THIN FILM RHEOLOGY OF POLYMER MODIFIED BINDERS

A discussion prepared for the Association of Modified Asphalt Producers 11th Annual meeting

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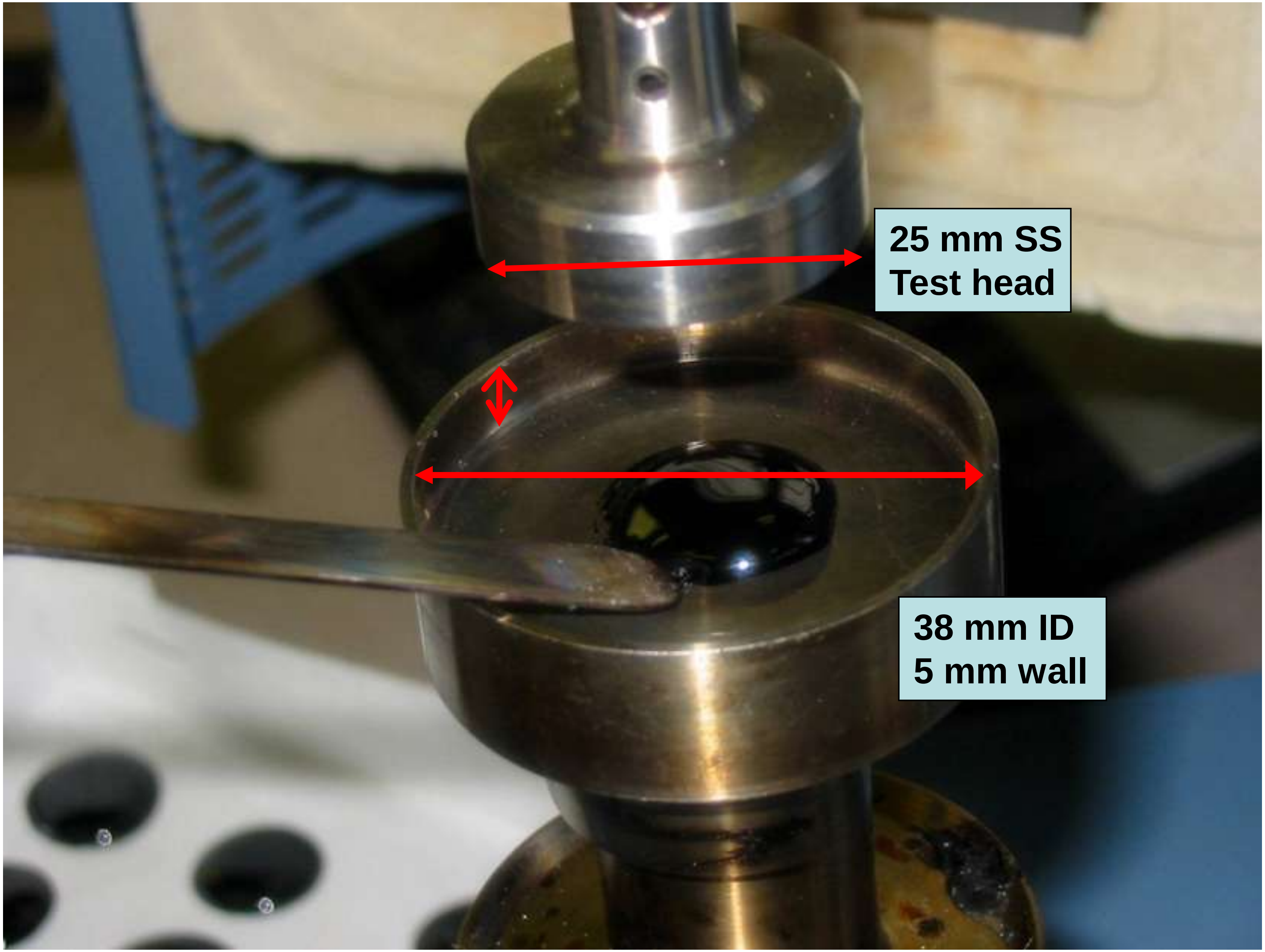
USE OF THIN FILM RHEOLOGY FOR INVESTIGATION OF MIXING & COMPACTION TEMPERATURE AND INVESTIGATION OF WARM MIX PERFORMANCE WHEN USING POLYMER MODIFIED BINDERS

PROCEDURE FOR THIN FILM RHEOLOGY

1. **A series of Steady State Flow Tests at a single temperature**
   1. **Temperatures determined by grade of binder being evaluated**
   2. **For PG 58-28 we use 95°C, 105°C, 115°C, and 125°C**
   3. **For PMA we use 105°C, 115°C, and 125°C, and 135°C**
2. **Uses a cup and plate assembly on a Dynamic Shear Rheometer equipped with a forced air oven**


25 mm SS
Test head
38 mm ID
5 mm wall

$$\dot{\gamma} = \Omega * r / H$$

$\dot{\gamma}$ = shear rate

Ω = angular velocity, rad/sec

r=radius, mm

H=gap, mm

Specimen brought to initial test gap

Gap is zeroed at test temp, be sure to all

PROCEDURE FOR THIN FILM RHEOLOGY

1. **Zero Gap and loading sample at test temperature**
   1. Allow at least 15 minutes at test temperature before zeroing gap--to be sure fixtures are at temp
   2. Load sample, bring head into contact and allow sample and equipment to equilibrate for ~15 minutes
   3. Set gap to initial test gap (500 μ), condition for 15 minutes
2. **Running the test (1)**
   1. Perform Steady shear flow test ramping angular velocity from 1 to 200 radians/sec using 10 points per log decade
   2. Sample period 10 seconds
   3. Steady state determined by 3 consecutive points within 5% tolerance, maximum time to reach steady state 8 minutes, then move on to next velocity step.
   4. When all angular velocity steps have been performed move on to next gap.

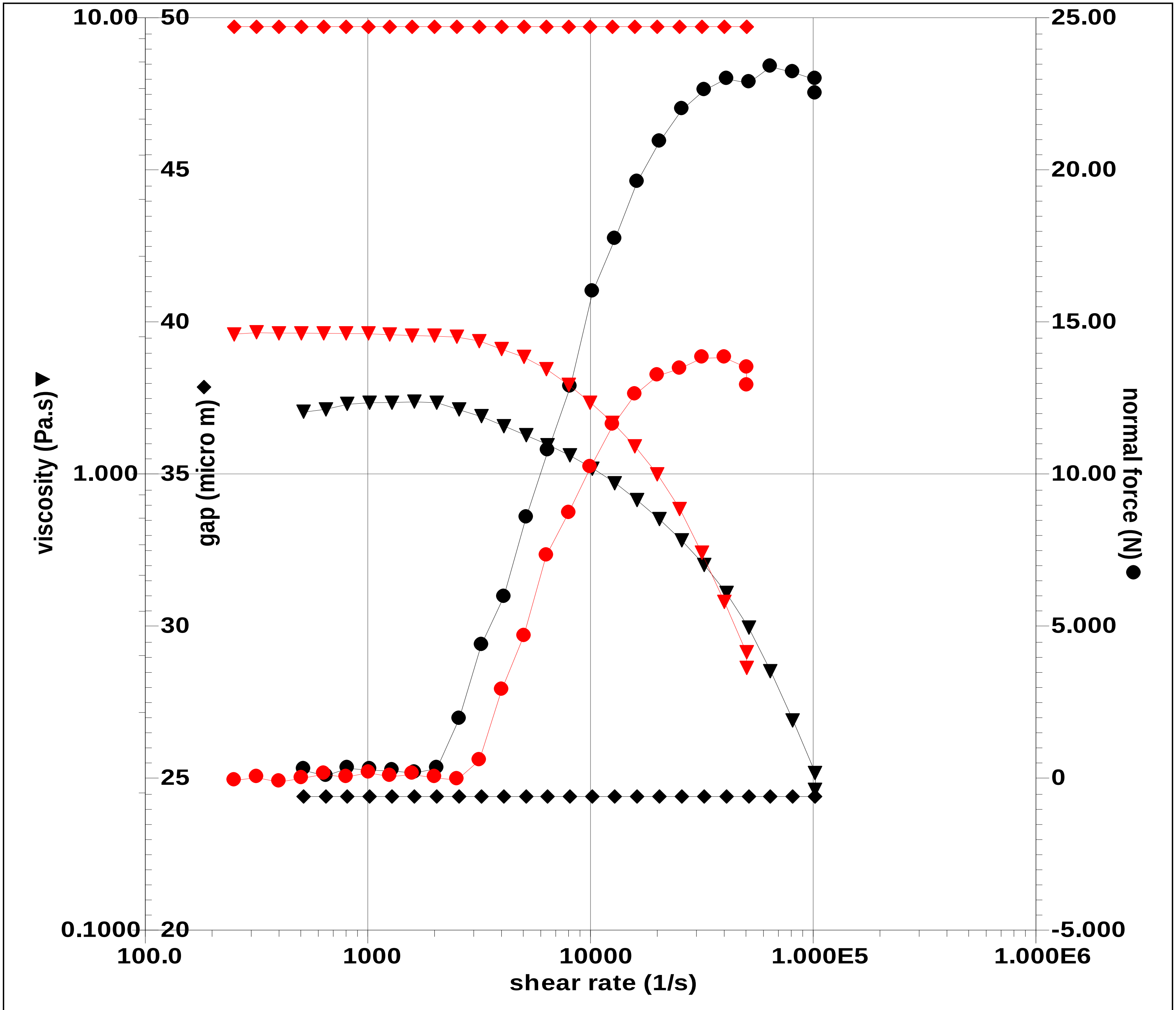
PROCEDURE FOR THIN FILM RHEOLOGY

Running the test (2)

5. To change gaps program a squeeze/pull off test following the first steady shear flow test.
    1. Parameters for the squeeze/pull off test are:
        1. gap speed 10 μ/sec in compression for a total distance of 400 μ.
        2. Changes gap from 500 μ to 100 μ.
        3. Sample delay time 10 sec although we ignore the data collected.
        4. Step is just to change the gap without having to do it manually and without opening the machine
6. The next step is another steady shear flow test following the same angular velocity parameters but set at the 100 μ gap.

