# A Discussion of Peltier Cooling Compared to Chilled Air Cooling and 4 mm DSR Results (and related issues)

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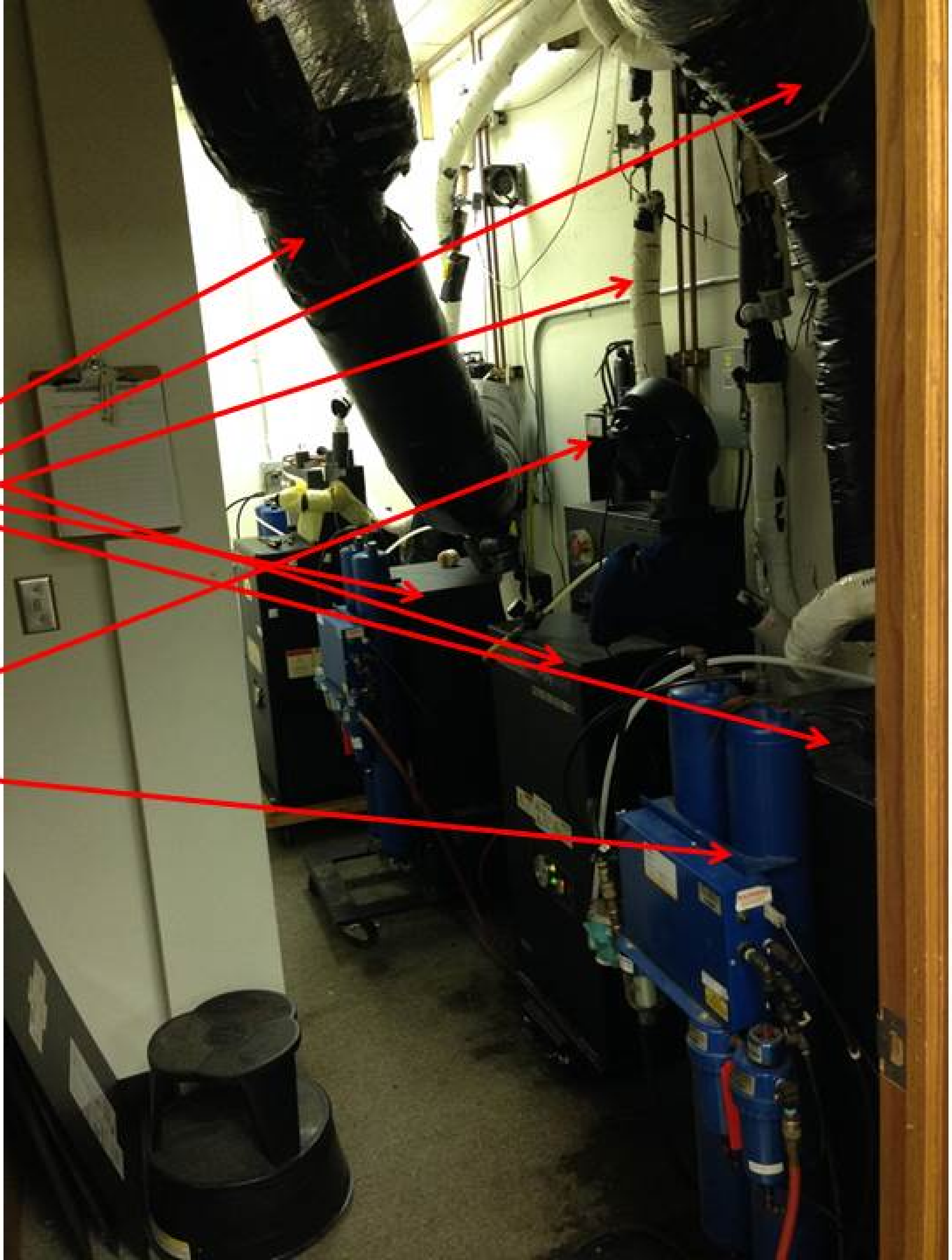
# STATEMENT OF PROBLEM

- In the course of working with a laboratory trying to implement the 4 mm DSR comparative testing between MTE and that lab was performed
- The laboratory was using a DSR with a Peltier temperature control system
- MTE DSR uses a chilled air system
- Significant variation in results when testing the same binder samples
- Investigation into factors causing the variation

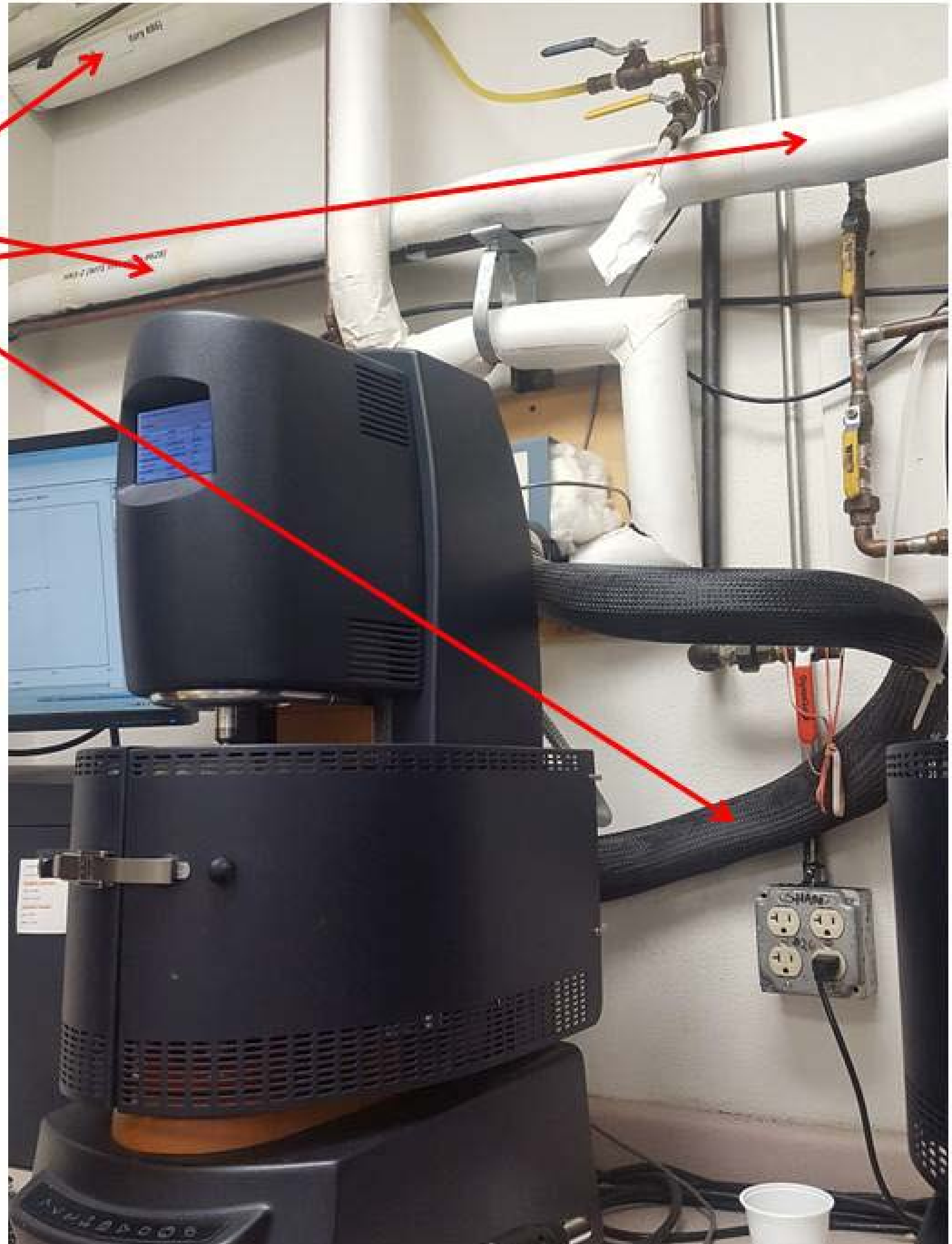
Forced Air Chillers, will cool to -70°C.

