METHOD OF TESTS FOR HIGH FLOAT EMULSIFIED ASPHALT

1. SCOPE

1.1 This method applies to liquid asphaltic materials in the form of aqueous emulsions of the anionic high float type.

1.2 High float emulsified asphalt consists essentially of paving asphalts dispersed in an aqueous phase. It may contain a light petroleum distillate. The residual bitumen has non-Newtonian flow characteristics. It exhibits resistance to flow, regardless of penetration of the asphalt.

2. RELEVANT DOCUMENTS

2.1 ASTM D 5

2.2 ASTM D 88

2.3 ASTM D139

2.4 ASTM D 244

2.5 ASTM D 2042

2.6 ASTM D 2171

2.7 ASTM E 1

2.8 ASTM E 77

3. PROCEDURE

3.1 Procedure of ASTM from each one of the relevant documents above shall be followed, except as noted below.

4. TEST METHOD EXCEPTIONS

4.1 RESIDUE BY DISTILLATION

4.1.1 Use an aluminum-alloy still only as outlined by ASTM D 244, a still with an expansion chamber, a 127 mm ring burner and a thermometer with a total length of approximately 500 mm.

The 127 mm adjustable burner is placed midway up the still at the expansion chamber joint. The amount of heat applied by this burner is regulated to prevent overheating.

4.1.2 Apply heat so that the first drop of distillate is collected within 10 ± 2 min. in the receiver. Continue heating until the temperature rises to approximately 215°C. At this point, move the ring burner approximately level with the bottom of the still, and increase the temperature to 260 ± 5°C maintaining it at this temperature for 15 min. Complete the total distillation in 60 ± 15 min. from the first application of heat.

4.1.3 When pouring the residue from the still, it is essential that contents of the still be swirled for 10 s just prior to pouring. After completing the distillation procedure, the contents of the still must be poured out within 3 min. into the other test apparatus required for further testing (penetration can, 30 mL glass beaker, and float test thimbles). Such test apparatus should be readied and preheated to 260°C (Figure 1), except float test thimbles, beforehand so that the residue and still do not cool below 232°C before pouring. The residue shall not be poured through the sieve as recommended in ASTM.

4.2 PENETRATION

4.2.1 Follow procedure of ASTM D 5, except that: The sample container shall be 55 mm in diameter and 70 mm deep. For penetration above 350, a special penetration needle is required. The needle shall meet all the requirements of ASTM D 5 under Penetration Needle for dimensions and mass except that the minimum exposed length of the needle shall be 50 mm.

4.2.2 Pour the residual asphalt into the preheated penetration container , filling approximately one-half of the container, and then pour approximately 25 mL into a second preheated pouring container. The second container is then used to fill float thimbles and viscosity tubes. Pour remaining residue into the penetration container to fill it to a depth of approximately 60 mm. Immediately remove penetration can from the hot plate, and place on a paper towel on top of approximately 20 mm thick plywood, all on a counter top. Cover the can with a 600 mL, low form beaker with spout.

4.2.3 These containers should be on a hot plate so that there is no jelling of the high float residue. Split sample into containers directly from still as shown in Fig. 1. After pouring into penetration container, do not stir sample.

4.2.4 Follow the general procedure for the penetration test as outlined in ASTM D 5, except that time for penetration sample to cool shall be 1.5 to 2 h in air, and 1.5 to 2 h in water.

4.3 FLOAT TEST

4.3.1 Follow the general procedure for the float test as outlined ASTM D 139, except that the residue from the distillation shall be poured into a 30 mL glass beaker, and not through a 300 µm sieve, and then immediately into the float thimble. If the residue has been allowed to cool, it shall be reheated to 260°C, and poured into the collar. It may be necessary to cap off the collar with a second pour because of possible shrinkage. Float test should be completed on the same day as the distillation test.