PROCEDURE FOR THIN FILM RHEOLOGY

7. **Following the steady shear flow test at 100 µ the gap is changed with another squeeze/pull off test, this time compressing by 50 µ for a new test gap of 50 µ. The final squeeze/pull off test compresses the sample from 50 µ to 25 µ.**
8. **Steps are programmed so that the test can run unattended at a single test temperature.**
9. **All 4 steps take about 4600 seconds to run**
10. **Evaluate Normal Force response**


10.00
1.000
0.1000
viscosity (Pa.s)
50
45
40
35
30
25
20
gap (micro m)
25.00
20.00
15.00
10.00
5.000
0
-5.000
normal force (N)
100.0
1000
10000
1.000E5
1.000E6
shear rate (1/s)

PROCEDURE FOR THIN FILM RHEOLOGY CAVEATS

1. This test is difficult to run correctly
    1. Issues of temperature equilibrium
    2. Issues of gaping
    3. Issues of co-planarity
    4. Issues of optimum sample size
2. There are different approaches to get to similar information
    1. Apply a normal force and measure a friction response—MIT annular ring, Bahia UW-Madison
    2. Atomic Force Microscopy to measure rheology at nanoscale—numerous researchers in literature, Pauli-WRI

Source: “Tribo-rheometry: from Gap Dependent rheology to tribology”, Kavehpour and McKinley, MIT


LOW
SMART
RETURN

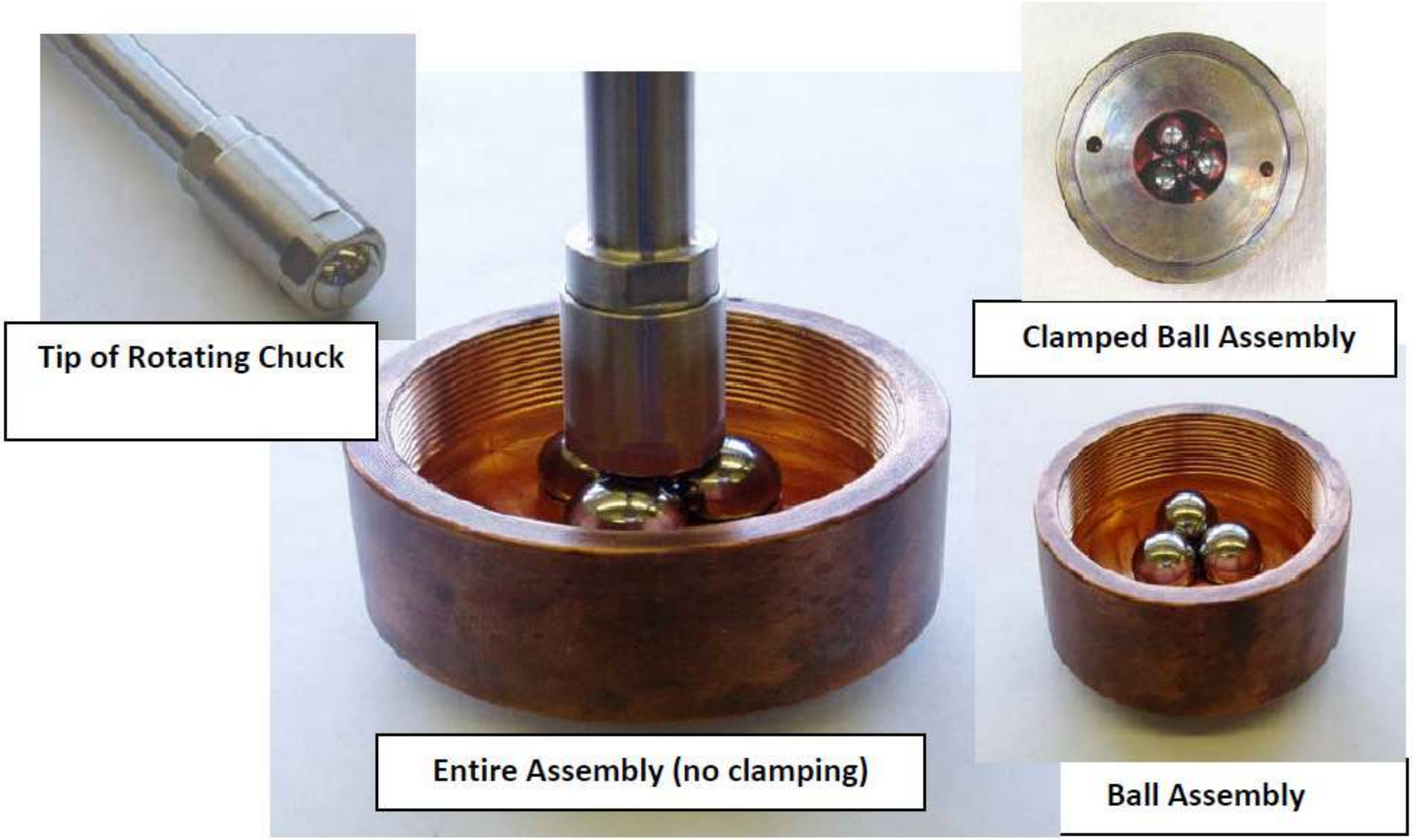
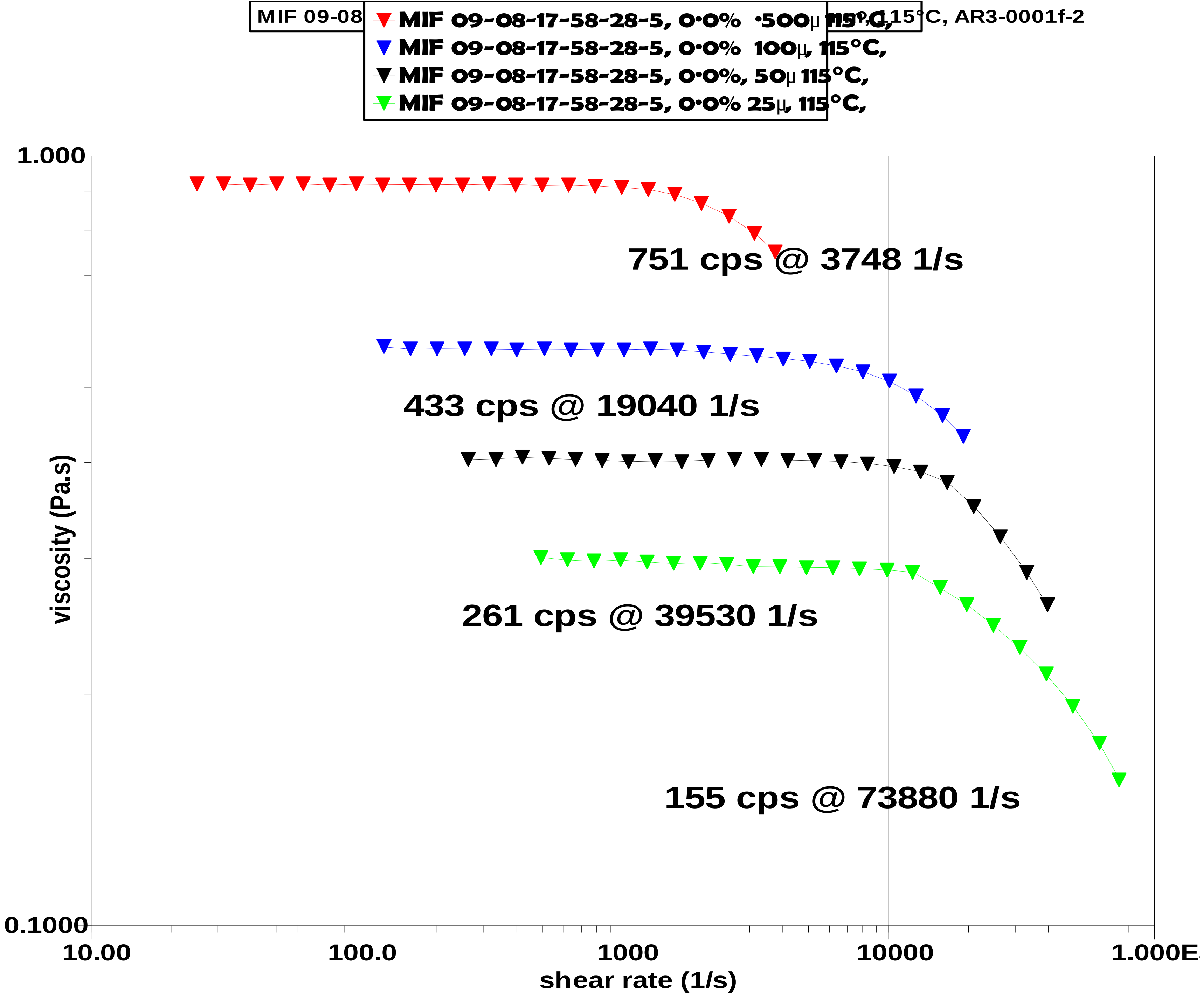
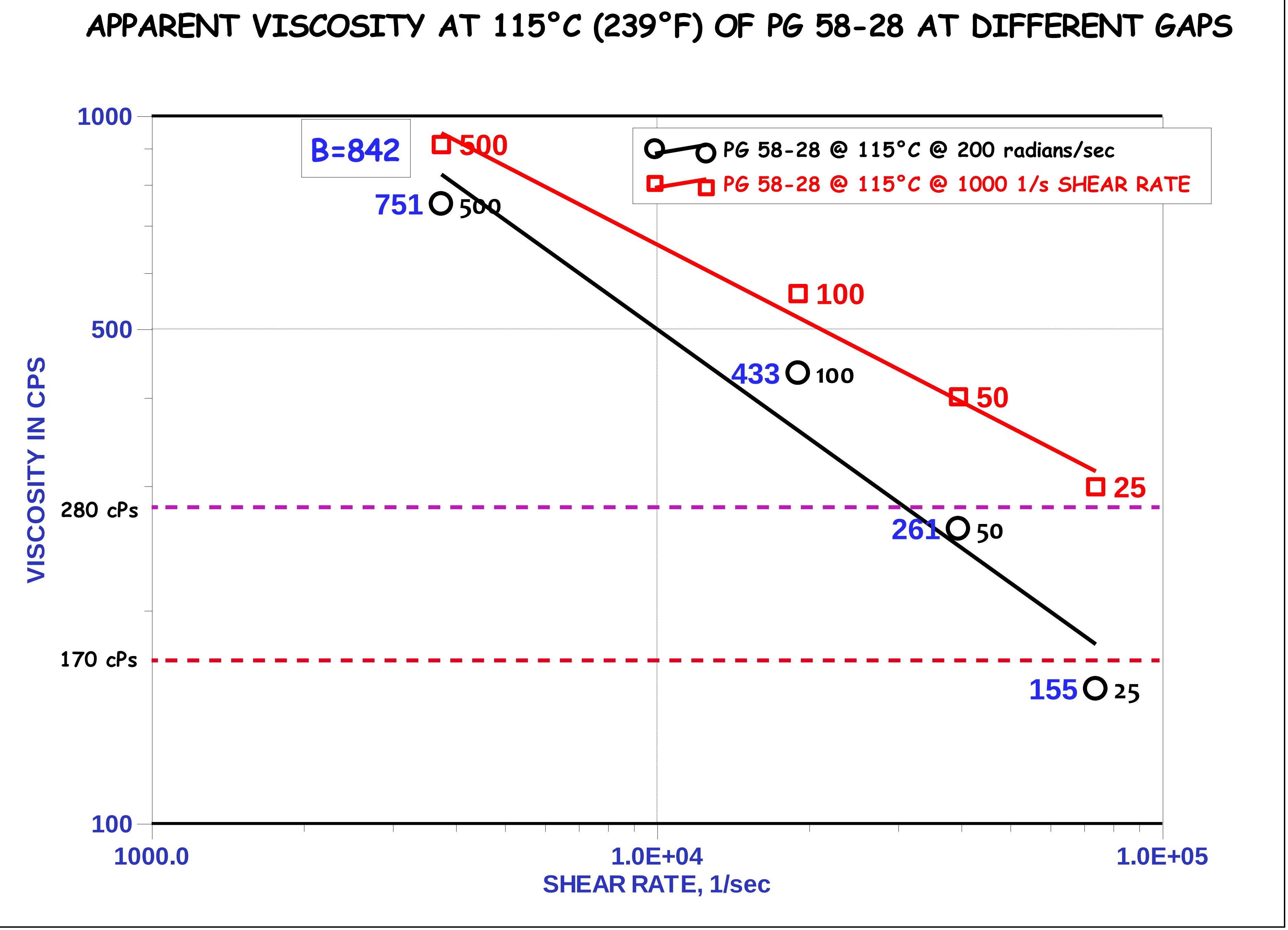