Air lines insulated,

Final Desiccant dryers on all chillers to make sure the air feeding the DSR is dry



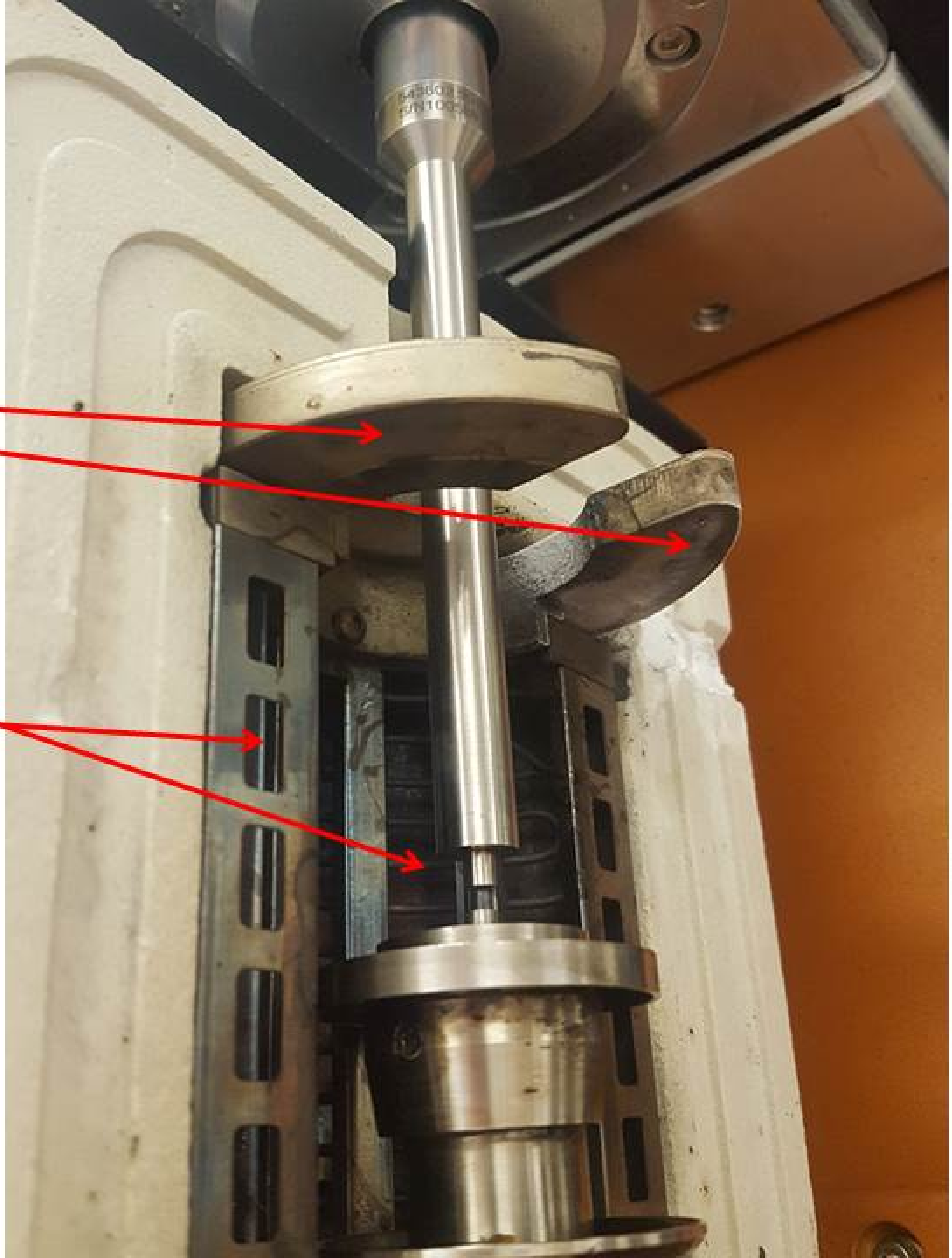
Insulated chilled air lines to cool the DSR test chamber



Chilled air flows out of small holes in these rings

Heat from coils counter the cold air and the electronics balance the chilled air and heated air to control the specimen temperature

Proper calibration essential



| RHEA G(t) @-12°C 1649, 01-20-17-D, 64-22 PAV, 4mm, HR3-4 | | | | | | |
|---|---|---|---|---|---|---|
| Critical temps based on 4 mm DSR test | | | | | | |
| TEMP | G)t) | m, creep | Log(G(t) | -24 | 231.47 | -0.1971 |
| **-24** | **231.47** | | 2.364503 | -18 | 130.96 | -0.2397 |
| -18 | 130.96 | -0.2397 | 2.117148 | -12 | 58.73 | -0.2922 |
| -12 | 58.73 | -0.2922 | | | | |
| -6 | | | | | | |
| **Critical values** | **143** | **-0.275** | 2.155336 | | | |
| slope | intercept | critical temps | | | | ΔTc |
| -0.041225836 | 1.375083 | -18.9263 | G(t) critical | **-28.926** | **-28.93** | **-4.96** |
| -0.008755333 | -0.3973 | -13.9686 | m critical | **-23.969** | **-23.97** | |

| RHEA G(t) @-24°C 1649, 01-20-17-D, 64-22 PAV, 4mm, HR3-2 | | | | | | |
|---|---|---|---|---|---|---|
| Critical temps based on 4 mm DSR test | | | | | | |
| TEMP | G)t) | m, creep | Log(G(t) | -24 | 235.46 | -0.1982 |
| **-24** | **235.46** | | 2.371923 | -18 | 133.16 | -0.2402 |
| -18 | 133.16 | -0.2402 | 2.1243581 | -12 | 58.93 | -0.2940 |
| -12 | 58.93 | -0.2940 | | | | |
| -6 | | | | -6 | | |
| **Critical values** | **143** | **-0.275** | 2.155336 | | | |
| slope | intercept | critical temps | | | | ΔTc |
| -0.041260824 | 1.381663 | -18.7508 | G(t) critical | **-28.751** | **-28.75** | **-4.63** |
| -0.0089671 | -0.40161 | -14.1195 | m critical | **-24.119** | **-24.12** | |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **Std Dev** | | **ΔTc Std dev** | **COV** | | **ΔTc COV** | | **Average** | | **ΔTc Avg** |
| | **0.1241** | **0.2308** | **$T_{S\text{-}Critical}$** | **0.4%** | **4.8%** | | **$T_{S\text{-}Critical}$** | **-28.84** | **-4.79** |
| **$T_{m\text{-}Critical}$** | **0.1067** | | **$T_{m\text{-}Critical}$** | **0.4%** | | | **$T_{m\text{-}Critical}$** | **-24.04** | |

