE LABS AND THE ERROR  
 LABS 4, 14 AND 15 ARE REMOVED FOR BOTH TEMPERATURES  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

----- TEMP=64 -----

Nested Random Effects Analysis of Variance for Variable GSTAR

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	53	791946			
LAB	15	675315			
ERROR	38	116631			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	14942	15562	100.0000
LAB	45021	12493	80.2776
ERROR	3069.243421	3069.243421	19.7224

Mean 1457.64814815  
 Standard error of mean 29.94258749

----- TEMP=70 -----

Nested Random Effects Analysis of Variance for Variable GSTAR

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	53	143984			
LAB	15	111714			
ERROR	38	32270			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	2716.684906	2814.177559	100.0000
LAB	7447.596000	1964.957559	69.8235
ERROR	849.220000	849.220000	30.1765

Mean 702.10000000  
 Standard error of mean 12.15736949

# **PRELIMINARY STATISTICAL ANALYSIS OF DELTA EXPERIMENT PA-U-1&42 (UNAGED PSU1 & 4)**

3. This experiment provides the data by which the total noise in the measured values of Delta may be assigned to the Laboratory and the measurement error. Accordingly, an appropriate model for the measured values is;

$$Y(i,j) = \mu + \text{Lab}(i) + \text{Error}(i,j) \quad \text{where}$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB),  
Error(i,j) is a random variable with mean of 0 and standard deviation of SIG(ERROR).

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation at both temperatures and an additional laboratory had excessive within laboratory variation at a temperature of 76C. Accordingly, their data for the appropriate temperatures were omitted. With these omitted the estimated values for the standard deviations, means, and coefficients of variation (%) are given below. A total of 18 laboratories had acceptable data at 70C and 17 had acceptable data at 76C.

	Temperature					
	70C			76C		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	.3357	.44%	75.856	.4779	.61%	78.00
ERROR	.7979	1.1%		.3430	.44%	

The analyses upon which the above were obtained are given in the next page.

THIS IS UNAGED PSU1 AND PSU4, DELTA AS THE VARIABLE.  
 WE HAVE THE COMPONENTS DUE TO THE LABS AND THE ERROR  
 LAB 17 REMOVED AT BOTH TEMP. LAB 16 REMOVED AT 76C  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

----- TEMP=70 -----

Nested Random Effects Analysis of Variance for Variable DELTA

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	63	46.917500			
LAB	18	18.265000			
ERROR	45	28.652500			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.744722	0.749418	100.0000
LAB	1.014722	0.112696	15.0378
ERROR	0.636722	0.636722	84.9622

Mean 75.85625000  
 Standard error of mean 0.12779647

----- TEMP=76 -----

Nested Random Effects Analysis of Variance for Variable DELTA

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	57	19.119310			
LAB	16	14.294310			
ERROR	41	4.825000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.335426	0.346065	100.0000
LAB	0.893394	0.228382	65.9940
ERROR	0.117683	0.117683	34.0060

Mean 78.00344828  
 Standard error of mean 0.12814719

# **PRELIMINARY STATISTICAL ANALYSIS OF DELTA EXPERIMENT PA-U-2&32 (UNAGED PSU2 & 3)**

4. This experiment provides the data by which the total noise in the measured values of Delta may be assigned to the Laboratory and the measurement error. Accordingly, an appropriate model for the measured values is;

$$Y(i,j) = \mu + \text{Lab}(i) + \text{Error}(i,j) \text{ where}$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB),  
Error(i,j) is a random variable with mean of 0 and standard deviation of SIG(ERROR).

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation at a temperature of 64C. A different laboratory had excessive within laboratory variation at a temperature of 70C. Accordingly, their data for the appropriate temperatures were omitted. With these omitted the estimated values for the standard deviations, means, and coefficients of variation (%) are given below. For each of the temperatures a total of 17 laboratories had acceptable data for these analyses.

	Temperature					
	64			70		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	.4098	.47%	86.413	.7348	.84%	87.75
ERROR	.2809	.33%		.2741	.31%	

The analyses upon which the above were obtained are given in the next page.

THIS IS UNAGED PSU2 AND PSU3, DELTA AS THE VARIABLE.  
 WE HAVE THE COMPONENTS DUE TO THE LABS AND THE ERROR  
 LABS 14(AT 70), 15(AT 64) ARE REMOVED FROM THE DATA SET  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

----- TEMP=64 -----

Nested Random Effects Analysis of Variance for Variable DELTA

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	59	14.129333			
LAB	17	10.814333			
ERROR	42	3.315000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.239480	0.246882	100.0000
LAB	0.636137	0.167953	68.0298
ERROR	0.078929	0.078929	31.9702

Mean	86.41333333
Standard error of mean	0.10673638

----- TEMP=70 -----

Nested Random Effects Analysis of Variance for Variable DELTA

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	59	34.889833			
LAB	17	31.734833			
ERROR	42	3.155000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.591353	0.615151	100.0000
LAB	1.866755	0.540032	87.7885
ERROR	0.075119	0.075119	12.2115

Mean	87.75166667
Standard error of mean	0.18345002

## PRELIMINARY STATISTICAL ANALYSIS OF G\*/SIN(DELTA) EXPERIMENT (UNAGED PSU1 & 4)

5. This experiment provides the data by which the total noise in the measured values of  $G^*/\sin(\Delta)$  may be assigned to the Laboratory and the measurement error. Accordingly, an appropriate model for the measured values is;

$$Y(i,j) = \mu + \text{Lab}(i) + \text{Error}(i,j) \quad \text{where}$$

$\text{Lab}(i)$  is a random variable with mean of 0 and standard deviation of  $\text{SIG}(\text{LAB})$ ,  
 $\text{Error}(i,j)$  is a random variable with mean of 0 and standard deviation of  $\text{SIG}(\text{ERROR})$ .

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that two laboratories had excessive within laboratory variation at both temperatures. Accordingly, their data were omitted. With these omitted the estimated values for the standard deviations, means, and coefficients of variation (%) are given below. A total of 17 laboratories had acceptable data at 70C and 16 had acceptable data at 76C.

	Temperature					
	70C			76C		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	86.81	6.0%	1438.6	44.09	5.6%	782.13
ERROR	54.48	3.8%		29.44	3.8%	

The analyses upon which the above were obtained are given in the next page.

THIS IS UNAGED PSU1 AND PSU4. GDBSD IS  $G^*/\sin(\Delta)$   
 WE HAVE THE COMPONENTS DUE TO THE LABS AND THE ERROR  
 LABS 14 AND 16 REMOVED AT BOTH TEMP.  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

----- TEMP=70 -----

Nested Random Effects Analysis of Variance for Variable GDBSD

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	57	578706			
LAB	16	457003			
ERROR	41	121704			
Variance Source	Mean Square	Variance Component		Percent of Total	
TOTAL	10153	10504		100.0000	
LAB	28563	7535.370342		71.7398	
ERROR	2968.383074	2968.383074		28.2602	
Mean			1438.55399774		
Standard error of mean			22.93599072		

----- TEMP=76 -----

Nested Random Effects Analysis of Variance for Variable GDBSD

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	53	143850			
LAB	15	110909			
ERROR	38	32941			
Variance Source	Mean Square	Variance Component		Percent of Total	
TOTAL	2714.147134	2810.586535		100.0000	
LAB	7393.948290	1943.729331		69.1574	
ERROR	866.857203	866.857203		30.8426	
Mean			782.13495209		
Standard error of mean			12.11203471		



## PRELIMINARY STATISTICAL ANALYSIS OF G\* EXPERIMENT FOR RTFOT PSU 1&4

This experiment provides the data by which the total noise in the measured values of G\* may be assigned to the Laboratory, the thin film oven aging, and the measurement error. Accordingly, an appropriate model for the measured values is;

$$Y(i,j,k) = \text{Lab}(i) + \text{RTFO}(i,j) + \text{Error}(i,j,k) \text{ where}$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB),  
RTFO(i,j) is a random variable with mean of 0 and standard deviation of SIG(RTFO),  
Error(i,j,k) is a random variable with mean of 0 and standard deviation of SIG(ERROR).

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that two laboratories had excessive within laboratory variation. Accordingly, the data for one laboratory were omitted for both temperatures and the data for the other laboratory were eliminated for the temperature of 76. With these omitted the estimated values for the standard deviations are given below.

	Temperature					
	70			76		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	95.7	2.9%	3298.5	68.9	3.9%	1783.6
RTFO	248.0	7.5%		150.1	8.4%	
ERROR	145.6	4.4%		65.2	3.7%	

The analyses upon which the above were obtained are given in the next page.

THIS IS FOR THE RTFOT G STAR FOR RTFOT PSU1&4  
 ALL DATA POINTS FOR 16 WERE OMITTED AND FOR 15 THE DATA POINTS AT 76  
 DUE TO EXCESSIVE NOISE WITHIN THE LAB  
 WE HAVE THE COMPONENTS DUE TO THE LABS THE RTFOF AND THE ERR

Nested Random Effects Analysis of Variance for Variable GSTAR

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	59	5342599			
LAB	16	2585459			
RTFOF	17	2206008			
ERROR	26	551133			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	90553	91881	100.0000
LAB	161591	9161.533452	9.9711
RTFOF	129765	61522	66.9583
ERROR	21197	21197	23.0706

Mean 3298.51666667  
 Standard error of mean 53.26678511

----- TEMP=76 -----

Nested Random Effects Analysis of Variance for Variable GSTAR

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	50	1543934			
LAB	14	827594			
RTFOF	14	622842			
ERROR	22	93498			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	30879	31524	100.0000
LAB	59114	4740.410149	15.0374
RTFOF	44489	22534	71.4811
ERROR	4249.886364	4249.886364	13.4814

Mean 1783.64705882  
 Standard error of mean 35.36695327

## PRELIMINARY STATISTICAL ANALYSIS OF DELTA EXPERIMENT (RTFO AGED PSU1 & 4)

6. This experiment provides the data by which the total noise in the measured values of Delta may be assigned to the Laboratory, the RTFO aging, and the measurement error. Accordingly, an appropriate model for the measured values is;

$$Y(i,j,k) = \mu + \text{Lab}(i) + \text{RTFO}(i,j) + \text{Error}(i,j,k) \quad \text{where}$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB),  
RTFO(i,j) is a random variable with mean of 0 and standard deviation of SIG(RTFO),  
Error(i,j,k) is a random variable with mean of 0 and standard deviation of SIG(ERROR).

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation at both temperatures. Accordingly, their data were omitted. With these omitted the estimated values for the standard deviations, means, and coefficients of variation(%) are given below. At 70C 15 laboratories had acceptable data and at 76C 16 laboratories had acceptable data.

	Temperature					
	70C			76C		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	.3293	.46%	70.98	.3413	.47%	73.24
RTFO	.4225	.60%		.4561	.62%	
ERROR	.3954	.56%		.3997	.55%	

The analyses upon which the above were obtained are given in the next page.

INDIV AGED PSU1 & PSU4 VARIABLE DELTA  
 WE HAVE THE COMPONENTS DUE TO THE LABS, RTFO, AND THE ERROR  
 LAB 16 REMOVED AT BOTH TEMP.  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

----- TEMP=70 -----

Nested Random Effects Analysis of Variance for Variable DELTA

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	59	25.703333			
LAB	16	13.625833			
RTFO	17	8.012500			
ERROR	26	4.065000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.435650	0.443296	100.0000
LAB	0.851615	0.108463	24.4674
RTFO	0.471324	0.178487	40.2636
ERROR	0.156346	0.156346	35.2690

Mean 70.98333333  
 Standard error of mean 0.12209634

----- TEMP=76 -----

Nested Random Effects Analysis of Variance for Variable DELTA

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	54	25.649818			
LAB	15	13.802318			
RTFO	15	8.012500			
ERROR	24	3.835000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.474997	0.484240	100.0000
LAB	0.920155	0.116462	24.0505
RTFO	0.534167	0.207986	42.9510
ERROR	0.159792	0.159792	32.9984

Mean 73.23818182  
 Standard error of mean 0.13371470

## PRELIMINARY STATISTICAL ANALYSIS OF G\*/SIN(DELTA) EXPERIMENT (INDIV RTFO AGED PSU1 & 4)

This experiment provides the data by which the total noise in the measured values of  $G^*/\sin(\Delta)$  may be assigned to the Laboratory, the RTFO aging, and the measurement error. Accordingly, an appropriate model for the measured values is;

$$Y(i,j,k) = \mu + \text{Lab}(i) + \text{RTFO}(i,j) + \text{Error}(i,j,k) \quad \text{where}$$

$\text{Lab}(i)$  is a random variable with mean of 0 and standard deviation of  $\text{SIG}(\text{LAB})$ ,  
 $\text{RTFO}(i,j)$  is a random variable with mean of 0 and standard deviation of  $\text{SIG}(\text{RTFO})$ ,  
 $\text{Error}(i,j,k)$  is a random variable with mean of 0 and standard deviation of  $\text{SIG}(\text{ERROR})$ .

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation at both temperatures. Another laboratory had excessive within laboratory variation at 76C. Accordingly, their data were omitted. With these omitted the estimated values for the standard deviations, means, and coefficients of variation (%) are given below. For each temperature, a total of 15 laboratories had acceptable data.

	Temperature					
	70C			76C		
	STD	CV%	MEAN	STD	CV%	MEAN
LAB	106.1	3.0%	3490.2	73.5	3.9%	1863.1
RTFO	270.8	7.8%		160.3	8.6%	
ERROR	159.9	4.6%		70.3	3.8%	

The analyses upon which the above were obtained are given in the next page.

INDIV AGED PSU1 & PSU4 VARIABLE GDBSD IS  $G^*/\sin(\Delta)$   
 WE HAVE THE COMPONENTS DUE TO THE LABS, RTFO, AND THE ERROR  
 LAB 16 REMOVED AT BOTH TEMP. 15 out at 76C  
 DUE TO EXCESSIVE WITHIN LAB VARIATION

----- TEMP=70 -----

Nested Random Effects Analysis of Variance for Variable GDBSD

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	59	6406249			
LAB	16	3106347			
RTFO	17	2635147			
ERROR	26	664754			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	108580	110180	100.0000
LAB	194147	11262	10.2217
RTFO	155009	73350	66.5731
ERROR	25567	25567	23.2053

Mean 3490.22731348  
 Standard error of mean 58.38565826

----- TEMP=76 -----

Nested Random Effects Analysis of Variance for Variable GDBSD

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	50	1766178			
LAB	14	945307			
RTFO	14	711764			
ERROR	22	109107			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	35324	36060	100.0000
LAB	67522	5406.787909	14.9941
RTFO	50840	25693	71.2526
ERROR	4959.396801	4959.396801	13.7534

Mean 1863.12102859  
 Standard error of mean 37.79625831

**PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS  
PSU1 AND PSU4 WHEN AGED BY THE RTFOT AND THE PAV AND TESTED AT  
-12C**

This experiment provides the data by which the noise in the measured stiffness of the bending beam at 60 seconds (S60) may be assigned to the Laboratory, the Aging and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j,k) = \text{Lab}(i) + \text{PAV}(i,j) + \text{Error}(i,j,k)$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB)

PAV(i,j) is a random variable with mean of 0 and standard deviation of SIG(PAV)

Error(i,j,k) is a random variable with mean of 0 and standard deviation of SIG(ERROR)

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation. Accordingly, the data for one laboratory were omitted.

With these omitted the estimated values for the standard deviations, CV% and mean are given below.

	Temperature -12C		
	STD	CV%	MEAN
LAB	8.3	6.2%	134.4
PAV	6.2	4.6%	
ERROR	4.6	3.4%	

The analyses upon which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH PAV AGED PSU1 & PSU4  
 ALL DATA POINTS FOR 15 WERE OMITTED  
 DUE TO EXCESSIVE NOISE WITHIN THE LAB  
 THIS IS FOR THE BBR AT TEMP -12C AND STIFFNESS AT 60 SECONDS

Coefficients of Expected Mean Squares

Source	LAB	PAV	ERROR
LAB	3.940298507	1.980099502	1.000000000
PAV	0.000000000	1.960784314	1.000000000
ERROR	0.000000000	0.000000000	1.000000000

Nested Random Effects Analysis of Variance for Variable S60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	66	8252.447761			
LAB	16	5915.531095			
PAV	17	1650.916667			
ERROR	33	686.000000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	125.037087	128.707341	100.0000
LAB	369.720693	68.993781	53.6052
PAV	97.112745	38.925682	30.2436
ERROR	20.787879	20.787879	16.1513

Mean 134.43283582  
 Standard error of mean 2.35297243



**PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS  
PSU1 AND PSU4 WHEN AGED BY THE RTFOT AND THE PAV AND TESTED AT  
-12C (m60)**

This experiment provides the data by which the noise in the measured  $m(60)$  of the bending beam at 60 seconds ( $m60$ ) may be assigned to the Laboratory, the Aging and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j,k) = MU + Lab(i) + PAV(i,j) + Error(i,j,k)$$

$Lab(i)$  is a random variable with mean of 0 and standard deviation of  $SIG(LAB)$

$PAV(i,j)$  is a random variable with mean of 0 and standard deviation of  $SIG(PAV)$

$Error(i,j,k)$  is a random variable with mean of 0 and standard deviation of  $SIG(ERROR)$

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that no laboratory had excessive within laboratory variation. Accordingly no data were omitted. The estimated values for the standard deviations, CV% and mean for  $m(60)$  are given below.

Temperature -12C			
	STD	CV%	MEAN
LAB	.008118	2.5%	.3268
PAV	.005158	1.6%	
ERROR	.005718	1.7%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH IND PAV AGED PSU1 & PSU4  
 THIS IS FOR THE BBR AT TEMP -12C AND m(60)  
 NO OUTLIERS NEEDED TO BE OMITTED

# Coefficients of Expected Mean Squares

Source	LAB	PAV	ERROR
LAB	3.943661972	1.981220657	1.000000000
PAV	0.000000000	1.962962963	1.000000000
ERROR	0.000000000	0.000000000	1.000000000

## Nested Random Effects Analysis of Variance for Variable M60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	70	0.008555			
LAB	17	0.005876			
PAV	18	0.001532			
ERROR	35	0.001147			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.000122	0.000125	100.0000
LAB	0.000346	0.000065940	52.5921
PAV	0.000085106	0.000026654	21.2587
ERROR	0.000032786	0.000032786	26.1492

Mean 0.32680282  
 Standard error of mean 0.00220974

# **PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS PSU2 AND PSU3 WHEN AGED BY THE RTFOT AND THE PAV AND TESTED AT -12C**

This experiment provides the data by which the noise in the measured stiffness of the bending beam at 60 seconds (S60) may be assigned to the Laboratory, the Aging and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j,k) = MU + Lab(i) + PAV(i,j) + Error(i,j,k)$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB)  
PAV(i,j) is a random variable with mean of 0 and standard deviation of SIG(PAV)  
Error(i,j,k) is a random variable with mean of 0 and standard deviation of SIG(ERROR)

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation. Accordingly, the data for one laboratory were omitted. With these omitted the estimated values for the standard deviations, CV% and mean are given below.

	Temperature -12C		
	STD	CV%	MEAN
LAB	6.3	4.2%	149.0
PAV	5.9	4.0%	
ERROR	6.2	4.2%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH PAV AGED PSU2 & PSU3  
 ALL DATA POINTS FOR 15 WERE OMITTED  
 DUE TO EXCESSIVE NOISE WITHIN THE LAB  
 THIS IS FOR THE BBR AT TEMP -12C AND STIFFNESS AT 60 SECONDS

# Coefficients of Expected Mean Squares

Source	LAB	PAV	ERROR
LAB	4	2	1
PAV	0	2	1
ERROR	0	0	1

## Nested Random Effects Analysis of Variance for Variable S60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	67	7425.941176			
LAB	16	4271.441176	2.465	0.0369	PAV
PAV	17	1841.500000	2.805	0.0051	ERROR
ERROR	34	1313.000000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	110.834943	113.130974	100.0000
LAB	266.965074	39.660386	35.0571
PAV	108.323529	34.852941	30.8076
ERROR	38.617647	38.617647	34.1353

Mean	148.97058824
Standard error of mean	1.98140278

**PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS  
PSU2 AND PSU3 WHEN AGED BY THE RTFOT AND THE PAV AND TESTED AT  
-12C (m60)**

This experiment provides the data by which the noise in the measured stiffness of the bending beam at 60 seconds (m60) may be assigned to the Laboratory, the Aging and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j,k) = MU + Lab(i) + PAV(i,j) + Error(i,j,k)$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB)

PAV(i,j) is a random variable with mean of 0 and standard deviation of SIG(PAV)

Error(i,j,k) is a random variable with mean of 0 and standard deviation of SIG(ERROR)

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that no laboratory had excessive within laboratory variation. Accordingly no data were omitted. The estimated values for the standard deviations, CV% and mean for m(60) are given below.

Temperature -12C			
	STD	CV%	MEAN
LAB	.0104	3.0%	.3514
PAV	.00367	1.0%	
ERROR	.00697	2.0%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH IND PAV AGED PSU2 & PSU3  
 THIS IS FOR THE BBR AT TEMP -12C AND m(60)  
 NO OUTLIERS NEEDED TO BE OMITTED

Coefficients of Expected Mean Squares

Source	LAB	PAV	ERROR
LAB	3.943661972	1.981220657	1.000000000
PAV	0.000000000	1.962962963	1.000000000
ERROR	0.000000000	0.000000000	1.000000000

Nested Random Effects Analysis of Variance for Variable M60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	70	0.011635			
LAB	17	0.008580			
PAV	18	0.001353			
ERROR	35	0.001701			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.000166	0.000171	100.0000
LAB	0.000505	0.000109	63.6527
PAV	0.000075194	0.000013541	7.9184
ERROR	0.000048614	0.000048614	28.4288

Mean 0.35139437  
 Standard error of mean 0.00267039

**PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS  
PSU1 AND PSU4 WHEN AGED BY THE RTFOT AND THE PAV AND TESTED AT  
-18C**

This experiment provides the data by which the noise in the measured stiffness of the bending beam at 60 seconds (S60) may be assigned to the Laboratory, the Aging and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j,k) = MU + Lab(i) + PAV(i,j) + Error(i,j,k)$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB)

PAV(i,j) is a random variable with mean of 0 and standard deviation of SIG(PAV)

Error(i,j,k) is a random variable with mean of 0 and standard deviation of SIG(ERROR)

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that two laboratories had excessive within laboratory variation. Accordingly, the data for two laboratories were omitted. With these omitted the estimated values for the standard deviations, CV% and mean are given below.

	Temperature -18C		
	STD	CV%	MEAN
LAB	28.2	10.0%	279.3
PAV	7.1	2.5%	
ERROR	10.7	3.8%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH PAV AGED PSU1 & PSU4  
 ALL DATA POINTS FOR PECA AND VTRC WERE OMITTED  
 DUE TO EXCESSIVE NOISE WITHIN THE LABS  
 THIS IS FOR THE BBR AT TEMP -18C AND STIFFNESS AT 60 SECONDS

# Coefficients of Expected Mean Squares

Source	LAB	PAV	ERROR
LAB	3.936507937	1.978835979	1.000000000
PAV	0.000000000	1.958333333	1.000000000
ERROR	0.000000000	0.000000000	1.000000000

# Nested Random Effects Analysis of Variance for Variable S60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	62	57025			
LAB	15	50033			
PAV	16	3411.916667			
ERROR	31	3580.500000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	919.762417	958.311796	100.0000
LAB	3335.523545	792.899562	82.7392
PAV	213.244792	49.912234	5.2084
ERROR	115.500000	115.500000	12.0524

Mean	279.30158730
Standard error of mean	7.29032120



# **PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS PSU1 AND PSU4 WHEN AGED BY THE RTFOT AND THE PAV AND TESTED AT -18C (m60)**

This experiment provides the data by which the noise in the measured  $m(60)$  of the bending beam at 60 seconds ( $m60$ ) may be assigned to the Laboratory, the Aging and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j,k) = MU + Lab(i) + PAV(i,j) + Error(i,j,k)$$

$Lab(i)$  is a random variable with mean of 0 and standard deviation of  $SIG(LAB)$

$PAV(i,j)$  is a random variable with mean of 0 and standard deviation of  $SIG(PAV)$

$Error(i,j,k)$  is a random variable with mean of 0 and standard deviation of  $SIG(ERROR)$

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation. Accordingly the data from that laboratory were omitted. The estimated values for the standard deviations, CV% and mean for  $m(60)$  are given below.

	Temperature -18C		
	STD	CV%	MEAN
LAB	.009418	3.3%	.2813
PAV	.003225	1.1%	
ERROR	.004099	1.5%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH IND PAV AGED PSU1 & PSU4  
 THIS IS FOR THE BBR AT TEMP -18C AND M(60)  
 LAB VTRC OMITTED DUE TO EXCESSIVE WITHIN LAB NOISE

Coefficients of Expected Mean Squares

Source	LAB	PAV	ERROR
LAB	3.940298507	1.980099502	1.000000000
PAV	0.000000000	1.960784314	1.000000000
ERROR	0.000000000	0.000000000	1.000000000

Nested Random Effects Analysis of Variance for Variable M60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	66	0.007389			
LAB	16	0.006197			
PAV	17	0.000635			
ERROR	33	0.000556			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.000112	0.000116	100.0000
LAB	0.000387	0.000088758	76.4588
PAV	0.000037382	0.000010465	9.0144
ERROR	0.000016864	0.000016864	14.5268

Mean 0.28134328  
 Standard error of mean 0.00240862

**PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS  
PSU2 AND PSU3 WHEN AGED BY THE RTFOT AND THE PAV AND TESTED AT  
-18C**

This experiment provides the data by which the noise in the measured stiffness of the bending beam at 60 seconds (S60) may be assigned to the Laboratory, the Aging and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j,k) = MU + Lab(i) + PAV(i,j) + Error(i,j,k)$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB)

PAV(i,j) is a random variable with mean of 0 and standard deviation of SIG(PAV)

Error(i,j,k) is a random variable with mean of 0 and standard deviation of SIG(ERROR)

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation. Accordingly, the data for two laboratories were omitted. With these omitted the estimated values for the standard deviations, CV% and mean are given below.

	Temperature -18C		
	STD	CV%	MEAN
LAB	31.3	9.7%	322.6
PAV	8.6	2.7%	
ERROR	11.1	3.4%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH PAV AGED PSU2 & PSU3  
 ALL DATA POINTS FOR 15 AND 20 WERE OMITTED  
 DUE TO EXCESSIVE NOISE WITHIN THE LABS  
 THIS IS FOR THE BBR AT TEMP -18C AND STIFFNESS AT 60 SECONDS

Coefficients of Expected Mean Squares

Source	LAB	PAV	ERROR
LAB	3.873118280	1.957706093	1.000000000
PAV	0.000000000	1.916666667	1.000000000
ERROR	0.000000000	0.000000000	1.000000000

Nested Random Effects Analysis of Variance for Variable \$60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	61	68871			
LAB	15	60939			
PAV	16	4238.416667			
ERROR	30	3694.000000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	1129.036753	1176.839841	100.0000
LAB	4062.588351	979.740747	83.2518
PAV	264.901042	73.965761	6.2851
ERROR	123.133333	123.133333	10.4630

Mean 322.56451613  
 Standard error of mean 8.12486053

**PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS  
PSU2 AND PSU3 WHEN AGED BY THE RTFOT AND THE PAV AND TESTED AT  
-18C (m60)**

This experiment provides the data by which the noise in the measured  $m(60)$  of the bending beam at 60 seconds ( $m60$ ) may be assigned to the Laboratory, the Aging and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j,k) = MU + Lab(i) + PAV(i,j) + Error(i,j,k)$$

$Lab(i)$  is a random variable with mean of 0 and standard deviation of  $SIG(LAB)$

$PAV(i,j)$  is a random variable with mean of 0 and standard deviation of  $SIG(PAV)$

$Error(i,j,k)$  is a random variable with mean of 0 and standard deviation of  $SIG(ERROR)$

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation. Accordingly the data from that laboratory were omitted. The estimated values for the standard deviations, CV% and mean for  $m(60)$  are given below.