FIGURE 1 Photographs of Asphalt Lubricity Tester

Source: Measuring Effects of Warm Mix Additives Using a Newly Developed Asphalt Binder Lubricity Test for the DSR, Hanz et al, TRB 2010

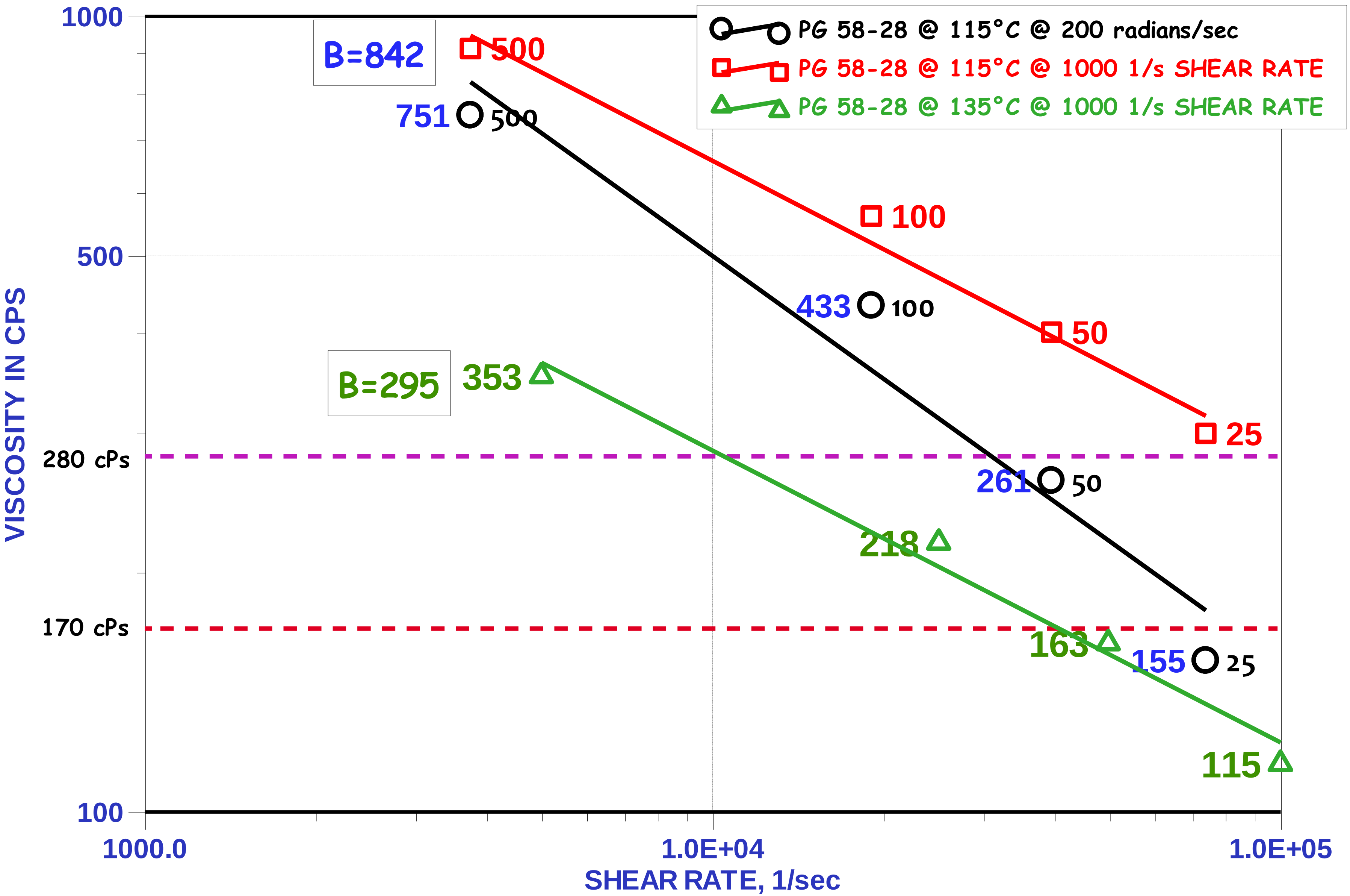


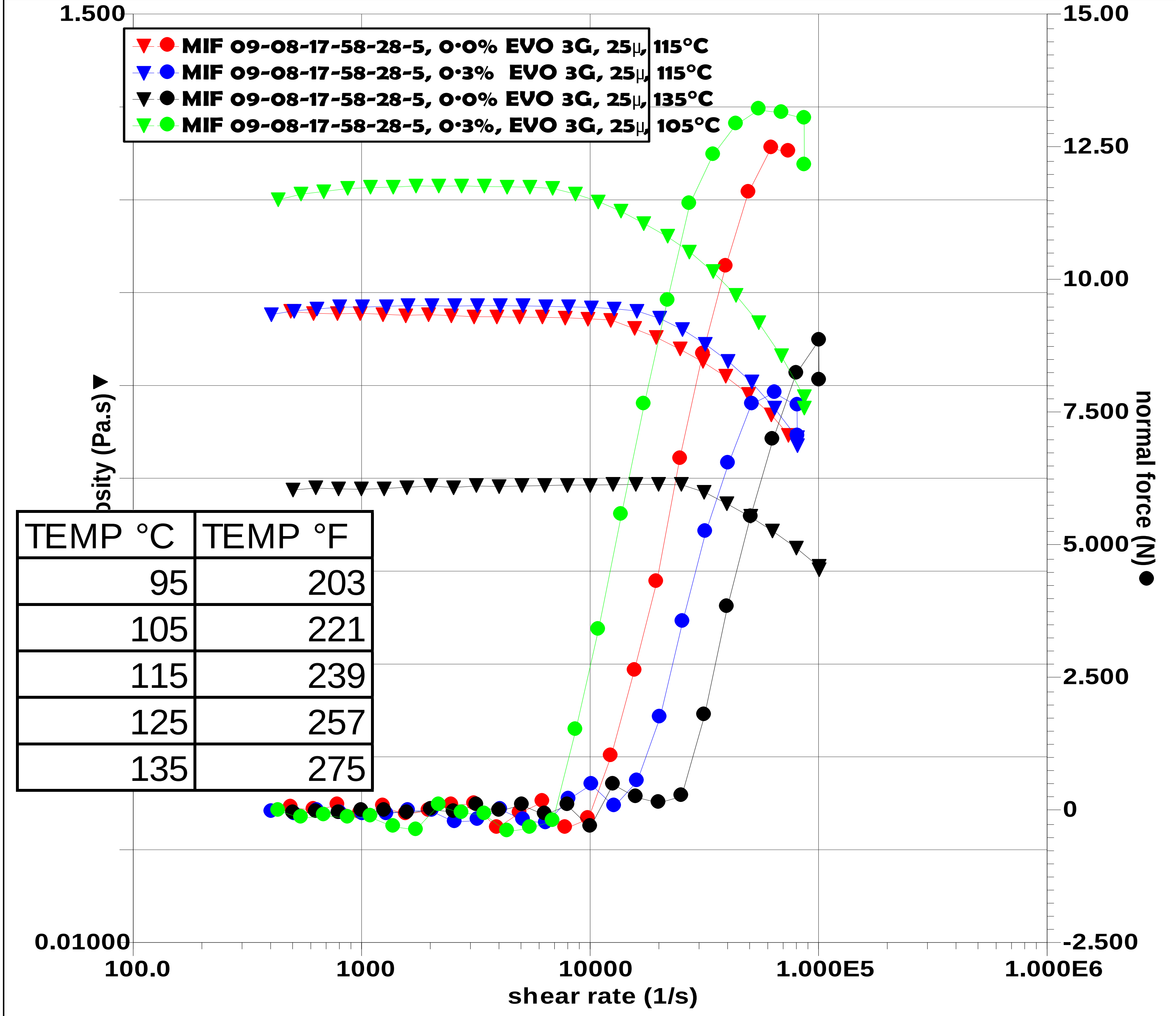
MIF 09-08
°C, 115°C, AR3-0001f-2
MIF 09-08-17-58-28-5, 0·0% ·500µ 115°C,
MIF 09-08-17-58-28-5, 0·0% 100µ, 115°C,
MIF 09-08-17-58-28-5, 0·0%, 50µ 115°C,
MIF 09-08-17-58-28-5, 0·0% 25µ, 115°C,
751 cps @ 3748 1/s
433 cps @ 19040 1/s
261 cps @ 39530 1/s
155 cps @ 73880 1/s
1.000
0.1000
viscosity (Pa.s)
10.00
100.0
1000
10000
1.000E
shear rate (1/s)



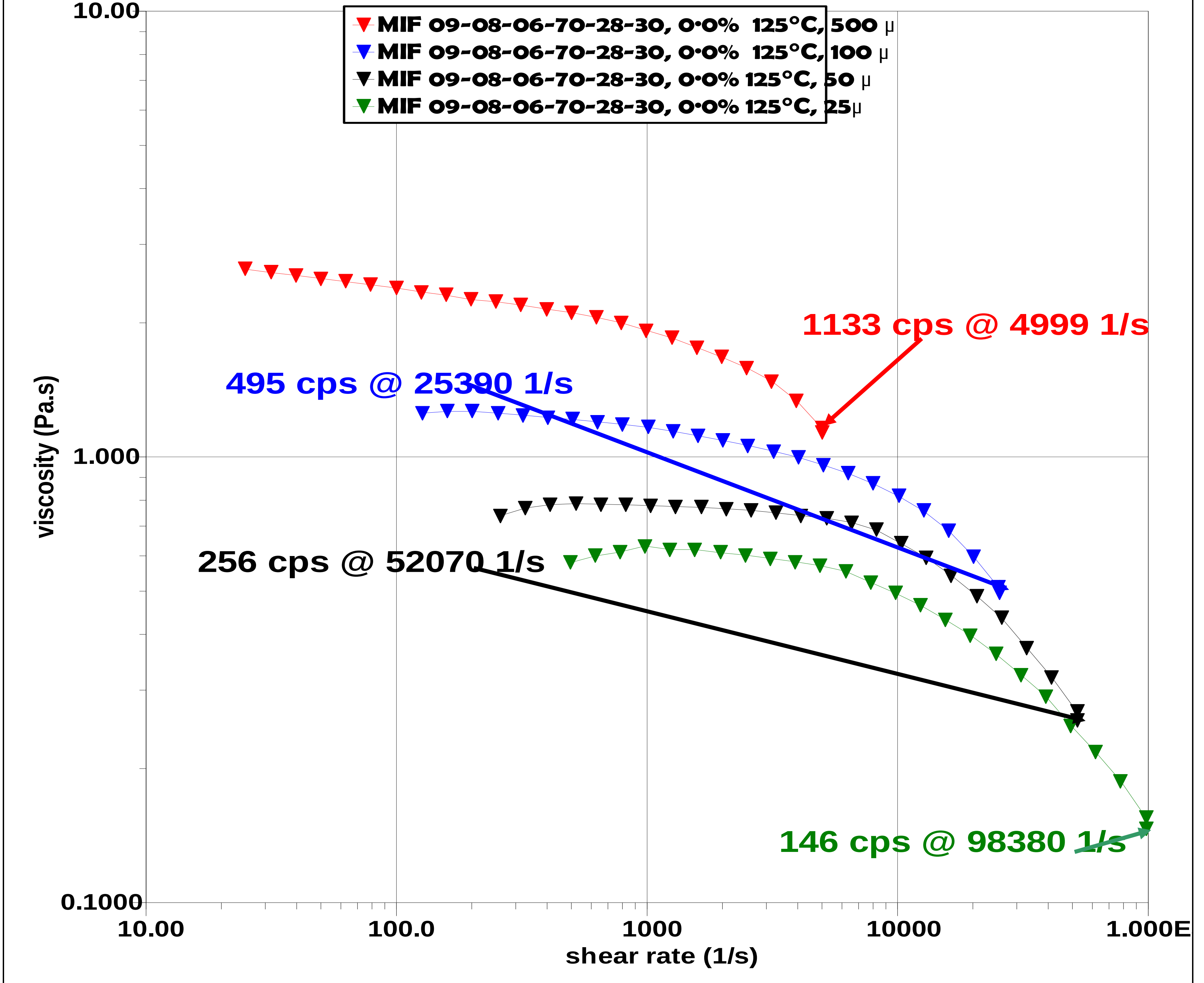
APPARENT VISCOSITY AT 115°C (239°F) OF PG 58-28 AT DIFFERENT GAPS
B=842
PG 58-28 @ 115°C @ 200 radians/sec
PG 58-28 @ 115°C @ 1000 1/s SHEAR RATE
500
100
50
25
751
433
261
155
280 cPs
170 cPs
1000
500
100
1000.0
1.0E+04
1.0E+05
VISCOSITY IN CPS
SHEAR RATE, 1/sec