5. APPARENT VISCOSITY OF HIGH FLOAT RESIDUES

5.1 SCOPE: This method describes procedures primarily designed to determine apparent viscosities of residues obtained by distilling High Float Emulsified Asphalts.

The test is carried out at 60°C. It is applicable to materials having viscosity in the range from 20 to 3000 + Pa·s. The modified Koppers Viscometer is the instrument used for the determination of the apparent viscosity.

5.2 DEFINITIONS: Viscosity is the resistance to deformation or internal friction of a liquid expressed as the ratio of the shear stress to shear rate, whether this ratio is constant or not. The SI unit of viscosity is 1 Pa·s (PASCAL-SECOND), and is equivalent to 10 P (Poise).

5.3 SUMMARY OF METHOD: The time is measured for a fixed volume of the liquid to be drawn up through a straight open-end capillary tube by means of vacuum, under closely controlled conditions of vacuum and temperature. The apparent viscosity in Poise is calculated by multiplying the flow time in seconds by the appropriate viscometer calibration factor or calculated viscometer constant. This value divided by 10 will give the apparent viscosity in Pascal seconds (Pa·s).

5.4 APPARATUS

5.4.1 Calibrated-Modified Koppers Vacuum Viscometers And Holders: Required viscometer sizes: 25, 50, 100, 150, 200, 400 (Fig. 2).

5.4.2 Thermometers: Calibrated liquid-in-glass thermometers ASTM 47C in accordance with the requirements of ASTM E-1.

5.4.3 Constant Temperature Bath: A bath suitable for immersion of the viscometer so that the liquid reservoir or the top of the capillary, whichever is uppermost, is at least 20 mm below the upper surface of the bath liquid and with provisions for visibility of the viscometer and thermometer. The efficiency of the stirring and the balance between heat losses and heat input shall be such that the temperature of the bath medium does not vary by more than ± 0.03°C over the length of the viscometer or from viscometer to viscometer in the various bath positions at 60°C.

5.4.4 Vacuum System: A vacuum system capable of maintaining a vacuum to within ±0.5 mm of the desired level up to and including 300 mm Hg. The essential system is shown schematically in Fig. 3. A vacuum pump is suitable for the vacuum source.

5.4.5 Timer: A stopwatch or other timing device graduated in divisions of 0.1 s or less, and accurate to within 0.05% when tested over intervals of not less than 15 min.

5.4.6 Beakers: 30 mL, low form with spout.

5.4.7 Aspirator

5.4.8 Vacuum Tubing

5.4.9 Bath: Capable of maintaining temperature at 135 ± 10°C.

5.4.10 Graph Paper: Full logarithmic 1 x 1, 2 x 2 cycles.

5.4.11 Sample Preparation Oven: A suitable oven for semi-continuous operations with control of temperature up to 202 ± 2°C. It should have a fast heating rate capability in order not to delay testing when needed on short notice, and capable of maintaining temperature at 195 ± 2°C (Fig. 6).

5.5 PREPARATION OF SAMPLE

5.5.1 Pour a suitable portion of the total distillation residue at 260°C into a 30 mL beaker, and allow to cool to 180 ± 5°C. Stir this portion of the sample at 1 rev/s for 10 s. At the end of a 10 s period, charge the visometer filling tube as in 7.6.

5.5.2 If the residue is at room temperature, cut a test sample of about 20 to 35 g from the source container with a warm spatula, and place in an oven at 195 ± 2°C, and heat until contents are at 180 ± 5°C. Stir the sample at 1 rev/s for 10 s. At the end of a 10 s period, charge the visometer filling tubes as in 3.7.

5.5.3 If the residue does not have sufficient fluidity at 180 ± 5°C, it may be heated to minimum temperature necessary to achieve fluidity for pouring. This temperature must then be reported with the test data. Select the viscometer size that will be suitable for the material to be tested.