	Temperature -18C		
	STD	CV%	MEAN
LAB	.01483	5.1%	.2931
PAV	.003912	1.3%	
ERROR	.006557	2.2%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH IND PAV AGED PAU2 & PSU3  
 THIS IS FOR THE BBR AT TEMP -18C AND M(60)  
 LAB 3 OMITTED DUE TO EXCESSIVE WITHIN LAB NOISE

Coefficients of Expected Mean Squares

Source	LAB	PAV	ERROR
LAB	3.821153846	1.940384615	1.000000000
PAV	0.000000000	1.882352941	1.000000000
ERROR	0.000000000	0.000000000	1.000000000

Nested Random Effects Analysis of Variance for Variable M60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	64	0.017142			
LAB	16	0.014585			
PAV	17	0.001222			
ERROR	31	0.001335			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.000268	0.000278	100.0000
LAB	0.000912	0.000220	78.9901
PAV	0.000071902	0.000015320	5.5129
ERROR	0.000043065	0.000043065	15.4969

Mean 0.29310769  
 Standard error of mean 0.00376344

**PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS  
PSU1 AND PSU4 WHEN PREAGED AND TESTED AT -18C (S60)**

This experiment provides the data by which the noise in the measured  $s(60)$  of the bending beam at 60 seconds ( $s(60)$ ) may be assigned to the Laboratory and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j) = MU + Lab(i) + Error(i,j)$$

$Lab(i)$  is a random variable with mean of 0 and standard deviation of  $SIG(LAB)$

$Error(i,j)$  is a random variable with mean of 0 and standard deviation of  $SIG(ERROR)$

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that one laboratory had excessive within laboratory variation. Accordingly the data from that laboratory were omitted. The estimated values for the standard deviations, CV% and mean for  $m(60)$  are given below.

Temperature -18C			
	STD	CV%	MEAN
LAB	16.15	9.8%	164.26
ERROR	7.348	4.5%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH PRE AGED PSU1 & PSU4  
 THIS IS FOR THE BBR AT TEMP -18C AND S(60)  
 LAB 15 OMITTED DUE TO EXCESSIVE WITHIN LAB NOISE

Coefficients of Expected Mean Squares

Source	LAB	ERROR
LAB	3.887394958	1.000000000
ERROR	0.000000000	1.000000000

Nested Random Effects Analysis of Variance for Variable S60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	69	20955			
LAB	17	18148			
ERROR	52	2807.416667			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	303.701035	314.712989	100.0000
LAB	1067.526751	260.724207	82.8451
ERROR	53.988782	53.988782	17.1549

Mean	164.25714286
Standard error of mean	3.91797656



## PRELIMINARY STATISTICAL ANALYSIS OF m(60s) DATA FOR PREAGED MATERIALS PSU1 AND PSU4

This experiment provides the data by which the noise in the measured m(60s) at temperature of -18c of the preaged materials PSU1 and PSU4 may be assigned to the laboratory and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j) = MU + Lab(i) + Error(i,j)$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB)

Error(i,j) is a random variable with mean of 0 and standard deviation of SIG(ERROR)

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

None of the laboratories were judged to be outliers. The estimated values for the standard deviations, CV% and means are given below.

	STD	CV%	MEAN
LAB	.01307	3.7%	.352
ERROR	.005822	1.7%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR PREAGED PSU1 AND PSU4 PREAGED S(60S)  
 TEMPERATURE OF -18C  
 NO OUTLIERS REMOVED  
 PREAGED MATERIALS WERE SENT TO LABS

Coefficients of Expected Mean Squares

Source	LAB	ERROR
LAB	3.893393393	1.000000000
ERROR	0.000000000	1.000000000

Nested Random Effects Analysis of Variance for Variable M60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	73	0.014454			
LAB	18	0.012588			
ERROR	55	0.001866			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.000198	0.000205	100.0000
LAB	0.000699	0.000171	83.4380
ERROR	0.000033924	0.000033924	16.5620

Mean 0.35231081  
 Standard error of mean 0.00308373

## PRELIMINARY STATISTICAL ANALYSIS OF BBR DATA FOR MATERIALS PSU2 AND PSU3 WHEN PREAGED AND TESTED AT -18C (S60)

This experiment provides the data by which the noise in the measured  $s(60)$  of the bending beam at 60 seconds ( $s_{60}$ ) may be assigned to the Laboratory and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j) = MU + Lab(i) + Error(i,j)$$

$Lab(i)$  is a random variable with mean of 0 and standard deviation of  $SIG(LAB)$

$Error(i,j)$  is a random variable with mean of 0 and standard deviation of  $SIG(ERROR)$

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

When the data in this experiment were first analyzed it was clear that two laboratories had excessive within laboratory variation. Accordingly the data from those laboratories were omitted. The estimated values for the standard deviations, CV% and mean for  $m(60)$  are given below.

Temperature -18C			
	STD	CV%	MEAN
LAB	32.26	10.9%	296.54
ERROR	9.889	3.3%	

The analyses from which the above were obtained are given in the next page.

THIS IS FOR THE BENDING BEAM WITH PRE AGED PSU2 & PSU3  
 THIS IS FOR THE BBR AT TEMP -18C AND S(60)  
 LABS 15 AND 16 OMITTED DUE TO EXCESSIVE WITHIN LAB NOISE

# Coefficients of Expected Mean Squares

Source	LAB	ERROR
LAB	3.878787879	1.000000000
ERROR	0.000000000	1.000000000

## Nested Random Effects Analysis of Variance for Variable S60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	65	70928			
LAB	16	66137			
ERROR	49	4791.750000			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	1091.205594	1138.256978	100.0000
LAB	4133.538352	1040.466162	91.4087
ERROR	97.790816	97.790816	8.5913

Mean 296.54545455  
 Standard error of mean 7.97400788

## PRELIMINARY STATISTICAL ANALYSIS OF m(60s) DATA FOR PREAGED MATERIALS PSU2 AND PSU3

This experiment provides the data by which the noise in the measured m(60s) at temperature of -18c of the preaged materials PSU2 and PSU3 may be assigned to the laboratory and the measurement errors. Accordingly an appropriate model for the measured values is;

$$Y(i,j) = MU + Lab(i) + Error(i,j)$$

Lab(i) is a random variable with mean of 0 and standard deviation of SIG(LAB)

Error(i,j) is a random variable with mean of 0 and standard deviation of SIG(ERROR)

All components are assumed to be independent random variables. The purpose of a components of variance analysis is to estimate each of the above standard deviations and to provide the basis for the estimation of other important quantities such as the coefficients of variation, the usual D1S, D2S for the between and within Laboratories variation. In this preliminary analysis only the estimated standard deviations and associated coefficients of variation will be given.

None of the laboratories were judged to be outliers. The estimated values for the standard deviations, CV% and means are given below.

	STD	CV%	MEAN
LAB	.0173	5.6%	.310
ERROR	.00713	2.3%	

The analyses from which the above were obtained are given in the attached files BEAM12.LST and OUT12.LST.

THIS IS FOR BBR m(60s) FOR PSU2 AND PSU3 PREAGED  
 TEMPERATURE OF -18C  
 NO OUTLIERS REMOVED  
 PREAGED MATERIALS WERE SENT TO LABS

Coefficients of Expected Mean Squares

Source	LAB	ERROR
LAB	3.891891892	1.000000000
ERROR	0.000000000	1.000000000

Nested Random Effects Analysis of Variance for Variable m60

Variance Source	Degrees of Freedom	Sum of Squares	F Value	Pr > F	Error Term
TOTAL	73	0.024783			
LAB	18	0.021982			
ERROR	55	0.002801			

Variance Source	Mean Square	Variance Component	Percent of Total
TOTAL	0.000339	0.000352	100.0000
LAB	0.001221	0.000301	85.5170
ERROR	0.000050927	0.000050927	14.4830

Mean 0.30962162  
 Standard error of mean 0.00408937

# PRELIMINARY STATISTICAL ANALYSIS OF THE REFERENCE FLUID DATA AT 64C

Number of observations in data set = 112

NOTE: Due to missing values, only 69 observations can be used in this analysis.

RESULTS FOR TEMP = 64, AVERAGE OF 264.5 USED

## General Linear Models Procedure

Dependent Variable: DIFF

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	26	6590.5028	253.4809	12.67	0.0001
Error	42	840.1413	20.0034		
Corrected Total	68	7430.6441			

R-Square	C.V.	Root MSE	DIFF Mean
0.886936	99.68130	4.4725	4.4868

Source	DF	Type I SS	Mean Square	F Value	Pr > F
LAB	13	4745.8415	365.0647	18.25	0.0001
REP (LAB)	10	1343.4050	134.3405	6.72	0.0001
FREQ	1	405.5552	405.5552	20.27	0.0001
STRAIN	1	76.4586	76.4586	3.82	0.0573
FREQ*STRAIN	1	19.2424	19.2424	0.96	0.3323

Source	DF	Type III SS	Mean Square	F Value	Pr > F
LAB	13	4747.5826	365.1987	18.26	0.0001
REP (LAB)	10	1315.2283	131.5228	6.58	0.0001
FREQ	1	392.1439	392.1439	19.60	0.0001
STRAIN	1	84.9672	84.9672	4.25	0.0455
FREQ*STRAIN	1	19.2424	19.2424	0.96	0.3323

# PRELIMINARY STATISTICAL ANALYSIS OF THE REFERENCE FLUID DATA AT 76C

Number of observations in data set = 112

NOTE: Due to missing values, only 69 observations can be used in this analysis.

RESULTS FOR TEMP = 76C. AVERAGE OF 130.87 USED FOR REF

## General Linear Models Procedure

Dependent Variable: DIFF

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	25	2465.4011	98.6160	18.39	0.0001
Error	43	230.5418	5.3614		
Corrected Total	68	2695.9429			

R-Square	C.V.	Root MSE	DIFF Mean
0.914486	9999.99	2.3155	0.0068

Source	DF	Type I SS	Mean Square	F Value	Pr > F
LAB	13	2139.4437	164.5726	30.70	0.0001
REP (LAB)	9	240.1467	26.6830	4.98	0.0001
FREQ	1	53.5739	53.5739	9.99	0.0029
STRAIN	1	30.2951	30.2951	5.65	0.0220
FREQ*STRAIN	1	1.9417	1.9417	0.36	0.5505

Source	DF	Type III SS	Mean Square	F Value	Pr > F
LAB	13	1960.2640	150.7895	28.12	0.0001
REP (LAB)	9	228.5273	25.3919	4.74	0.0002
FREQ	1	51.1113	51.1113	9.53	0.0035
STRAIN	1	32.1688	32.1688	6.00	0.0185
FREQ*STRAIN	1	1.9417	1.9417	0.36	0.5505





## Appendix III

### Pre-molded DSR Samples Information

## Pre-molded DSR Sample Instructions

July 29, 1998

2 Pre-molded samples will arrive by express mail. Please store samples at room temperature in their molds, until the samples are placed in the DSR.

All testing will be performed at 64 C

Testing will be performed between 1:00 PM and 3:00 PM on July 29, 1998.

Late Morning

Set DSR software for original binder (12% Strain and 10 rad/sec).

Set the equilibrium time to 900 sec (15 minutes). Preheat the DSR to 64 C with upper spindle loose so as not to create pressure on the machine as it warms to the test temperature. Start preheating DSR at such a time so that the DSR is ready to test at 1:00 PM.

1:00 PM

On data sheet please note room temperature in space provided.

1:10 PM

Set zero gap at 64 C

1:15 PM

With sample still in mold mount sample #1 at room temperature in DSR.

Note time sample mounted in DSR on data sheet

Compress sample to 1.05 mm

Trim sample immediately with heated trimming tool

Compress sample to 1.00 mm

Note time on data sheet

Start test using 900 second equilibrium time

After test, printout results

Clean up machine and preheat as before to 64 C

1:50 PM

Reset zero gap at 64 C

Note room temperature on data sheet

2:00 PM

With sample still in mold transfer second sample to DSR

Note time mounted on data sheet

Compress sample to 1.05 mm

Trim immediately with heated trimming tool

Compress sample to 1.00 mm

Note time on data sheet

Start test using 900 second equilibrium

After test, print out results

After testing please fax or mail the data sheets as well as the printouts from your tests back to the CAP Lab (fax 860-486-2399)

Jim Mahoney

University of Connecticut

CAP Lab - Transportation Institute

179 Middle Turnpike, U 202

Storrs, CT 06269-5202

## Pre-Molded Sample Data Sheet

Laboratory Name \_\_\_\_\_

Date & Time Sample Arrived at Lab \_\_\_\_\_

### Sample #1

Room Temperature \_\_\_\_\_

Time Mounted in Machine \_\_\_\_\_

Time Sample Compressed to 1.00 mm \_\_\_\_\_

### Sample #2

Room Temperature \_\_\_\_\_

Time Mounted in Machine \_\_\_\_\_

Time Sample Compressed to 1.00 mm \_\_\_\_\_

How did you heat your trimming tool? \_\_\_\_\_

DSR Manufactured by? \_\_\_\_\_

Any irregularities while testing? \_\_\_\_\_

Please mail or fax (860) 486 - 2399 this sheet along with copies of your DSR test printouts.

Jim Mahoney  
University of Connecticut  
CAP Lab - Transportation Institute  
179 Middle Turnpike, U 202  
Storrs, CT 06269-5202

## NETC Pre-molded DSR Sample Participants

New Hampshire DOT

Vermont AOT

Mass Highway Department

Rhode Island DOT

New York State DOT

Connecticut DOT

Delaware DOT

Hudson Asphalt

Sun Company

NECEPT

CAP Lab

Appendix IV  
Round Robin 99 Report

New England Transportation Consortium Project 96-1

Superpave Implementation

Report on  
PG Graded Binder Round Robin 99

Prepared by:

James Mahoney and Jack Stephens

From

The Connecticut Advanced Pavement Laboratory

June 7, 1999



## NETC Round Robin 99

The Connecticut Advanced Pavement Laboratory as part of a New England Transportation Consortium research project conducted a Round Robin of Asphalt Binder Samples. The samples were distributed to the six New England State Departments of Transportation, the New York State Department of Transportation and to the asphalt binder suppliers active in the New England area.

The Round Robin consisted of four binder samples in 1-quart paint cans. Detailed handling instructions were submitted with the samples. The instructions were intended to reduce the variability of sample handling. The participants were also asked to document, on the enclosed data sheets, the time each step was completed. A floppy disk was also included with the samples on which the participants would enter the test and time data and return to the CAP Lab. The instructions and sample data sheets are included in Appendix A.

The four asphalt samples used during this Round Robin were identical to each other. This was done to evaluate the reproducibility of the equipment and the operator.

### Dynamic Shear Rheometer (DSR)

DSR testing on the samples was performed for this Round Robin on both original and RTFO material. This testing was performed using the 25 millimeter parallel plate configuration for the DSR. Testing was not performed on the 8 millimeter plates due to the uncertain future of this configuration. The binder used for the Round Robin was a borderline PG 64-28. The testing was concentrated on this grade since it is the predominate grade used throughout New England.

The Round Robin participants were asked to mount two DSR specimens from the same sample. The first specimen was to be tested at both 58°C and 64°C. The second sample was to be tested at 64°C only. There were several purposes for this sequence of testing. The first reason for this sequence of testing was to investigate the effects of the bulge disappearing during the testing process. The bulge is created by squeezing the specimen by 50 microns after the sample is trimmed. It has been observed that during the testing process that the bulge was tending to creep down the plates. The extended time that the sample tested at 58°C and 64°C was held at the warmer temperature was assumed to be enough to cause the bulge to creep. The second purpose for this testing was to determine if there were differences caused by determining the zero gap space between the 25 mm plates at different temperatures.

The raw data from the Round Robin is shown in Appendix B. For data analysis, values that deviated from the average more than three standard deviations were dropped from the analysis. AMRL utilizes three standard deviations for

eliminating outliers. The data after removing the outliers can be seen in Appendix C. None of the 142 reported  $G^*$  or phase angle values for the original binder were dropped from the analysis. DSR values of  $G^*$  for the RTFO material have a larger spread of values. Only 3 of the 143 reported RTFO  $G^*$  values exceeded the three standard deviation allowance. For the RTFO DSR phase angle data, the three phase angle values corresponding to the dropped  $G^*$  values also exceeded three standard deviations. This increase in  $G^*$  values can be seen in figure 1.

The increase in the spread of the data for the DSR values of  $G^*$  for the RTFO material would be best accounted for by the increase in the handling of the material. The material tested as DSR-RTFO material had undergone several extra handling steps as well as an aging process. This allowed for the increase in the variability of the test data. Another measure of the spread in test data is the percentage of the average represented by one standard deviation (%1S). The %1S  $G^*$  value for the original binder using all of the data reported can be seen in Table 1. The values for the %1S  $G^*$  values after the outliers have been removed can also be seen in Table 1. Both sets of %1S  $G^*$  values for the RTFO material can be seen in Table 2. The RTFO material has a %1S  $G^*$  value about 1.6% higher than the original material, after the outliers have been removed from the computations. The RTFO material has a %1S  $G^*$  value about 4.95% higher than the original material when all of the test results are used.

Table 1 – DSR Original Material %1S  $G^*$  Values

Temperature Tested	%1S including Outliers	%1S excluding Outliers
58° C	4.90%	No Outliers Found
64° C after 58° C testing	5.71%	No Outliers Found
64° C	5.83%	No Outliers Found

Table 2 – DSR RTFO Material %1S  $G^*$  Values

Temperature Tested	%1S including Outliers	%1S excluding Outliers
58° C	10.03%	7.17%
64° C after 58° C testing	10.36%	8.03%
64° C	10.85%	8.48%

The %1S values for  $G^*$  obtained for this Round Robin are smaller than those gathered from the latest published data from AMRL's proficiency samples. The majority of the effort for the data analysis was placed upon the complex modulus  $G^*$  due to its very large impact upon the test results. While the phase angle is important, the amount of variation in test results caused by the phase angle is limited. Table 3 shows the %1S DSR phase angle data for the original binder samples.

Figure 1 - NETC Round Robin 99  
%1S Values for G\*

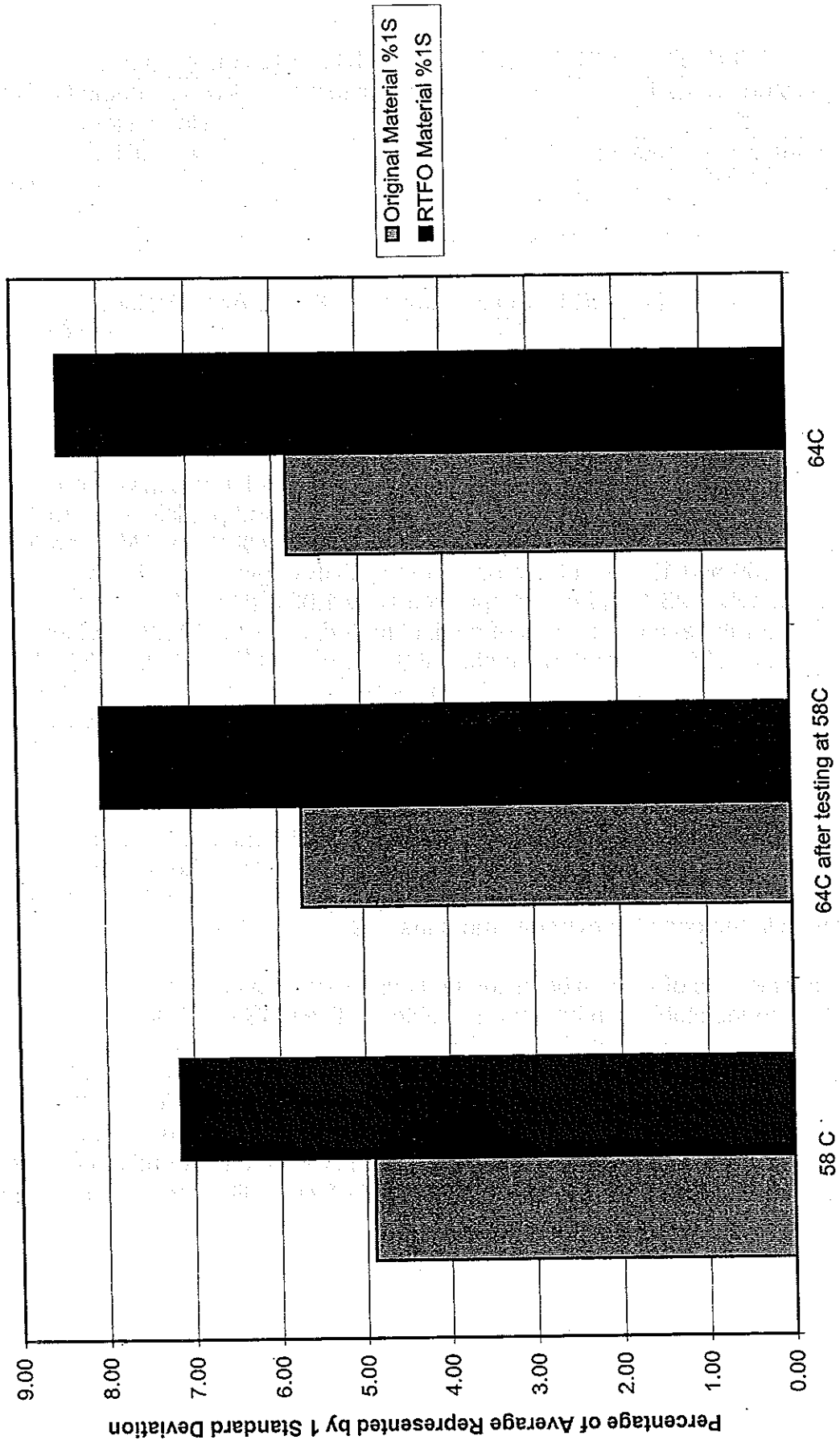


Table 3 – DSR Original Binder %1S Phase Angle Values

Temperature Tested	%1S including Outliers	%1S excluding Outliers
58° C	0.25%	No Outliers Found
64° C after 58° C testing	0.54%	No Outliers Found
64° C	0.75%	No Outliers Found

Table 4 shows the %1S DSR phase angle data for the RTFO material.

Table 4 – DSR RTFO Material %1S Phase Angle Values

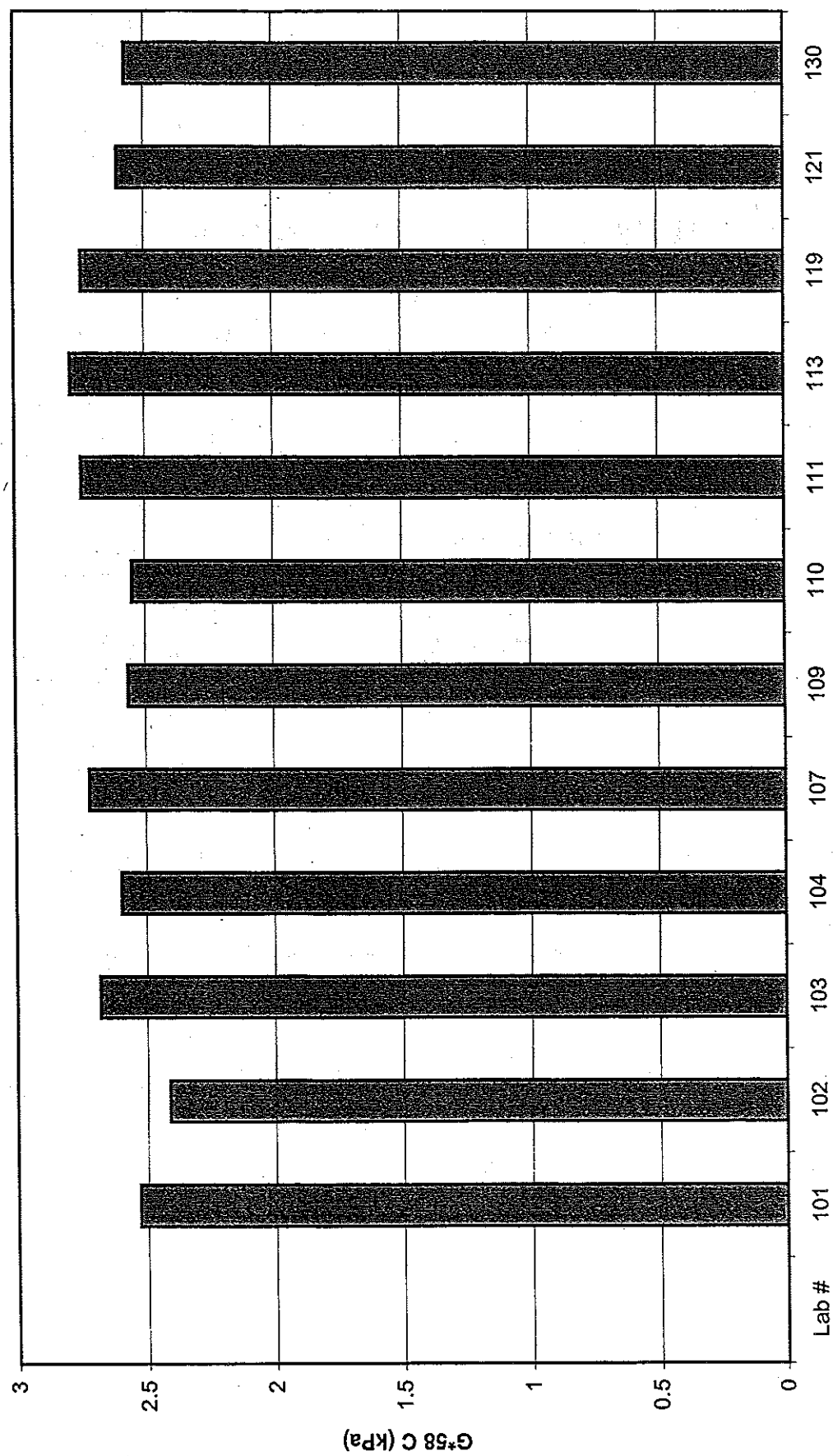
Temperature Tested	%1S including Outliers	%1S excluding Outliers
58° C	0.82%	0.47%
64° C after 58° C testing	0.74%	0.47%
64° C	0.72%	0.46%

The %1S phase angle data collected for this Round Robin is considerably lower than that collected by AMRL during their latest published proficiency sample results. The multilaboratory precision as measured by %1S for AMRL proficiency samples 169 and 170, for phase angle of the DSR original material was approximately 1.25% and for  $G^*$  approximately 1.05%. (Ref. #2) The average  $G^*$  at 64° C for the second sample of original material was 0.012 kPa higher than the average  $G^*$  for the material tested at 64° C after 58° C. 8 of the labs had higher averages for the specimen tested at 64° C only. The average values for  $G^*$  at 64° C of the RTFO material were virtually the same for both specimens. The extra holding time in the silicone molds did not appear to have any effect on the  $G^*$  values of the RTFO material.

Figures 2-9 show the  $G^*$  values and phase angles for the original and RTFO material. The phase angle figures demonstrate how close the phase angle results are to each other. Thus, the majority of the variability in the test results appears in the complex dynamic modulus –  $G^*$ .

