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Committee D02 on PETROLEUM PRODUCTS AND LUBRICANTS

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April 10, 2006

Reply to:
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ASTM D02.B0.03 L-60-1 Surveillance Panel
Members and Guests:

Attached for your review and comment are the unconfirmed minutes of the November 2, 2005 L-60-1 Surveillance Panel meetings held at the PRI Headquarters, Warrendale, PA. Please direct any corrections or comments to my attention.

Sincerely,

Chris Schenkenberger, Chairman
L-60-1 Surveillance Panel

Attachments

Report of Meeting
L-60-1 Surveillance Panel
PRI Headquarters, Apollo Room, Warrendale, Pa.
November 02, 2005

Sign-in/Review of Membership: The meeting was called to order at 8:06am. The sign-in sheet is *Attachment 1*. A review of membership was not performed.

Meeting Agenda

In order to preserve enough time for the L-42-1 SP meeting, the L-60-1 SP meeting agenda (*Attachment 2*) focused on the TMC proposal for updating reference oil test targets. Items 3 and 5 were tabled for a future meeting.

Summary of Meeting Discussions

TMC Proposal for Updating Targets for TMC 148-1 and TMC 151-2

Mr. Lind presented the background to the TMC proposal for updating test targets. During the August L-60-1 Surveillance Panel (SP) meeting, the open action item of bringing TMC 133 into the system was revisited. The SP has historically desired a reference fluid that would yield an end of test viscosity increase near the SAE J2360 pass/fail limit of 100%. Significant laboratory differences were observed in the initial matrix conducted many years ago. The SP Chairman had noted that many significant findings from L-60-1 Task Force visits conducted in 2002 were uncovered. With these issues now addressed, his thought was that reproducibility in TMC 133 should be improved. However the TMC mentioned that current reference oil targets were a concern and should be addressed before bringing this into the system.

The TMC proposal for updating the reference bands is similar to methods taken in other industry test types that utilize the LTMS system. The proposal is based on a feeling that test targets are not applicable for the current situation of the L-60-1 test. The situation can be summarized as follows:

- An industry severity trend with average carbon varnish was observed in the 1999 time frame with TMC 148. During the severity trend, many changes were made to improve test precision but the cause of the severity trend could not be identified.
- Stand differences in severity, while still present, have been minimized.
- Industry severity has leveled off.

At present, the current reference fluids consist of TMC 148-1 and TMC 151-2 which were introduced in the same time frame during the severe trend. TMC 148-1 was introduced as a reblend to TMC 148. At the time, an industry matrix was conducted which showed TMC 148-1 to be slightly more severe than TMC 148 in some parameters. The L-60-1 SP elected to not issue new LTMS Shewhart reference acceptance bands for TMC 148-1 because new bands could give a false impression that a severity issue did not exist when looking at control charts. It was also thought to be wise to wait until the results from an L-60-1 Task Force conducting lab visits to investigate the severity trend were known.

Similar to TMC 148-1, TMC 151-2 was introduced as a reblend to TMC 151. However the TMC 151 was brought into the L-60-1 referencing system after the reference trend began. An acceptance matrix was conducted for TMC 151 but the L-60-1 SP used a pooled standard deviation across all oils for calculating LTMS Shewhart reference acceptance bands. This is a common approach that many surveillance panels will use to minimize the financial burden for labs to generate enough data to calculate reference acceptance bands with a new oil. The pooled standard deviation using all oils is typically replaced with the single oils standard deviation once 10 valid reference tests have been completed. The bands are then updated after 20 tests and locked after the n-size reaches 30 tests. Since TMC 151 was introduced in the L-60-1 during the severity trend, the pooled standard deviation across all reference fluids continues to be in use.