5.6 TEST METHOD

5.6.1 Select a calibrated clean dry viscometer that will give a flow time between 50 and 200 s for the Bulb C.

5.6.2 An anticipated MK Viscometer required is as follows:

HF-250-s	-	size 50
HF-150-s	-	size 100, 150 or 200
HF-100-s	-	size 150, 200

5.6.3 Maintain the bath at the test temperature to within ± 0.03°C.

5.6.4 Apply the necessary corrections, if any, to all thermometer readings.

5.6.5 Insert the viscometer in a holder, and place the viscometer filling tube and capillary tube separately into an oven which is maintained at 195 ± 2°C, and preheat for 5 min. to assist in eliminating air bubbles when the sample is poured.

5.6.6 Pour the prepared sample slowly from the 30 mL beaker into the viscometer filling tube to within ± 2 mm of the fill line.

5.6.7 Allow the charged viscometer filling tube to stay in an oven maintained at 195 ± 2°C for a period of 10 ± 2 min. to allow large air bubbles to escape.

5.6.8 Remove the viscometer filling tube and capillary tube from the oven, and then properly position the capillary tube into the filling tube (Note 1).

5.6.9 Transfer the viscometer into an oil bath maintained at 60 ± 0.03°C, and position the viscometer in the bath so that the uppermost timing mark is at least 20 mm below the surface of the bath liquid.

5.6.10 Establish a 300 ± 0.5 mm Hg vacuum in the vacuum system, and connect the vacuum system to the viscometer with the toggle valve or stopcock closed in the line leading to the viscometer.

Note 1: Capillary action on certain small diameter viscometer tubes may cause a rise of the sample above the first timing mark. If this is a problem, the capillary tube should be withheld from contact with the sample for a few minutes prior to running the test, then positioned quickly, and allowed to equilibrate a minute or two.

5.6.11 After the viscometer has been in the bath for 30 ± 5 min., start the flow of asphalt in the viscometer by opening the toggle valve or stopcock in the line leading to the vacuum system.

5.6.12 Measure, to within 0.1 s, the time required for the leading edge of the meniscus to pass between successive pairs of timing marks.

5.6.13 Upon completion of the test, remove the viscometer from the bath, and place it in an oven or bath maintained at 135 ± 5°C. Clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent (Note 2). Dry the tube by passing a slow stream of filtrated dry air through the capillary for 2 min., or until the last trace of solvent is removed.

Note 2: A mixture of equal parts of stoddard solvent and 1.1.1 trichloroethane is recommended. Withdraw specimen and solvent with the aid of the vacuum, flushing more solvent until tube is clean. Rinse with acetone.

5.7 REPORTING OF RESULTS

5.7.1 It is suggested that a form sheet, as shown in Figure 4, be used to record the measured and calculated data.

5.7.2 Viscosity for each bulb is calculated as follows:

$$V = (t \times f) / 10$$

where: V = viscosity in pascal seconds, Pa·s
t = fill time for bulb in question, s
f = viscometer constant, P/s

5.7.3 Shear rate is calculated for each bulb as follows:

$$SR = \frac{SC}{t}$$

where: SR = shear rate in reciprocal seconds, s^{-1}
SC = shear constant
t = fill time for bulb in question, s

5.7.4 Plot the viscosity versus shear rate for the different bulbs on log paper as shown by the rheogram in Figure 5.

5.7.5 Report the Apparent Viscosity test result obtained from the rheogram at the appropriate shear rate, which is,

0.5 s^{-1} for HF-100S and HF-150S, and
1.0 s^{-1} for HF-250S

5.7.6 Always report the test temperature and vacuum with the test result. For example, apparent viscosity, in Pa·s, at 60°C and 300 mm Hg vacuum.