APPARENT VISCOSITY AT 115°C (239°F) OF PG 58-28 AT DIFFERENT GAPS

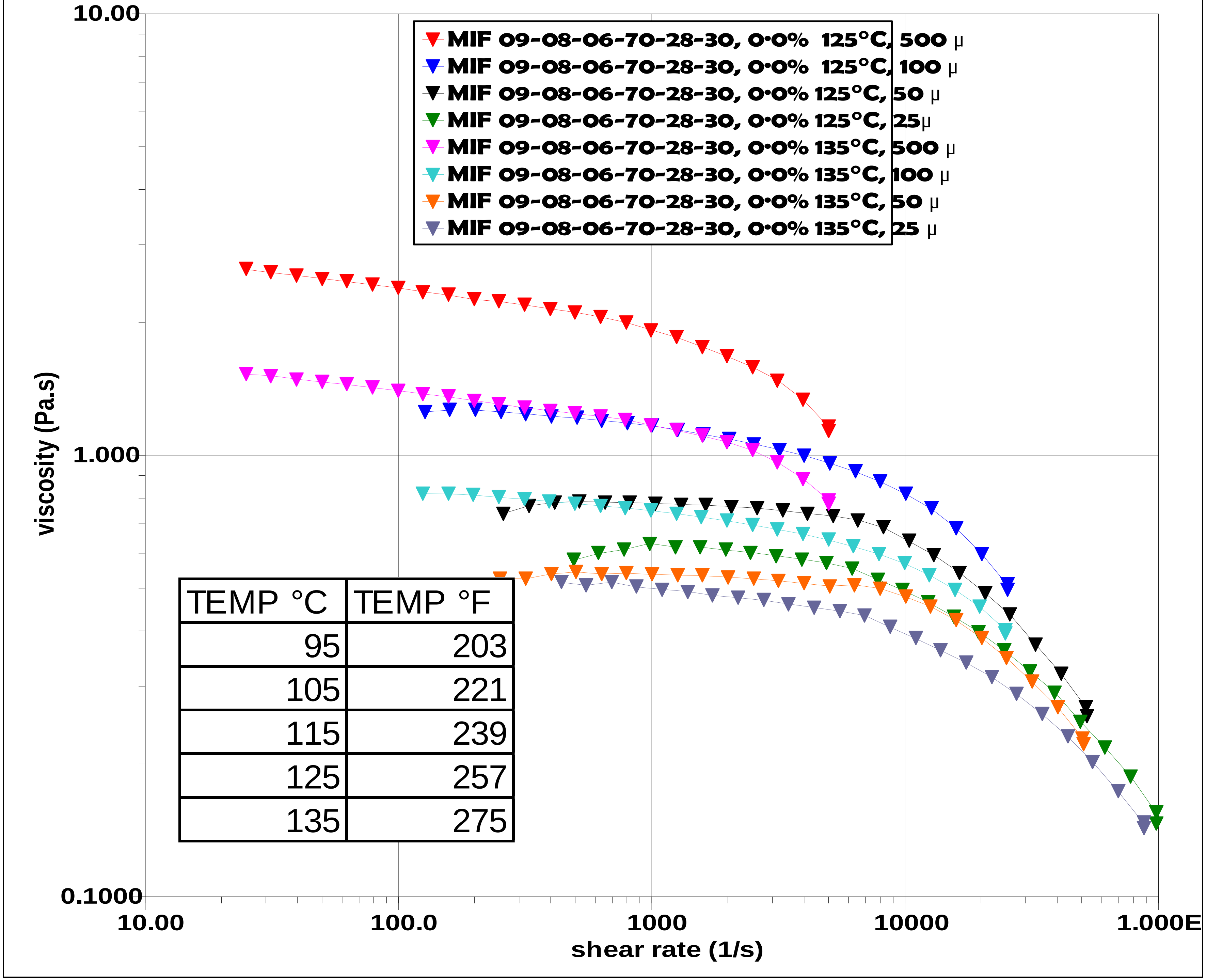




TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275

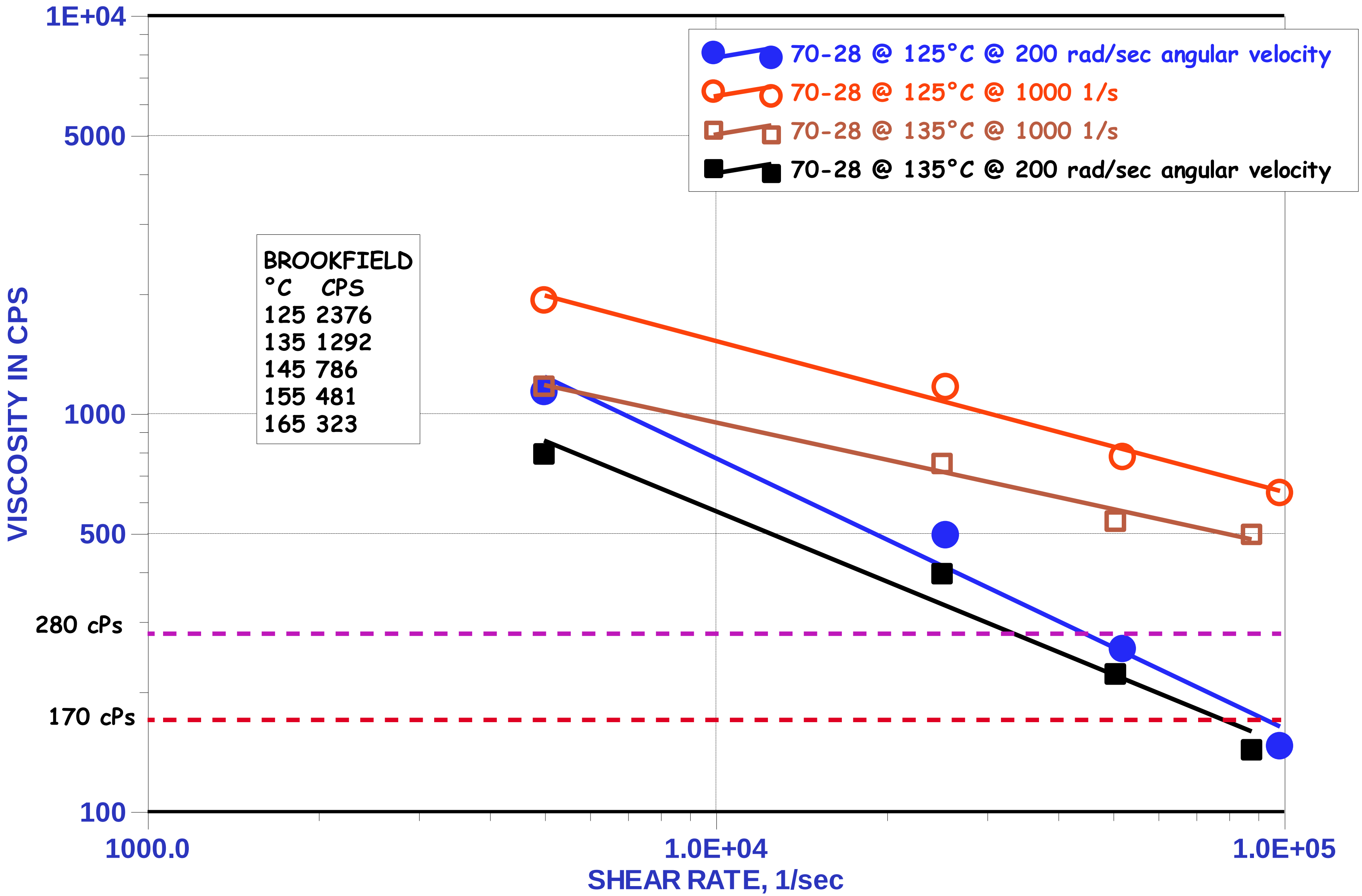


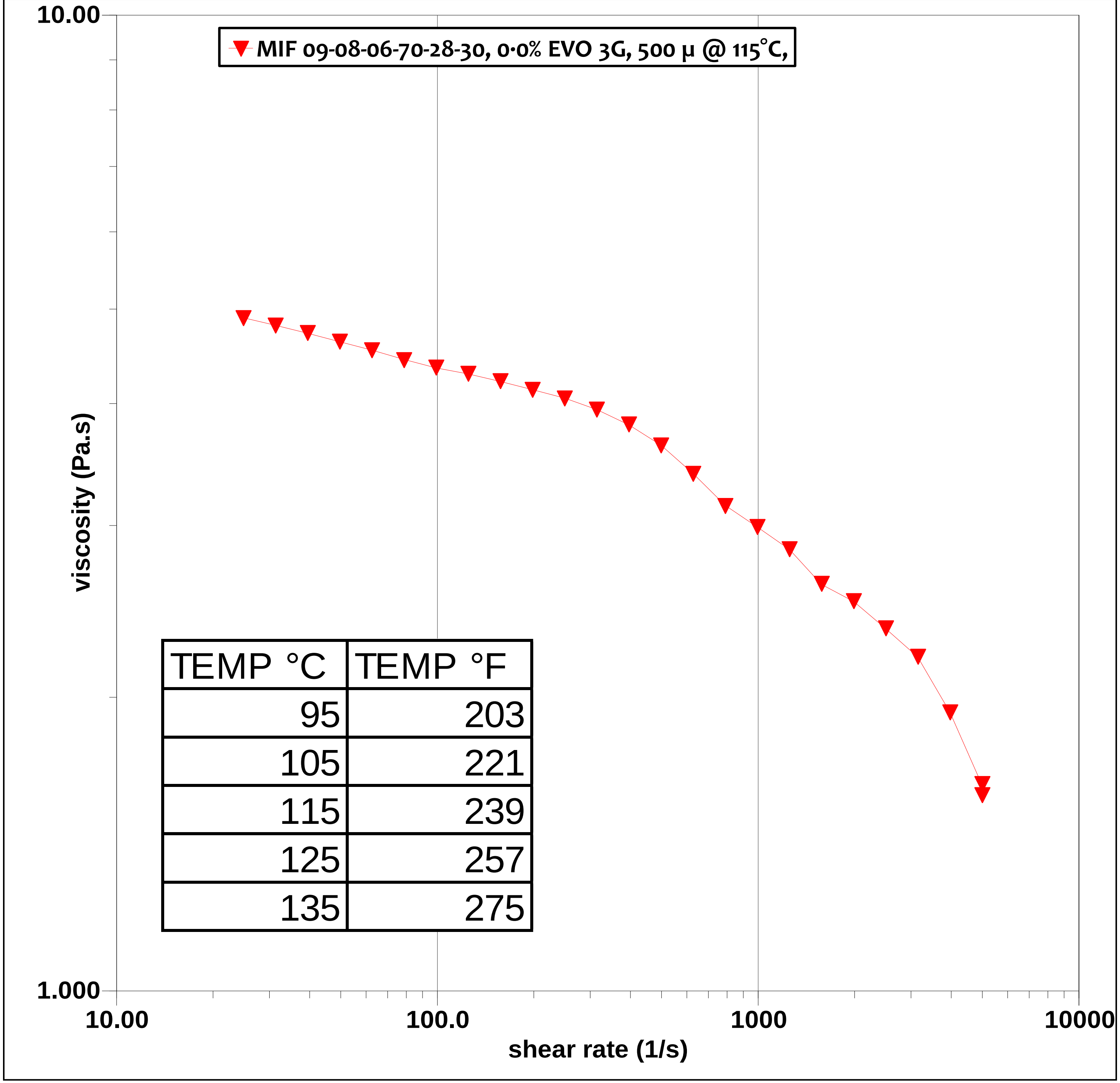
MIF 09-08-06-70-28-30, 0·0% 125°C, 500 µ
MIF 09-08-06-70-28-30, 0·0% 125°C, 100 µ