**Data at RHEA G(t) @-18°C Peltier cooling 64-22 PAV - C 0_02 #1**

Critical temps based on 4 mm DSR test

| | | | | | | |
|---|---|---|---|---|---|---|
| TEMP | G)t) | m, creep | Log(G(t) | -30 | 271.14 | -0.1858 |
| -30 | 271.14 | | | -24 | 171.67 | -0.2205 |
| **-24** | **171.67** | | 2.234703 | -18 | 93.07 | -0.2588 |
| -18 | 93.07 | -0.2588 | 1.9688055 | -12 | 46.16 | -0.3043 |
| -12 | 46.16 | -0.3043 | | -6 | | |
| **Critical values** | **143** | **-0.275** | 2.155336 | | | |
| slope | intercept | critical temps | | | | ΔTc |
| -0.044316249 | 1.171113 | -22.2091 | $T_{S-Critical}$ | **-32.209** | **-32.21** | **-6.34** |
| -0.00758635 | -0.39538 | -15.8673 | $T_{m-Critical}$ | **-25.867** | **-25.87** | |

# SUMMARY PG 64-22 PAV

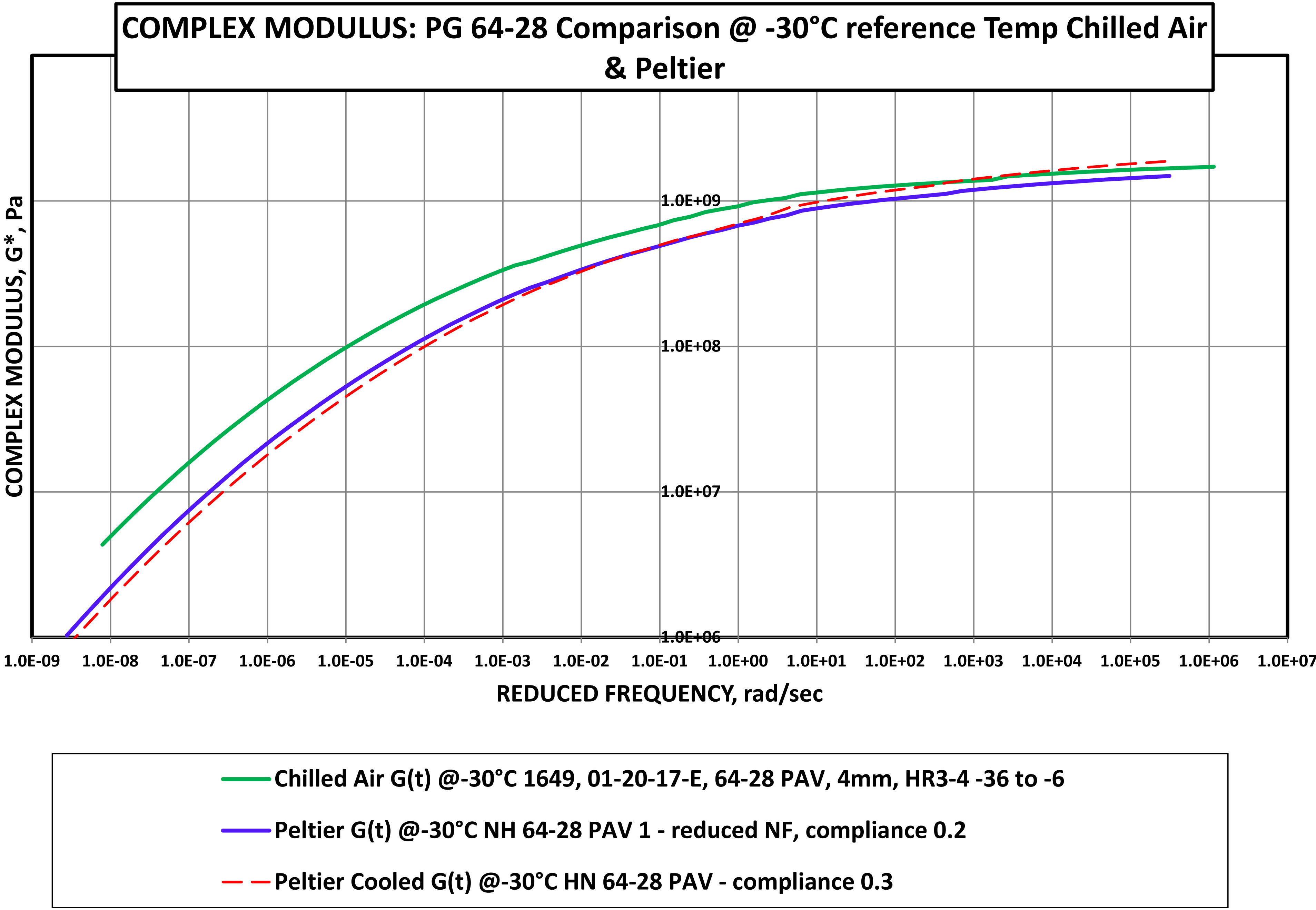
| COOLING TYPE | $\Delta T_{S\text{-critical}}$ | $\Delta T_{m\text{-critical}}$ | ΔTc |
|---|---|---|---|
| PELTIER | -32.21 | -25.87 | -6.34 |
| CHILLED AIR | -28.84 | -24.04 | -4.79 |

| $\Delta(\Delta T_{S\text{-critical}})$ | $\Delta(\Delta T_{m\text{-critical}})$ | Δ(ΔTc) |
|---|---|---|
| -3.37 | -1.83 | -1.55 |