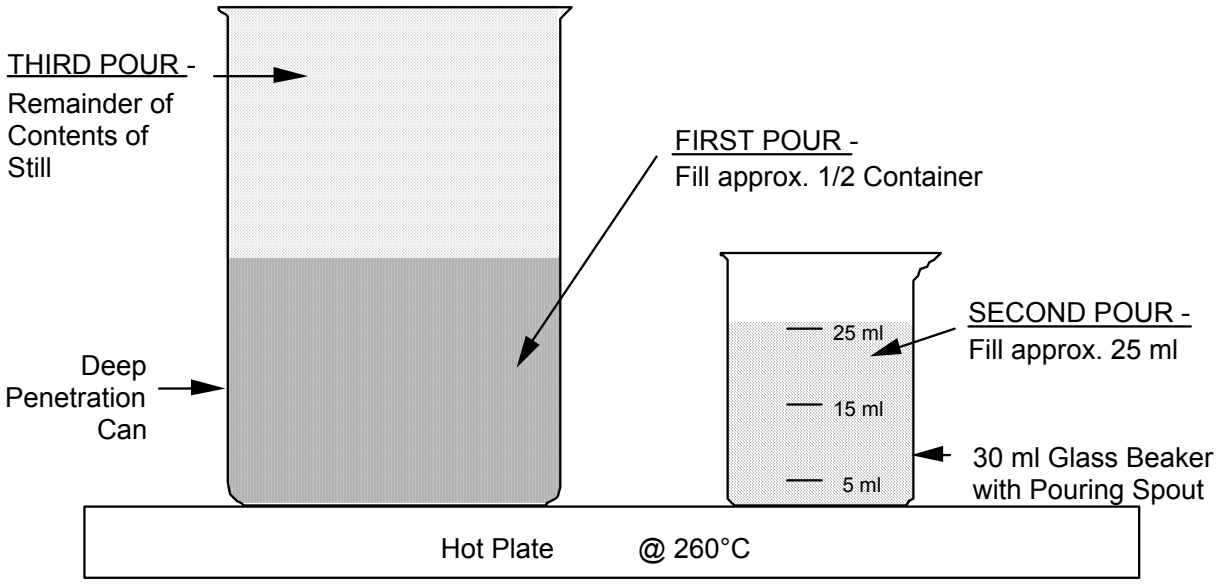