MIF 09-08-06-70-28-30, 0·0% 125°C, 50 µ
MIF 09-08-06-70-28-30, 0·0% 125°C, 25µ
1133 cps @ 4999 1/s
495 cps @ 25390 1/s
256 cps @ 52070 1/s
146 cps @ 98380 1/s
10.00
1.000
0.1000
viscosity (Pa.s)
10.00
100.0
1000
10000
1.000E
shear rate (1/s)



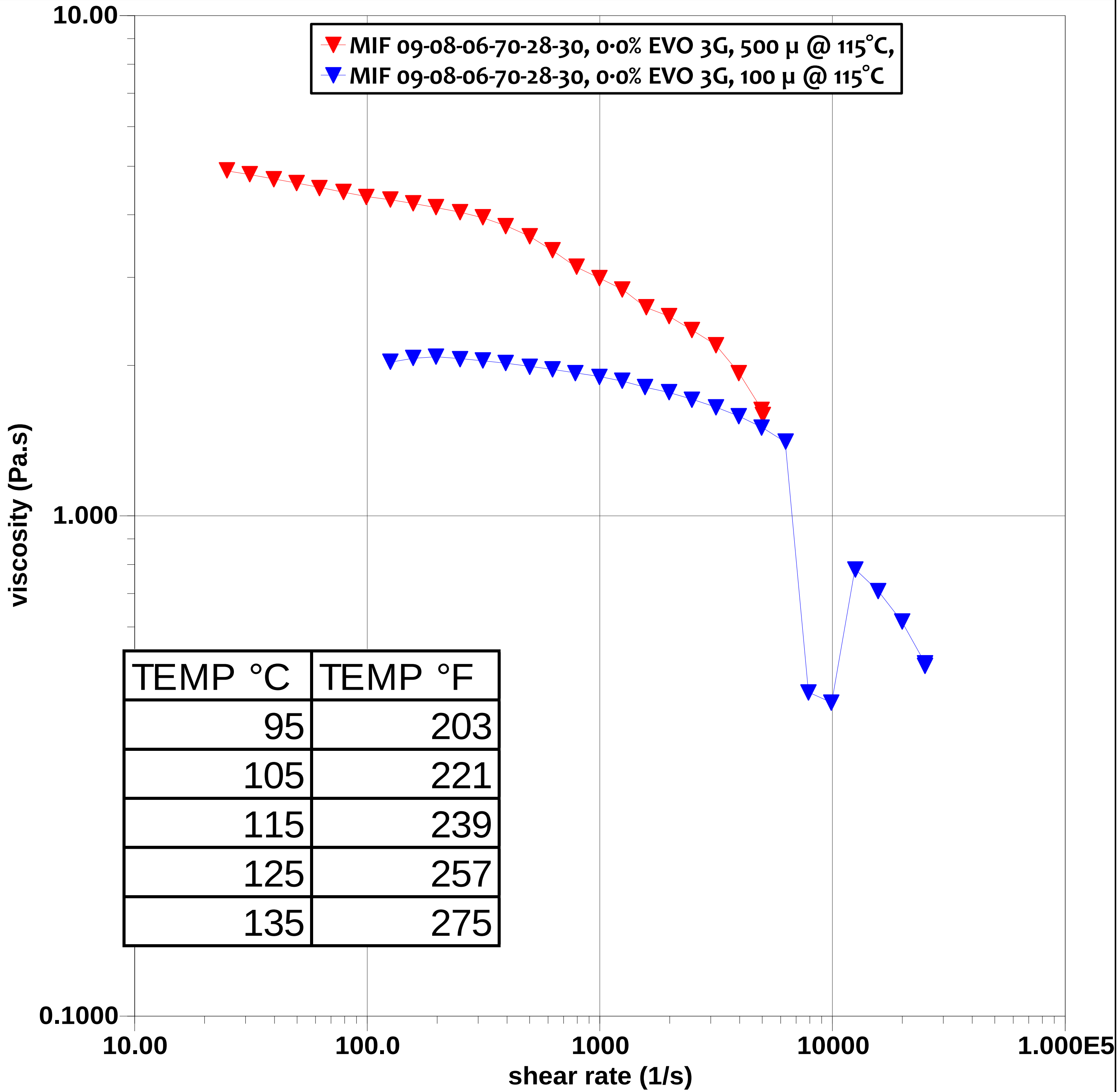
TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275