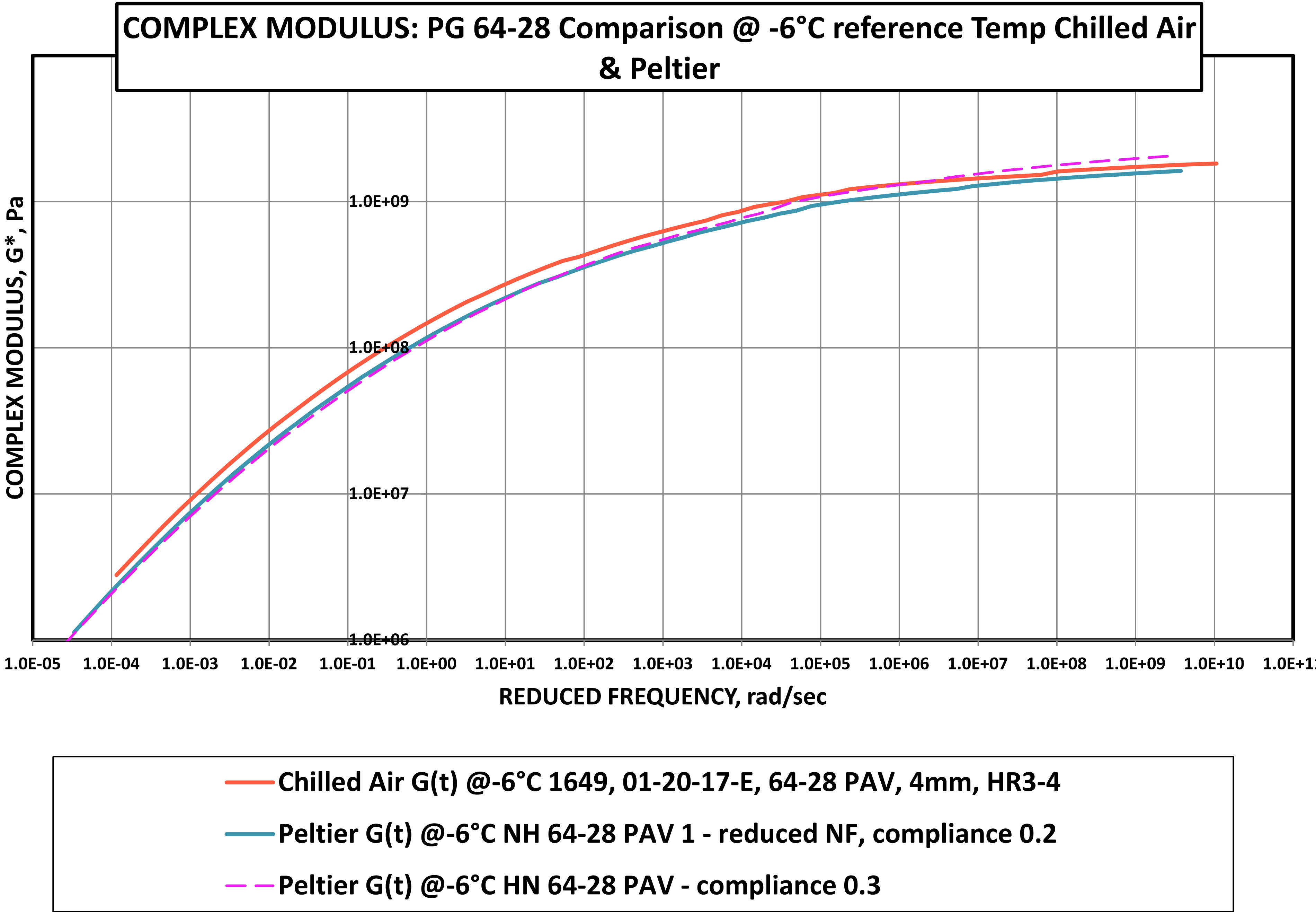
# PG 58-28 PAV Residue

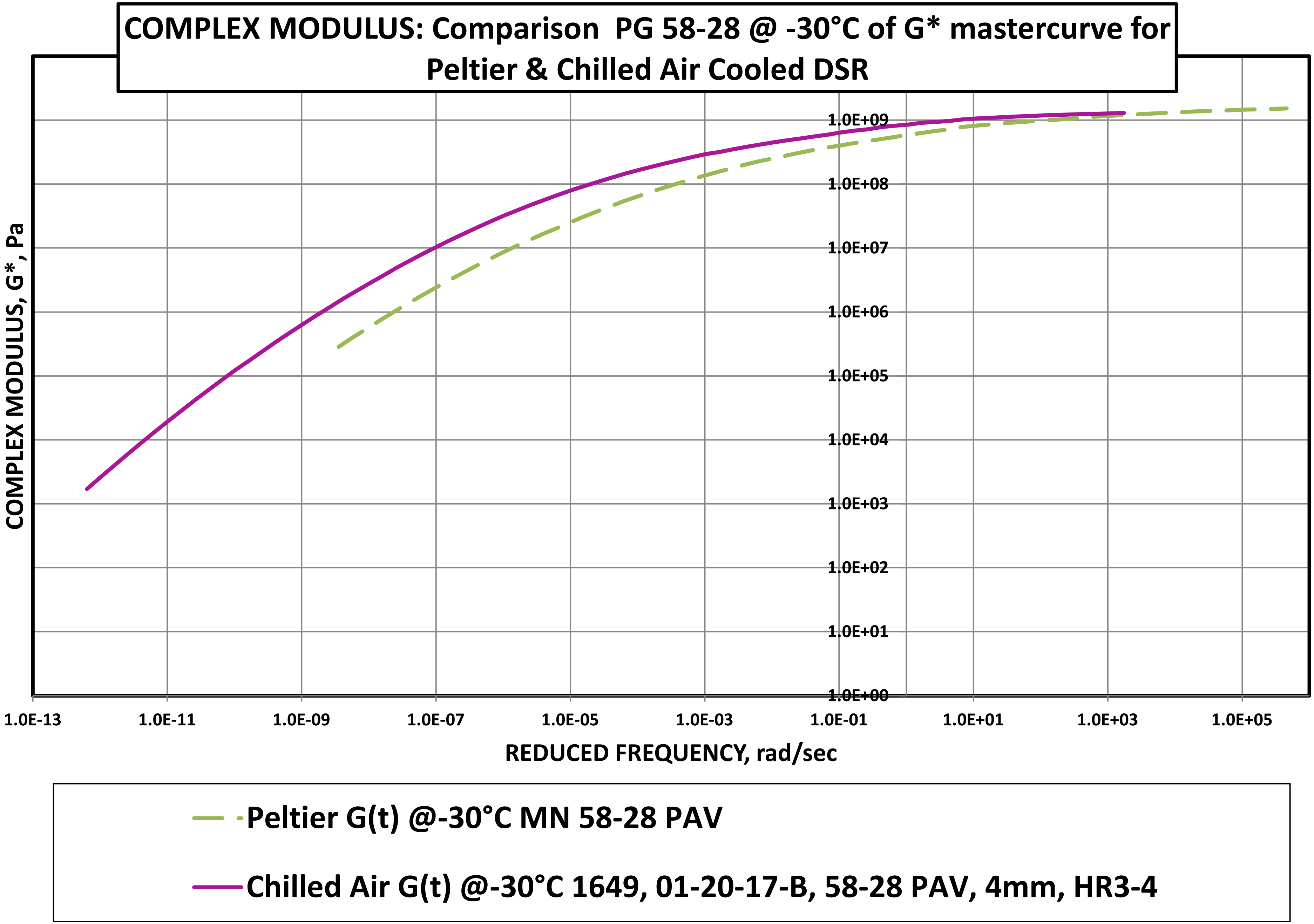
| | | | | | | Relative to BBR Results | | Peltier relative to Air Results | |
|---|---|---|---|---|---|---|---|---|---|
| Test Device | $T_{S\text{-critical}}$ | $T_{m\text{-critical}}$ | ΔTc | Δ(ΔTc) | Limiting Grade | $\Delta T_{S\text{-critical}}$ | $\Delta T_{m\text{-critical}}$ | $\Delta T_{S\text{-critical}}$ | $\Delta T_{m\text{-critical}}$ |
| **BBR** | **-29.91** | **-31.02** | **1.11** | | **-29.9** | | | | |
| **4 mm DSR using chilled air system** | **-29.90** | **-29.59** | **-0.32** | **1.43** | **-29.6** | **0.01** | **-1.43** | | |
| **4 mm Peltier circulator at -15** | **-34.16** | **-33.16** | **-1.00** | **2.11** | **-33.2** | **-4.25** | **-2.14** | **-4.26** | **-3.57** |
| **4 mm Peltier circulator at -18** | **-33.16** | **-33.25** | **0.09** | **1.02** | **-33.2** | **-3.25** | **-2.23** | **-3.26** | **-3.66** |

| DSR Unit | $T_{S\text{-Critical}}$ | $T_{m\text{-Critical}}$ | ΔTc |
|---|---|---|---|
| HR3-3 | -29.92 | -29.75 | -0.16 |
| HR3-2 | -29.89 | -29.42 | -0.47 |
| Average | -29.90 | -29.59 | -0.32 |