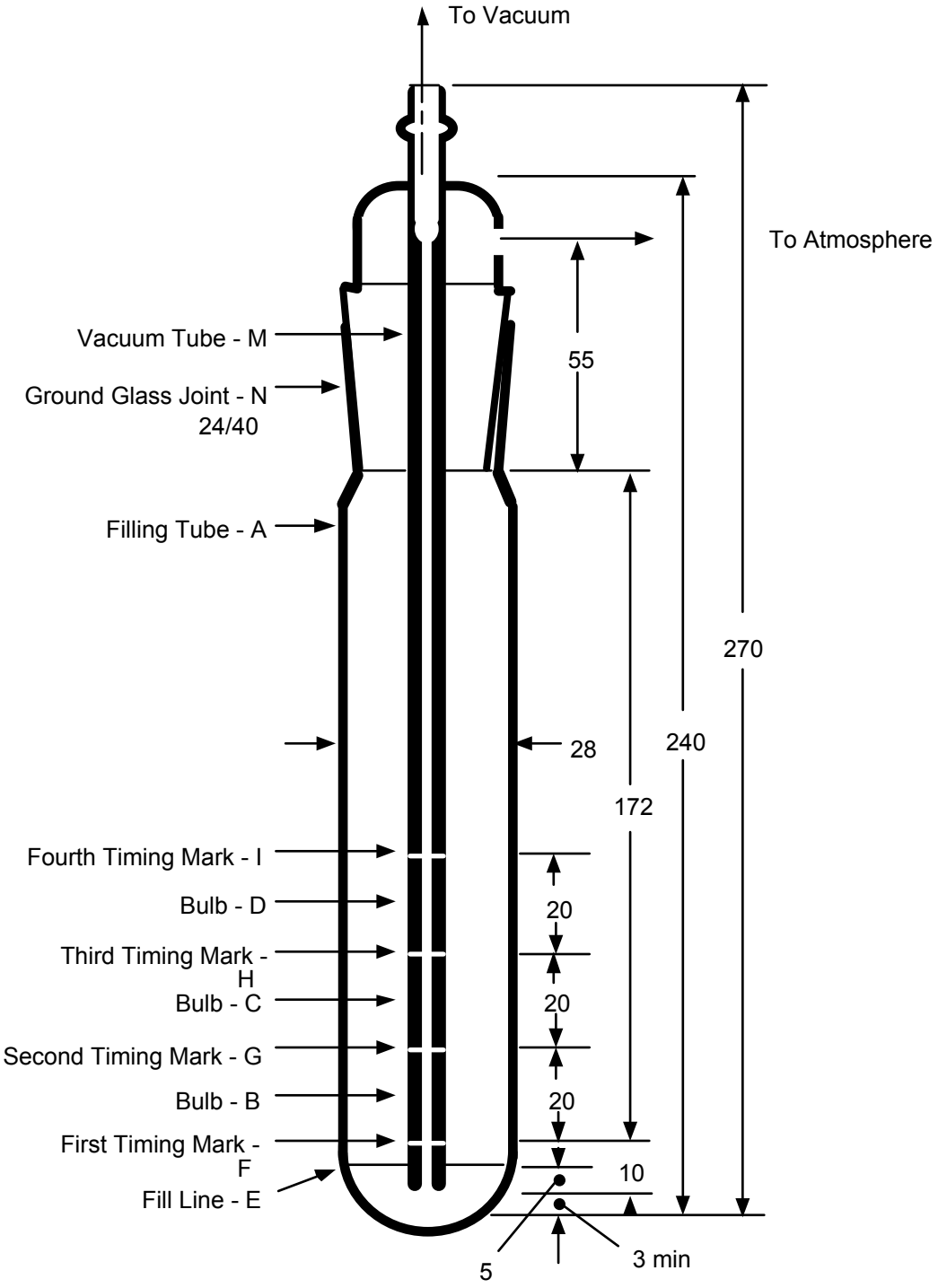