APPARENT VISCOSITY AT 125° & 135°C OF PG 70-28 AT DIFFERENT GAPS

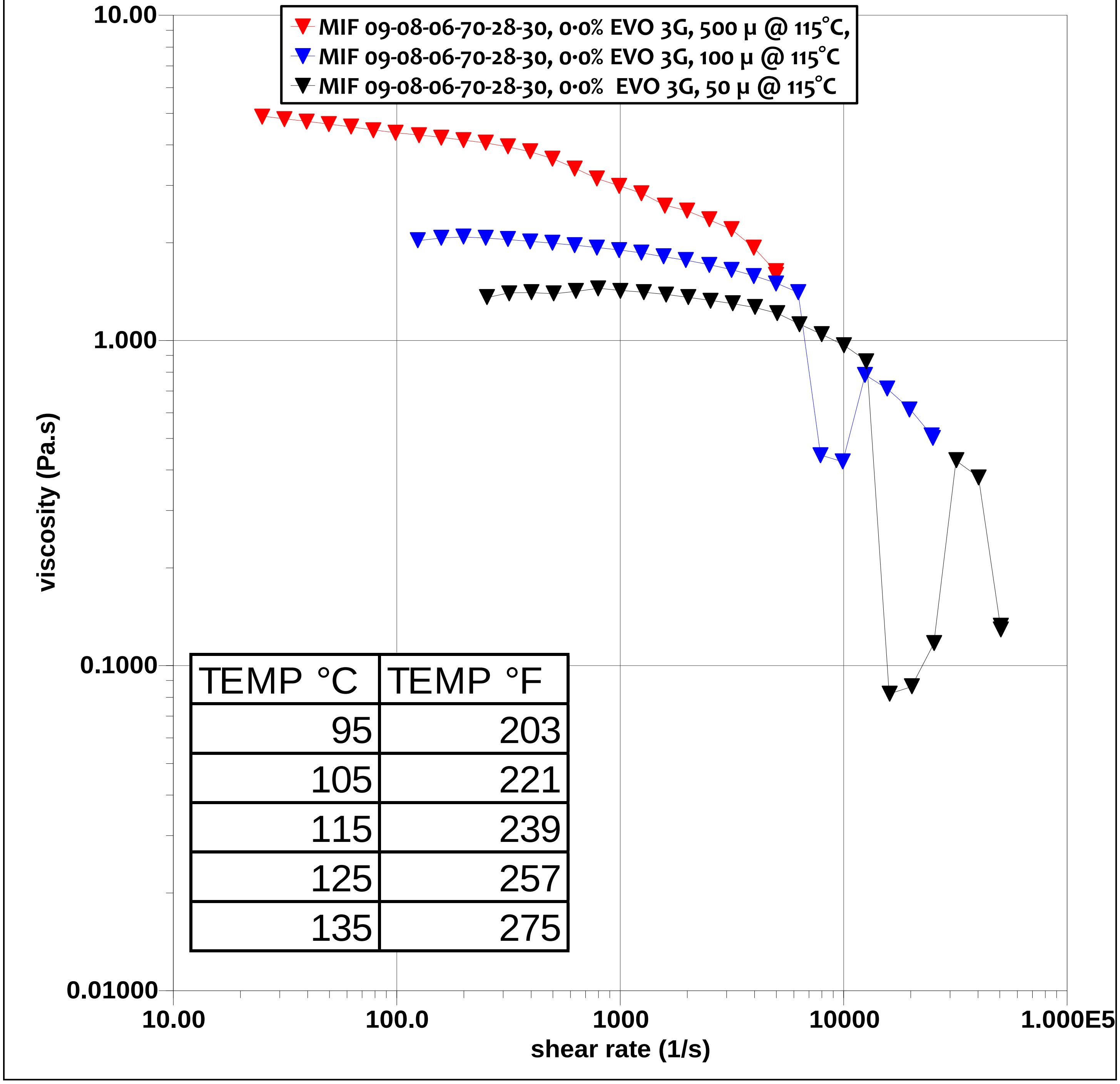




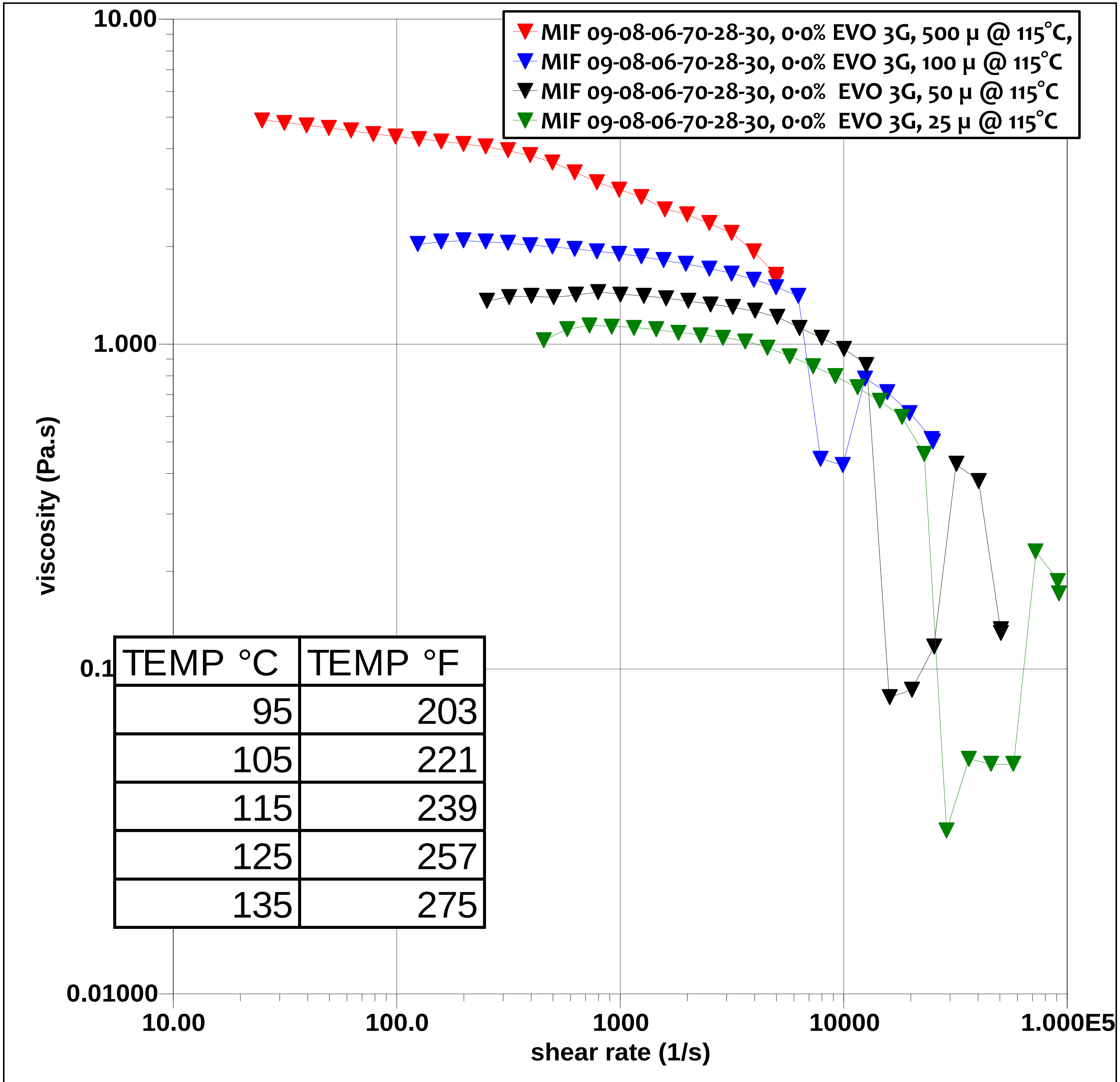
TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275



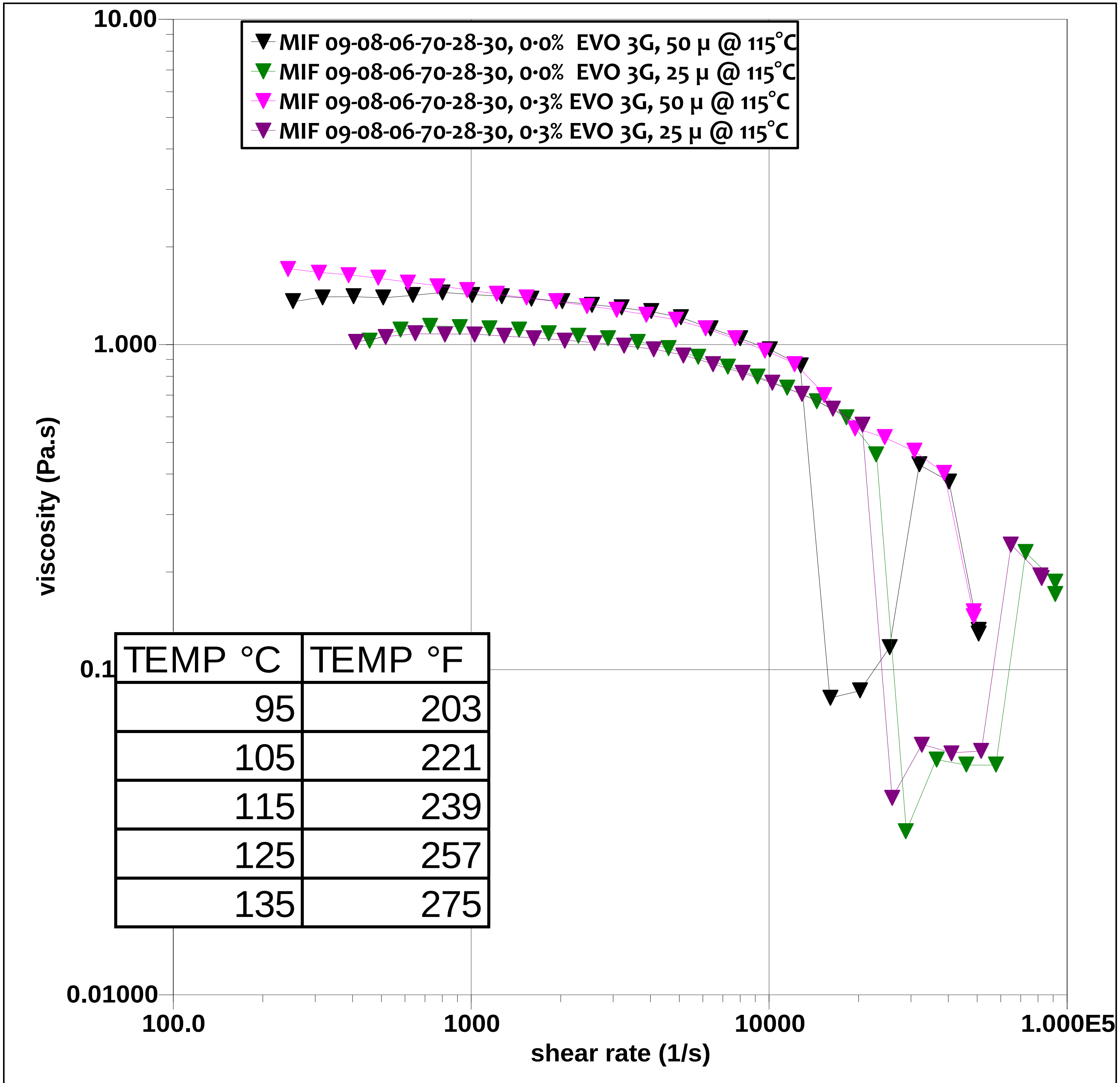
TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275



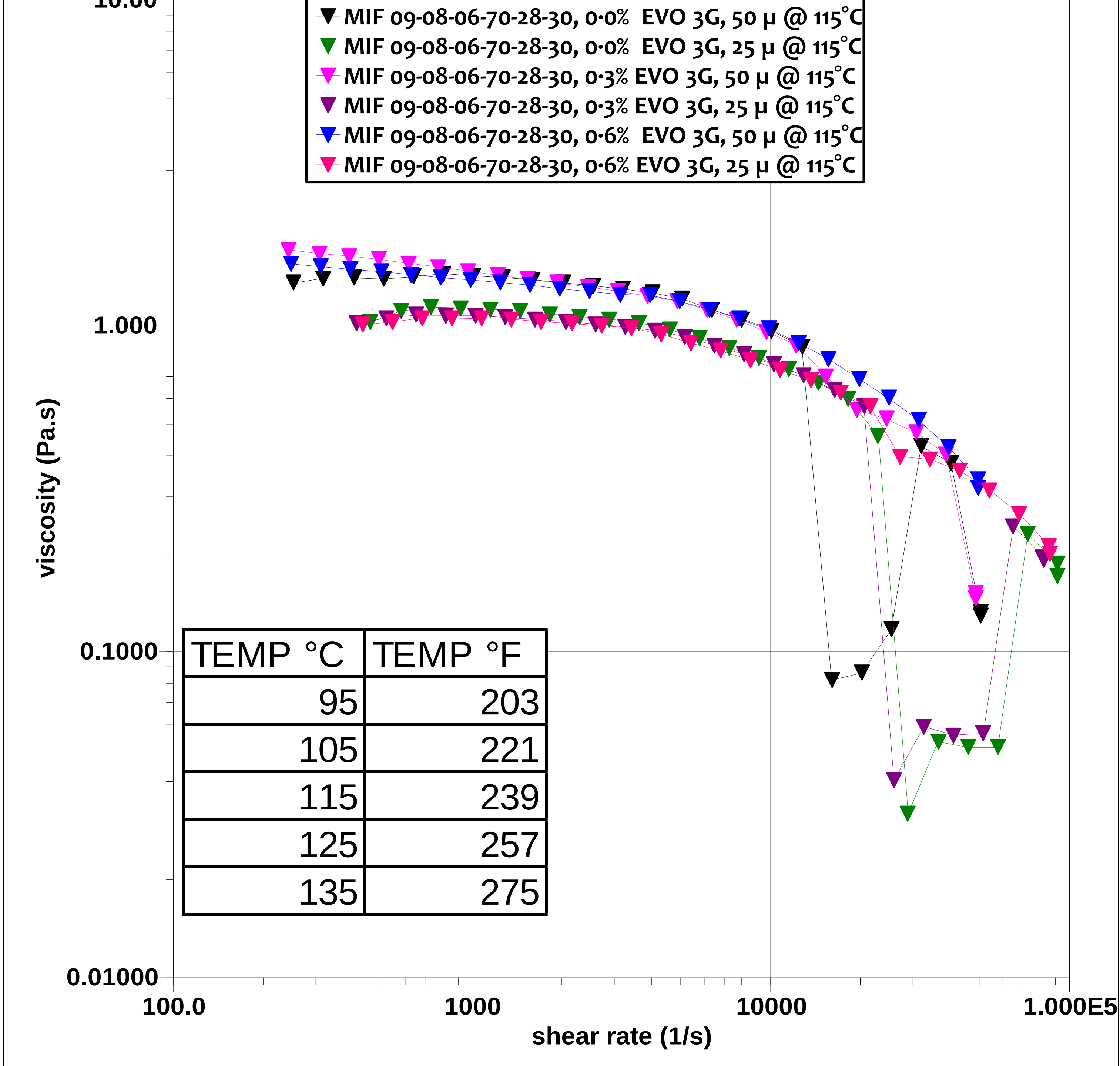
TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275