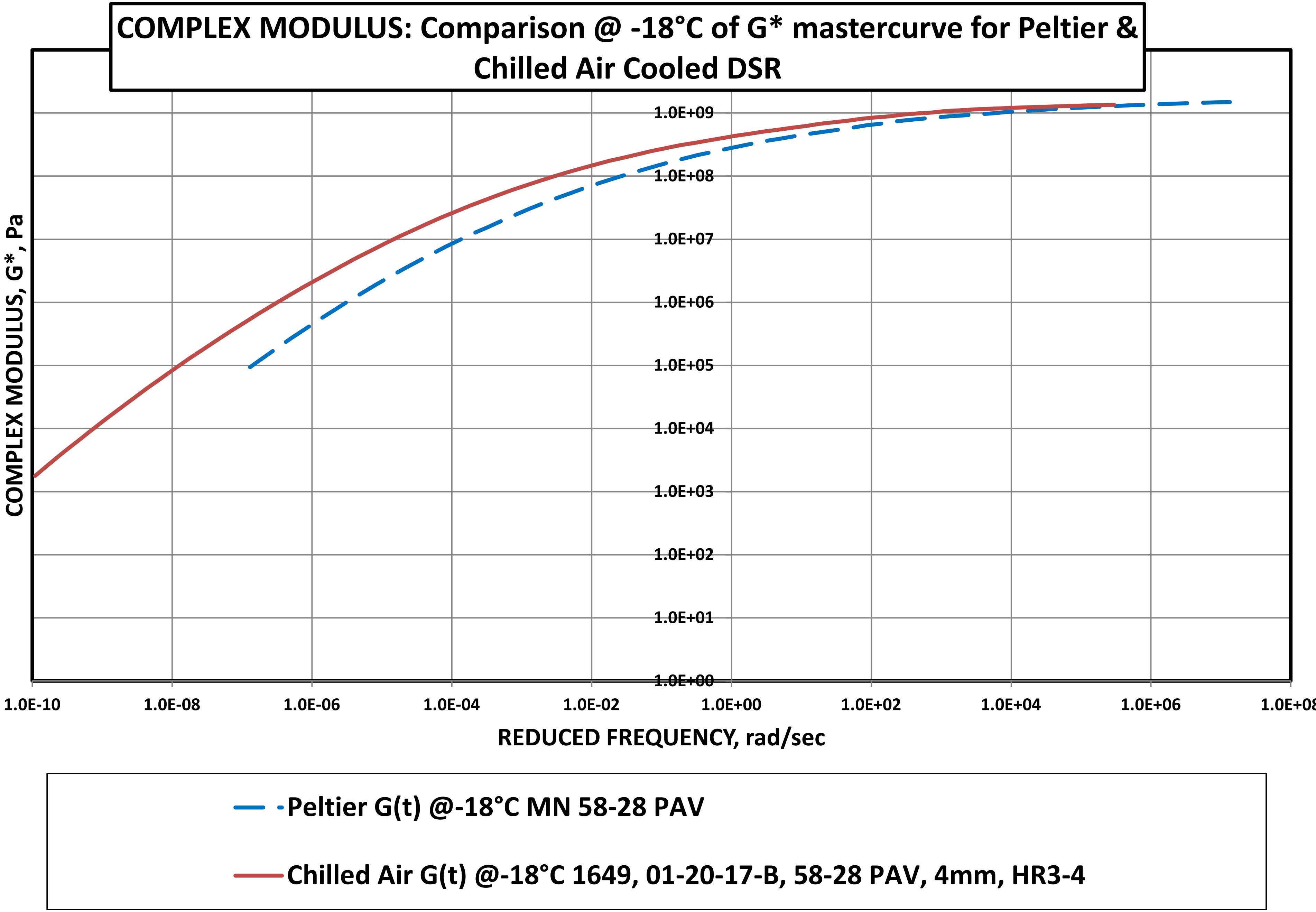


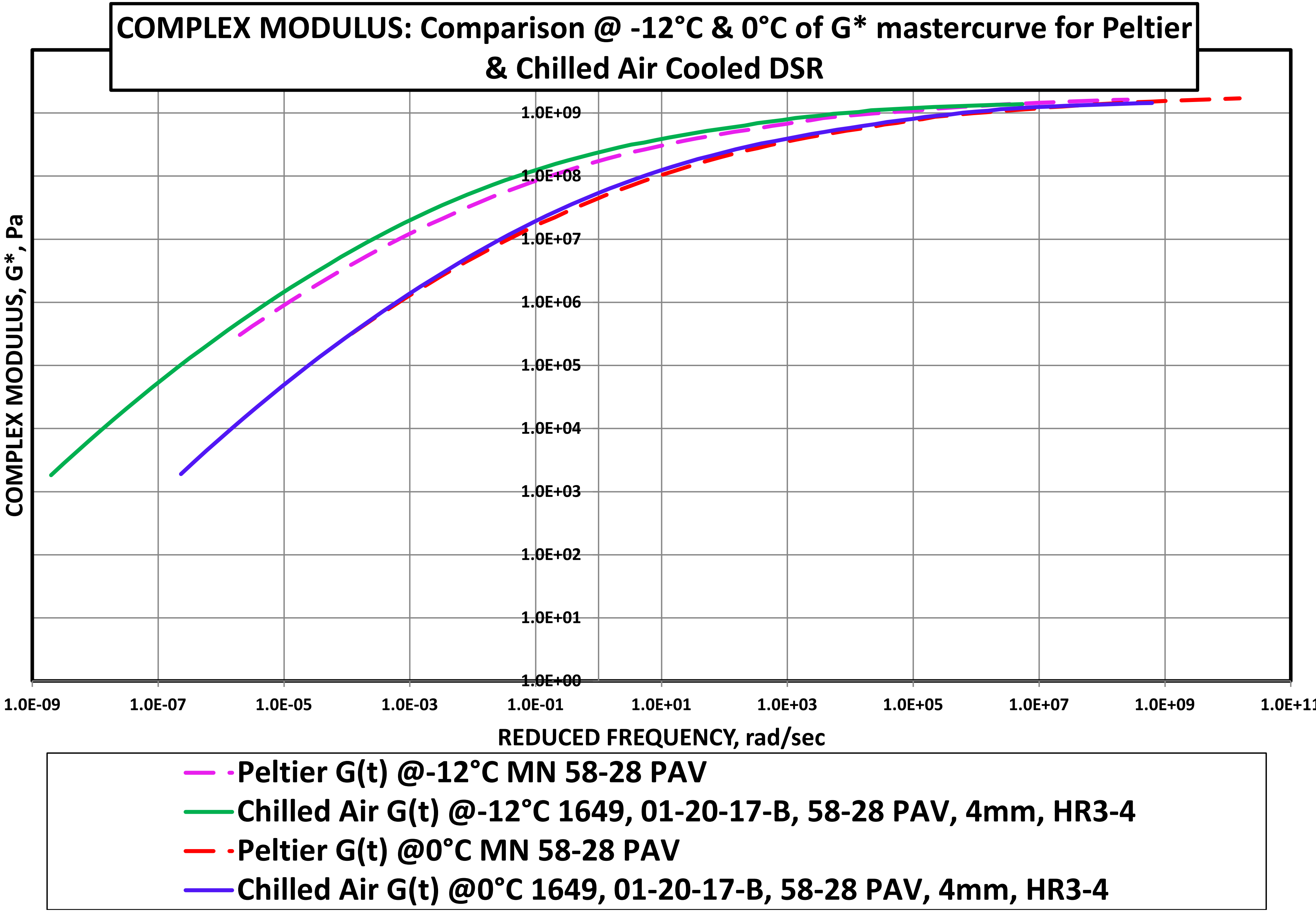
COMPLEX MODULUS: PG 64-28 Comparison @ -30°C reference Temp Chilled Air & Peltier
COMPLEX MODULUS, G*, Pa
1.0E+09
1.0E+08
1.0E+07
1.0E+06
1.0E-09
1.0E-08
1.0E-07
1.0E-06
1.0E-05
1.0E-04
1.0E-03
1.0E-02
1.0E-01
1.0E+00
1.0E+01
1.0E+02
1.0E+03
1.0E+04
1.0E+05
1.0E+06
1.0E+07
REDUCED FREQUENCY, rad/sec
Chilled Air G(t) @-30°C 1649, 01-20-17-E, 64-28 PAV, 4mm, HR3-4 -36 to -6
Peltier G(t) @-30°C NH 64-28 PAV 1 - reduced NF, compliance 0.2
Peltier Cooled G(t) @-30°C HN 64-28 PAV - compliance 0.3


COMPLEX MODULUS: PG 64-28 Comparison @ -6°C reference Temp Chilled Air & Peltier
COMPLEX MODULUS, G*, Pa
1.0E+09
1.0E+08
1.0E+07
1.0E+06
1.0E-05
1.0E-04
1.0E-03
1.0E-02