The coefficients of variability for the DSR and BBR testing performed by each lab for this Round Robin can be seen in Table 5. The DSR coefficients were computed by determining the coefficient of variability for each test temperature and then averaging the three coefficients. The BBR coefficients were computed by comparing all of the m-value data for each lab with itself. The coefficient for the Stiffness was determined in a manner similar to the m-value. The coefficient of variability of  $G^*$  increased for the RTFO material for 8 labs and decreased for only 3 labs. The magnitude of the coefficient of variability increases far outweigh the decreases which were observed.

Figure 2 - NETC Round Robin 99  
Average  $G^*$  for Original Binder at 58 C



**Figure 3 - NETC Round Robin 99**  
**Average G\* for Original Binder Tested at 64C**

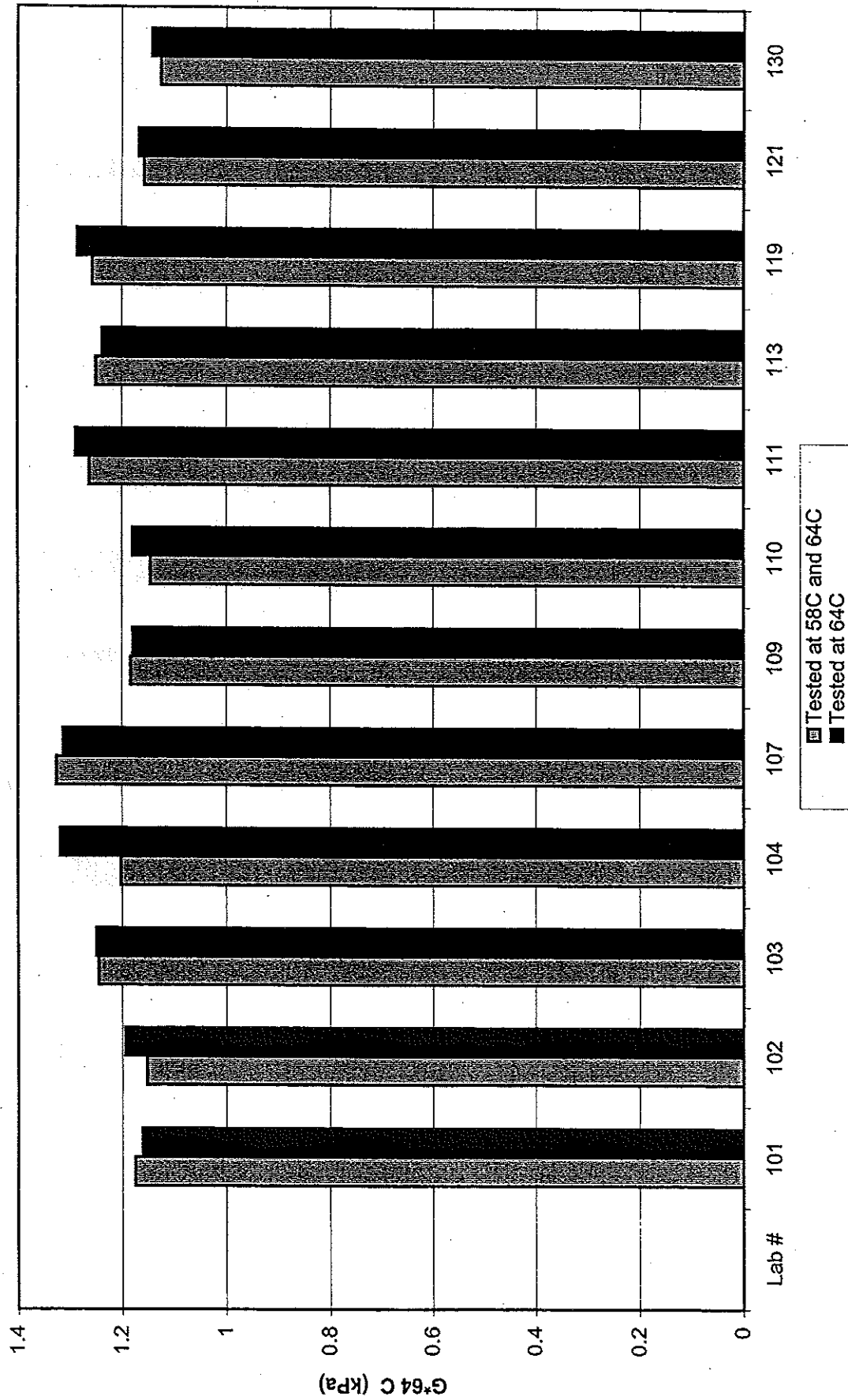


Figure 4 - NETC Round Robin 99  
Average Phase Angle for Original Binder Tested at 58C

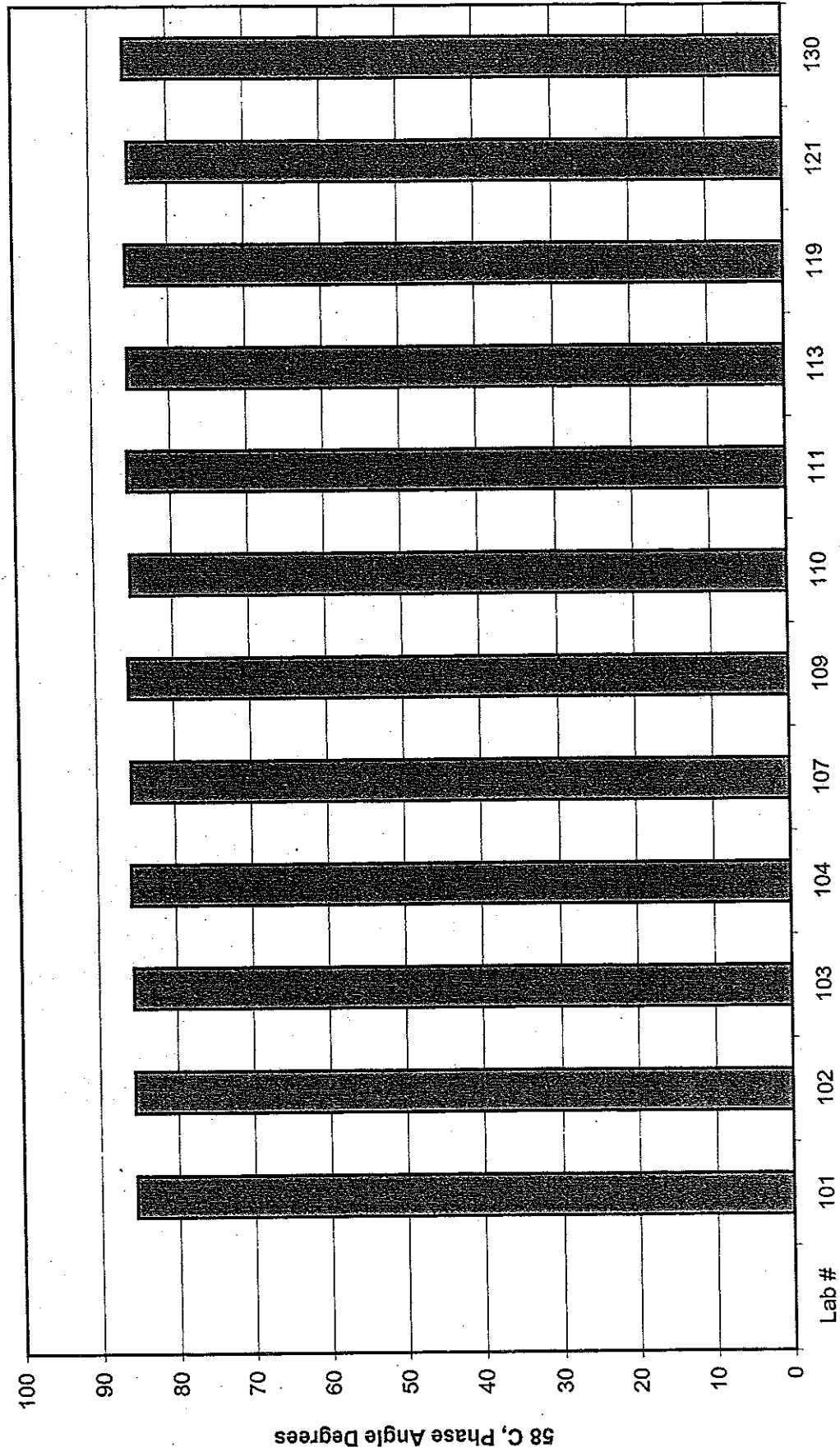


Figure 5 - NETC Round Robin 99  
Average Phase Angles for Original Binder Tested at 64C

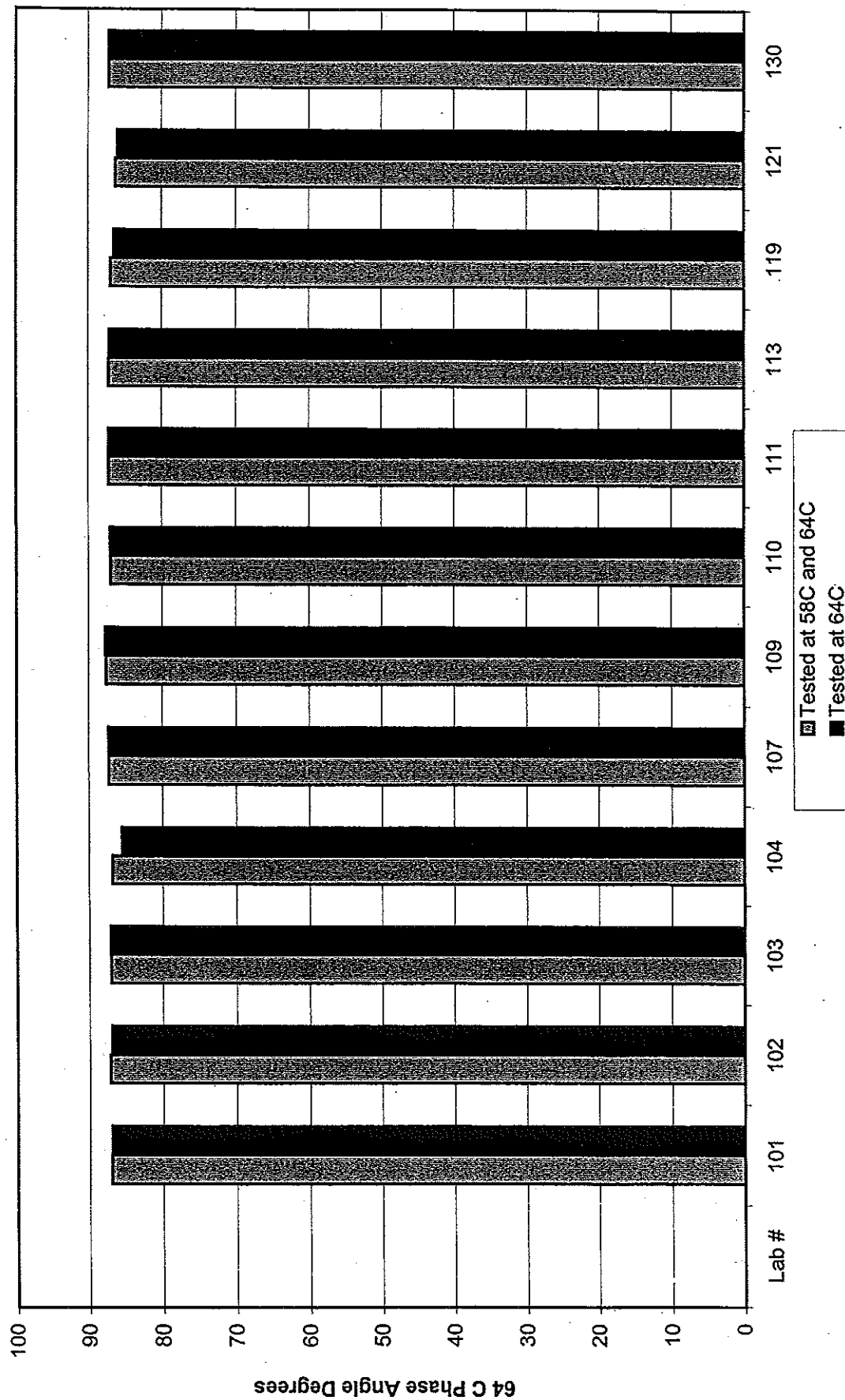
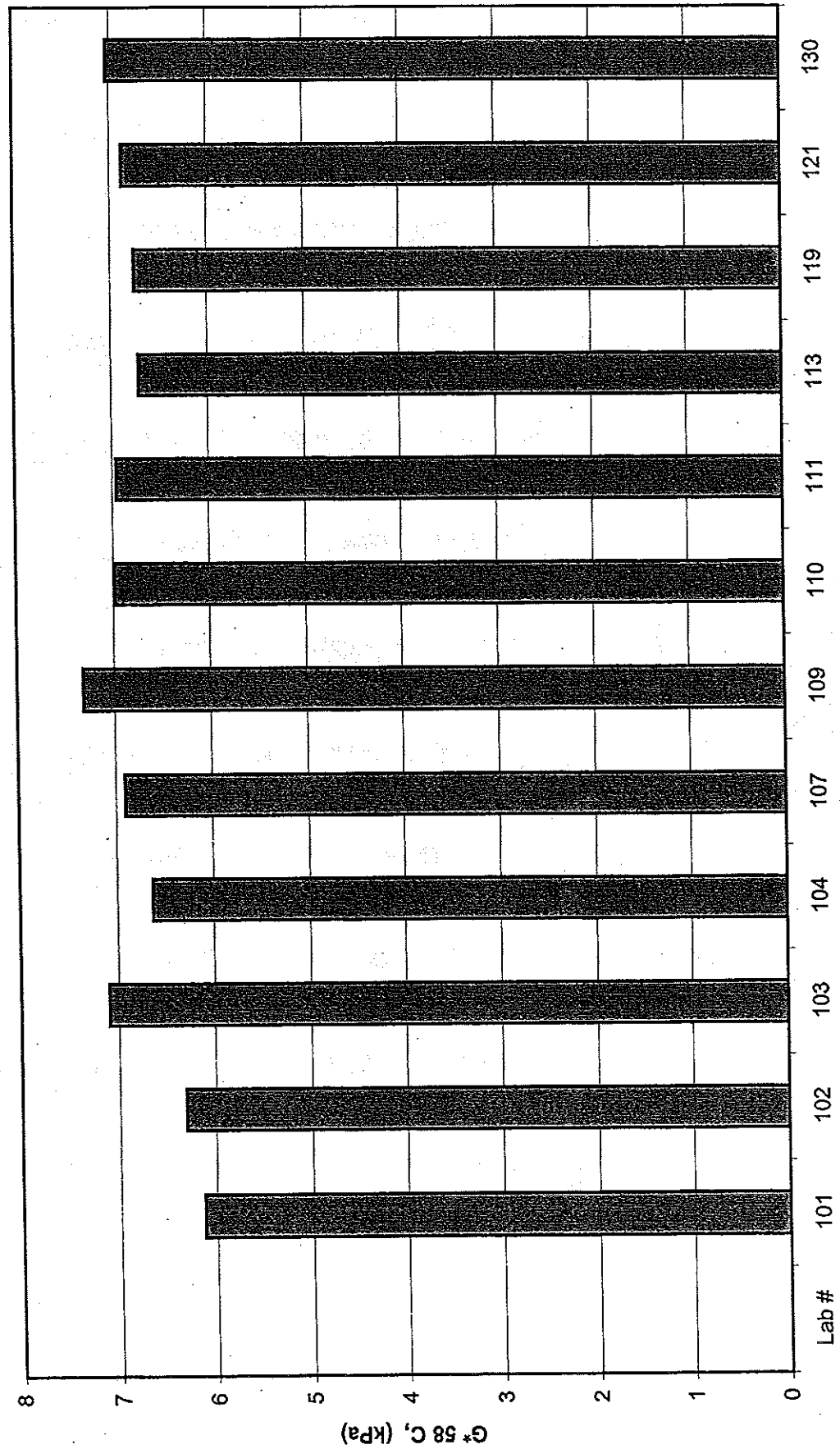
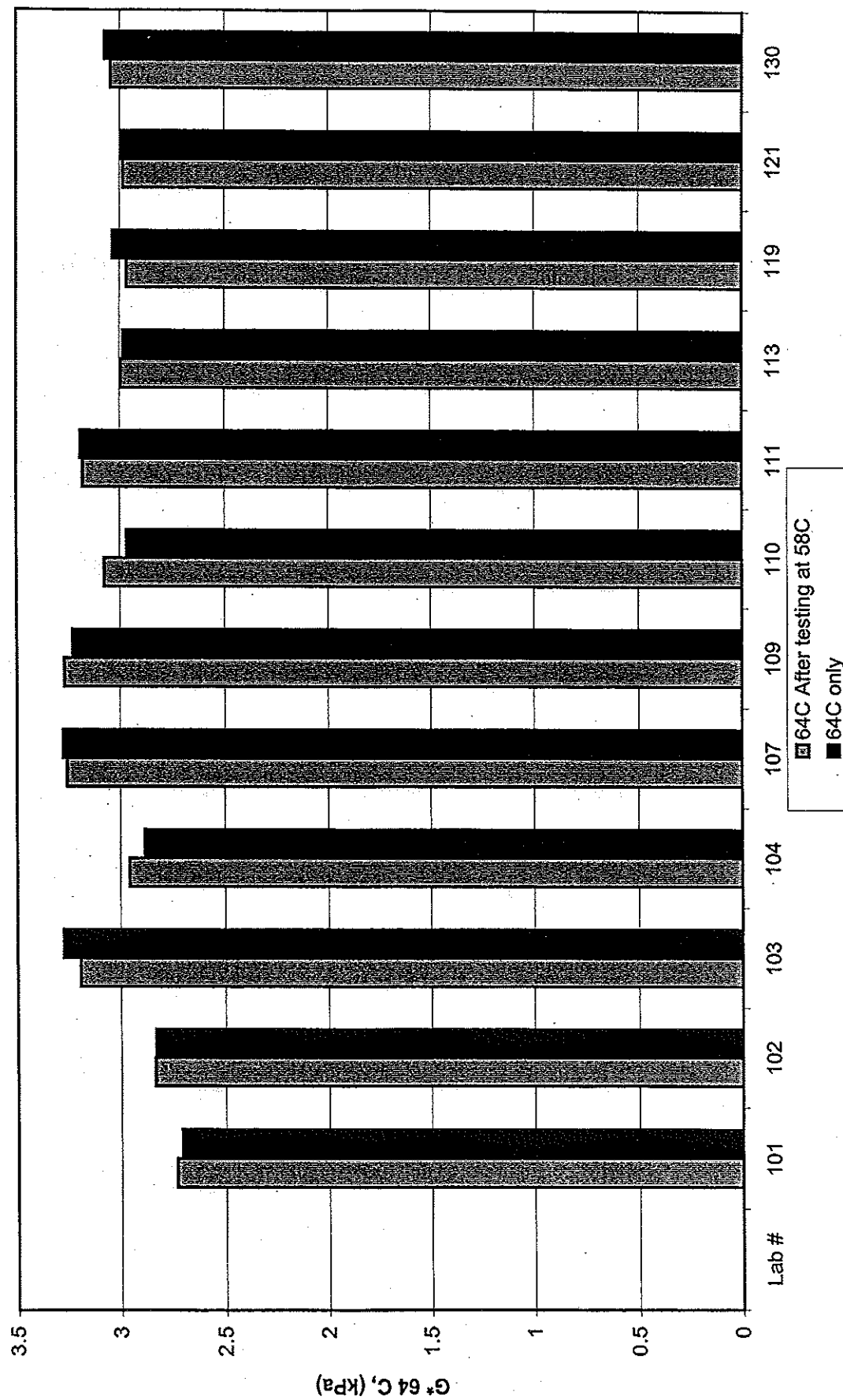




Figure 6 - NETC Round Robin 99  
Average RTFO G\* Values for 58C



**Figure 7 - NETC Round Robin 99**  
**Average G\* Values for RTFO Material at 64C**



**Figure 8 - NETC Round Robin 99**  
**Average Phase Angle Values for RTFO Material at 58C**

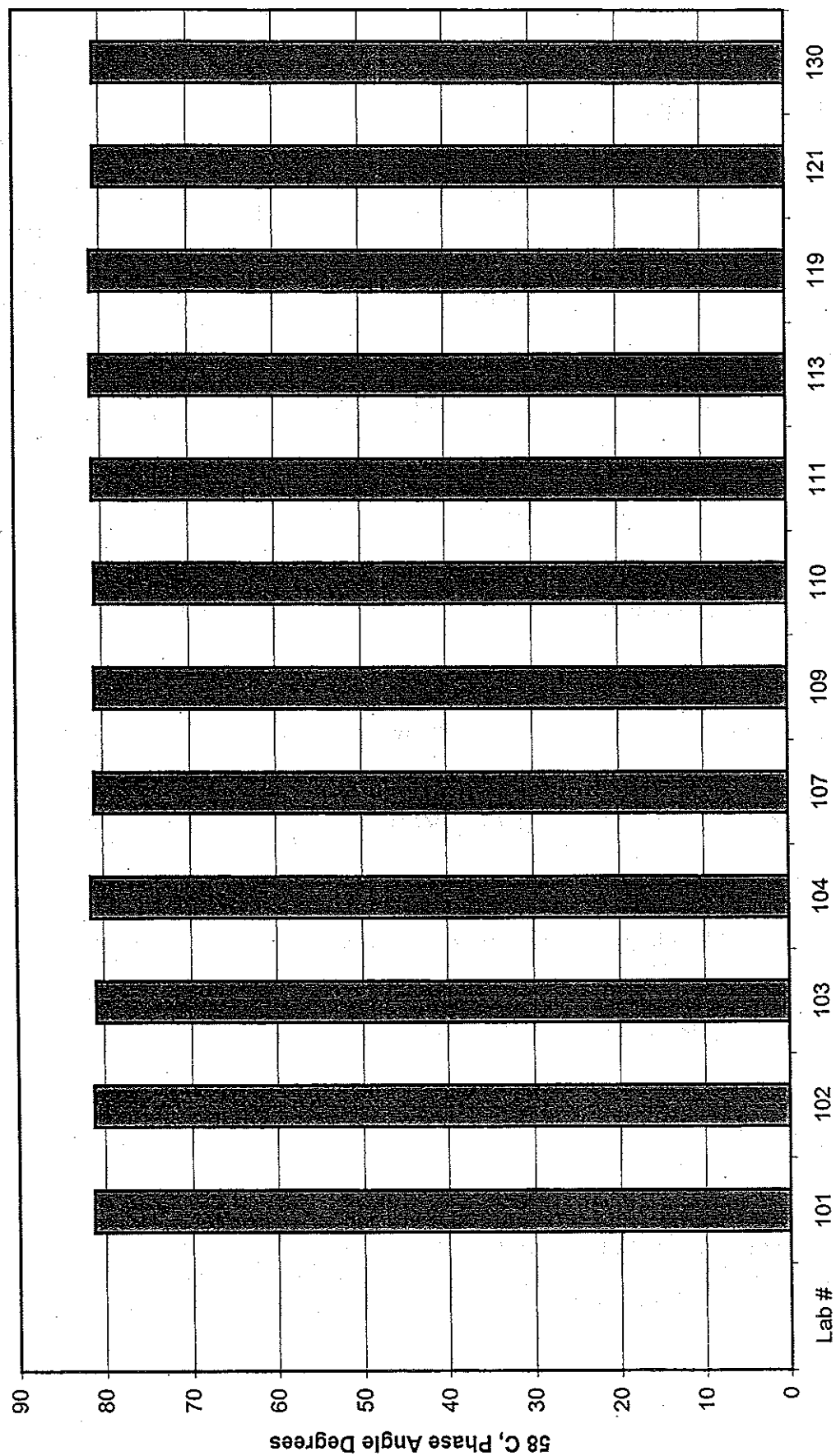


Table 5 – Average Coefficients of Variability for DSR and BBR Testing

Lab Number	Original Material G*, %	Original Material, Phase Angle, %	RTFO Material G*, %	RTFO Material Phase Angle, %	BBR m-value, %	BBR Stiffness, %
101	1.90	0.07	3.39	0.07	1.21	2.58
102	2.42	0.15	2.10	0.26	0.92	1.10
103	4.73	0.09	8.44	0.28	5.51	3.45
104	5.61	1.10	10.99	0.99	17.60	9.95
107	1.50	0.08	9.61	0.66	1.33	2.74
109	3.42	0.12	30.00	2.11	1.33	2.74
110	4.22	0.03	3.38	0.09	3.42	6.17
111	1.71	0.05	4.44	0.23	2.00	2.04
113	1.35	0.07	3.05	0.15	1.43	3.26
119	1.82	0.54	3.27	0.16	1.25	3.04
121	4.16	0.25	9.36	0.38	1.31	3.55
130	2.29	0.04	1.85	0.14		

Figures 10-13 show the coefficients of variability for the various stages of testing.

#### Bending Beam Rheometer (BBR)

The testing performed using the BBR was performed at one temperature  $-18^{\circ}\text{C}$ . The testing was performed at this temperature because it correlates with PG Grades of  $-28^{\circ}\text{C}$ . The material tested was subjected to aging in the RTFO and the Pressure Aging Vessel (PAV). Vacuum degassing was not used for this round robin. The BBRs had not been upgraded with the new supports at the time this Round Robin was performed.

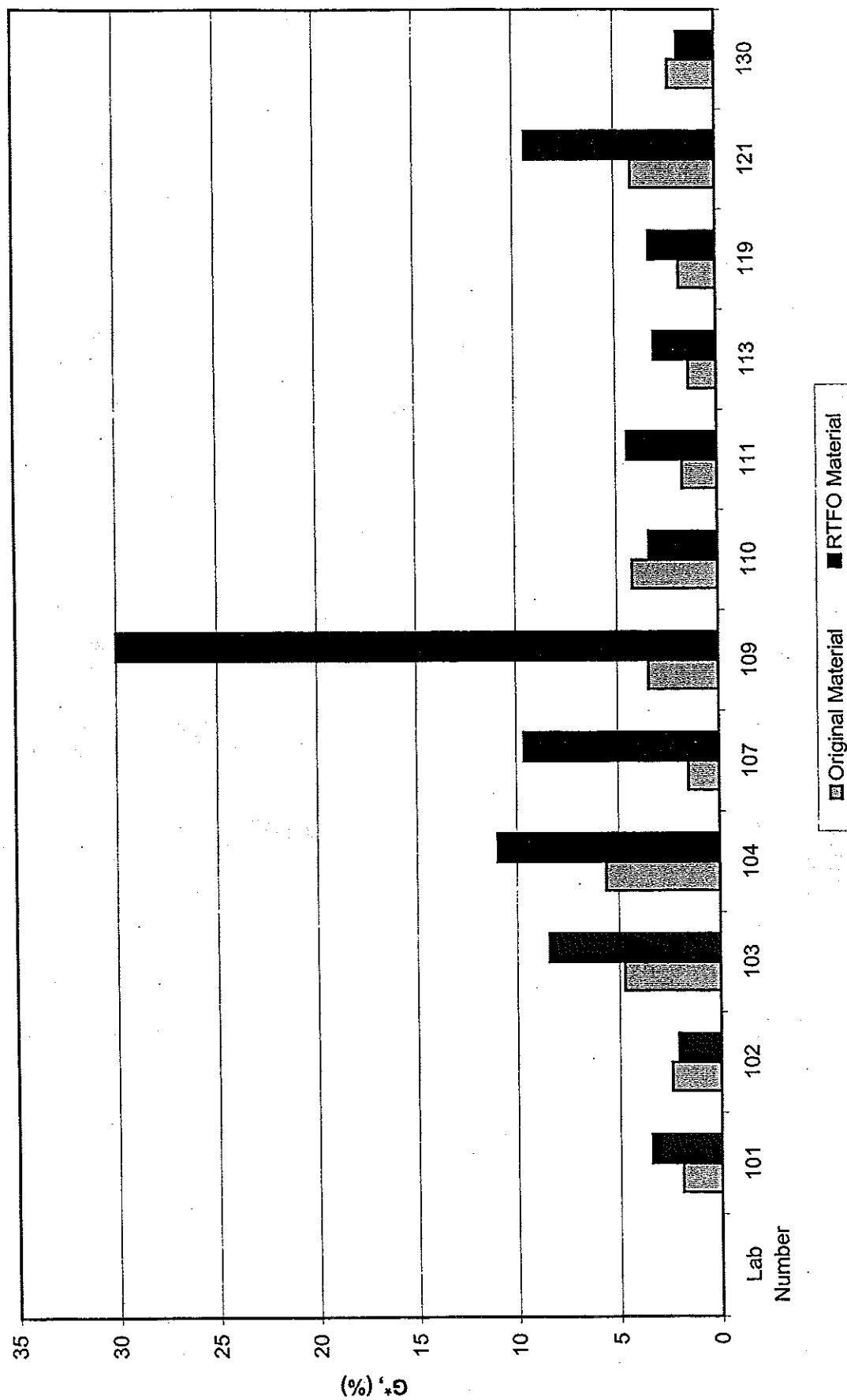
The Round Robin participants were asked to use only the data from the first two beams tested. Data from any extra beams tested was not to be included as long as data was collected from the first two beams. The participants also were to use the aluminum beam molds with the appropriate plastic strips.