As previously documented, the average carbon varnish severity trend had been identified as starting in January of 1999. It appears that the severity has somewhat leveled off with respect to average carbon varnish especially. With the test being in control, the TMC proposed an option for the L-60-1 SP to consider if it is desired to update reference targets. The current and proposed targets are shown in ***Attachment 3***. The proposal involves a one time severity adjustment which uses the new reference bands as compared to the current reference bands. This is a method which surveillance panels have used in other test types within LTMS. The method allows changing reference targets and having a severity adjustment application for the subsequent stand reference period. This is a one time correction in the reference oil targets on TMC 148 to the 30 initial reference oil tests on TMC 148-1. The adjustment is for the 30 tests to get us back to the initial targets for future references and then use the appropriate severity adjustments for future candidates.

In order to understand the effect of the possible change, many questions surfaced from surveillance panel members on the mechanics behind the application of this one-time severity adjustment. As a way of gaining a better understanding for the SP, Mr. Buitrago commented that it might be wise to take the most recent 30 reference tests and plot them in the LTMS control charts by the current and proposed reference bands. This should be done for all parameters (sludge, average carbon varnish, viscosity increase, pentane and toluene). Test lab representatives voiced concerns over the affect of new reference bands on the pentane and toluene reference results. Particularly for one lab, Mr. Lind felt concerns with pentane, toluene, and viscosity are valid. In general, the SP wants to fully understand the impact of this change. The SP requested the following action item for the TMC to help in their level of understanding the proposed changes to the reference bands.

Action Item: TMC to chart all the reference data since August 1, 2005 with the proposed bands and identify references tests that would now fail under the new targets. The data is also requested to be shown in non-transformed units (merit numbers) with an estimate of a possible stand severity adjustment. In this hypothetical case, an example of how the severity adjusted reference might affect a future candidate test was also requested.

Procedural Clarification to the annex of D5704

Mr. Lyle Bowman had requested the SP resolve an issue which he identified in annex A10.9 of the D5704. Mr. Bowman's request, which was addressed to Mr. Lind in an e-mail, is ***Attachment 4***. The

issue is in regards to clarifying the only varnish rating scales as being scales A, B, or C for non-rubbing parts in CRC Manual No. 20.

Motion 1 (Motion ⇒ Mr. Sullivan, Second ⇒ Mr. Layton)
Revise D5704, annex A10.9 to read as follows: Use any of the three CRC Rating Scales (A,B, or C) for Non-Rubbing Parts in CRC Manual No. 20 to determine the varnish rating.

Motion Results: Passed
In favor: 3
Opposed: 0
Abstain: 1

Automated Kinematic Viscometers and the ASTM D445
For the purpose of general awareness, the SP chairman notified the L-60-1 SP of a letter ballot for changes to the ASTM D445-04, Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids. This is Item 10 in Ballot D02.07 and is included as ***Attachment 5***. The D445 test method is used as a subtest for calculating the viscosity increase for the L-60-1 D5704 ASTM test method. Much of the former D445 method was written around the use of manual instruments and the ballot provides additions specific to automated kinematic viscometers.

Some L-60-1 SP members were aware of the ballot and could not guess whether the changes would affect results for L-60-1 viscosity increase. The SP chairman mentioned that this change would be documented in the meeting minutes and the LTMS system used to monitor any outcomes. If necessary in the future, the SP also discussed the possibility of requesting the TMC to contact the analytical laboratory managers to schedule future lab audits. As was reminded by the TMC, a motion from the L-60-1 SP would be required for this to happen because the D445 is not a test that falls underneath the scope of the TMC. For this to happen, the subcommittee overseeing the D445 (ASTM Subcommittee D02.07.A) would need to contact the Test Monitoring Board (TMB) to request services from the TMC. The mechanisms for requesting the TMC as a resource was only discussed and the SP chose not to pursue action on contacting D02.07.A representatives or the TMB.

The meeting was adjourned at 9:30 am (Mr. Layton/Mr. Bartlett).