1.0E-01
1.0E+00
1.0E+01
1.0E+02
1.0E+03
1.0E+04
1.0E+05
1.0E+06
1.0E+07
1.0E+08
1.0E+09
1.0E+10
1.0E+11
REDUCED FREQUENCY, rad/sec
Chilled Air G(t) @-6°C 1649, 01-20-17-E, 64-28 PAV, 4mm, HR3-4
Peltier G(t) @-6°C NH 64-28 PAV 1 - reduced NF, compliance 0.2
Peltier G(t) @-6°C HN 64-28 PAV - compliance 0.3

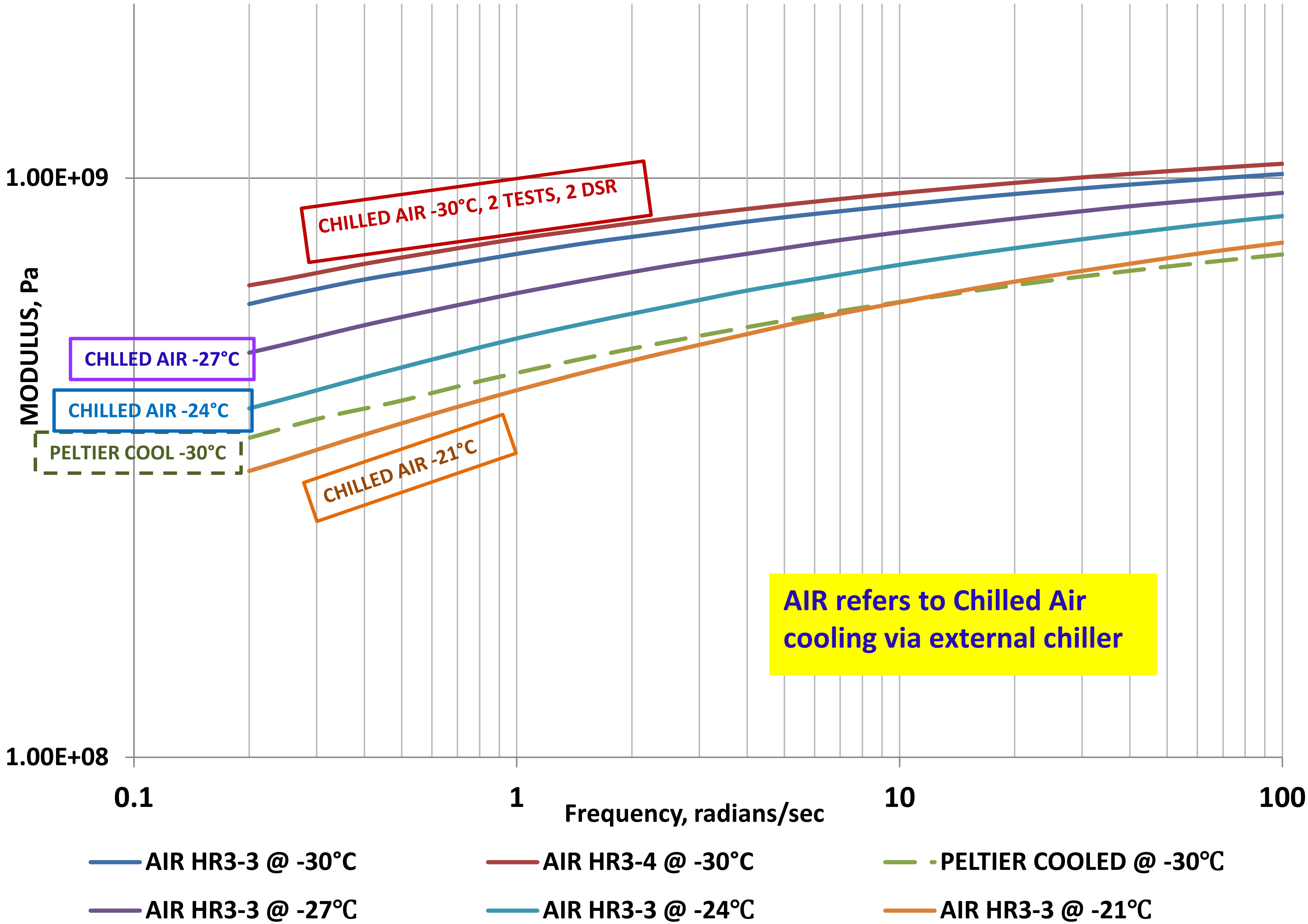


COMPLEX MODULUS: Comparison PG 58-28 @ -30°C of G* mastercurve for
Peltier & Chilled Air Cooled DSR
COMPLEX MODULUS, G*, Pa
1.0E+09
1.0E+08
1.0E+07
1.0E+06
1.0E+05
1.0E+04
1.0E+03
1.0E+02
1.0E+01
1.0E+00
1.0E-13
1.0E-11
1.0E-09
1.0E-07
1.0E-05
1.0E-03
1.0E-01
1.0E+01
1.0E+03
1.0E+05
REDUCED FREQUENCY, rad/sec
Peltier G(t) @-30°C MN 58-28 PAV
Chilled Air G(t) @-30°C 1649, 01-20-17-B, 58-28 PAV, 4mm, HR3-4