BBR data from lab 109 was excluded in all of the data analysis. Upon investigation it was determined that their BBR was not functioning properly.

Table 6 shows the results from the %1S BBR data. Figures 14 and 15 show the m-value averages and the stiffness averages for each lab. A three standard deviation cutoff was used for eliminating outliers. All of the outliers for the BBR data occurred from the same lab. Two of the eliminated data points were m-values and the third point eliminated was a stiffness value.

The %1S value obtained for the m-value for this Round Robin after dropping 2 outliers was one-third lower than the published values from AMRL for their latest

Figure 10 - NETC Round Robin 99  
Average Coefficient of Variation for G\* for Original and RTFO Material



**Figure 11 - NETC Round Robin 99**  
**Average Coefficient of Variation for Phase Angle Original and RTFO Material**

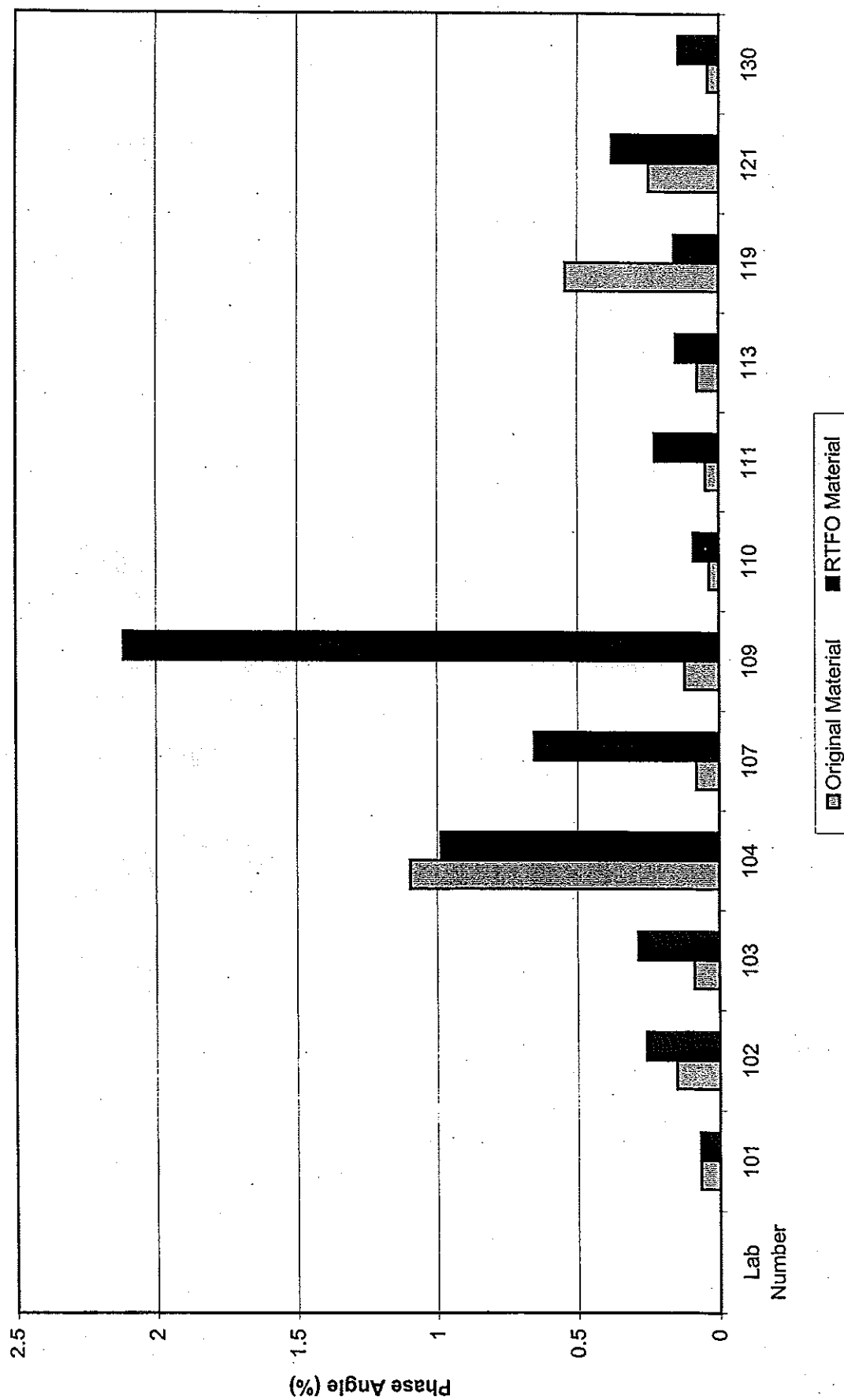
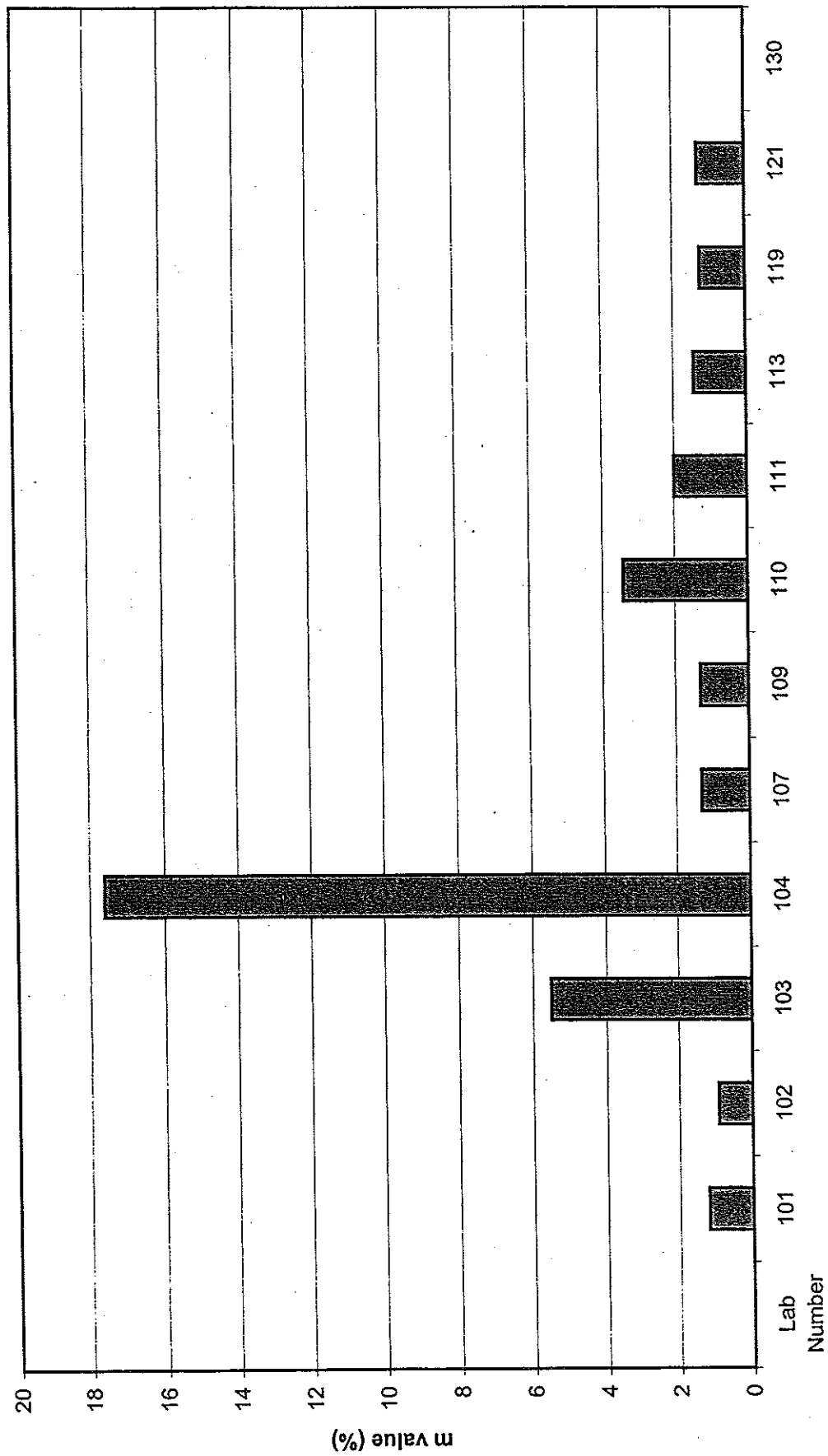


Figure 12 - NETC Round Robin 99  
Coefficient of Variation for BBR m-value



**Figure 13 - NETC Round Robin 99  
Coefficient of Variation for BBR Stiffness**

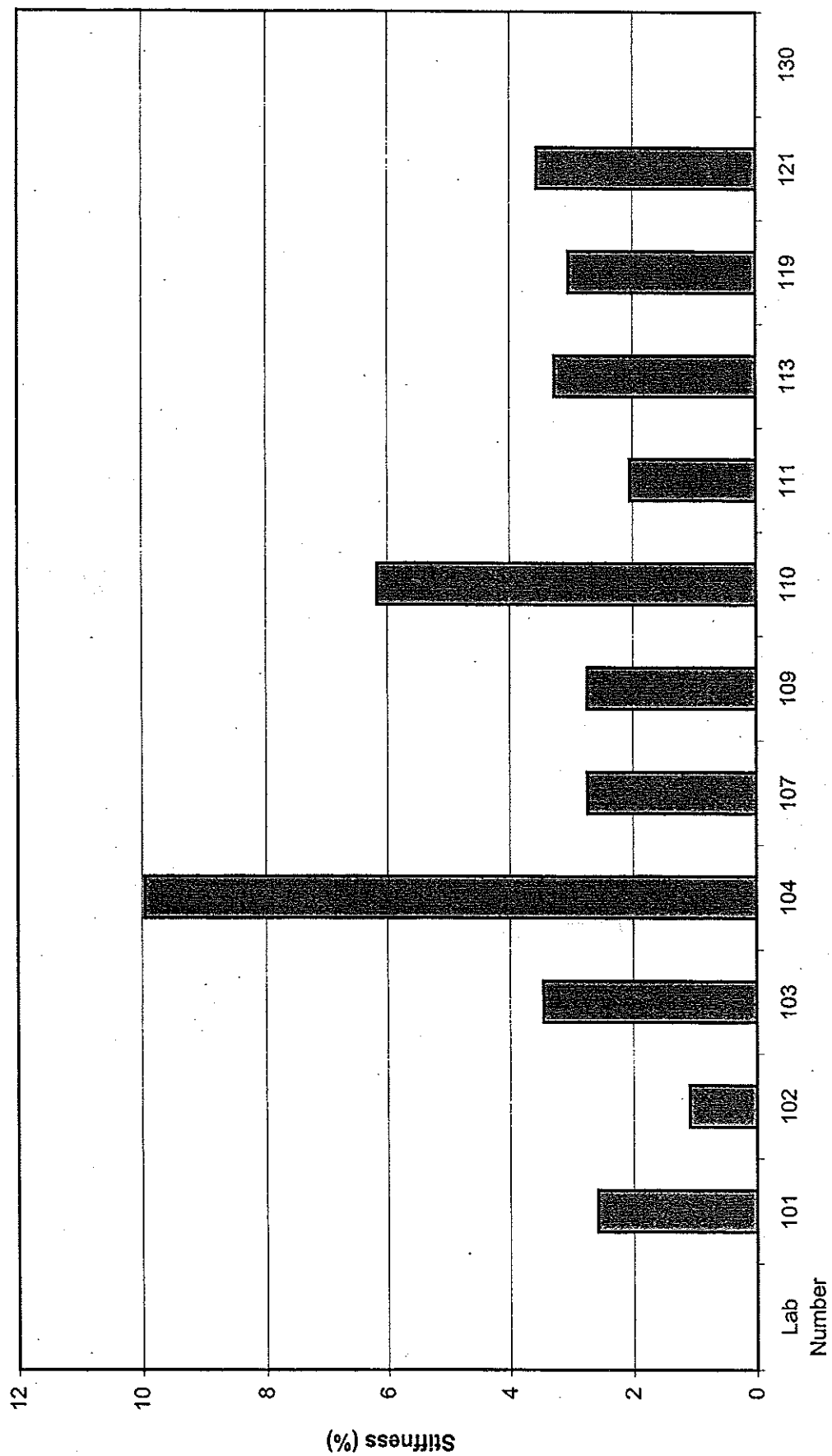




Figure 9 - NETC Round Robin 99  
Average Phase Angle Values for RTFO Material at 64C

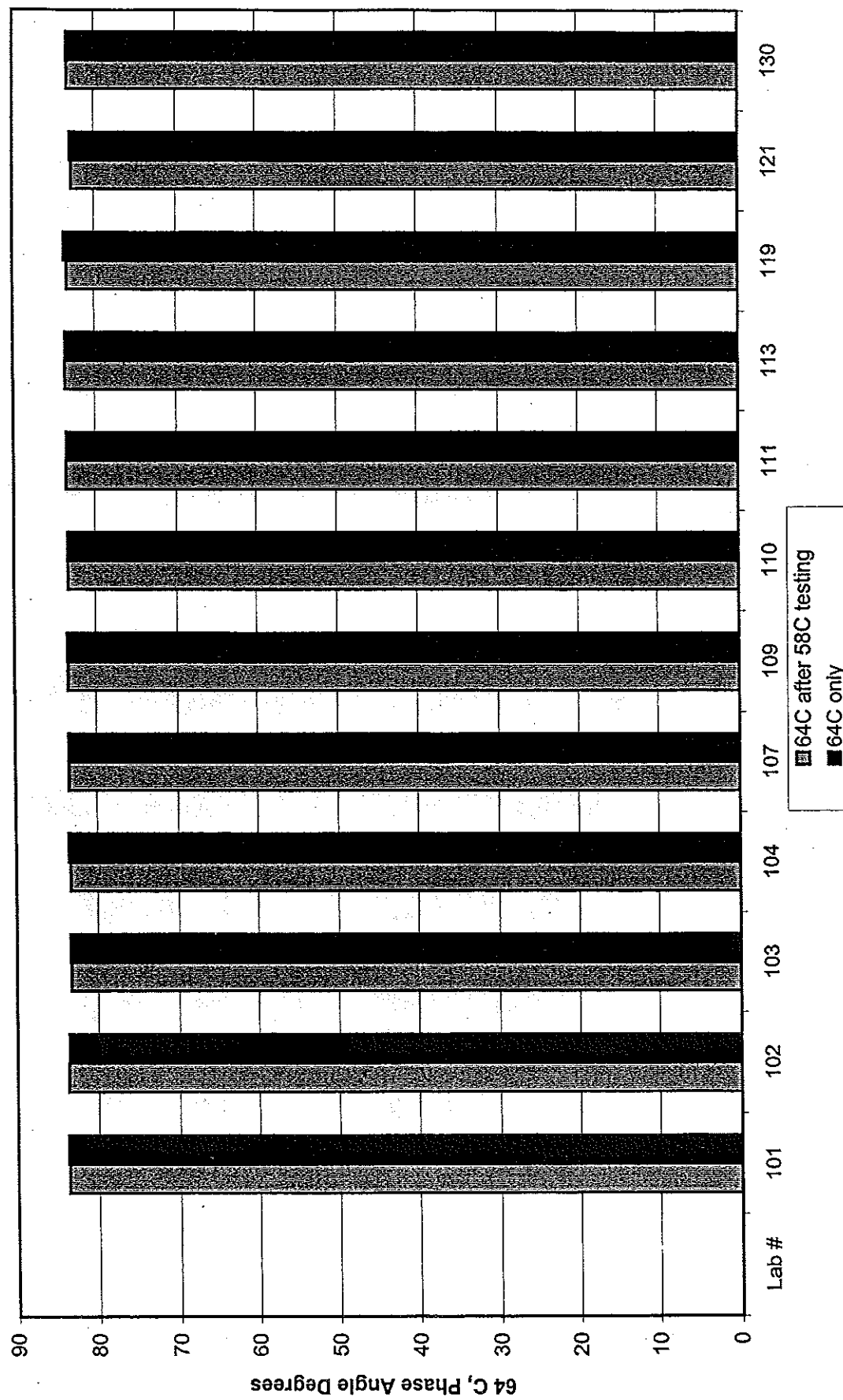


Figure 14 - NETC Round Robin 99  
Average m-value for BBR Testing at -18C

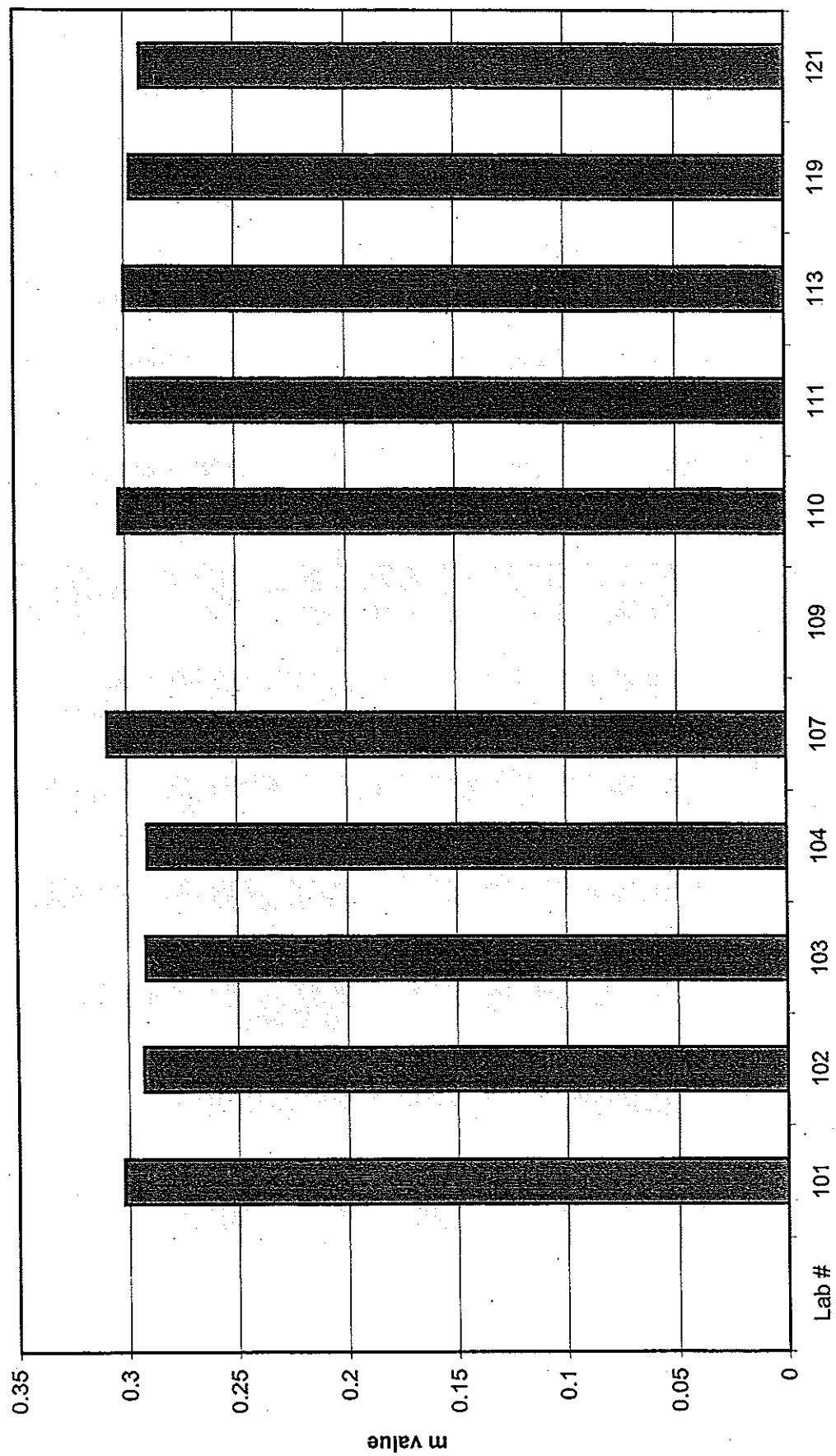
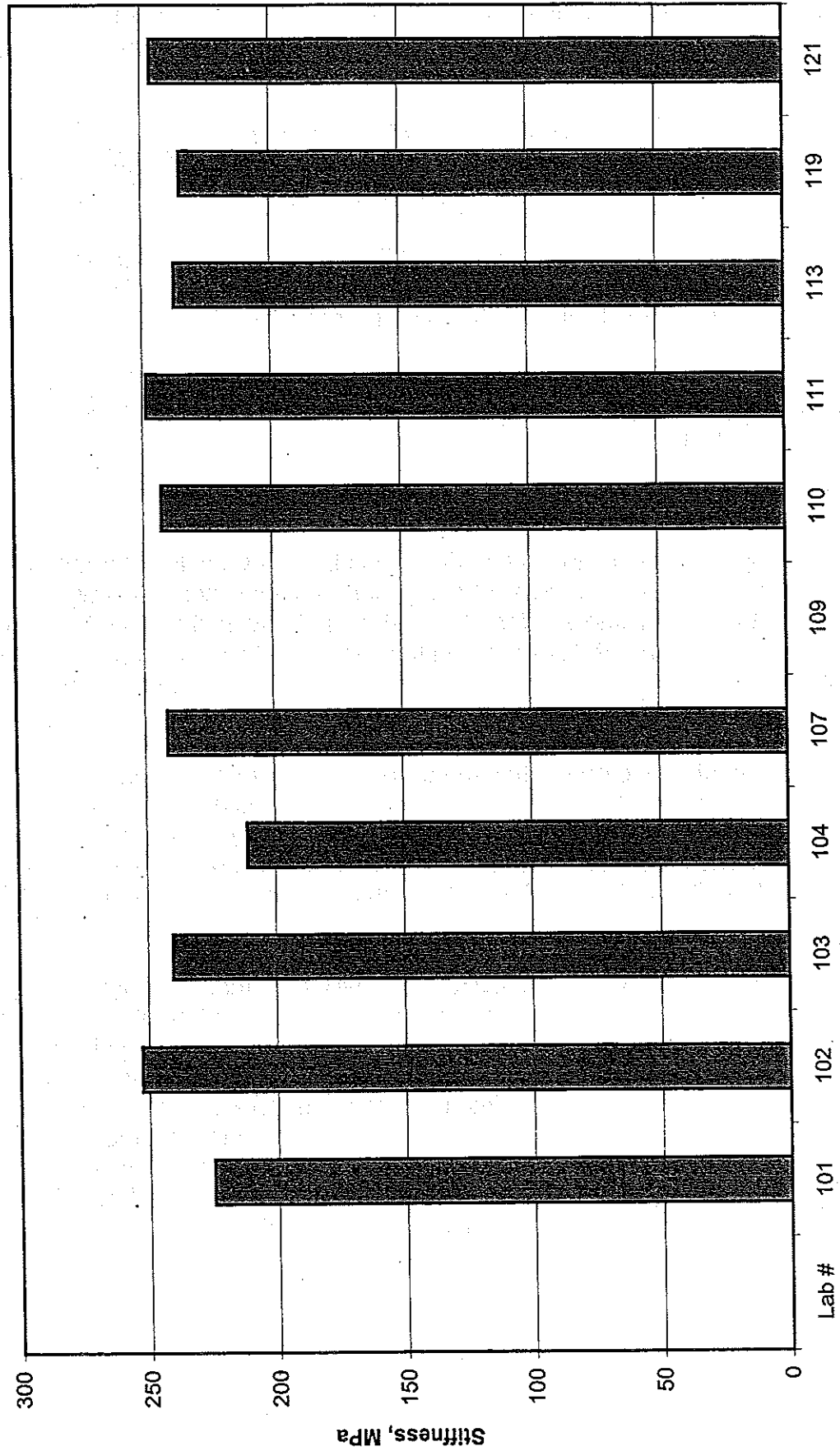


Figure 15 - NETC Round Robin 99  
Average Stiffness for BBR Testing at -18C



proficiency samples. Table 6 shows the %1S for the m-value for this Round Robin. The average published value of the %1S for AMRL samples number 169 and 170 is 4.15%.

Table 6 – BBR %1S for m-value

Temperature Tested	%1S including Outliers	%1S excluding Outliers
-18° C	6.38%	2.80%

The %1S value obtained for the BBR stiffness is in Table 7. Again these values are lower than the published AMRL proficiency sample values for AMRL samples 169 and 170. The published %1S value for samples 169 and 170 is 10.85%.

Table 7 – BBR %1S for Stiffness

Temperature Tested	%1S including Outliers	%1S excluding Outliers
-18° C	6.10%	5.65%

### Conclusions

There appeared to be virtually no difference in DSR  $G^*$  values for material tested over two temperatures versus a freshly mounted specimen. This would tend to indicate that the meltdown of the bulge does not adversely affect the results. Also, with no apparent differences in test results between the specimen tested at both 58C and 64C and the specimen tested only at 64C, the difference in setting the gap, 58C versus 64C does not appear to matter.

The results obtained during this round robin are more consistent than those obtained from the AMRL proficiency samples. This is due in part to the very specific instructions which were provided to the participants. Following such detailed handling instructions would not be practical, but some improvement in sample handling uniformity from lab to lab may decrease the variation between labs.

The variation observed in the BBR results will more than likely improve with the introduction of the vacuum degassing oven. The procedure used for this will standardize the sample handling. As the current AASHTO specification is currently written, there is a lot of room for interpretation as to how to determine the proper amount of heating time to remove entrapped air in the Pressure Aging Vessel (PAV) residue. The procedure for vacuum degassing the PAV residue is very specific as to heating times and length of time the material should be exposed to the vacuum. This should reduce variability caused by different techniques currently in use to remove entrapped air.

### References

1. AASHTO, *June 1998 – AASHTO Provisional Standards*, 1998.
2. AASHTO Materials Reference Laboratory, Performance-Graded Binder Samples, [www.nist.gov/aashto/amrl/services/sect3b.html](http://www.nist.gov/aashto/amrl/services/sect3b.html), June 1, 1999.
3. Anderson, David and Marasteanu, Mihai, *Manual of Practice*, NECEPT, 1999.