Respectfully submitted,

Chris Schenkenberger
L-60-1 Surveillance Panel Chairman

ASTM L-60-1 Surveillance Panel Membership/Mailing List

Meeting Date: November 2, 2005

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Attachment 1
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ASTM L-60-1 Surveillance Panel Membership/Mailing List

Meeting Date: November 2, 2005

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Attachment 1
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ASTM L-60-1 Surveillance Panel Membership/Mailing List

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Attachment 1
Page Page 5 of 6
Reference L-60-1

ASTM L-60-1 Surveillance Panel Membership/Mailing List

Meeting Date: November 2, 2005

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Attachment 1
Page Page 6 of 6
Reference L-60-1

L-60-1 Surveillance Panel

November 2, 2005
PRI Apollo Room – Warrendale, PA

Agenda

I. Call to order/Review Membership

II. Review agenda

III. Review LTMS Charts after Recent Data Dictionary Change

IV. TMC Proposal for Updating Targets for TMC 148-1 and TMC 151-2

V. TMC 133 Reference Fluid

VI. New Business

VII. Adjournment

REFERENCE OIL 148-1 (30 Tests)				
	Current Targets		Updated Severity Adjusted Targets	
	Mean	S.D.	Mean	S.D.
Average Carbon Varnish	1.59	0.47	1.44	0.28
Average Sludge	0.76	0.19	0.73	0.21
Viscosity Increase	3.61	0.15	3.70	0.07
Pentane	-0.95	0.39	-0.73	0.28
Toluene	-1.36	0.49	-1.12	0.49

REFERENCE OIL 151-2 (30 Tests)				
	Current Targets		Updated Severity Adjusted Targets	
	Mean	S.D.	Mean	S.D.
Average Carbon Varnish	1.81	0.40	1.66	0.45
Average Sludge	0.54	0.23	0.47	0.13
Viscosity Increase	3.62	0.15	3.57	0.10
Pentane	0.75	0.37	0.69	0.17
Toluene	0.26	0.50	0.19	0.26

Attachment 3
Page 1 of 1
Reference L-60-1

-----Original Message-----
From: Lyle Bowman [mailto:jbfoodie@comcast.net]
Sent: Monday, October 31, 2005 10:57 AM
To: Don Lind
Subject: possible clarification of A10.9

Don,

Assuming that there are no other A, B, or C Scales in CRC Manual 20, if the phrase "rust/varnish/lacquer" was deleted from A10.9, there would be no possible confusion or inferences drawn (as I did). What do you think about the following revised A10.9?

A10.9 Use any of the three CRC Rating Scales (A,B, or C) for Non-Rubbing Parts in CRC Manual No. 20 to determine the
varnish rating.

Lyle

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ITEM 10

To: Members of Committee D02 and Subcommittee D02.07

Attached for letter ballot are changes to **ASTM D 445-04$^{\varepsilon 1}$, Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)** (Work Item WK9028). These changes provide additions specific to automated kinematic viscosity measurement in the method. Many commercially available automated kinematic viscometers employ sensing and timing technologies which exceed the capabilities and performance of manual instruments. However, much of the current D 445 method is prescribed around the use of manual instruments. The prose being presented in this ballot is intended to provide additional guidance to users with automated equipment on the use of that type of equipment in making viscosity measurements per D 445.

For many years, users of automated D 445 kinematic viscometers have reported the successful use of measurement efflux times significantly less than 200 seconds; the prescribed minimum in D 445. ASTM D02.07.A was tasked (Task Group 7A.5) with evaluating the effects (if any) of using flow times below this minimum threshold of 200 seconds. Approximately a year and half ago, a set of data was collected using both automated capillary instruments and manual viscometers. The flow times for automated capillary instruments ranged from a little less than 20 seconds to a little over 200 seconds. The manual measurements had flow times ranging from 200 seconds to approximately 800 seconds. The primary purpose of this study was to determine the minimum allowable flow time for automated equipment and whether there was any bias relative to manual determinations.

In summary, an analysis of variance was done on this set of data by an independent statistical consultant using *Minitab*® software to fit a general linear model to determine if a relationship exists between percent difference from reference viscosity (PDfRV) to average flow time and instrument. The analysis showed a significant relationship between instrument and PdfRV. Upon review of the plotted data, significant differences were seen for a number of manual measurements. {Data is included in the SC7A minutes for the June 2005 meeting.} These differences compound the fact that the data set is unbalanced. Unfortunately, this data and analysis does not provide sufficient information to confirm (nor refute) any apparent bias between manual and automated measurement techniques.