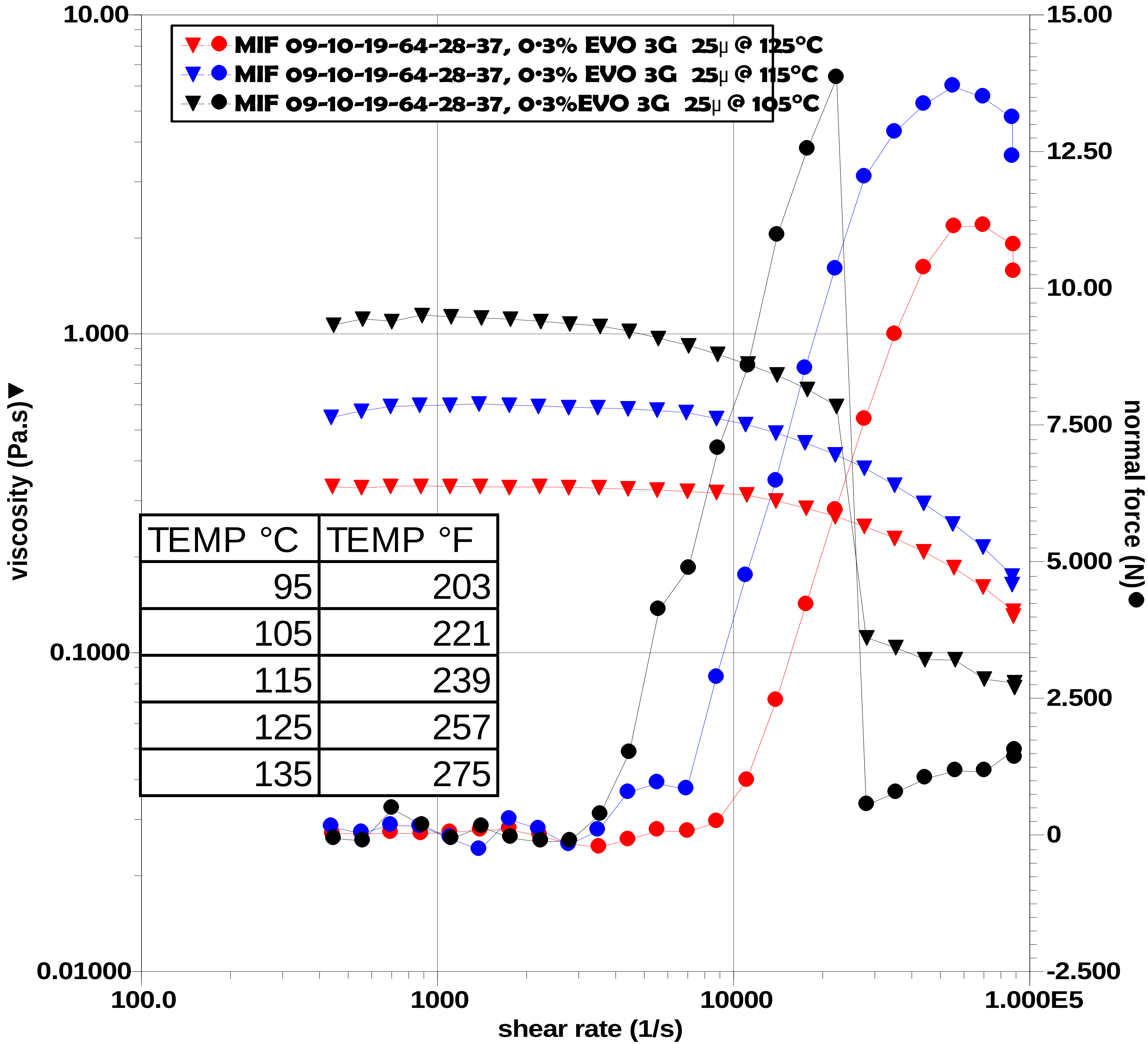


TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275

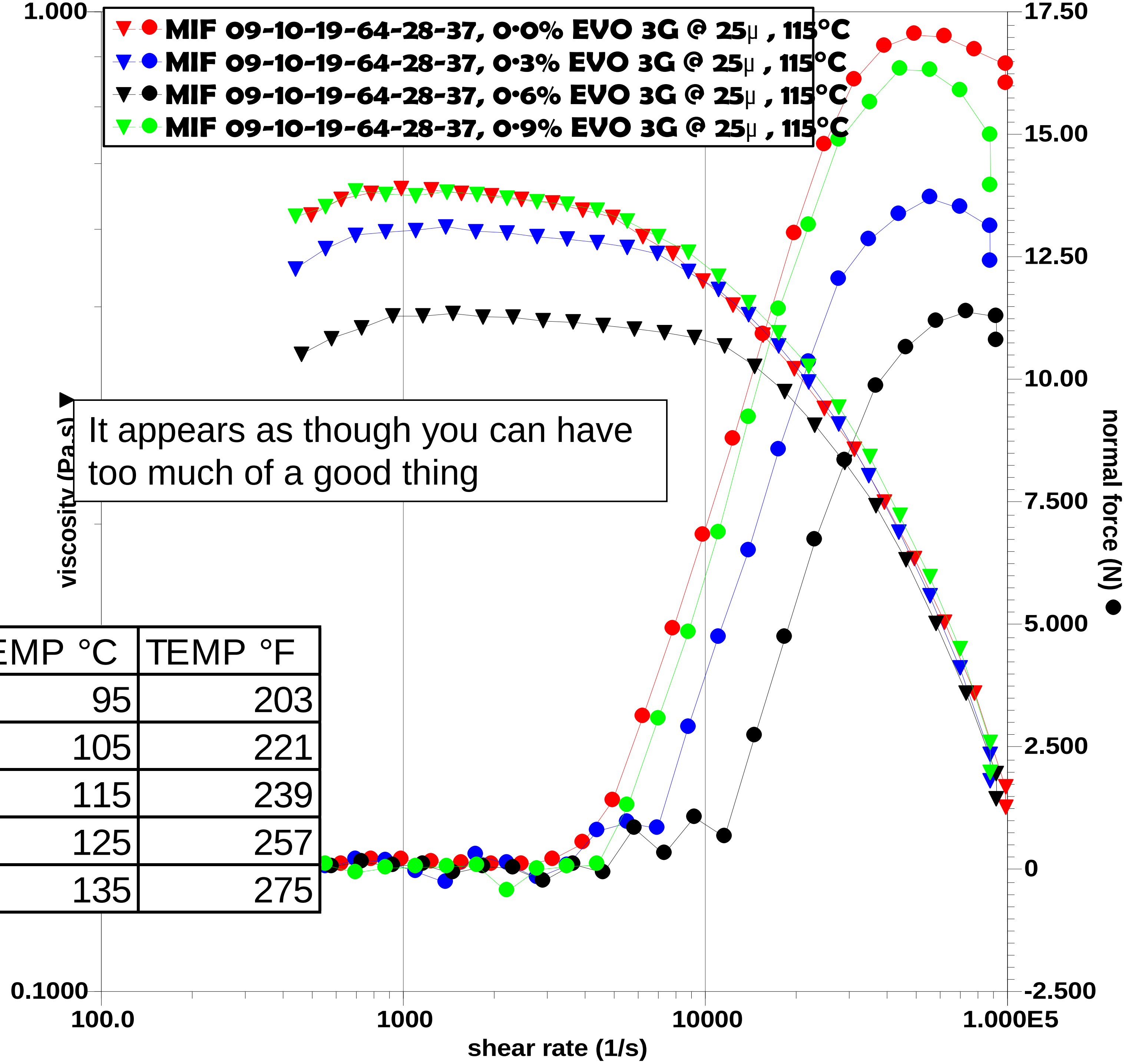


TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275


10.00
1.000
0.1000
0.01000
viscosity (Pa.s)
100.0
1000
10000
1.000E5
shear rate (1/s)
MIF 09-08-06-70-28-30, 0·0% EVO 3G, 50 μ @ 115°C
MIF 09-08-06-70-28-30, 0·0% EVO 3G, 25 μ @ 115°C
MIF 09-08-06-70-28-30, 0·3% EVO 3G, 50 μ @ 115°C
MIF 09-08-06-70-28-30, 0·3% EVO 3G, 25 μ @ 115°C
MIF 09-08-06-70-28-30, 0·6% EVO 3G, 50 μ @ 115°C
MIF 09-08-06-70-28-30, 0·6% EVO 3G, 25 μ @ 115°C
TEMP °C TEMP °F
95 203
105 221
115 239
125 257
135 275