## Appendix A

### Round Robin 99 Instructions and Sample Data Sheets

## NETC Round Robin 1999

There are four 1-quart cans containing asphalt samples. They are labeled 99A, 99B, 99C and 99D. These sample should be subjected to the full battery of PG binder tests with the exception of the flash point. Below are the detailed instructions regarding the handling procedures for these samples. Please adhere to these instructions as closely as possible and **note deviations** from the instructions. We are trying to determine the effect of handling by different labs on the asphalt samples, so adhering to the instructions as closely as possible is critical. Please note: there are better handling procedures available, but for consistency please follow the instructions as given.

**For each sample** the following tests will be performed:

- 1 Rotational Viscosity (Original Binder 135 C)
- 1 DSR Original (58 C and 64 C)
- 1 DSR Original (64 C only)
- 1 Rotational Viscosity (RTFO Residue 165 C)
- 1 DSR RTFO (58 C and 64 C)
- 1 DSR RTFO (64 C only)
- 1 BBR Test (-18 C) 2 Beams per test on PAV material

The data should be entered into the spreadsheets provided. Please also record the time data in the provided time data sheets. The time data may allow us to discover why some data is more scattered than other data.

Before beginning any testing, please verify the temperature calibrations for both the DSR and BBR. If during the testing of the 4 samples you have to change the temperature calibrations, please note when you did it on the spreadsheet provided.

Instructions for testing:

Please test only one sample at a time.

All equipment should be brought to temperature far enough in advance so as not to effect the timing of the testing.

### **Original Binder Material**

- Loosen lid on quart can but do not remove.
- Place can with loose lid in 135 C oven.
- Allow can to heat for 1 hour 45 minutes
- Stir sample
- Pour a minimum of 3 DSR original samples, 1 Rotational Viscosity Sample and 8 RTFO bottles (35 g +/- 0.5 g).

- Cover DSR samples to prevent dust or other contaminants from getting into the samples.
- Begin testing DSR samples as soon as possible.
  - Before loading each DSR sample please warm the DSR to the loading temperature and zero the gap with warm plates – not room temperature plates.
  - Use the standard DSR testing parameters for original binder such as 12% strain, 10 rad/sec oscillation frequency, 1 mm thick sample and creating the bulge by squeezing the sample 0.050 mm.
  - Use the equilibrium time you normally use for your equipment
  - The trimming tool should be heated prior to trimming sample.
- Load and trim one sample at **58 C** and test it at both **58 C and 64 C**.
- Load and trim second sample at **64 C** and test it at **64 C only**.
- Please complete DSR original testing within 2 hours of pouring the samples.
- Place the Rotational Viscosity sample into the conditioning chamber as soon as possible after pouring and begin testing sample at 135 C.
- Allow the RTFO bottles to stand for 30 minutes in desiccator after pouring and then place bottles into RTFO oven and run for 85 minutes.

#### **RTFO Material**

- After running the RTFO for 85 minutes remove bottles and combine all of the RTFO residue into a single container. **Do not scrape bottles!!**
- While material in the container is still warm, place container into 163 C oven for 20 minutes.
- Remove container from oven and stir.
- Pour a minimum of 3 DSR RTFO samples, 1 Rotational Viscosity and as many PAV pans as possible.
- Begin the DSR RTFO testing as soon as possible.
  - Before loading each sample please zero the gap with plates warmed to the loading temperature.
  - Use standard DSR testing parameters for RTFO material such as 10% strain, 10 rad/sec oscillation frequency, 1 mm thick sample and creating the bulge by squeezing the sample 0.050 mm.
  - Use the equilibrium time you normally use for your equipment
  - Trimming tool should be heated prior to trimming sample.
- Load and trim one of the DSR samples at **58 C** and test it at **both 58 C and 64 C**.
- Load and trim the second DSR sample at **64 C** and test it at **64 C only**.
- Please complete the DSR RTFO within 2 hours of pouring samples.
- Place the rotational viscosity sample in the sample conditioning chamber as soon after pouring as possible.
- Begin testing material at 165 C.
- Cover the PAV pans (1 gallon paint can lids work nicely) and allow the PAV pans to cool on the countertop for a minimum of 30 minutes.



### PAV Material

- PAV should already be preheated and the PAV pan rack should be preheated in the PAV
- Place samples into PAV pan rack and place into the PAV.
- Start PAV.
- When PAV is finished allow it to depressurize in 9+/-1 minutes.
- Place PAV pans into **163 C oven for 15 minutes.**
- Combine contents of PAV pans into a **single container.**
- Place covered container back into **163 C oven for 1 hour** – stirring every twenty minutes.
- Remove from oven and stir once more.
- Pour a minimum of 2 asphalt beam specimens.
- Allow asphalt beams to stand on countertop for 45-60 minutes.
- Trim beams.
- Place beams into freezer no colder than -8 C (if possible, if not please note on spread sheet) or ice bath with 10 minutes time separation between beams.
- Allow beams to stay in freezer or ice bath for 7 minutes.
- At this time, the BBR bath should be at -18 C.
- De-mold and place beams into BBR bath and allow each of them to soak for exactly 60 minutes.
- Set standard test loads on the non-compliant beam. No load adjustment should be made once an asphalt beam is placed in the supports.
  - If your BBR has trouble maintaining the test loads for the duration of the testing – please forward a copy of the printout for each beam with your data.
- Test beams. If more than three beams are poured only use data from the first two beams tested – unless there is a malfunction of the equipment. Do not choose the data from the two beams with the closest values.

Repeat these instructions for each of the four samples. If you have any questions please contact Jim Mahoney at 860-486-5956.

# NETC Asphalt Binder Round Robin 1999

Appendix IV

Lab Number

Sample ID

	Original Binder	RTFO Material	PAV Material	Deviations from testing instructions
G*, kPa 58C				
Phase Angle 58C				
Strain 58C				
G*, kPa 64C				
Phase Angle 64C				
Strain 64C				
G*, kPa 64C				
Phase Angle 64C				
Strain 64C				
Rotat Vis, Pa-S 135C				
Rotat Vis, Pa-S 165C				
Beam 1 -18C @ 60 S				
Stiffness, MPa				
m-value				
Beam 2 -18C @ 60 S				
Stiffness, MPa				
m-value				

Note: For deviations from testing instructions - you type as much as you need to type - It may exceed the cell size but the information will be stored on the disk.

## Appendix B

### The Raw Data from Round Robin 99

NETC Round Robin 99 - Raw Data  
G\* Values for DSR - Original Testing

Lab #	Sample #	Single specimen mounted			Lab Averages Including Outliers			Average Coefficient of Variability for each lab, %
		G* 58 C, kPa	G* 64 C, kPa	New specimen G* 64 C, kPa	Single specimen mounted G* 58 C, kPa	New specimen G* 64 C, kPa	G* 64 C, kPa	
101	99A	2.477	1.157	1.177	2.531	1.175	1.161	1.90
101	99B	2.517	1.169	1.188				
101	99C	2.581	1.196	1.120				
101	99D	2.547	1.178	1.160				
102	99A	2.371	1.125	1.122	2.411	1.152	1.194	2.42
102	99B	2.441	1.165	1.202				
102	99C	2.388	1.156	1.222				
102	99D	2.443	1.163	1.230				
103	99A	2.516	1.157	1.155	2.679	1.246	1.250	4.73
103	99B	2.709	1.262	1.295				
103	99C	2.714	1.273	1.274				
103	99D	2.778	1.290	1.276				
104	99A	2.764	1.246	1.406	2.595	1.202	1.319	5.61
104	99B	2.526	1.161	1.321				
104	99C	2.590	1.272	1.350				
104	99D	2.499	1.130	1.199				
107	99A	2.701	1.315	1.297	2.716	1.326	1.314	1.50
107	99B	2.769	1.357	1.339				
107	99C	2.705	1.322	1.296				
107	99D	2.689	1.311	1.323				
109	99A	2.470	1.116	1.147	2.565	1.184	1.179	3.42
109	99B	2.501	1.188	1.184				
109	99C	2.682	1.188	1.179				
109	99D	2.607	1.242	1.207				
110	99A	2.593	1.187	1.162	2.549	1.145	1.180	4.22
110	99B	2.380	1.048	1.161				
110	99C	2.688	1.160	1.205				
110	99D	2.534	1.185	1.190				
111	99A	2.690	1.258	1.320	2.741	1.263	1.289	1.71
111	99B	2.691	1.247	1.264				
111	99C	2.768	1.267	1.279				
111	99D	2.814	1.279	1.292				
113	99A		1.250	1.228	2.783	1.250	1.238	1.35
113	99B	2.818	1.273	1.266				
113	99C	2.778	1.243	1.232				
113	99D	2.754	1.233	1.226				
119	99A	2.745	1.262	1.305	2.740	1.256	1.285	1.82
119	99B	2.701	1.237	1.272				
119	99C	2.777	1.301	1.302				
119	99D	2.736	1.225	1.263				
121	99A	2.660		1.102	2.600	1.157	1.167	4.16
121	99B	2.580	1.199	1.165				
121	99C	2.444	1.117	1.177				
121	99D	2.716	1.156	1.226				
130	99A	2.485	1.099	1.128	2.571	1.126	1.142	2.29
130	99B	2.513	1.109	1.152				
130	99C	2.692	1.158	1.145				
130	99D	2.594	1.137	1.141				
Average		2.620	1.208	1.227				
Standard Deviation		0.128	0.069	0.072				
Std. Dev % of Avg.		4.90	5.71	5.83				

NETC Round Robin 99 - Raw Data  
Phase Angle Values for DSR - Original Testing

Lab #	Sample #	Lab Averages Including Outliers						Average Coefficient of Variability for each lab, %
		Single specimen mounted		New specimen	Single specimen mounted		New specimen	
		58 C, Phase Angle Degrees	64 C, Phase Angle Degrees	64 C, Phase Angle Degrees	58 C, Phase Angle Degrees	64 C, Phase Angle Degrees	64 C, Phase Angle Degrees	
101	99A	85.54	86.93	86.97	85.51	86.92	86.88	0.07
101	99B	85.50	86.91	86.73				
101	99C	85.53	86.95	86.92				
101	99D	85.46	86.87	86.90				
102	99A	85.40	87.00	87.00	85.63	87.10	86.88	0.15
102	99B	85.70	87.20	86.90				
102	99C	85.70	87.00	86.70				
102	99D	85.70	87.20	86.90				
103	99A	85.75	86.96	86.93	85.60	86.94	87.00	0.09
103	99B	85.55	86.99	87.10				
103	99C	85.58	86.92	87.02				
103	99D	85.50	86.90	86.94				
104	99A		84.70	84.70	85.83	86.73	85.43	1.10
104	99B	85.80	87.40	84.60				
104	99C	85.70	87.40	87.40				
104	99D	86.00	87.40	85.00				
107	99A	85.71	87.24	87.22	85.69	87.20	87.24	0.08
107	99B	85.62	87.13	87.15				
107	99C	85.65	87.15	87.29				
107	99D	85.78	87.28	87.28				
109	99A	85.99	87.62	87.73	85.87	87.55	87.66	0.12
109	99B	85.81	87.52	87.68				
109	99C	85.75	87.35	87.60				
109	99D	85.91	87.70	87.63				
110	99A	85.42	86.93	86.98	85.41	86.92	86.95	0.03
110	99B	85.40	86.97	86.93				
110	99C	85.39	86.85	86.96				
110	99D	85.43	86.91	86.94				
111	99A	85.61	87.13	87.09	85.57	87.14	87.15	0.05
111	99B	85.58	87.13	87.15				
111	99C	85.61	87.17	87.20				
111	99D	85.49	87.12	87.15				
113	99A		87.12	87.06	85.42	87.09	87.07	0.07
113	99B	85.33	87.03	86.98				
113	99C	85.44	87.08	87.08				
113	99D	85.49	87.13	87.14				
119	99A	85.60	86.70	87.00	85.53	86.85	86.40	0.54
119	99B	85.60	86.70	87.00				
119	99C	85.50	87.00	84.70				
119	99D	85.40	87.00	86.90				
121	99A	85.30		85.90	85.10	86.13	85.85	0.25
121	99B	85.30	86.20	85.90				
121	99C	84.90	86.40	85.90				
121	99D	84.90	85.80	85.70				
130	99A	85.53	87.13	87.08	85.57	87.11	87.12	0.04
130	99B	85.58	87.09	87.13				
130	99C	85.53	87.11	87.14				
130	99D	85.65	87.12	87.14				
Average		85.56	86.99	86.80				
Standard Deviation		0.22	0.47	0.75				
Std. Dev % of Avg.		0.25	0.54	0.86				

NETC Round Robin 99 - Raw Data  
G\* Values for DSR - RTFO Testing

Lab #	Sample #	Single specimen mounted			Lab Averages Including Outliers			Average Coefficient of Variability for each lab, %
		G* 58 C, kPa	G* 64 C, kPa	New specimen G* 64 C, kPa	Single specimen mounted G* 58 C, kPa	New specimen G* 64 C, kPa	New specimen G* 64 C, kPa	
101	99A	6.435	2.897	2.714	6.126	2.737	2.713	3.39
101	99B	6.081	2.672	2.671				
101	99C	5.794	2.628	2.769				
101	99D	6.194	2.749	2.698				
102	99A	6.178	2.739	2.759	6.299	2.838	2.835	2.10
102	99B	6.405	2.934	2.868				
102	99C	6.264	2.830	2.877				
102	99D	6.350	2.849	2.837				
103	99A	6.988	3.132	3.059	7.094	3.198	3.278	8.44
103	99B	7.082	3.215	3.143				
103	99C	7.930	3.559	3.624				
103	99D	6.374	2.886	3.287				
104	99A	7.046	3.155	3.168	6.632	2.956	2.883	10.99
104	99B	6.000	2.749	2.367				
104	99C	7.360	3.270	2.900				
104	99D	6.120	2.650	3.097				
107	99A	7.748	3.608	3.634	6.909	3.260	3.279	9.61
107	99B	6.695	3.150	3.084				
107	99C	6.967	3.365	3.499				
107	99D	6.224	2.916	2.899				
109	99A	3.556	1.671	1.623	6.253	2.878	2.860	30.00
109	99B	6.824	3.297	3.353				
109	99C	7.902	3.605	3.572				
109	99D	6.731	2.937	2.892				
110	99A	7.393	3.291	2.973	6.989	3.077	2.967	3.38
110	99B	6.830	2.989	2.906				
110	99C	6.952	3.030	3.010				
110	99D	6.781	2.998	2.977				
111	99A	6.704	3.160	3.081	6.961	3.181	3.193	4.44
111	99B	6.734	3.058	3.058				
111	99C	7.002	3.170	3.223				
111	99D	7.403	3.335	3.409				
113	99A		3.177	3.083	6.721	2.994	2.983	3.05
113	99B	6.655	2.883	2.913				
113	99C	6.886	2.986	3.012				
113	99D	6.621	2.929	2.924				
119	99A	6.624	2.989	2.920	6.758	2.965	3.033	3.27
119	99B	6.968	3.040	3.214				
119	99C	6.847	2.939	2.895				
119	99D	6.592	2.894	3.104				
121	99A	6.817	2.912	2.849	6.875	2.984	2.992	9.36
121	99B	6.327	2.644	2.797				
121	99C	6.750	2.912	2.951				
121	99D	7.606	3.466	3.373				
130	99A	6.962	3.045	2.995	7.031	3.043	3.073	1.85
130	99B	7.012	2.968	3.105				
130	99C	7.095	3.082	3.002				
130	99D	7.055	3.076	3.190				
Average		6.720	3.009	3.007	Includes Outlier			
Standard Deviation		0.674	0.312	0.326	Includes Outlier			
Std. Dev % of Avg.		10.03	10.36	10.85	Includes Outlier			

NETC Round Robin 99 - Raw Data  
Phase Angle Values for DSR - RTFO Testing

Lab #	Sample #	Lab Averages Including Outliers						Average Coefficient of Variability for each lab, %
		Single specimen mounted		New specimen	Single specimen mounted		New specimen	
		58 C, Phase	64 C, Phase	64 C, Phase	58 C, Phase	64 C, Phase	64 C, Phase	
		Angle Degrees	Angle Degrees	Angle Degrees	Angle Degrees	Angle Degrees	Angle Degrees	
101	99A	81.20	83.59	83.73	81.28	83.67	83.76	0.07
101	99B	81.29	83.73	83.72				
101	99C	81.36	83.69	83.77				
101	99D	81.27	83.66	83.82				
102	99A	81.40	83.60	83.70	81.23	83.70	83.60	0.26
102	99B	81.00	83.50	83.60				
102	99C	81.40	84.00	83.80				
102	99D	81.10	83.70	83.30				
103	99A	80.96	83.20	83.60	80.89	83.29	83.37	0.28
103	99B	80.99	83.42	83.41				
103	99C	80.50	83.07	83.02				
103	99D	81.11	83.47	83.46				
104	99A	80.60	82.30	82.30	81.43	83.33	83.53	0.99
104	99B	81.80	83.00	84.60				
104	99C	81.40	83.60	83.90				
104	99D	81.90	84.40	83.30				
107	99A	80.32	82.89	82.90	80.98	83.39	83.43	0.66
107	99B	81.26	83.64	83.73				
107	99C	80.65	83.08	83.08				
107	99D	81.68	83.95	84.01				
109	99A	84.74	86.73	86.66	81.86	84.26	84.29	2.11
109	99B	81.16	83.50	83.50				
109	99C	80.22	82.95	83.09				
109	99D	81.32	83.86	83.92				
110	99A	80.93	83.41	83.42	80.78	83.33	83.36	0.09
110	99B	80.77	83.32	83.39				
110	99C	80.69	83.30	83.34				
110	99D	80.72	83.28	83.30				
111	99A	81.10	83.62	83.53	80.97	83.51	83.46	0.23
111	99B	81.16	83.66	83.63				
111	99C	80.93	83.47	83.44				
111	99D	80.68	83.27	83.24				
113	99A		83.42	83.33	81.09	83.58	83.58	0.15
113	99B	81.15	83.68	83.67				
113	99C	80.99	83.55	83.61				
113	99D	81.14	83.66	83.69				
119	99A	81.20	83.40	83.80	81.10	83.35	83.68	0.16
119	99B	80.90	83.20	83.60				
119	99C	81.20	83.40	83.80				
119	99D	81.10	83.40	83.50				
121	99A	81.00	82.60	83.00	80.70	82.73	82.90	0.38
121	99B	80.80	82.70	83.10				
121	99C	80.90	83.20	82.70				
121	99D	80.10	82.40	82.80				
130	99A	80.77	83.27	83.53	80.67	83.33	83.36	0.14
130	99B	80.62	83.42	83.24				
130	99C	80.75	83.38	83.43				
130	99D	80.53	83.23	83.22				
Average		81.08	83.45	83.53	Includes Outlier			
Standard Deviation		0.66	0.62	0.60	Includes Outlier			
Std. Dev % of Avg.		0.82	0.74	0.72	Includes Outlier			

# NETC Round Robin 99 - Raw Data BBR Values

Lab #	Sample #	Beam 1		Beam 2		Lab Average		Average Coefficient of Variability for each lab, %	Average Coefficient of Variability for each lab, %
		m-value	Stiffness, MPa	m-value	Stiffness, MPa	m-value	Stiffness	m-value	Stiffness
101	99A	0.301	232.0	0.301	229.0	0.302	224.9	1.21	2.58
101	99B	0.301	223.0	0.301	227.0				
101	99C	0.297	216.0	0.303	229.0				
101	99D	0.310	217.0	0.302	226.0				
102	99A	0.294	252.0	0.293	253.0	0.293	252.6	0.92	1.10
102	99B	0.293	256.0	0.288	248.0				
102	99C	0.293	251.0	0.292	252.0				
102	99D	0.296	254.0	0.297	255.0				
103	99A	0.301	239.0	0.303	255.0	0.292	240.1	5.51	3.45
103	99B	0.295	246.0	0.294	227.0				
103	99C	0.253	234.0	0.299	242.0				
103	99D	0.295	252.0	0.296	226.0				
104	99A	0.308	199.0	0.288	210.0	0.324	207.9	17.60	9.95
104	99B	0.408	206.0	0.406	213.0				
104	99C	0.290	219.0						
104	99D	0.288	189.0	0.282	219.0				
107	99A	0.305	245.1	0.310	244.2	0.309	241.3	1.33	2.74
107	99B	0.302	238.1	0.311	238.1				
107	99C	0.305	258.6	0.311	243.1				
107	99D	0.314	241.5	0.311	221.8				
109								3.42	6.17
109									
109									
109									
110	99A	0.311	250.5	0.310	249.2	0.303	242.9	2.00	2.04
110	99B	0.303	237.8	0.303	240.7				
110	99C	0.294	244.1	0.296	238.0				
110	99D	0.300	243.3	0.305	239.4				
111	99A	0.298	259.0	0.304	250.0	0.298	248.5	1.43	3.26
111	99B	0.296	232.0	0.294	240.0				
111	99C	0.296	251.0	0.300	255.0				
111	99D	0.294	260.0	0.305	241.0				
113	99A	0.299	243.2	0.295	250.1	0.300	237.3	1.25	3.04
113	99B	0.305	242.7						
113	99C	0.298	229.2	0.302	228.7				
113	99D	0.304	238.1	0.297	229.2				
119	99A	0.301	244.9	0.290	220.7	0.297	235.0	1.31	3.55
119	99B	0.295	230.2	0.294	248.4				
119	99C	0.300	238.9	0.299	221.1				
119	99D	0.301	231.6	0.299	244.5				
121	99A	0.296	255.0	0.294	254.0	0.293	246.4	0.57	2.13
121	99B	0.293	248.0	0.291	239.0				
121	99C	0.291	247.0	0.292	237.0				
121	99D	0.293	240.0	0.292	251.0				

Overall Average		Std. Dev.	
m-value	0.301	0.019	Includes Outliers
Stiffness, MPa	238.077	14.532	Includes Outliers
Std. Dev % of Avg.	m-value	6.38	Includes Outliers
	Stiffness	6.10	Includes Outliers

Equipment not functioning properly, excluded from overall average



## Appendix C

### Amended Data for Round Robin 99

NETC Round Robin 99 - Refined  
Phase Angle Values for DSR - Original Testing

Lab #	Sample #	Lab Averages Excluding Outliers					
		Single specimen mounted		New specimen	Single specimen mounted		New specimen
		58 C, Phase Angle Degrees	64 C, Phase Angle Degrees	64 C, Phase Angle Degrees	58 C, Phase Angle Degrees	64 C, Phase Angle Degrees	64 C, Phase Angle Degrees
101	99A	85.54	86.93	86.97	85.51	86.92	86.88
101	99B	85.50	86.91	86.73			
101	99C	85.53	86.95	86.92			
101	99D	85.46	86.87	86.90			
102	99A	85.40	87.00	87.00	85.63	87.10	86.88
102	99B	85.70	87.20	86.90			
102	99C	85.70	87.00	86.70			
102	99D	85.70	87.20	86.90			
103	99A	85.75	86.96	86.93	85.60	86.94	87.00
103	99B	85.55	86.99	87.10			
103	99C	85.58	86.92	87.02			
103	99D	85.50	86.90	86.94			
104	99A		84.70	84.70	85.83	86.73	85.43
104	99B	85.80	87.40	84.60			
104	99C	85.70	87.40	87.40			
104	99D	86.00	87.40	85.00			
107	99A	85.71	87.24	87.22	85.69	87.20	87.24
107	99B	85.62	87.13	87.15			
107	99C	85.65	87.15	87.29			
107	99D	85.78	87.28	87.28			
109	99A	85.99	87.62	87.73	85.87	87.55	87.66
109	99B	85.81	87.52	87.68			
109	99C	85.75	87.35	87.60			
109	99D	85.91	87.70	87.63			
110	99A	85.42	86.93	86.98	85.41	86.92	86.95
110	99B	85.40	86.97	86.93			
110	99C	85.39	86.85	86.96			
110	99D	85.43	86.91	86.94			
111	99A	85.61	87.13	87.09	85.57	87.14	87.15
111	99B	85.58	87.13	87.15			
111	99C	85.61	87.17	87.20			
111	99D	85.49	87.12	87.15			
113	99A		87.12	87.06	85.42	87.09	87.07
113	99B	85.33	87.03	86.98			
113	99C	85.44	87.08	87.08			
113	99D	85.49	87.13	87.14			
119	99A	85.60	86.70	87.00	85.53	86.85	86.40
119	99B	85.60	86.70	87.00			
119	99C	85.50	87.00	84.70			
119	99D	85.40	87.00	86.90			
121	99A	85.30		85.90	85.10	86.13	85.85
121	99B	85.30	86.20	85.90			
121	99C	84.90	86.40	85.90			
121	99D	84.90	85.80	85.70			
130	99A	85.53	87.13	87.08	85.57	87.11	87.12
130	99B	85.58	87.09	87.13			
130	99C	85.53	87.11	87.14			
130	99D	85.65	87.12	87.14			
Average		85.56	86.99	86.80			
Standard Deviation		0.22	0.47	0.75			
Std. Dev % of Avg.		0.25	0.54	0.86			

 Excluded from average because it differed from the average by more than 3 Standard Deviations

NETC Round Robin 99 - Refined  
G\* Values for DSR - Original Testing

		Lab Averages Excluding Outliers					
		Single specimen mounted		New specimen	Single specimen mounted		New specimen
Lab #	Sample #	G* 58 C, kPa	G* 64 C, kPa	G* 64 C, kPa	G* 58 C, kPa	G* 64 C, kPa	G* 64 C, kPa
101	99A	2.477	1.157	1.177	2.531	1.175	1.161
101	99B	2.517	1.169	1.188			
101	99C	2.581	1.196	1.120			
101	99D	2.547	1.178	1.160			
102	99A	2.371	1.125	1.122	2.411	1.152	1.194
102	99B	2.441	1.165	1.202			
102	99C	2.388	1.156	1.222			
102	99D	2.443	1.163	1.230			
103	99A	2.516	1.157	1.155	2.679	1.246	1.250
103	99B	2.709	1.262	1.295			
103	99C	2.714	1.273	1.274			
103	99D	2.778	1.290	1.276			
104	99A	2.764	1.246	1.406	2.595	1.202	1.319
104	99B	2.526	1.161	1.321			
104	99C	2.590	1.272	1.350			
104	99D	2.499	1.130	1.199			
107	99A	2.701	1.315	1.297	2.716	1.326	1.314
107	99B	2.769	1.357	1.339			
107	99C	2.705	1.322	1.296			
107	99D	2.689	1.311	1.323			
109	99A	2.470	1.116	1.147	2.565	1.184	1.179
109	99B	2.501	1.188	1.184			
109	99C	2.682	1.188	1.179			
109	99D	2.607	1.242	1.207			
110	99A	2.593	1.187	1.162	2.549	1.145	1.180
110	99B	2.380	1.048	1.161			
110	99C	2.688	1.160	1.205			
110	99D	2.534	1.185	1.190			
111	99A	2.690	1.258	1.320	2.741	1.263	1.289
111	99B	2.691	1.247	1.264			
111	99C	2.768	1.267	1.279			
111	99D	2.814	1.279	1.292			
113	99A		1.250	1.228	2.783	1.250	1.238
113	99B	2.818	1.273	1.266			
113	99C	2.778	1.243	1.232			
113	99D	2.754	1.233	1.226			
119	99A	2.745	1.262	1.305	2.740	1.256	1.285
119	99B	2.701	1.237	1.272			
119	99C	2.777	1.301	1.302			
119	99D	2.736	1.225	1.263			
121	99A	2.660		1.102	2.600	1.157	1.167
121	99B	2.580	1.199	1.165			
121	99C	2.444	1.117	1.177			
121	99D	2.716	1.156	1.226			
130	99A	2.485	1.099	1.128	2.571	1.126	1.142
130	99B	2.513	1.109	1.152			
130	99C	2.692	1.158	1.145			
130	99D	2.594	1.137	1.141			
Average		2.620	1.208	1.227			
Standard Deviation		0.128	0.069	0.072			
Std. Dev % of Avg.		4.90	5.71	5.83			