The Task Group then turned to evaluating any apparent bias between measurements made on automated capillary instruments with sample efflux times ranging between 35 and 200+ seconds.

This analysis was conducted using a liner model which showed any bias between measurement efflux time and viscosity to be in fact insignificant. The relationship developed from this analysis is presented below:

Percent Difference from Reference Viscosity = 0.013 – 0.0000512 * Flow time

Further analysis of the data set using ASTM E691, showed poorer repeatability than that currently published in the method. However, the study was not properly designed to fit the precision calculations by E-691.

As a result, SC 7A formed a new Task Group (7A.13) to reassess the precision of D 445 when measuring the viscosity of base stocks. The scope of the project is to include automated/automatic as well as manual instruments.

Rationale:

This ballot was initiated by Task Group 7A.5 under the direction of D02.07 section A (work item WK5226).

Technical Contact(s):

Janet Lane, (856) 224-3302, Fax: (856) 224-3616, janet.l.lane@exxonmobil.com
Ken Henderson: (814)353-8000 x265, fax: (814)353-8007, kenohenderson@worldnet.att.net

Precision and Bias Section: This change has no impact on the precision and bias section.

Recent Ballot History:

This new ballot contains the original balloted text with revisions to address negative voters concerns as was previously balloted in April 2005 (45 Affirmatives, 2 Negatives, 58 Abstentions). This prior text was balloted in conjunction with a research report which was subsequently withdrawn.

Research Report: None is required as there is no change to the method precision and bias section.

Detailed summary of changes to D 445-04$^{\varepsilon 1}$: see attached

Sincerely,

Fred W. Girshick, Secretary, D02.07

C. Patrick Maggi, Chairman, D02.07A

Text changes to D 445-04 to accommodate automated viscometers and short flow times.

Revised – August 10, 2005

Only new paragraphs and those with changes are shown

6. Apparatus

Add following:

Note xx—Guidance is given in Appendix xx to determine whether an automated viscosity assembly meets the precision given in Section 17.

Add following:

6.1.2.1 *Automated Capillary Assemblies*—shall be designed in a manner which mimics the physical geometries or operational characteristics of the manual viscometers described in D 446. Specifically, the viscometer shall be oriented vertically and contain one or more timing bulbs, a working capillary, fixed timing locations or timing sensors for detection of the sample meniscus. Typical automated sensor technologies may employ thermal or optical means for detection of the liquid meniscus. The design of the viscometer shall be such as to allow the liquid sample under test to flow freely (impeded only by the restriction of the working capillary) and in a vertical orientation such that the only motivating force is the hydrostatic head developed by the column of sample. The pressure above the column of sample and at the exit of the viscometer tube shall be equal (typically at atmospheric pressure). The capillary diameter and length shall be appropriate for this hydrostatic head and the kinematic viscosity of the liquid being measured. The tubes shall be designed so that a Reynolds number is always less than 300 over the tube range.

Revise following:

CURRENT 6.5:

6.5 *Timing Device*—Use any timing device that is capable of taking readings with a discrimination of 0.1 s or better and has an accuracy within 0.07 % (see Annex A3) of the reading when tested over the minimum and maximum intervals of expected flow times.

PROPOSED REVISION 6.5:

6.5 *Timing Device*—For manual determinations, use any timing device that is capable of taking readings with a discrimination of 1 part in 2000 or better of the minimum flow time, and has an accuracy within ±0.07 % (see Annex A3) of the reading when tested over the minimum and maximum intervals of expected flow times. For a flow time of 200 seconds the discrimination would be 0.1 seconds. For an automatic or automated instrument, the same clock timing device shall be used for both calibration and measurement. For flow times of less than 200 sec, the discrimination shall be 1 part in 2000.

9. Calibration and Verification

Proposed addition:

9.4 Automated Capillary Assemblies– for those assemblies where the manufacturer's instructions require the use of calibrated viscometers, use 9.1. Alternatively, for those automated capillary assemblies which require the glass capillary tubes (viscometers) to be calibrated in situ in the apparatus, follow the operating instructions specified by the manufacturer in order to carry out the following calibration procedure.