THIRD POUR -
Remainder of Contents of Still
FIRST POUR -
Fill approx. 1/2 Container
SECOND POUR -
Fill approx. 25 ml
25 ml
15 ml
5 ml
Deep Penetration Can
30 ml Glass Beaker with Pouring Spout
Hot Plate @ 260°C

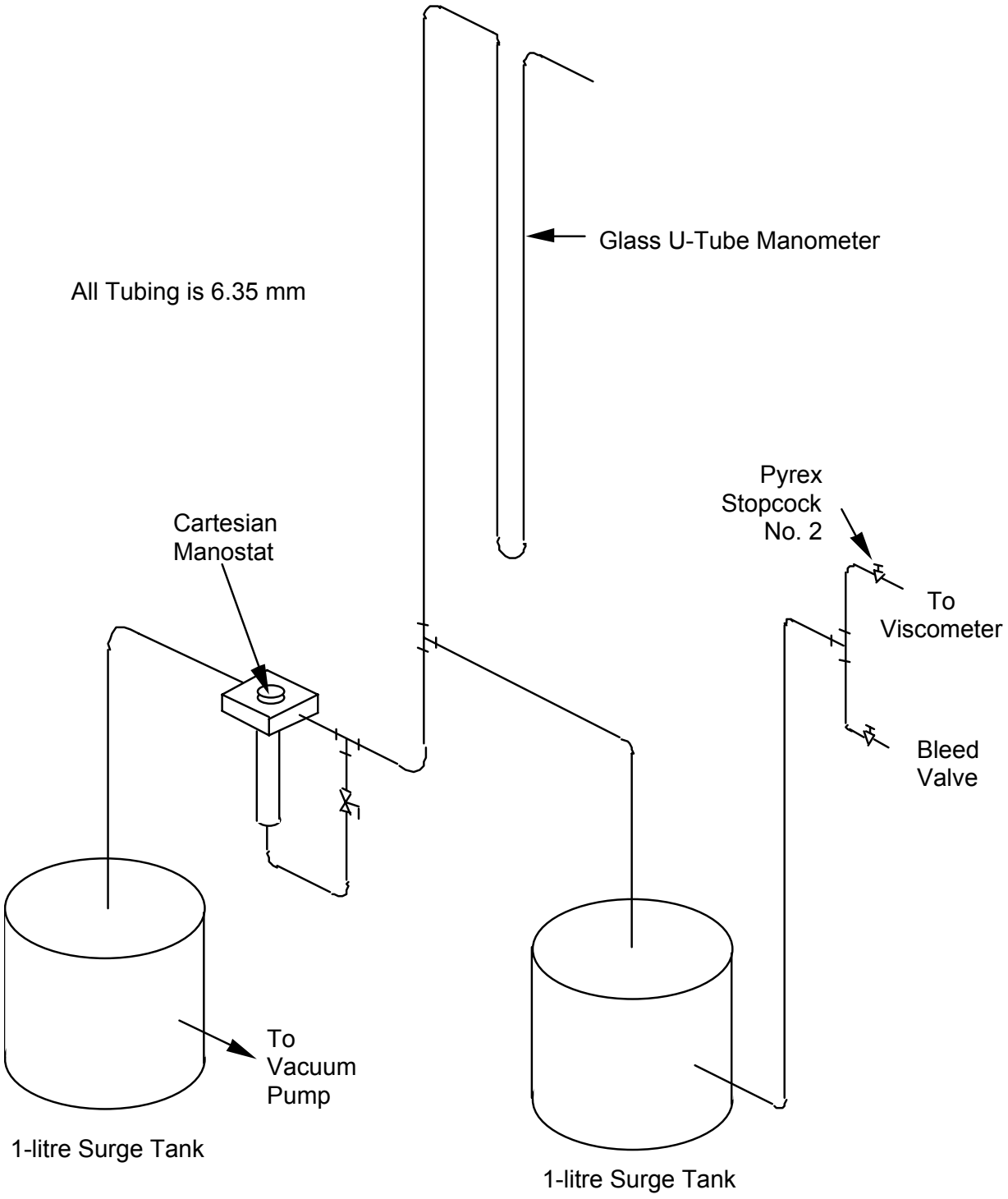
Figure 1



All dimensions are in millimetres.

Modified Koppers Vacuum Capillary Viscometer

Figure 2



Vacuum System for Vacuum Capillary Viscometer

Figure 3

TEST DATA REPORT FORM

Sample No. 124 Viscometer No. K188 Vacuum Hg 300 mm Date 89 02 09

Bulb	Time s	Viscometer Constant $P{\cdot}s^{-1}$	Viscosity P·s	Shear Constant	Shear Rate s^{-1}
B	46	126.5	582	80	1.7
C	106.2	62.7	666	80	0.75
D	176.3	41.4	730	80	0.45
E	258.0	31.0	800	80	0.31
F	346.9	24.79	860	80	0.23

Temp. 60°C Type Viscometer MK Size 200

Remarks:

by:

TEST DATA REPORT FORM

Sample No. 123 Viscometer No. K443 Vacuum Hg 300 mm Date 89 02 09

Bulb	Time s	Viscometer Constant $P{\cdot}s^{-1}$	Viscosity P·s	Shear Constant	Shear Rate s^{-1}
B	85.3	7.86	67	320	3.75
C	206.6	3.92	81	320	1.55
D	376.0	2.611	98	320	0.85
E	600.9	1.975	119	320	0.53
F	894.4	1.592	142	320	0.36

Temp. 60°C Type Viscometer MK Size 50

Remarks:

by:

Figure 4

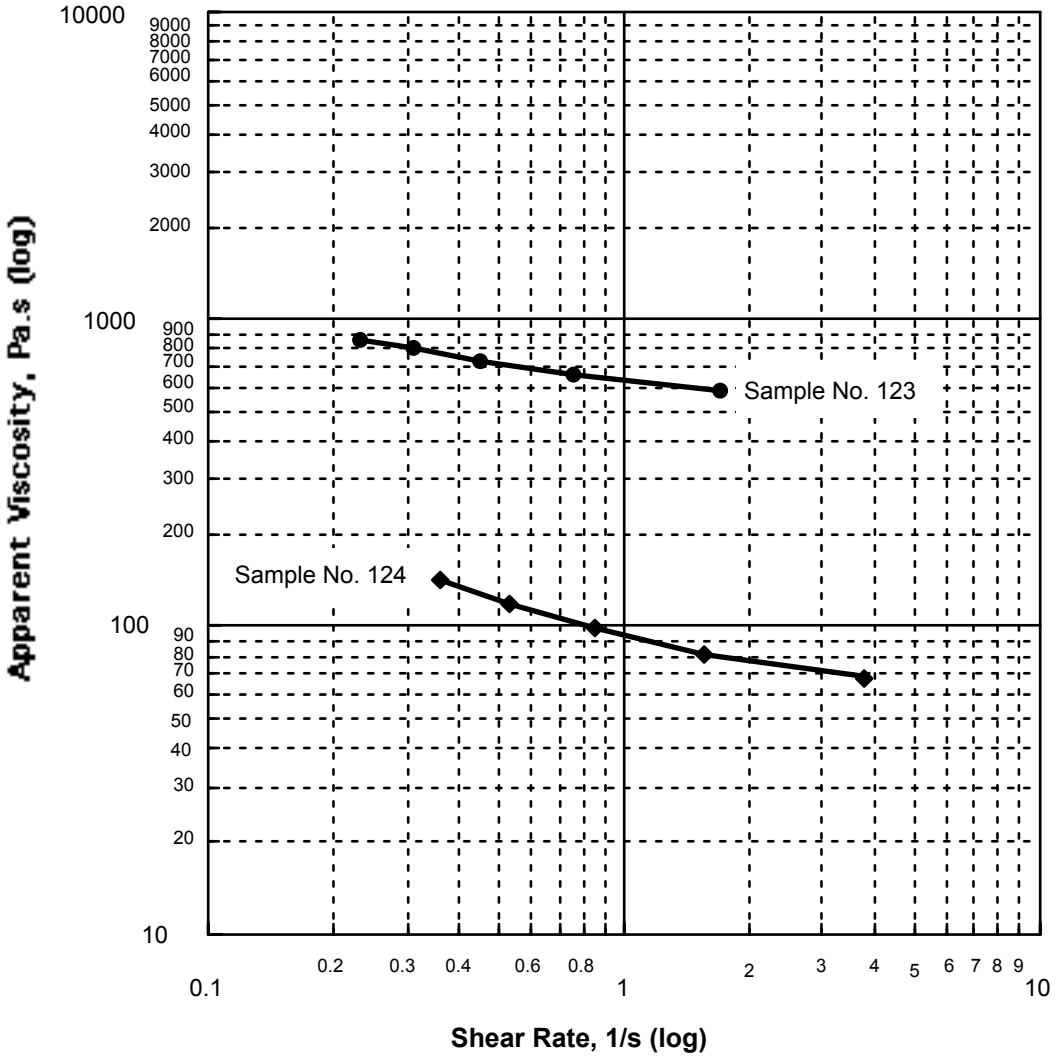

Apparent Viscosity, Pa.s (log)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
900
800
700
600
500
400
300
200
100
90
80
70
60
50
40
30
20
10
Sample No. 123
Sample No. 124
0.1
0.2
0.3
0.4
0.6
0.8
1
2
3
4
5
6
7
8
9
10
Shear Rate, 1/s (log)

Figure 5

Figure 6 Oven with tube installed.