 Excluded from average because it differed from the average by more than 3 Standard Deviations

NETC Round Robin 99 - Refined  
Phase Angle Values for DSR - RTFO Testing

Lab #	Sample #	Single specimen mounted			Lab Averages Including Outliers		
		58 C, Phase	64 C, Phase	New specimen	58 C, Phase	64 C, Phase	New specimen
		Angle Degrees	Angle Degrees	Angle Degrees	Angle Degrees	Angle Degrees	Angle Degrees
101	99A	81.20	83.59	83.73	81.28	83.67	83.76
101	99B	81.29	83.73	83.72			
101	99C	81.36	83.69	83.77			
101	99D	81.27	83.66	83.82			
102	99A	81.40	83.60	83.70	81.23	83.70	83.60
102	99B	81.00	83.50	83.60			
102	99C	81.40	84.00	83.80			
102	99D	81.10	83.70	83.30			
103	99A	80.96	83.20	83.60	80.89	83.29	83.37
103	99B	80.99	83.42	83.41			
103	99C	80.50	83.07	83.02			
103	99D	81.11	83.47	83.46			
104	99A	80.60	82.30	82.30	81.43	83.33	83.53
104	99B	81.80	83.00	84.60			
104	99C	81.40	83.60	83.90			
104	99D	81.90	84.40	83.30			
107	99A	80.32	82.89	82.90	80.98	83.39	83.43
107	99B	81.26	83.64	83.73			
107	99C	80.65	83.08	83.08			
107	99D	81.68	83.95	84.01			
109	99A	80.74	83.73	83.66	80.90	83.44	83.50
109	99B	81.16	83.50	83.50			
109	99C	80.22	82.95	83.09			
109	99D	81.32	83.86	83.92			
110	99A	80.93	83.41	83.42	80.78	83.33	83.36
110	99B	80.77	83.32	83.39			
110	99C	80.69	83.30	83.34			
110	99D	80.72	83.28	83.30			
111	99A	81.10	83.62	83.53	80.97	83.51	83.46
111	99B	81.16	83.66	83.63			
111	99C	80.93	83.47	83.44			
111	99D	80.68	83.27	83.24			
113	99A		83.42	83.33	81.09	83.58	83.58
113	99B	81.15	83.68	83.67			
113	99C	80.99	83.55	83.61			
113	99D	81.14	83.66	83.69			
119	99A	81.20	83.40	83.80	81.10	83.35	83.68
119	99B	80.90	83.20	83.60			
119	99C	81.20	83.40	83.80			
119	99D	81.10	83.40	83.50			
121	99A	81.00	82.60	83.00	80.70	82.73	82.90
121	99B	80.80	82.70	83.10			
121	99C	80.90	83.20	82.70			
121	99D	80.10	82.40	82.80			
130	99A	80.77	83.27	83.53	80.67	83.33	83.36
130	99B	80.62	83.42	83.24			
130	99C	80.75	83.38	83.43			
130	99D	80.53	83.23	83.22			
Average		81.00	83.38	83.46	Excludes Outlier		
Standard Deviation		0.38	0.39	0.39	Excludes Outlier		
Std. Dev % of Avg.		0.47	0.47	0.46	Excludes Outlier		

NETC Round Robin 99 - Refined  
G\* Values for DSR - RTFO Testing

Lab #	Sample #	Single specimen mounted			Lab Averages Excluding Outliers		
		G* 58 C, kPa	G* 64 C, kPa	New specimen	G* 58 C, kPa	G* 64 C, kPa	G* 64 C, kPa
101	99A	6.435	2.897	2.714	6.126	2.737	2.713
101	99B	6.081	2.672	2.671			
101	99C	5.794	2.628	2.769			
101	99D	6.194	2.749	2.698			
102	99A	6.178	2.739	2.759	6.311	2.838	2.835
102	99B	6.405	2.934	2.868			
102	99C	6.284	2.830	2.877			
102	99D	6.350	2.849	2.837			
103	99A	6.988	3.132	3.059	7.094	3.198	3.278
103	99B	7.082	3.215	3.143			
103	99C	7.930	3.559	3.624			
103	99D	6.374	2.886	3.287			
104	99A	7.046	3.155	3.168	6.632	2.956	2.883
104	99B	6.000	2.749	2.367			
104	99C	7.360	3.270	2.900			
104	99D	6.120	2.650	3.097			
107	99A	7.748	3.608	3.634	6.909	3.260	3.279
107	99B	6.695	3.150	3.084			
107	99C	6.967	3.365	3.499			
107	99D	6.224	2.916	2.899			
109	99A	3.556	3.167	3.1623	7.317	3.271	3.232
109	99B	6.824	3.297	3.353			
109	99C	7.902	3.605	3.572			
109	99D	6.731	2.937	2.892			
110	99A	7.393	3.291	2.973	6.989	3.077	2.967
110	99B	6.830	2.989	2.906			
110	99C	6.952	3.030	3.010			
110	99D	6.781	2.998	2.977			
111	99A	6.704	3.160	3.081	6.961	3.181	3.193
111	99B	6.734	3.058	3.058			
111	99C	7.002	3.170	3.223			
111	99D	7.403	3.335	3.409			
113	99A		3.177	3.083	6.721	2.994	2.983
113	99B	6.655	2.863	2.913			
113	99C	6.886	2.986	3.012			
113	99D	6.621	2.929	2.924			
119	99A	6.624	2.989	2.920	6.758	2.965	3.033
119	99B	6.968	3.040	3.214			
119	99C	6.847	2.939	2.895			
119	99D	6.592	2.894	3.104			
121	99A	6.817	2.912	2.849	6.875	2.984	2.992
121	99B	6.327	2.644	2.797			
121	99C	6.750	2.912	2.951			
121	99D	7.606	3.466	3.373			
130	99A	6.962	3.045	2.995	7.031	3.043	3.073
130	99B	7.012	2.968	3.105			
130	99C	7.095	3.082	3.002			
130	99D	7.055	3.076	3.190			
Average		6.789	3.038	3.037	Excludes Outliers		
Standard Deviation		0.487	0.244	0.257	Excludes Outliers		
Std. Dev % of Avg.		7.17	8.03	8.48	Excludes Outliers		


 Excluded from average because it differed from the average by more than 3 Standard Deviations


# NETC Round Robin 99 - Refined BBR Data

Lab #	Sample #	Beam 1		Beam 2		Lab Average	
		m-value	Stiffness, MPa	m-value	Stiffness, MPa	m-value	Stiffness
101	99A	0.301	232.0	0.301	229.0	0.302	224.9
101	99B	0.301	223.0	0.301	227.0		
101	99C	0.297	216.0	0.303	229.0		
101	99D	0.310	217.0	0.302	226.0		
102	99A	0.294	252.0	0.293	253.0	0.293	252.6
102	99B	0.293	256.0	0.288	248.0		
102	99C	0.293	251.0	0.292	252.0		
102	99D	0.296	254.0	0.297	255.0		
103	99A	0.301	239.0	0.303	255.0	0.292	240.1
103	99B	0.295	246.0	0.294	227.0		
103	99C	0.253	234.0	0.299	242.0		
103	99D	0.295	252.0	0.296	226.0		
104	99A	0.308	199.0	0.288	210.0	0.291	211.0
104	99B	0.308	206.0	0.308	213.0		
104	99C	0.290	219.0				
104	99D	0.288	189.0	0.282	219.0		
107	99A	0.305	245.1	0.310	244.2	0.309	241.3
107	99B	0.302	238.1	0.311	238.1		
107	99C	0.305	258.6	0.311	243.1		
107	99D	0.314	241.5	0.311	221.8		
109							
109							
109							
109							
110	99A	0.311	250.5	0.310	249.2	0.303	242.9
110	99B	0.303	237.8	0.303	240.7		
110	99C	0.294	244.1	0.296	238.0		
110	99D	0.300	243.3	0.305	239.4		
111	99A	0.298	259.0	0.304	250.0	0.298	248.5
111	99B	0.296	232.0	0.294	240.0		
111	99C	0.296	251.0	0.300	255.0		
111	99D	0.294	260.0	0.305	241.0		
113	99A	0.299	243.2	0.295	250.1	0.300	237.3
113	99B	0.305	242.7				
113	99C	0.298	229.2	0.302	228.7		
113	99D	0.304	238.1	0.297	229.2		
119	99A	0.301	244.9	0.290	220.7	0.297	235.0
119	99B	0.295	230.2	0.294	248.4		
119	99C	0.300	238.9	0.299	221.1		
119	99D	0.301	231.6	0.299	244.5		
121	99A	0.296	255.0	0.294	254.0	0.293	246.4
121	99B	0.293	248.0	0.291	239.0		
121	99C	0.291	247.0	0.292	237.0		
121	99D	0.293	240.0	0.292	251.0		

Overall Average		Std. Dev.
m-value	0.298	0.008
Stiffness, MPa	238.714	13.486

Std. Dev % of Avg.	m-value	2.80	Excludes Outliers
	Stiffness	5.65	Excludes Outliers

 Excluded from average because it differed from the average by more than 3 Standard Deviations

 Equipment not functioning properly, excluded from overall average

## Appendix D

### Round Robin 99 Participants

## NETC Round Robin 99 Participants

Maine DOT

New Hampshire DOT

Vermont AOT

Mass Highway Department

Rhode Island DOT

New York State DOT

Hudson Asphalt

Sun Company

Chevron

Citgo

Bitumar

CAP Lab



## Appendix V

### Lab Visits – Observations

## State DOT Binder Lab Visits

Visits have been made to all of the New England State Departments of Transportation except Massachusetts. A visit was also made to the New York State Department of Transportation. The visit was made to NYSDOT since they are a "Lead State" for the implementation of Superpave. A visit to Massachusetts has not yet been scheduled since they are still in the process of installing the Superpave binder testing equipment.

These visits were intended to identify variations in testing techniques which might account for some of the variations observed in recent round robins. Emphasis was placed on trying to observe lab personnel performing binder testing as they would normally. Observations were also made during the visits for techniques which may save time. In order to protect the confidentiality of labs performing tests improperly, this information will be reported in a list form only – no agency names will be assigned to this list.

### Bending Beam Rheometer

1. Using silicone rubber molds rather than the aluminum molds.
2. Adjusting loads on asphalt beams rather than the steel beams. The test should begin immediately after placing asphalt beam in supports and lowering the load nose.
3. One BBR was modified significantly. It is not clear whether or not this is affecting the test results.
4. Using the glycerin-talc mixture over the entire inside of the aluminum beam molds rather than using the plastic strips.
5. Covering the end-spacers with Vaseline rather than the glycerin-talc mixture.
6. No thermometer capable of measuring the temperature of the BBR bath.
7. The cooling unit for the BBR bath should not be placed on the same counter top as the BBR due to vibrations.
8. Using the AASHTO Fluid (methanol, water and ethylene glycol) causes the beams to float. If the beams are not submerged in the fluid and are allowed to float around the surface of the fluid, the beams are probably not at the test temperature.

### Rolling Thin Film Oven (RTFO)

1. Not using the RTFO and instead using the Thin Film Oven.
2. Losing track of amount of time samples stayed in the RTFO.
3. Only one state scrapes their RTFO bottles.

### Pressure Aging Vessel (PAV)

1. The pressure regulator set either too low or too high.
2. The amount of time to de-air samples after removing samples from the PAV varies from 30 minutes to 120 minutes at 163 °C for similar materials.
3. Temperature which the de-airing was performed was close to 180 °C.
4. About half the labs visited de-aired their samples in the PAV pans while the other half of the labs transferred the samples (as soon as the samples were heated to pourable state) to a container which could be covered while it is in the oven.

### Rotational Viscometer

1. Only 1 Paar Physica Rotational Viscometer was observed to still be in service in the New England Region.
2. Labs only utilizing the #27 Brookfield Spindle can not usually achieved the torque required by the Brookfield Viscometer for high resolution of the viscosity at 20 RPM. This can be corrected by utilizing the #21 Brookfield Spindle. By increasing the spindle the viscosity reading will be better defined – although for the most part – most asphalts do not approach the failure criteria defined by AASHTO MP-1.

### Dynamic Shear Rheometer (DSR)

Note: Work performed with the 8mm DSR plates was not emphasized since they will be replaced with the 12 mm plates.

1. One lab did not have any ability to measure the temperature between the plates.
2. Daily temperature calibration to adjust for wide temperature fluctuations within a lab probably did not improve the equipment's temperature accuracy since the calibration was performed early in the morning and it was never rechecked as the room temperature increased.

3. Strong air drafts across the DSR, even with the oven closed on the machine, can effect the sample temperature.
4. Trimming of samples in DSR – many places tended to leave a ragged front side to the sample.
5. Amount of time sample was allowed to stand in the silicone mold. Some samples were allowed to stand open in the lab for several hours. These samples had the opportunity to be contaminated with dust or other impurities. It takes very little dust to begin to alter the properties of the asphalt on the DSR.
6. Cleaning the DSR plates with a solvent and then not rinsing the solvent off of the plates.
7. Zeroing the gap on the DSR and then removing the Spindle to mount the sample.
8. Zeroing the gap on the DSR without first warming the DSR plates.
9. About half of the labs used a heated trimming tool for trimming DSR samples of neat asphalts. All labs used a heated trimming tool for modified asphalts.

### Time Saving Techniques

1. Glass cleaning ovens are being used for their RTFO bottles. Ignition ovens are also employed to burn out the RTFO bottles. This technique appears to work very well as long as the bottles are allowed to warm up with the ovens. Placing the room temperature bottles into a hot oven has created some problems. Burning the contents out of the bottles tends to leave only a small amount of ash in the bottles which can be rinsed out with water. New Hampshire's glass cleaning oven had the largest capacity of any lab visited and it appeared to do the best job. The ignition ovens appeared to work adequately – although some modification may be required to hold the bottles properly. Maine has created a rack which sets on top of their baskets for their Thermolyne Ignition oven.
2. The plastic strips in BBR aluminum molds which are too tall can be trimmed down to size using a razor blade.
3. The plastic strips for the BBR molds can be sized by photocopying lines onto the plastic sheets for guides to cut the strips.

4. One lab does not use solvent for their BBR molds. Instead of soaking the molds, they scrape the excess binder off of the molds using a razor blade. This is performed immediately after the beam has been demolded and placed into the bath. The binder is still stiff from being frozen and is easily removed. Care must be used to avoid gouging the aluminum with the razor blade.
5. Rather than preheating rotational viscosity spindles in the machine – they can be preheated in an oven.
6. Placing ovens on timers so that they can be preheated before anyone arrives in the morning will save time.
7. The use of straight alcohol in the BBR will save time (If allowed by your regulations) because it changes temperature much quicker than the AASHTO Mixture does.

#### How particular things are being handled

Solvents being Utilized	
<u>Solvent</u>	<u># of States using</u>
Toluene	1
Xylene	2
Citrus	3

BBR Bath Fluids	
<u>Fluid</u>	<u># of States using</u>
Methanol	1
Ethanol	3
AASHTO Mix	2

#### Conclusions

For the most part, the labs visited were complying with the requirements of the AASHTO Standards. The largest recommendation I can make would be the use of time log sheets. On these sheets, the time is recorded for the beginning of each step of the test process. This would help to reduce uncertainties regarding the length of time samples are in each step. This may help to reduce questions which arise about certain samples.

The following information is provided for your information only. It is not intended to be used as a substitute for the information provided in the other sections of this binder. The information is provided for your information only. It is not intended to be used as a substitute for the information provided in the other sections of this binder.

The following information is provided for your information only. It is not intended to be used as a substitute for the information provided in the other sections of this binder.

The following information is provided for your information only. It is not intended to be used as a substitute for the information provided in the other sections of this binder.

The following information is provided for your information only. It is not intended to be used as a substitute for the information provided in the other sections of this binder.

## **Appendix VI**

### **NETC Binder Technician Workshops Presentations**

## 1999 NETC Binder Technician Workshop Executive Summary

February 8, 1999

NETC Project 96-1 "Superpave Implementation"

Dear Technical Committee Member,

On January 19, 1999, the CAP Lab hosted a one-day NETC Binder Technician Workshop. On January 20-21, 1999, the CAP Lab hosted a NECEPT/CAP Lab Binder Technician Workshop. The NETC Binder Technician Workshop focused on the progress that has been made so far during this NETC project. The NECEPT/CAP Lab Workshop focused on the Binder Technician Certification program under development jointly between the CAP Lab and NECEPT.

### NETC Binder Technician Workshop

During the NETC Binder Technician Workshop, presentations were made by Dr. Jack E. Stephens and James Mahoney. These presentations included data on the Round Robin #2 (March 1998), Pre-molded DSR samples, Observations made during lab visits, the '99 Round Robin and the Purpose of the 1999 Lab Visits to be made by the CAP Lab in Late February/March. I have included the presentations which were made during the workshop. I have also included a floppy disk containing all of the presentations – the graphs are much easier to see on the computer rather than the printouts. The presentations were made on PowerPoint/Windows 97. I have tried to save them in a lower format and all of the formatting ends up lost and makes the slides difficult to understand. I have also included print outs of all the data from Round Robin #2.

Round Robin #2 has been a joint effort between the CAP Lab and NECEPT. This was done to increase the number of participants and to increase the statistical value of the data. The final report on Round Robin #2 will be a joint effort between the CAP Lab and NECEPT since the entire Round Robin has been a joint effort. The CAP Lab has submitted information to NECEPT to assist in the preparation of the final report for Round Robin #2 and edit the final report as it is compiled.

Information regarding the Pre-Molded DSR samples was already circulated to all of the participants and to the Technical Committee members. The amount of data collected during the testing was small but it was the inspiration of the NETC 99 Round Robin. The samples used in the testing were prepared identically and the spread of the data was greatly reduced.

The presentation of the observations made during visits to the state labs were an opportunity to share what the different state DOT's were doing (both good and bad) without embarrassing anyone, since the observations were reported anonymously. Several participants commented that they appreciated the ability to share their technique problems with others while remaining anonymous.

The '99 NETC Round Robin testing procedures were discussed and samples distributed to the participants present. Samples and a detailed set of sample handling instructions were mailed to the participants unable to attend the workshop. The goals of the 99 Round Robin will be discussed following the submission of the data. This Round Robin is being handled solely by the CAP Lab as a NETC function. The goal is to be able to present results of the 99 Round Robin before the construction season gets too far underway.

The 99 Lab visits are in conjunction with the NETC 99 Round Robin. The CAP Lab will send Jim Mahoney out to each of the New England State DOT's with identical samples prepared at the CAP Lab. Jim Mahoney will run these samples on each DOT's equipment. The idea of these visits is to try to separate the differences between equipment and the differences caused by different operators of the equipment during the 99 Round Robin. These visits should commence in Late February/March. The CAP Lab would like to run these samples on each DOT's Dynamic Shear Rheometer prior to the DSR's 6 month preventive maintenance. This will enable the CAP Lab to better differentiate where the sources of variation are occurring.

### NECEPT/CAP Lab Binder Technician Workshop

This workshop was held in duplicate at the CAP Lab facility in Storrs, CT and at the NECEPT facility at Penn State University. During this workshop, presentations were made by Dr. Jack Stephens and Dr. David Anderson. The focus of this workshop was the binder technician certification program. The presentations made during this workshop were made mainly from the Manual of Practice which is being written by NECEPT. This Manual of Practice will be used to better clarify testing procedures for technician certification. The manual is in the process of being revised following the workshops. When the revision of the manual has been completed, a copy will be forwarded to the members of the technical committee for NETC Project 96-1.

Also during the NECEPT/CAP Lab Workshops, changes to binder testing specifications were discussed by Dr. David Anderson. The largest change proposed to the binder testing specification at this point is the need to vacuum de-gas the PAV residue. This change should come in the 1999 AASHTO revisions to the PAV specification. The equipment requirements are in the manual of practice.



The RTFO Specification will also see changes. In the future, the RTFO bottles will have to be scraped in order to remove at least 90% of the material in the bottles. The bottles will also have to be stored in a horizontal position immediately after pouring so that modified binders will have a better chance to coat the sides of the bottle.

Improved methods for making temperature measurements were discussed. The use of a laboratory standard against which temperature measuring devices could be calibrated was discussed as being the method of preference. The exact specifications for these laboratory standards has yet to be finalized. The Direct Tension was also discussed. The use of the Direct Tension is coming. There was speculation that the Direct Tension could be in use by next winter.

Several issues that were discussed with no conclusion include the use of the ignition or glass cleaning ovens to clean the RTFO bottles. Some places reported having the bottles turn a milky white color. NECEPT has requested that several of the labs having these problems send them a bottle so that they can have the milky color analyzed to determine if this discoloration would affect the test results. The temperature controller for the Brookfield Rotational Viscometer appears to not be able to maintain temperature as closely as the temperature specification requires. Further investigation is required to clarify this issue.

CC:

Michael Byrne  
Reid Kiniry  
Paul Matthews  
Nellie Perlov  
Nelio Rodrigues  
Bruce Yeaton

Rhode Island DOT  
Vermont AOT  
New Hampshire DOT  
Mass Highway  
Connecticut DOT  
Maine DOT

**NETC Binder Technician Workshop Agenda**  
**January 19, 1999 – CAP Lab**  
**Storrs, Connecticut**

10:30 - 11:00	Registration
11:00 - 11:15	Welcome
11:15 - 12:00	Discussion of March 1998 Round Robin Results
12:00 - 1:00	Lunch (Provided)
1:00 - 1:30	Discussion of Pre-molded DSR Sample Results
1:30 - 2:45	Discussion of Lab Observations from visits
2:45 - 3:00	Break
3:00 - 3:30	Explanation of NETC Round Robin 1999 and distribution of samples
3:30 - 4:00	Lab visits from CAP Lab during February-March 1999

## NETC Binder Technician Workshop

A one-day NETC Binder Technician Workshop will be held on January 19, 1999 at 11:00 AM. This workshop will only be held at the CAP Lab in Storrs, CT. (There will be a joint CAP Lab/NECEPT binder technician workshop held on January 20-21 at the CAP Lab. Details on the NECEPT/CAP Lab workshop will follow.) Topics to be discussed at the NETC workshop include: Results from the Second Round Robin (March 1998), the Pre-Molded DSR Sample Results, Observations from the Lab Visits to New England State DOTs, Lab Visits from the CAP Lab with Asphalt Binder Samples (February-March 1999) and instructions for the January '99 Round Robin.

The registration fee for the NETC workshop is \$20.00. Lunch and refreshments are included. This fee will be waived for New England State DOT employees. In order to limit the size of the group (to allow for better participation) registration is limited to 2 technicians from each company or agency.

To register for this workshop, please fax or mail this form back to Jim Mahoney at:

CAP Lab  
179 Middle Turnpike, U-202  
Storrs, CT 06269-5202  
Phone 860-486-5956 Fax 860-486-2399

Registrant1 \_\_\_\_\_

Registrant2 \_\_\_\_\_

Company or Agency \_\_\_\_\_

Address \_\_\_\_\_

Town \_\_\_\_\_ State \_\_\_\_\_ Zip \_\_\_\_\_

Work Phone \_\_\_\_\_ Fax \_\_\_\_\_

Home Phone (In the event of inclement weather) \_\_\_\_\_

## **Observations from Visits to New England State DOT Binder Labs**

---

**New England  
Transportation Consortium  
Project 96-1**

### **Labs Visited**

---

- 5 of the 6 DOT Binder Labs in New England were visited
- New York State DOT was also observed since they are a lead state for Superpave Implementation

## **Objectives of Visits**

---

- Attempt to identify sources of variation observed in Round Robins
- Observe actual techniques being utilized by testing agencies
- Look for problems with testing techniques
- Look for techniques which may save time or increase reproducibility

## **Overall Observations**

---

- Overall, labs were performing the testing in accordance with the AASHTO Specifications
- There were some deviations or interpretations of the specifications
- There also were some procedures being performed which could be altered or eliminated to save time

## **Overall Observations continued**

---

- Some facilities have created some good time saving procedures
- Every facility visited was very open to discuss their procedures and each wanted to improve their methods anyway possible

## **Rotational Viscometer**

---

## **Rotational Viscometer**

---

- Majority of States have switched to the Brookfield Viscometer
- Brookfield Viscometers should have at least two different spindles sizes available
  - #21 and #27 spindles
  - Larger spindles allow machine to develop larger torques at slower speeds - This increases the machine's data resolution

## **Rotational Viscometer**

---

- Spindles may be heated to the testing temperature in an oven prior to testing - this will decrease the amount of time required to heat sample
- Temperature calibration should be performed while the spindle or bob is spinning

## **Dynamic Shear Rheometer**

---

### **DSR Temperature**

---

- Strong air currents across plates can cause wide fluctuations in sample temperature
- One facility did not have the ability to measure the temperature between the plates
- Daily calibration of the temperature for the DSR was being performed early in the morning because of the variations in room temperature - temperature was not being checked later in day



## **DSR Plates**

---

- The DSR plates were being cleaned with a solvent and then not being rinsed
- Zeroing the gap without warming the plates
- Zeroing the gap and then removing the upper spindle to mount sample on it

## **DSR Trimming**

---

- Most people do a very good job trimming the sample in all locations except for the very front of the sample
- About half of the labs visited used a heated trimming tool for all binders. All labs used a heated tool for modified binders.

## **DSR Miscellaneous**

---

- The amount of time the sample was allowed to stand in the silicone mold varied greatly from location to location. The samples in most cases were not covered. This allowed contaminants such as dust to get into samples. It does not take much dust to get a substantial increase in the stiffness of the binder.

## **Rolling Thin Film Oven**

---

## **RTFO**

---

- Using the Thin Film Oven rather than the Rolling Thin Film Oven
- Not accurately tracking length of time samples are in the RTFO
- Only one state scrapes their RTFO bottles
- One facility uses bottled compressed air rather than house air

## **Pressure Aging Vessel**

---

## **PAV**

---

- Pressure regulator was set to high or too low - can cause timing troubles for PAV
- All states have the same ATS Pressure Aging Vessel Ovens

## **PAV De-Airing**

---

- Length of time labs are placing material in oven varied from 30 minutes to 2 hours at the same temperature
- Samples were being de-aired at a temperature close to 180 C
- About half of the labs de-aired in the PAV pans and the others heated the material until they could combine residue into a larger covered container and then de-aired

## **BBR Observations**

---

## **BBR Molds**

---

- Using Vaseline on end spacers rather than the glycerin-talc mixture
- Using glycerin-talc mixture over entire mold and not using plastic strips
- Using silicone rubber molds rather than the aluminum molds
  - silicone rubber molds are no longer allowed in AASHTO TP1

## **BBR Molds Cleaning**

- One facility does not use a solvent to clean their molds - they use a razor blade to clean the beam molds. Caution must be used not to gouge soft aluminum with razor blade
- The beam mold parts can be soaked in a can with holes in it. This can is allowed to stand in a larger can of solvent and then removed - the solvent drains out of holes in smaller can.