9.4.1. When flow times are greater than 200 s. or the kinetic energy factor is insignificant, select at least two certified viscosity reference standards, covering the range of each tube or bulb to be calibrated

9.4.1.1 Measure two successive flow times, to the nearest 0.1 s, if the flow times agree within 0.2%, calculate the viscometer constant, C, as follows:

$$C = \nu/t \qquad (3)$$

Where

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v is the kinematic viscosity, mm^2/s, for the certified viscosity reference standard;
t is the mean flow time, in seconds.

9.4.1.2 Repeat with a second certified viscosity reference standard whose flow times are at least 50% different than the first standard. If the two values of C or if the two values of t differ by less than 0.2 %, for direct flow viscometers or 0.3% for reverse flow viscometers, use the average C as the viscometer constant for the viscometer being calibrated. If the constants or times differ by more than this value, repeat the procedure, taking care to examine all possible sources of errors.

9.4.2 Where the automated viscometer is calibrated with flow times less than 200 seconds or the kinetic energy factor is not insignificant, follow the operating instructions specified by the manufacturer in order to carry out the following calibration procedure.

9.4.2.1. Select at least three certified viscosity reference standards, covering the range of each tube to be calibrated.

9.4.2.2 Measure two successive flow times, to the nearest 0.01 s (at least 1 part in 2000) for each of the certified viscosity reference standards. The flow times shall agree within 0.2%. Using the several values of flow-time and certified kinematic viscosity use ASTM D 446 to determine C and E or perform a regression to determine the best value of C and E as follows:

$$v = C*t - E/t^2 \quad (3)$$

Where

v is the kinematic viscosity, mm^2/s, for the certified viscosity reference standard;
t is the mean flow time, in seconds.
E is the kinetic energy coefficient, in mm^2s

9.4.2.3. The automated viscometer may determine these values automatically. Kinetic energy correction is not permitted to exceed 4% of the measured viscosity.

Note xx -- Where the kinetic energy term, E / t^2, is less than 0.05% of the certified viscosity reference standard, the flow-time data may not allow a value of E to be determined and the value of the kinetic energy term will be insignificant. In this case, only two certified standards are needed to perform the calibration.

9.5 Automated viscometers may be verified using certified viscosity reference standards as described in 9.1 to 9.2.2,

9.6 Use the determined values of C, E and flow times in the above equation to calculate the kinematic viscosity for each flow time. If the calculated viscosity differs by less than 0.2 % from the certified values, use the C and E values for the viscometer being calibrated. If the viscosities differ by more than 0.2 %, repeat the procedure, taking care to examine all possible sources of errors.

10. General Procedure for Kinematic Viscosity

Revised following:
CURRENT 10.2:
10.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time shall not be less than 200 s or the longer time noted in Specifications D 446.

PROPOSED REVISION 10.2:
10.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). For manual viscometers, the flow time shall not be less than 200 s or the longer time noted in Specifications D 446. For automated viscometers that meet the description in Section 6.1.2, the minimum flow time shall be greater than 35 s provided

that the kinetic energy correction (see ASTM D 446, section 7.23) is used where applicable and all precision criteria are met at the minimum flow time established for the automated instrument.

Add NEW Section 13

13. Procedure for Automated Viscometers

13.1 Sections 6 through 12 apply both to manual and automated viscometers. Sections 13 through xxxx apply to automated viscometers. Follow Section 11 or Section 12 as appropriate for the design of the automated viscometer.

13.1.1 The general procedures in Sections 10 and 12 are followed, taking note that the automated viscometer may automatically select the timing bulb(s) and have a special automatic timing procedure (see section 6). Cleaning procedures may also differ; follow the manufacturer's instructions. Ensure that the automated viscometer is designed to be used for the type of material(s), temperature range(s) and kinematic viscosity range selected. For example, the automated viscometer may not be suited for operating at –20°C for Jet Fuels, unless the viscometer is specially designed for this type of low temperature kinematic viscosity measurement.