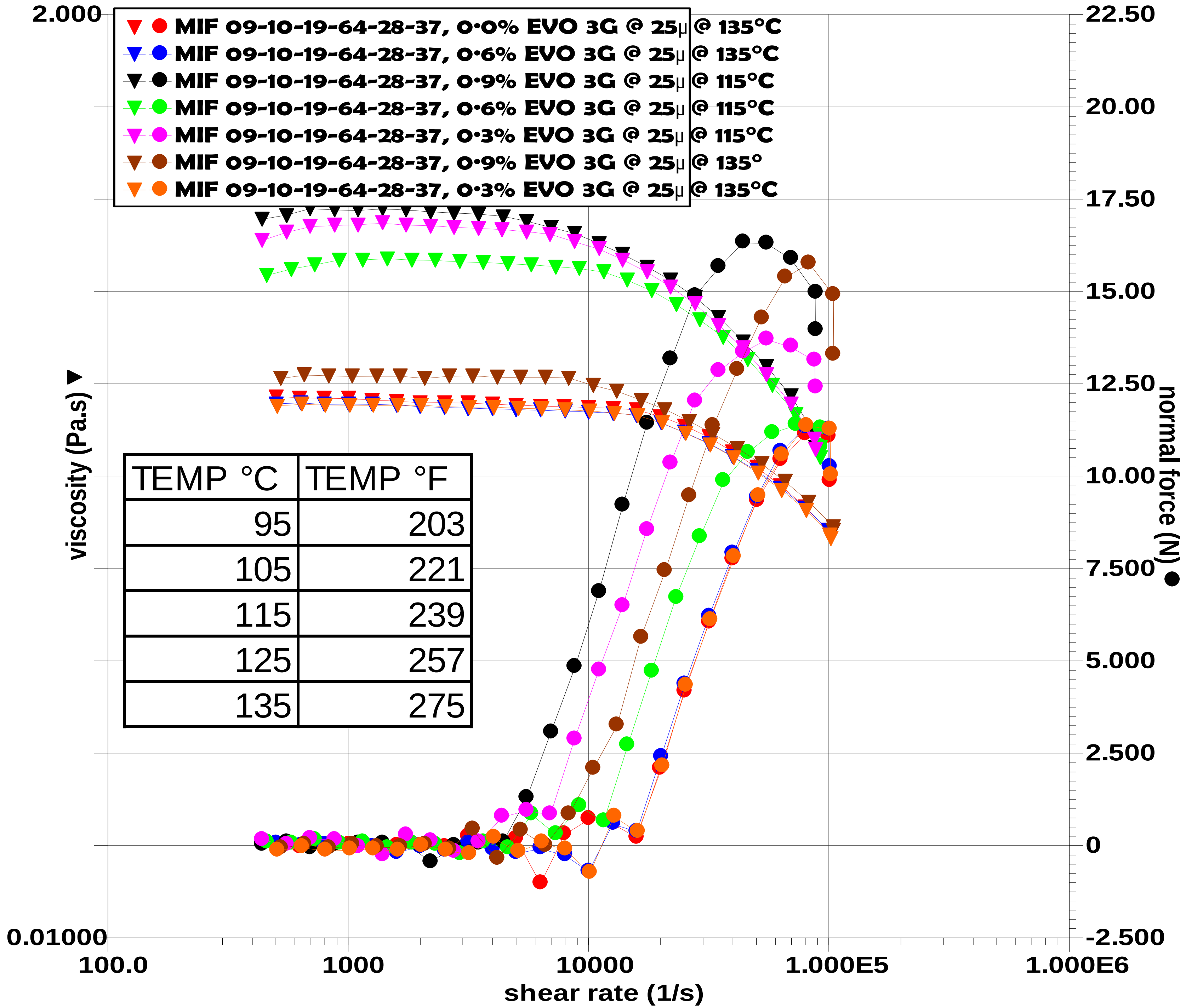


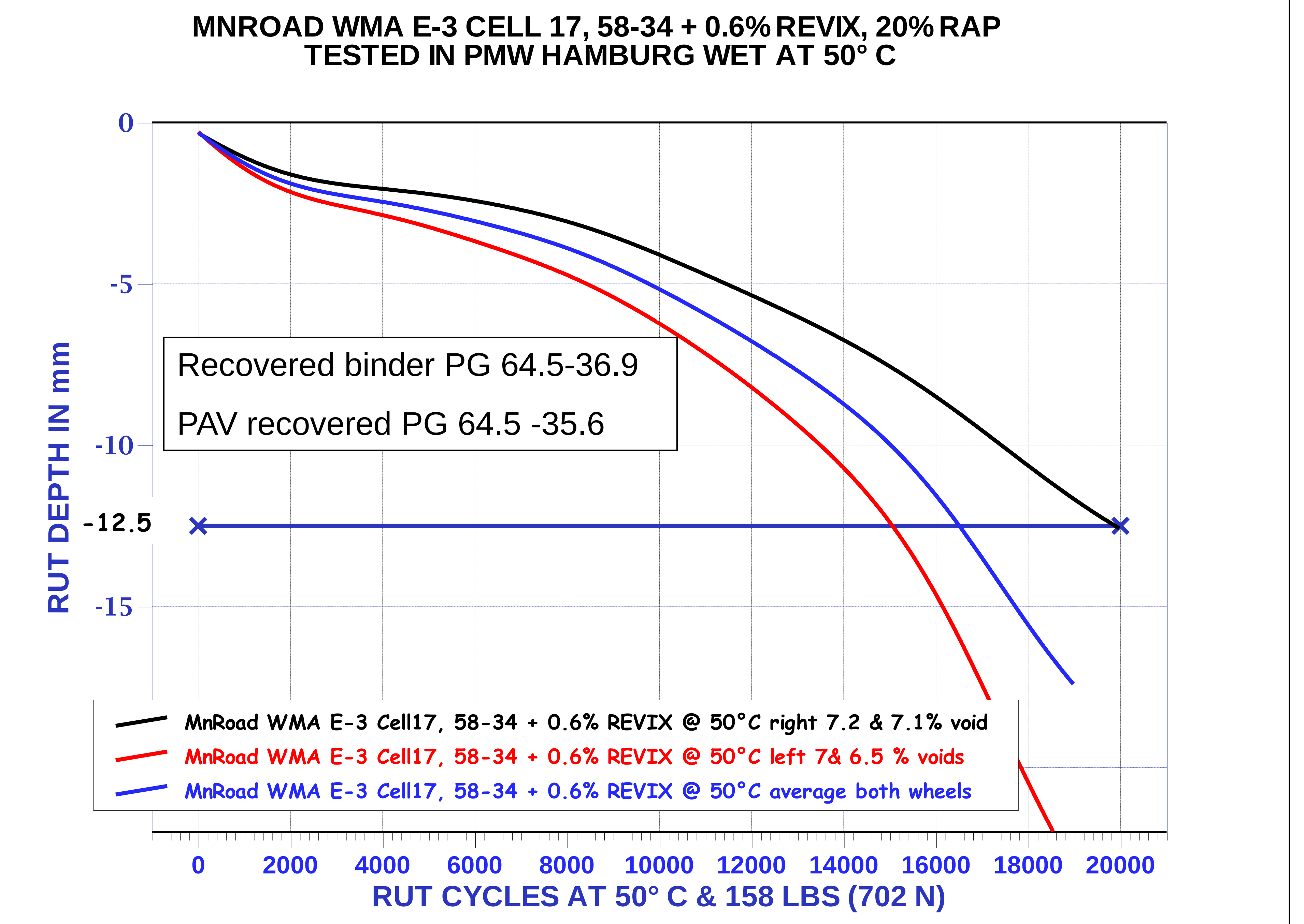
TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275

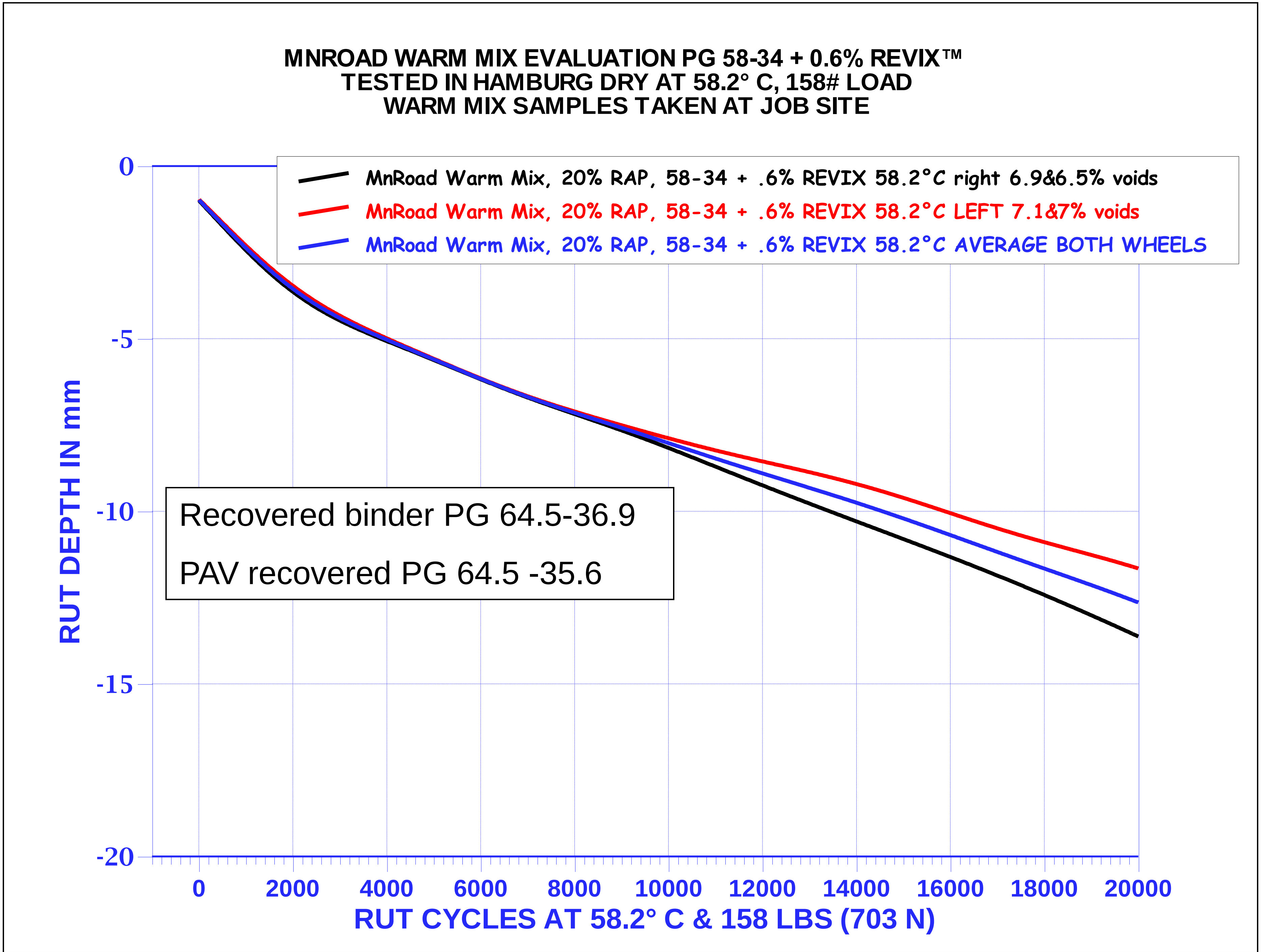


It appears as though you can have too much of a good thing

TEMP °C	TEMP °F
95	203
105	221
115	239
125	257
135	275


MIF 09-10-19-64-28-37, 0·0% EVO 3G @ 25μ @ 135°C
MIF 09-10-19-64-28-37, 0·6% EVO 3G @ 25μ @ 135°C
MIF 09-10-19-64-28-37, 0·9% EVO 3G @ 25μ @ 115°C
MIF 09-10-19-64-28-37, 0·6% EVO 3G @ 25μ @ 115°C
MIF 09-10-19-64-28-37, 0·3% EVO 3G @ 25μ @ 115°C
MIF 09-10-19-64-28-37, 0·9% EVO 3G @ 25μ @ 135°
MIF 09-10-19-64-28-37, 0·3% EVO 3G @ 25μ @ 135°C
TEMP °C TEMP °F
95 203
105 221
115 239
125 257
135 275
viscosity (Pa.s)
normal force (N)
shear rate (1/s)
2.000
0.01000
22.50
20.00
17.50
15.00
12.50
10.00
7.500
5.000
2.500
0
-2.500
100.0
1000
10000
1.000E5
1.000E6


MNROAD WMA E-3 CELL 17, 58-34 + 0.6% REVIX, 20% RAP
TESTED IN PMW HAMBURG WET AT 50° C
Recovered binder PG 64.5-36.9
PAV recovered PG 64.5 -35.6
0
-5
-10
-12.5
-15
RUT DEPTH IN mm
MnRoad WMA E-3 Cell17, 58-34 + 0.6% REVIX @ 50°C right 7.2 & 7.1% void
MnRoad WMA E-3 Cell17, 58-34 + 0.6% REVIX @ 50°C left 7& 6.5 % voids
MnRoad WMA E-3 Cell17, 58-34 + 0.6% REVIX @ 50°C average both wheels
0
2000
4000
6000
8000
10000
12000
14000
16000
18000
20000
RUT CYCLES AT 50° C & 158 LBS (702 N)


MNROAD WARM MIX EVALUATION PG 58-34 + 0.6% REVIX™
TESTED IN HAMBURG DRY AT 58.2° C, 158# LOAD
WARM MIX SAMPLES TAKEN AT JOB SITE
MnRoad Warm Mix, 20% RAP, 58-34 + .6% REVIX 58.2°C right 6.9&6.5% voids
MnRoad Warm Mix, 20% RAP, 58-34 + .6% REVIX 58.2°C LEFT 7.1&7% voids
MnRoad Warm Mix, 20% RAP, 58-34 + .6% REVIX 58.2°C AVERAGE BOTH WHEELS
Recovered binder PG 64.5-36.9
PAV recovered PG 64.5 -35.6
RUT DEPTH IN mm
0
-5
-10
-15
-20
0
2000
4000
6000
8000
10000
12000
14000
16000
18000
20000
RUT CYCLES AT 58.2° C & 158 LBS (703 N)

SOME FINAL THOUGHTS

1. Thin film rheology provides more information about PMA mix constructability than bulk rheology
2. Addition of surfactants can effectively enable the PMA binder to achieve higher shear rates at lower temperatures compared to untreated binder

SOME FINAL THOUGHTS

3. The normal force test is sensitive to surfactant type and surfactant loading level
4. This investigative approach requires additional work to fully understand the implications for identifying field performance

Thank you

QUESTIONS