## **BBR Test Procedures**

- Adjusting testing loads on the asphalt beam just prior to starting test

## **BBR Miscellaneous**

- One BBR was significantly modified - unsure if this affected the test results
- No thermometer capable of measuring the temperature of the BBR bath
- Cooling unit placed upon the same shelf as the BBR
- If you use the AASHTO fluid mixture you must ensure beams are submerged

## **BBR Bath Fluids**

- 1 State is using Methanol
- 3 States are using Ethanol
- 2 States are using the AASHTO Mixture
- Fluid level in bath does not have to be to the top of bath - just need sufficient fluid to cover samples

## **BBR Bath Fluids Continued**

---

- The use of straight alcohol in the BBR will reduce the amount of time required to bring the temperature bath down to test temperature

## **BBR Plastic Strips**

---

- Plastic Strips which extend above the aluminum molds once the molds are already assembled can be trimmed down to the correct size with a razor blade
- Lines for cutting the plastic strips can be photocopied onto the plastic sheets prior to cutting



## Round Robin 2 (March 98)

NETC Project 96-1  
Superpave Implementation

### 4 Binder Samples Were Sent

- Samples 1A & 4A were the same material
- Samples 2A & 3A were the same material
- Pre-aged samples 1 & 4 were the same
- Pre-aged samples 2 & 3 were the same



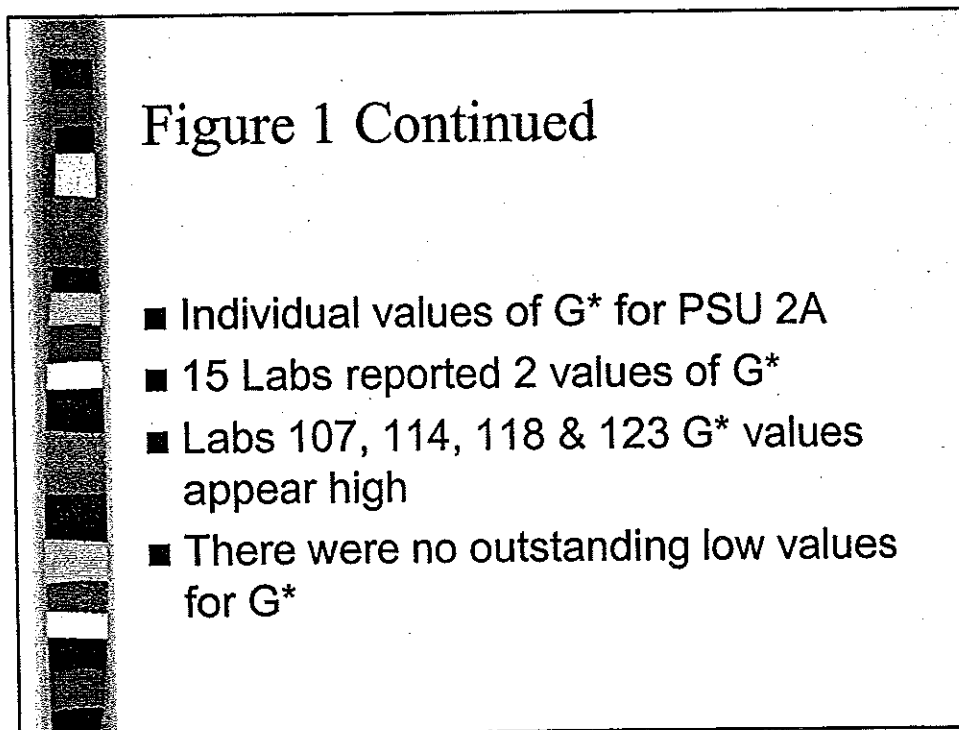
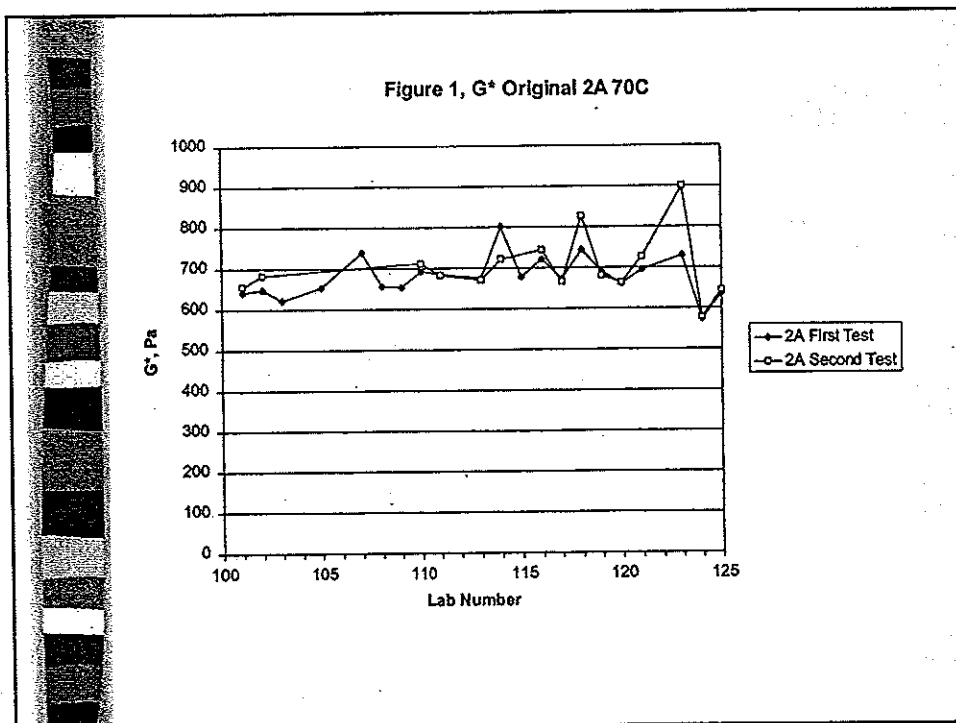
## DSR Testing

- Performed on Original and RTFO Material
- 1A and 4A tested at 70 & 76 C
- 2A and 3A tested at 64 & 70 C
- Duplicate samples were run of each material at each temperature



## Figure 1

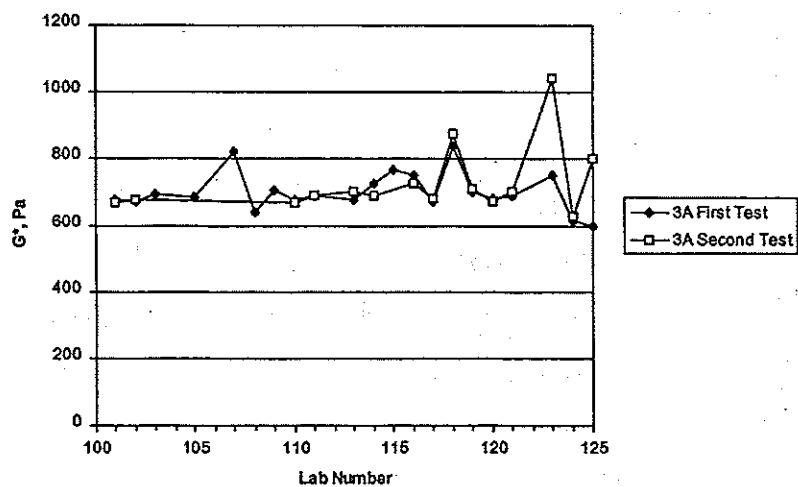
- PSU 2A Original @ 70 C
- Most data points are the average of two  $G^*$  values
- More erratic on the high side
- $G^*$  ranges from 625 to 815 Pa,
- The average was 686 Pa
- The range is 28% of the average



## Figure 2

- PSU 3A  $G^*$  @ 70 C
- Same material as PSU 2A
- For Figures 1 & 2, 4 of the highs are #2

Figure 2,  $G^*$  Original 3A 70C



# Figure 3

- Original PSU 2A & 3A @ 70 C
- The second G\* value was higher 17 out of the 21 times
- Labs 17, 118 and 123 were high for both the first and second tests for both 2A and 3A

## Figure 3, Original PSU 2&3 @70C

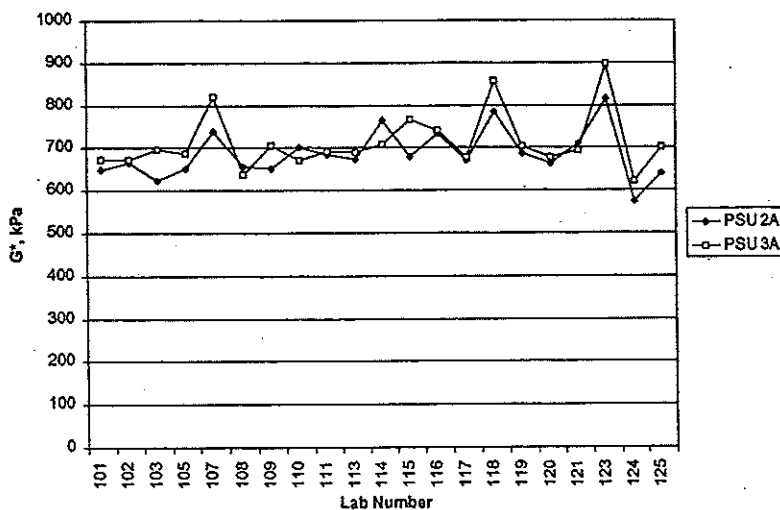


Figure 4

- $G^*$  Original 2A @ 64 C
- 34 out of 42 on line
- Labs 114, 118, 123 and 125 again out
- Range less at 64 C than at 70 C

Figure 4,  $G^*$  Original 2A 64C

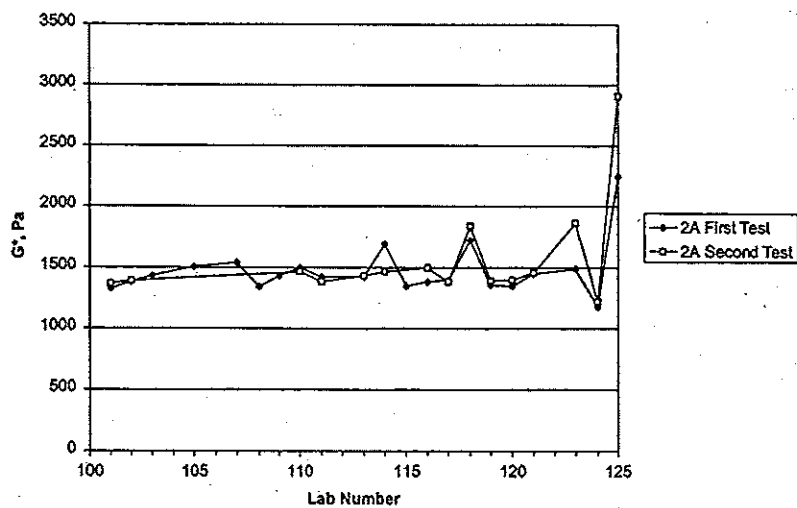


Figure 5

- G\* Original 1A & 4A @ 70 & 76 C
- Each Lab is in good agreement

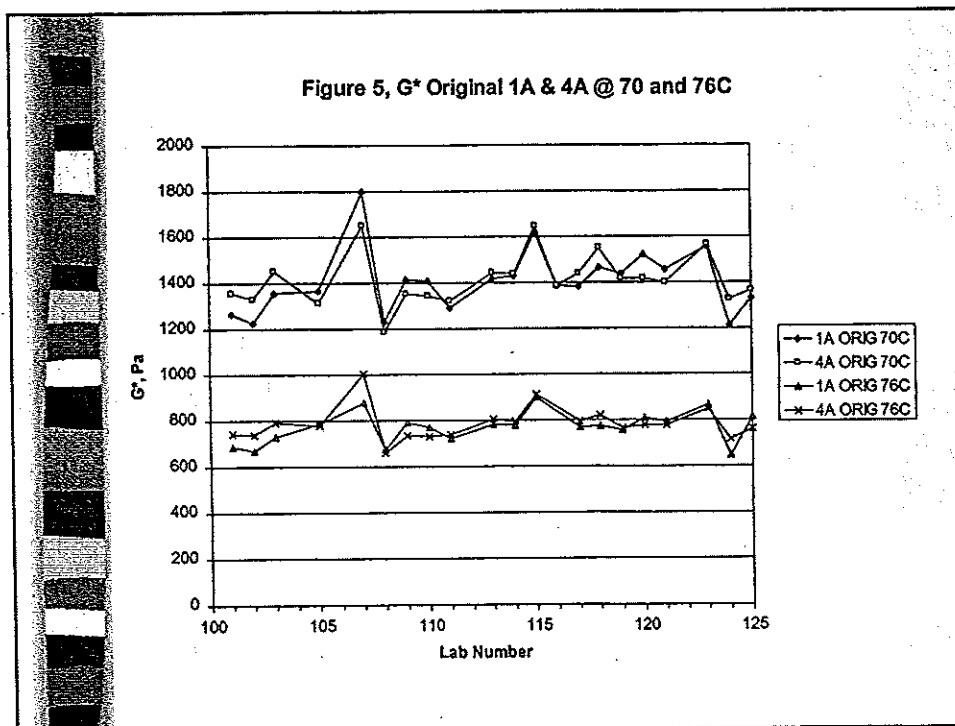


Figure 6

- $G^*$  Original 1A, 2A, 3A and 4A @ 70 C
- Labs 107 & 115 only reported one value for  $G^*$
- Labs 107, 115, 118 and 123 tend to be high

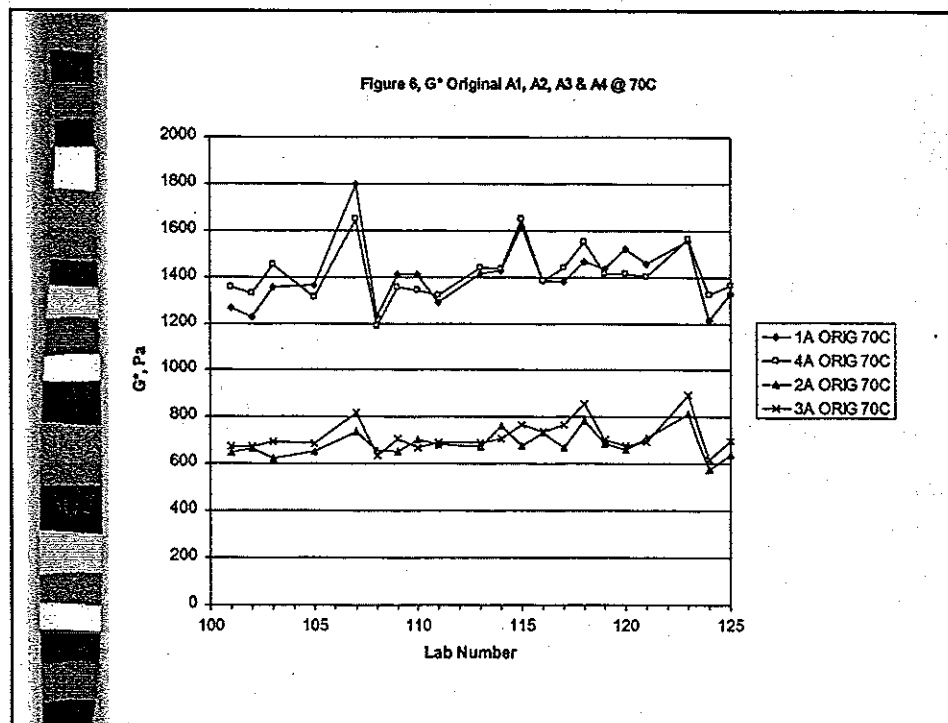
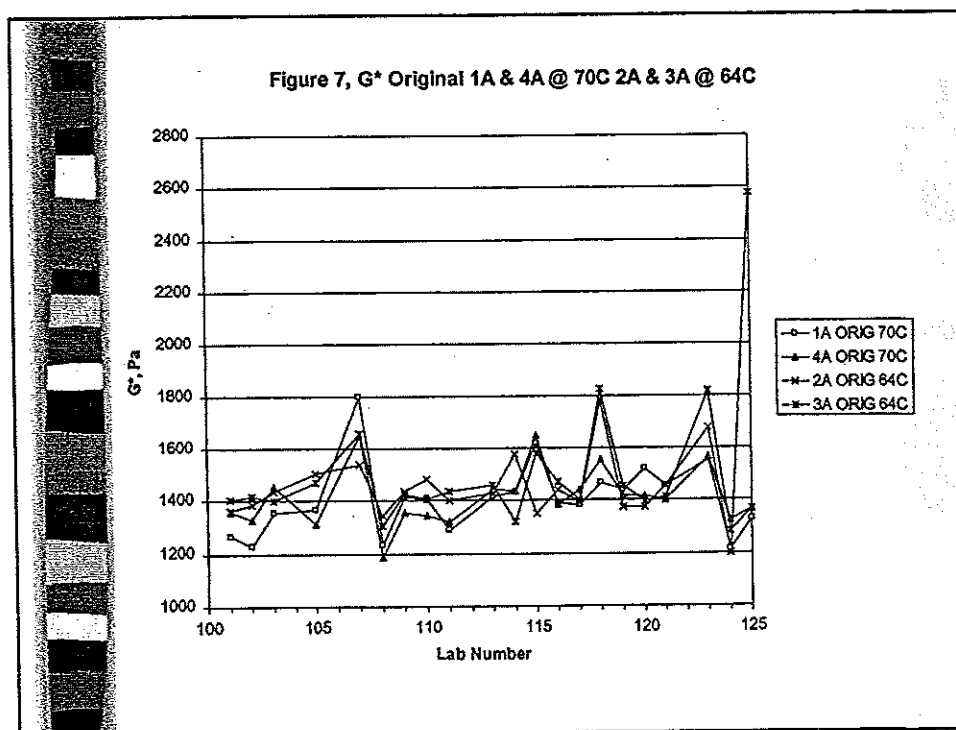




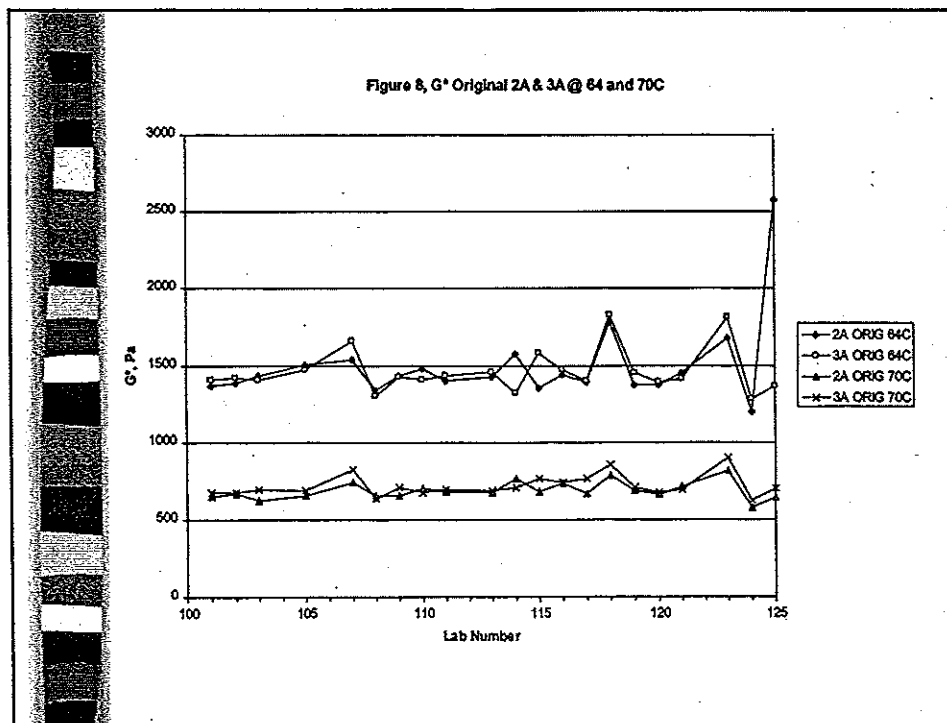
Figure 7

- G\* Original 1A & 4A @ 70 C and 2A & 3A @ 64 C
- Lab Pattern
- Labs 107, 115, 118 and 123 Consistently High
- Labs 108 and 124 are low
- Discard outlier at 125



# Figure 8

- G\* Original 2A and 3A @ 64 and 70 C
- Look at Lab Differences
- Note 107, 118 and 123 are consistently high
- Outlier at 125



# Figure 9

- G\* RTFO 2A & 3A @ 64 & 70C
- Lab 105 and 115 G\* values are low
- Lab 123 G\* continues to be high
- Lab 125 is erratic
- 64 C is more consistent than 70 C
- Compare variation of 1500 Pa to 3300 Pa
- Similar Percentages

Figure 9, G\* RTFO for 2A & 3A @ 64 and 70C

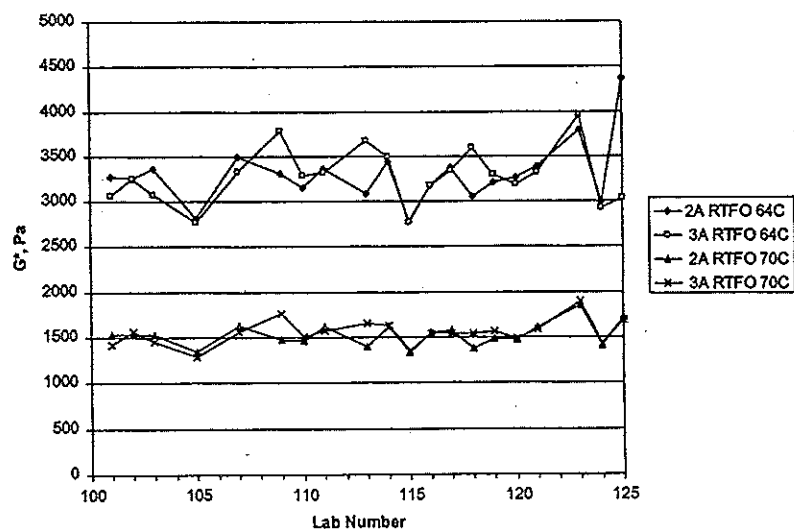


Figure 10

- $G^*$  RTFO 1A & 4A @ 70 C and 2A & 3A @ 64 C
- Average  $G^*$  for materials about the same at the two temperatures
- Lab 103, 105, 107, 109 and 115 reported only one value for  $G^*$
- Second test could temper the differences

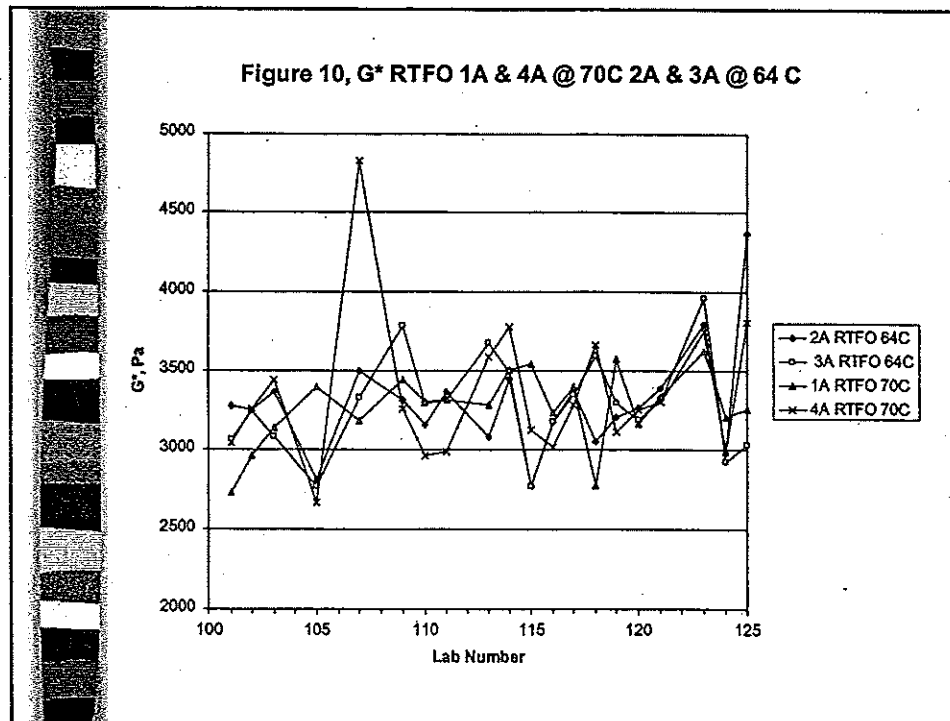
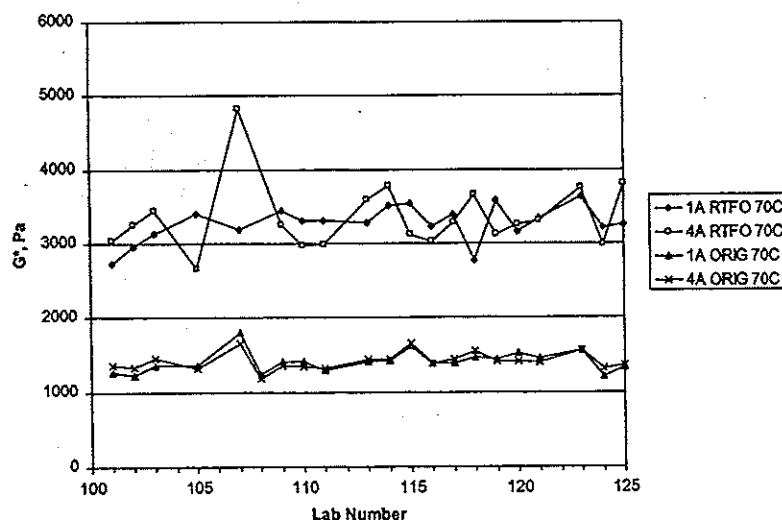


Figure 11

- $G^*$  Original & RTFO 1A & 4A @ 70C
- Why is  $G^*$  Original much more consistent than  $G^*$  RTFO?
- Labs 107, 115, 118 and 123 still tend to be high

Figure 11,  $G^*$  Original & RTFO A1 & A4 @ 70C



## Figure 12

- $G^*$  at Lowest Test Temp. per Sample Stage
- Lab 105, 107, 108, 109 and 115 only reported one value
- For RTFO Material Lab 105 tend to be low and Lab 123 tend to be High
- For Original Material Lab 107, 118 and 123 tend to be high and Lab 108 and 124 tend to be low

Figure 12, Lowest Test Temps Per Sample Stage

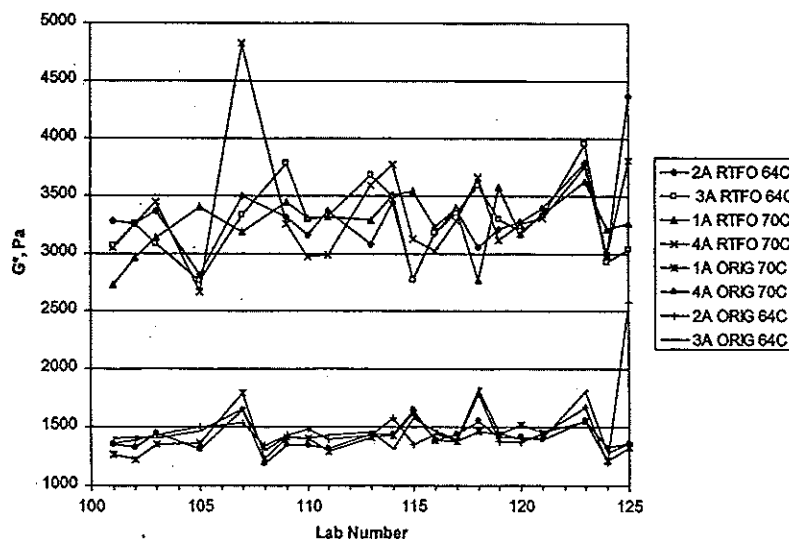


Figure 13

- PSU 2A @ 70 C First and Second Phase Angles
- Twelve Labs have less than 0.5 degree difference
- Lab 115 appears as outlier but is only 3.5 degrees from the average