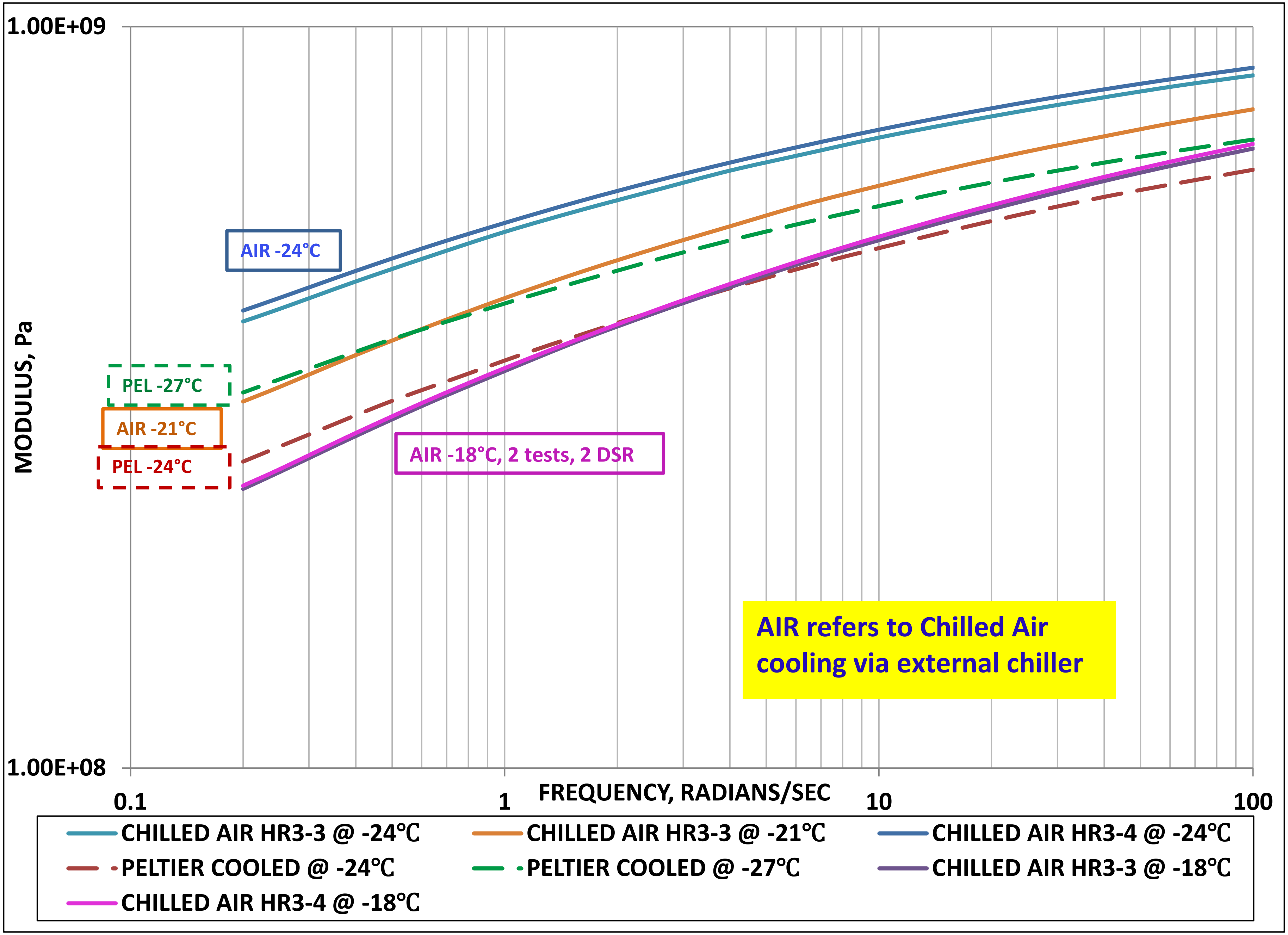

COMPLEX MODULUS: Comparison @ -18°C of G* mastercurve for Peltier & Chilled Air Cooled DSR
COMPLEX MODULUS, G*, Pa
1.0E+09
1.0E+08
1.0E+07
1.0E+06
1.0E+05
1.0E+04
1.0E+03
1.0E+02
1.0E+01
1.0E+00
1.0E-10
1.0E-08
1.0E-06
1.0E-04
1.0E-02
1.0E+00
1.0E+02
1.0E+04
1.0E+06
REDUCED FREQUENCY, rad/sec
Peltier G(t) @-18°C MN 58-28 PAV
Chilled Air G(t) @-18°C 1649, 01-20-17-B, 58-28 PAV, 4mm, HR3-4