13.2 The viscometer is charged in the manner dictated by the design of the instrument in conformity with that employed when the instrument was calibrated. If the sample is thought or known to contain fibers or solid particles, filter through a 75 μm screen, either prior to or during charging (see Specifications D 446).

NOTE xx—To minimize the potential of particles passing through the filter from aggregating, it is recommended that the time lapse between filtering and charging be kept to a minimum.

13.3 Depending on the design of the meniscus sensors and the nature of the test sample (e.g., transparent or opaque liquid) the procedures in Section 11 or Section 12 may apply.

13.3.1 With certain products which exhibit *gel-like* behavior, exercise care that flow time measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameters.

13.4 Allow the charged viscometer to remain in the bath long enough for the sample to reach the test temperature. Depending on the design of the automated viscometer the time required for the sample to reach the bath temperature may vary from less than one minute to 30 minutes.

13.4.1 This time will vary for different instruments, temperatures, materials, and kinematic viscosities. Establish a safe equilibrium time by trial.

13.5 Where required by the design of the viscometer, adjust the volume of the sample to the mark (sensor) while the sample reaches temperature equilibrium.

13.6 With the sample flowing freely under gravity, the automatic assembly records in seconds to within 0.1 s (1 part in 2000) for flow times greater than 200 s or 0.01 s for flow times less than 200 s, the time required for the meniscus to pass from the first to the second timing mark (sensor). If this flow time is less than the specified minimum (see 10.2), a capillary of smaller diameter is selected and the operation is repeated.

13.6.1 Repeat the procedure described in 13.6 to make a second measurement of flow time. Record both measurements.

13.6.2 From the two measurements of flow time, calculate two determined values of kinematic viscosity.

13.6.3 If the two determined flow times or values of kinematic viscosity calculated from the flow time measurements agree within the stated determinability figure (see 17.1.1) for the product, use the average of these determined values to calculate the kinematic viscosity result to be reported. Record the result. If not, repeat the measurements of flow times after an investigation and correction of possible sources or error. A thorough cleaning and drying of the viscometers and filtering (where required, see 13.2) of the sample may be necessary to obtain the calculated kinematic viscosity determinations within the stated determinability.

13.6.4 If the material or temperature, or both, is not listed in 17.1.1, for temperatures between 15 and 100°C, use as an estimate of the determinability 0.20%, and 0.35% for temperatures outside this range.

(Note: Renumber current sections 13 through 19 to 14 through 20)

20. Summary of Changes

20.1 Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 445-04$^{\varepsilon}$) that may impact the use of this standard.

20.2 Revised and added Sections relevant to automated viscometers: Section 6, 9, 10, 13.

APPENDIX

Add NEW Appendix X1

X1. Automated Viscometer Guidance

(Non-Mandatory Information)

X1.1 To demonstrate that an automated viscometer meets the precision of ASTM D 445, three criteria are reviewed as follows based on the type of material(s), kinematic viscosity, flow time and temperature range:

Determinability (precision section)

Repeatability (precision section)

Reproducibility (precision section)

X1.2 Test for Determinability: To meet the requirements of ASTM D 445, two measurements of the flow time are performed. The determinability can be determined, and compared to the determinability section of the D 445 precision. If the flow times do not meet the test determinability, the test is merely to be repeated. The automated viscometer software may perform this test automatically. For some materials, as many as five successive measurements may be required until two measurements are found to meet this requirement.

X1.3 Test for Repeatability: Determine the specific type of materials, flow-time (or viscosity) range and temperature for the automated viscometer. Select four or more samples of the material encompassing the flow-time (or viscosity) range and run each at least four times in the automated viscometer. The precision of the repeatability can be determined, and compared to the repeatability section of the D 445 precision.

X1.4 Test for Reproducibility: In a manner similar to X1.3, run the automated viscometer with certified viscosity reference standards (internal and/or traceable to NIST or other recognized NMI). Select four or more samples of the material encompassing the flow-time (or viscosity) range and run each at least four times in the automated viscometer. The precision of the reproducibility can be determined, and compared to the reproducibility section of the D 445 precision.