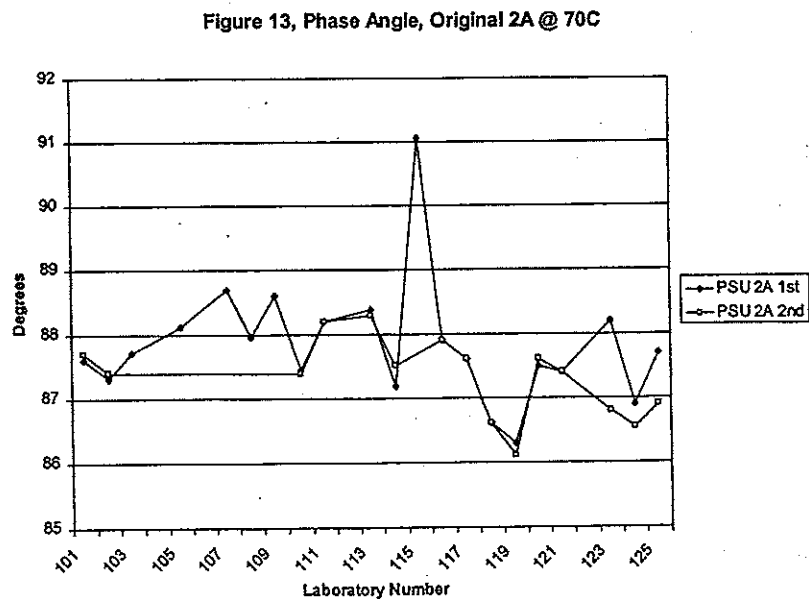


Figure 14

- PSU 3A, First and Second Phase Angles
- Eleven Labs with less than 0.5 Degree Difference
- Lab Number 115 appears as an outlier but is only 2.5 degrees from the average

Figure 14, Phase Angle, Original 3A @ 70C

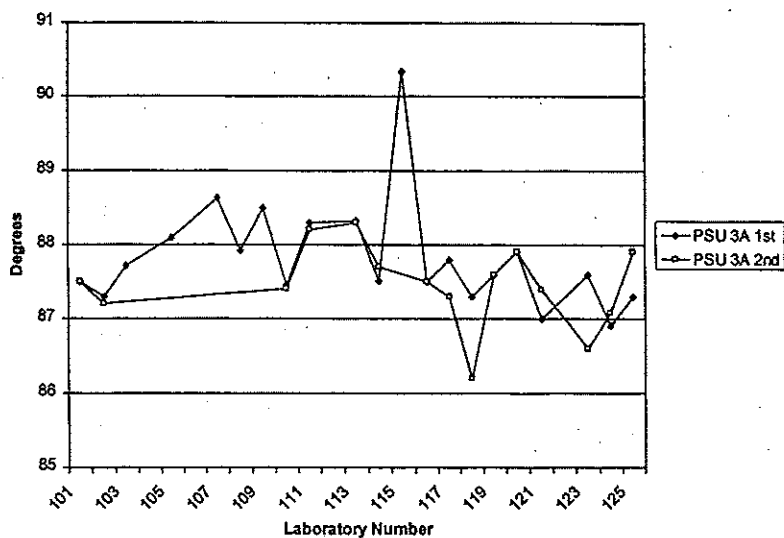
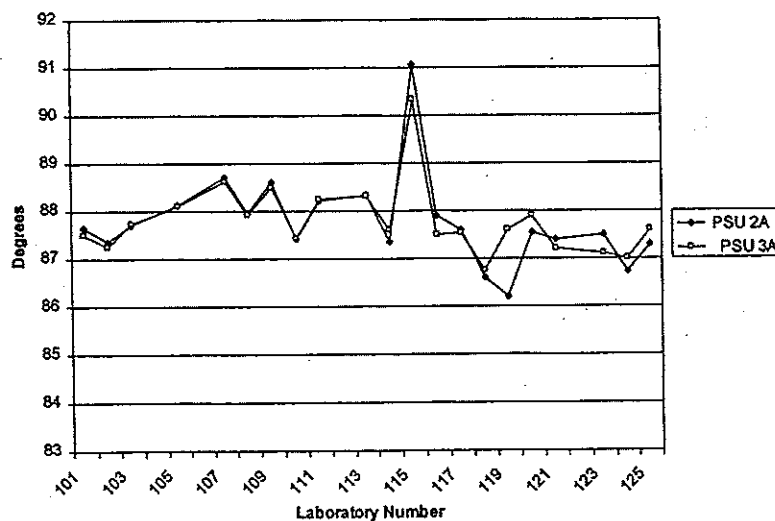




Figure 15

- PSU 2A & 3A @ 70C Average of both reported values - if two were reported
- Nineteen Labs with less than 0.5 degree difference
- Lab 115, only one test for each point

Figure 16, Phase Angle, Original 2A & 3A @ 70C



# Figure 16

■ Very little difference from lab-to-lab

## Figure 16, Phase Angle, Original Avg 1A & 4A, Avg 2A & 3A

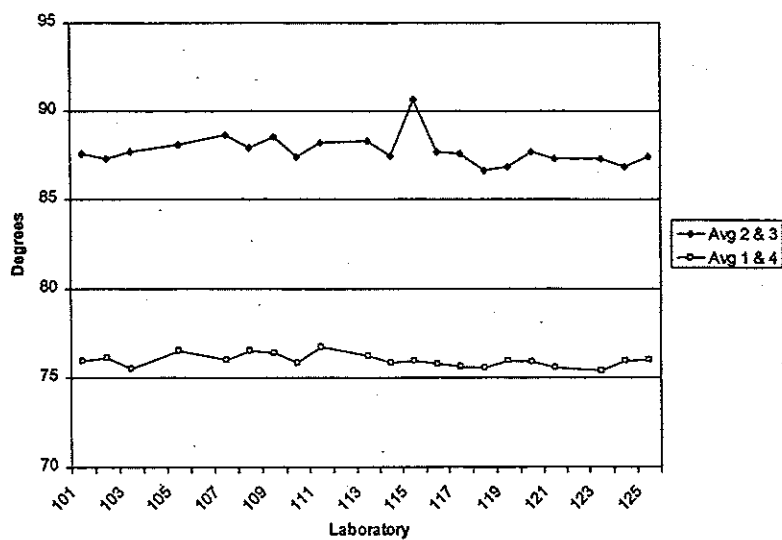


Figure 17

- Pre-molded Sample  $G^*$
- Only 11 Labs Participated
- For Lab 105, which data point is an outlier?
- Number 101 has two similar values but both are low

Figure 17, Premolded Sample  $G^*$

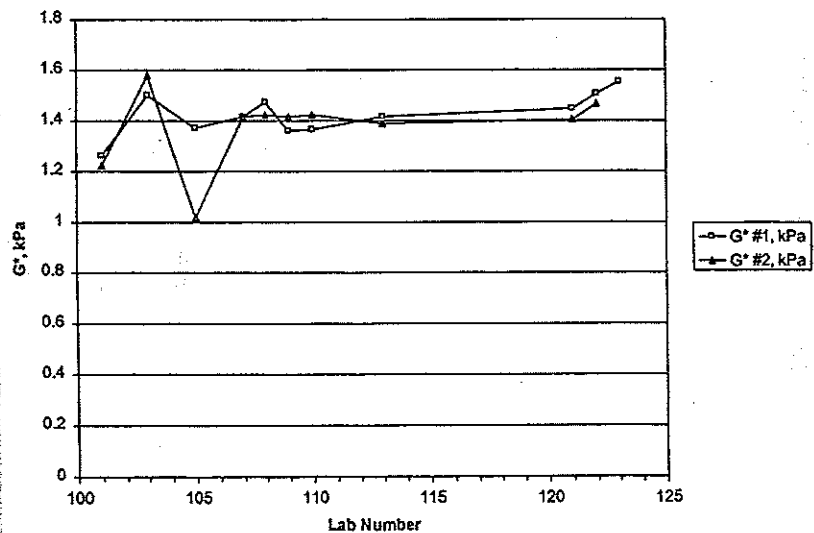
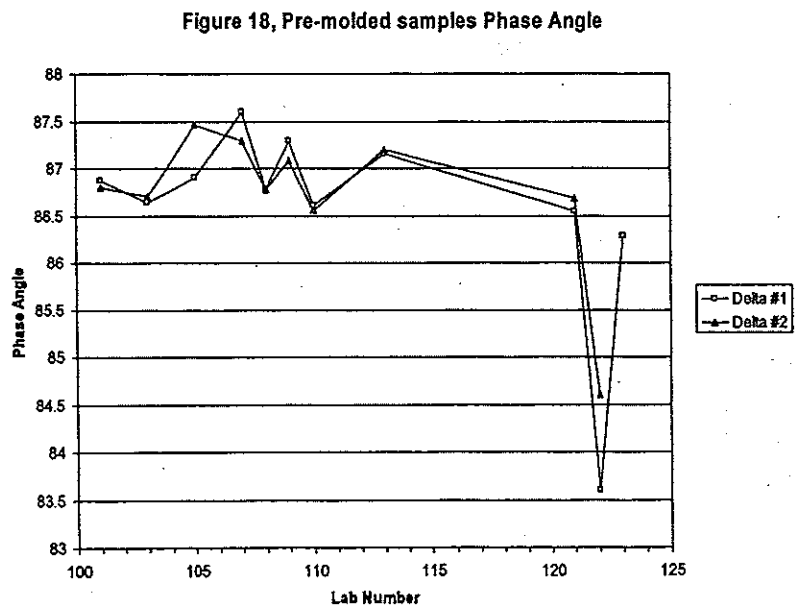


Figure 18

- Pre-molded Samples Phase Angle
- Both results from Lab Number 122 are Quite Low





## Summary Table

- Summation of all  $G^*$  for All Samples including the pre-molded samples
- All of the round robin samples had at least 3 labs with  $G^*$  more than 1 standard deviation away from the average - pre-molded samples had 1
- Every Round Robin sample had at least one lab more than 2 standard deviations away from the average - pre-molded none.



## DSR Results Summary

- Average  $G^*$  of Pre-molded samples approximately the same as original 1A & 4A @ 70 C and RTFO 2A and 3A - standard deviation of pre-molded samples less than any of these
- Repeated similarities in lab versus property curves indicate equipment differences.
- Random Differences Indicate Technique Differences

Figure 19

- BBR Stiffness 1A & 4A -18C @ 60 s
- Data very similar between labs except labs 109, 114 and 125

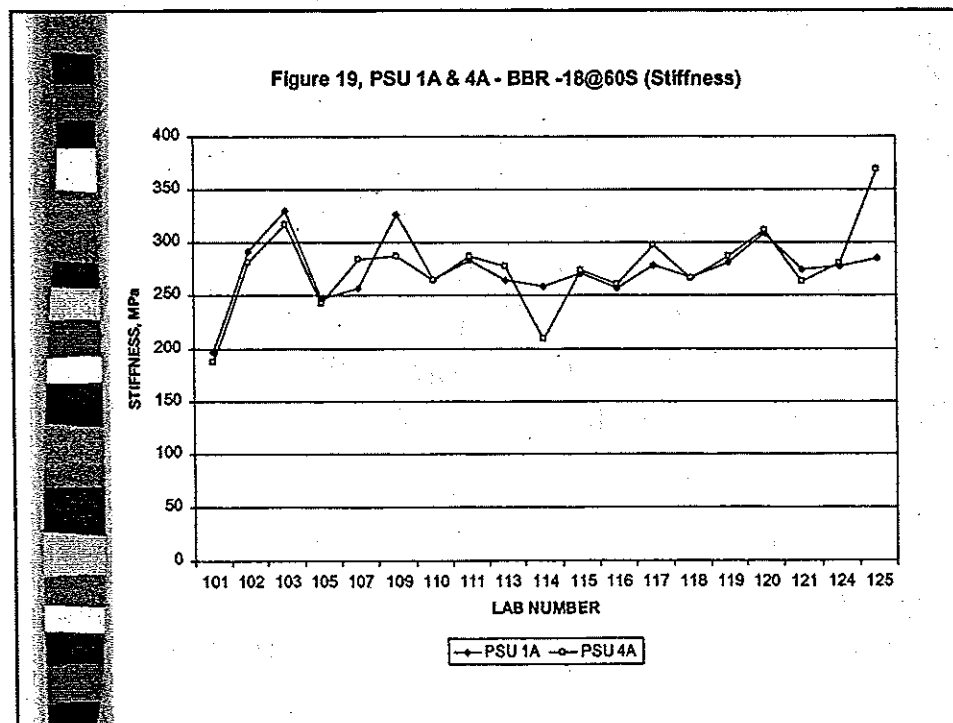


Figure 20

- BBR Average m-value PSU 2A & 3A - 12C
- Differences between labs very consistent
- For example lab 113 & 114 are low while lab 118 is high
- At -18C the within lab difference is small as well

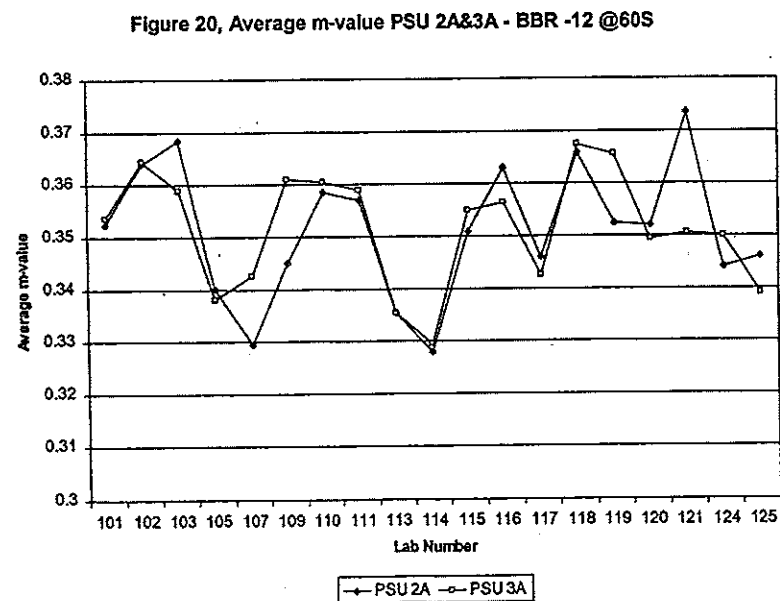


Figure 21

- BBR Stiffness 1A, 2A, 3A & 4A -12C @ 60s
- Note Similarity of curve shape from Lab 101 to Lab 110 & 115 to 124

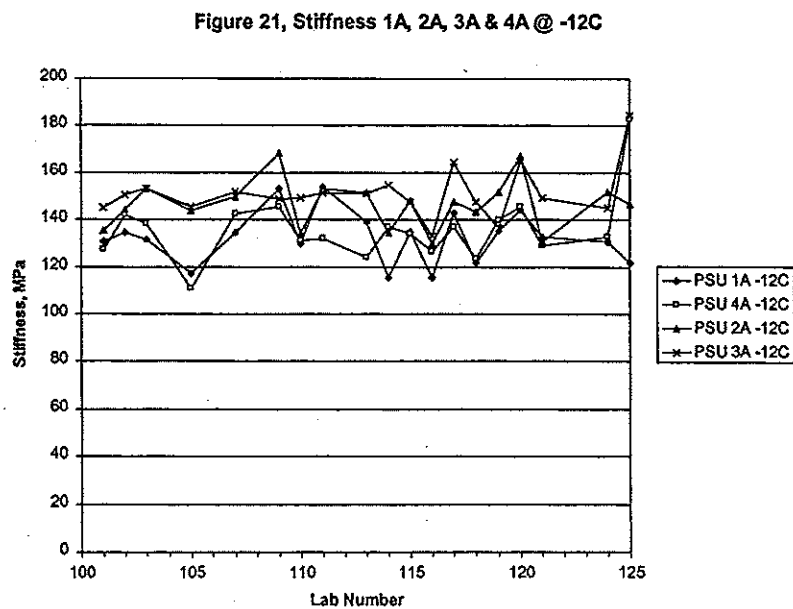




Figure 22

- BBR Stiffness 1A, 2A, 3A and 4A 12C @ 60s
- Differences between labs is similar throughout graph

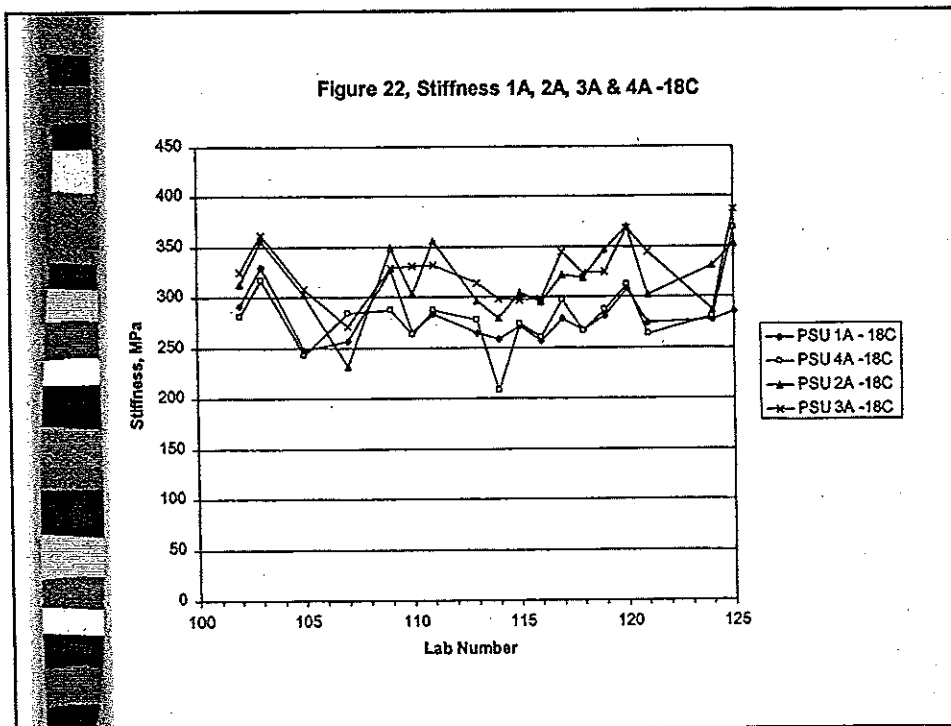
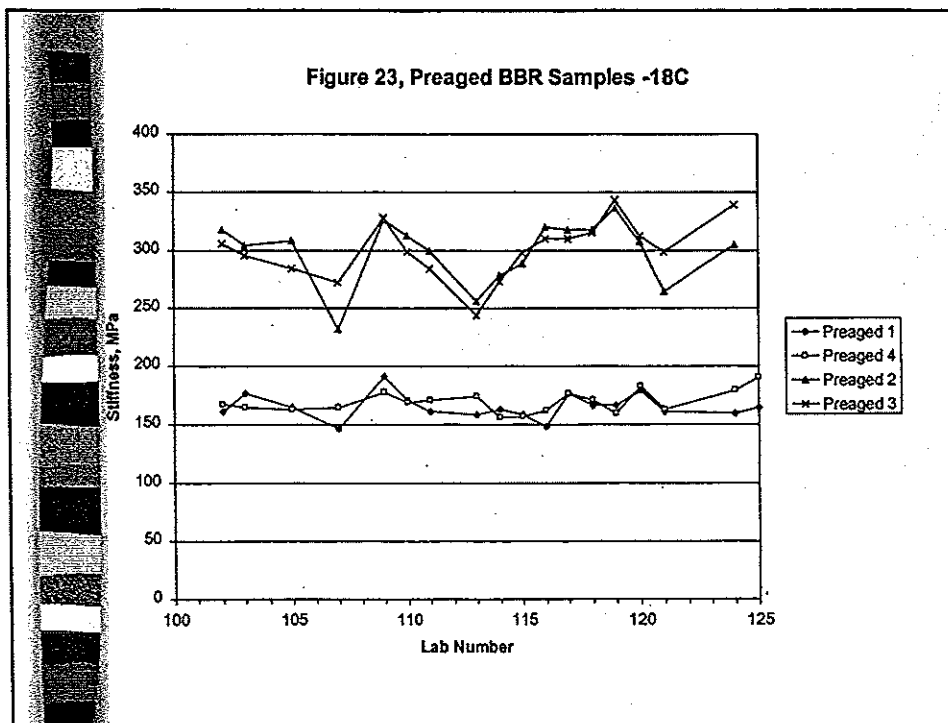
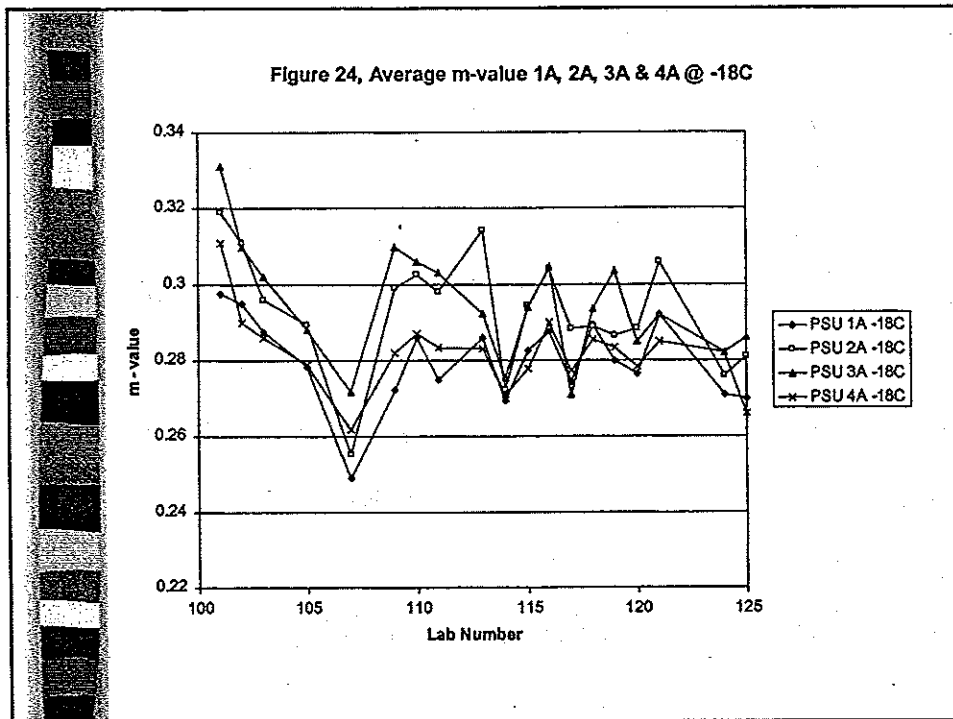


Figure 23

- BBR Stiffness Pre-aged Samples -18C @ 60 s
- Again lab-to-lab differences similar for all





NETC Binder Technician Workshop  
January 19, 1999  
Storrs, CT Attendance List

Name	Agency	Phone	Email
Alan Lugg	NH DOT	603-271-7932	
Nancy Tkach	NH DOT	603-271-7932	
Raymond Dulac	RI DOT	401-222-2524 X 4153	rdulac@dot.state.ri.us
Dave Clark	RI DOT	401-222-2524 X4153	dclark@dot.state.ri.us
Valerie Baseline	Sunoco	610-859-1896	valerie_a_baseline@sunoil.com
Nelio Rodrigues	CT DOT	860-258-0325	nelio.rodrigues@po.state.ct.us
Karissa Mooney	Hudson	401-781-5200	hudson@hudsoncompanies.com
Patrick J. Mitchell	Hudson	401-781-5200	hudson@hudsoncompanies.com
Denis Burt	Citgo	609-224-7420	dburt@citgo.com
Tom Waters	Citgo	609-224-7420	
Pete Hennessy	NYS DOT	518-457-5615	rmahoney@gw.dot.state.ny.us
Merlin Williams	ME DOT	207-941-4540	merlin.r.williams@state.me.us
Joseph Varhue	CT DOT	860-258-0341	
Lee C. Del Valle	CAP Lab	860-486-5956	

## Appendix VII

### Lab Visits – Equipment Comparisons Test Data

# CAP Lab Testing - Original Binder G\*

	Original G* @ 58C	Original G* @ 64C after 58C	Original G* @ 64 only
VT	2.976	1.298	1.218
	2.756	1.209	1.259
NH	2.951	1.305	1.259
	3.099	1.347	1.330
ME	3.162	1.41	1.369
	2.975	1.344	1.408
RI	2.784	1.354	1.347
	2.858	1.395	1.452
CT	3.221	1.402	1.419
	3.164	1.398	1.302
Excluding CT			
Average	2.945	1.333	1.330
St Dev	0.142	0.063	0.081
% Average	4.835	4.738	6.054
Including CT			
Average	2.995	1.346	1.336
St Dev	0.164	0.063	0.077
% Average	5.470	4.643	5.781

# CAP Lab Testing - Original Binder Phase Angle

	Original delta @ 58C	Original delta @ 64C after 58C	Original delta @ 64 only
VT	85.64	87.52	87.7
	85.80	87.66	87.77
NH	85.02	86.62	86.81
	84.59	86.56	86.56
ME	85.20	86.85	86.86
	85.28	86.90	86.91
RI	85.61	87.11	87.19
	85.56	87.13	86.92
CT	85.05	86.71	86.70
	85.01	86.68	85.77
Excluding CT			
Average	85.34	87.04	87.09
St Dev	0.40	0.39	0.43
% Average	0.47	0.45	0.50
Including CT			
Average	85.28	86.97	86.92
St Dev	0.37	0.38	0.57
% Average	0.44	0.43	0.66

# CAP Lab Testing - RTFO Residue G\*

	RTFO G* @ 58C	RTFO G* @ 64C after 58C	RTFO G* @ 64 only
VT	6.260	2.771	2.805
	6.637	2.893	2.734
NH	7.002	3.104	2.929
	6.843	2.993	3.015
ME	7.123	3.183	3.259
	7.122	3.097	3.13
RI	6.564	3.043	3.022
	6.694	3.096	3.066
CT	7.741	3.361	3.246
	8.032	3.534	3.486
Excluding CT			
Average	6.781	3.023	2.995
St Dev	0.301	0.133	0.170
% Average	4.434	4.415	5.681
Including CT			
Average	7.002	3.108	3.069
St Dev	0.541	0.218	0.224
% Average	7.723	7.023	7.299



# CAP Lab Testing - RTFO Residue Phase Angle

	RTFO delta @ 58C	RTFO delta @ 64C after 58C	RTFO delta @ 64 only
VT	81.39	83.96	84.04
	81.23	83.83	84.05
NH	80.58	83.19	83.29
	80.68	83.23	83.32
ME	80.78	83.35	83.38
	80.86	83.4	83.37
RI	81.37	83.72	83.77
	81.24	83.6	83.68
CT	80.47	83.13	83.27
	80.27	82.93	82.98
Excluding CT			
Average	81.02	83.54	83.61
St Dev	0.33	0.29	0.32
% Average	0.40	0.34	0.38
Including CT			
Average	80.89	83.43	83.52
St Dev	0.40	0.33	0.35
% Average	0.49	0.40	0.42

# CAP Lab Testing - BBR Testing

	Beam 1 Stiffness, MPa	Beam 1 m-value	Beam 2 Stiffness, MPa	Beam 2 m-value
VT	224.095	0.311	217.53	0.315
	218.39			
	221.15	0.316	224.04	0.320
NH	220.62	0.311	240.27	0.313
	198.91	0.313		
	218.33	0.309		
ME	223.09	0.310	214.4	0.307
	230.99	0.311	228.75	0.308
	213.45	0.309	224.58	0.307
RI	215.43	0.299	223.06	0.316
	230.34	0.312	220.49	0.306
	212.55	0.310	200.38	0.302
CT	210.89	0.298	195	0.306
	203.6	0.314	190.14	0.309
	199.29	0.305	199.86	0.313
Excluding CT				
Average	219.53	0.310	224.14	0.312
St Dev	8.76	0.004	7.88	0.005
% Average	3.99	1.421	3.52	1.618
Including CT				
Average	216.08	0.309	214.88	0.310
St Dev	9.91	0.005	15.27	0.005
% Average	4.59	1.647	7.10	1.632