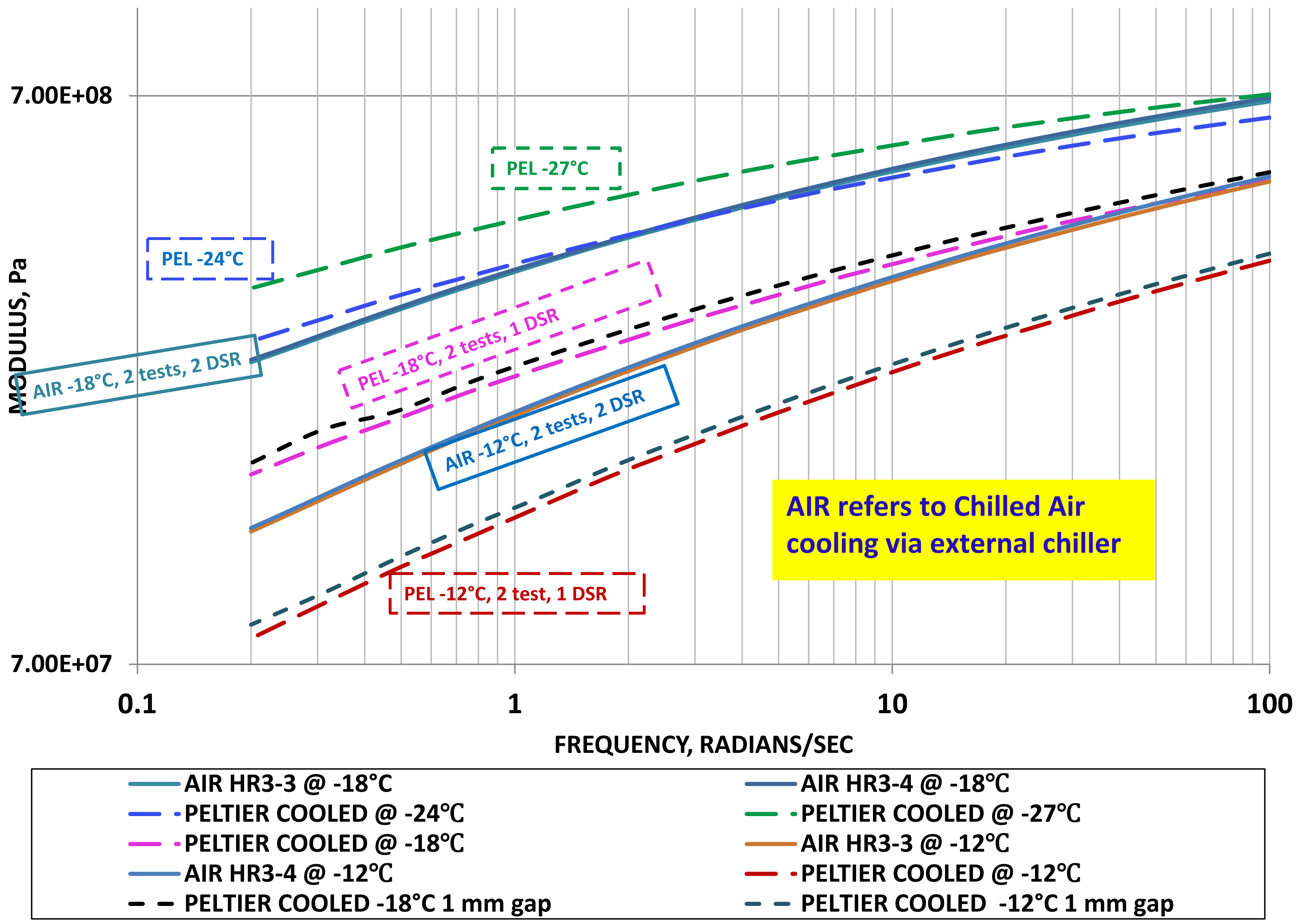
COMPLEX MODULUS: Comparison @ -12°C & 0°C of G* mastercurve for Peltier & Chilled Air Cooled DSR
COMPLEX MODULUS, G*, Pa
1.0E+09
1.0E+08
1.0E+07
1.0E+06
1.0E+05
1.0E+04
1.0E+03
1.0E+02
1.0E+01
1.0E+00
1.0E-09
1.0E-07
1.0E-05
1.0E-03
1.0E-01
1.0E+01
1.0E+03
1.0E+05
1.0E+07
1.0E+09
1.0E+11
REDUCED FREQUENCY, rad/sec
Peltier G(t) @-12°C MN 58-28 PAV
Chilled Air G(t) @-12°C 1649, 01-20-17-B, 58-28 PAV, 4mm, HR3-4
Peltier G(t) @0°C MN 58-28 PAV
Chilled Air G(t) @0°C 1649, 01-20-17-B, 58-28 PAV, 4mm, HR3-4



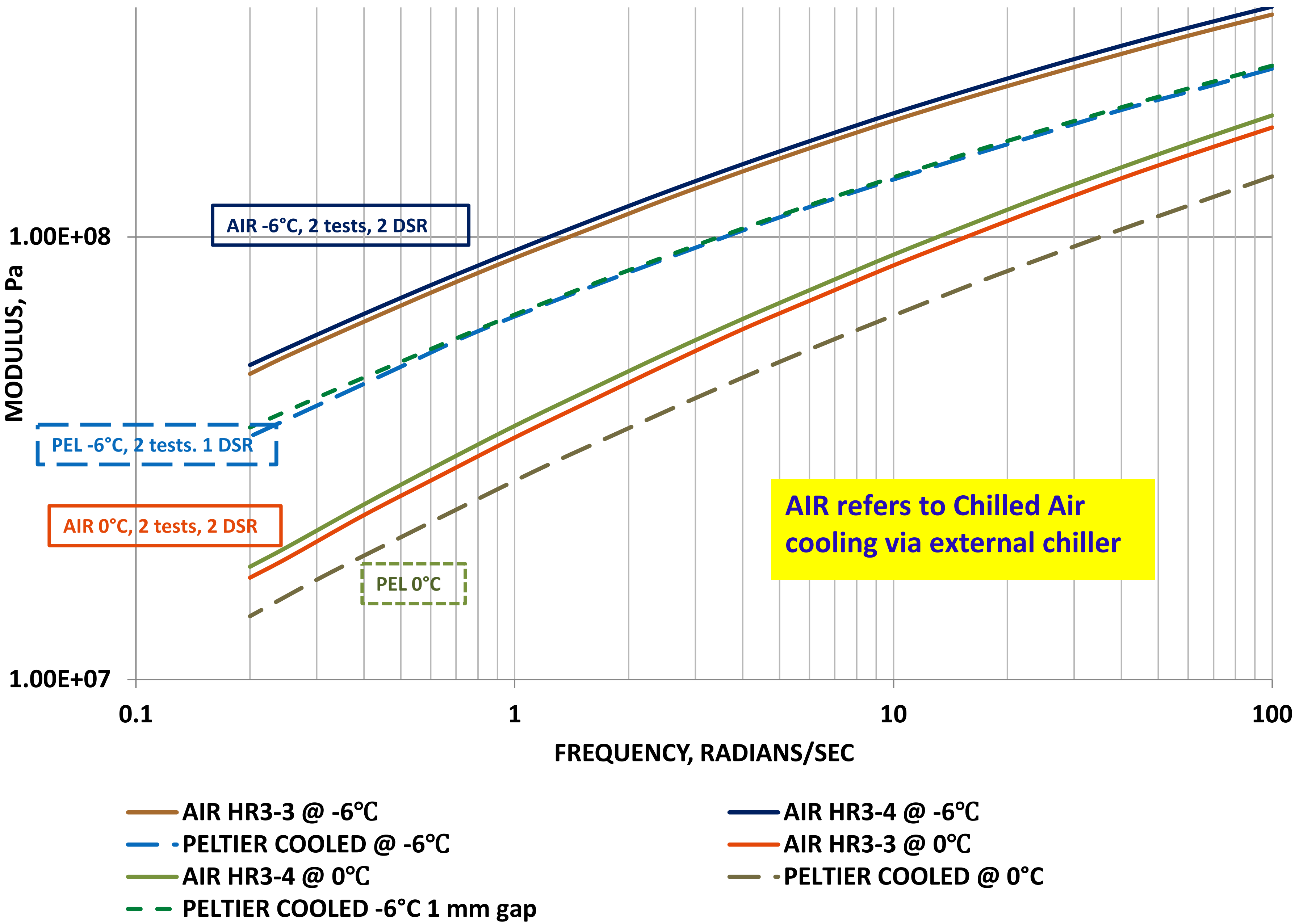
1.00E+09
1.00E+08
MODULUS, Pa
0.1
1
10
100
Frequency, radians/sec
CHILLED AIR -30°C, 2 TESTS, 2 DSR
CHLLED AIR -27°C
CHILLED AIR -24°C
PELTIER COOL -30°C
CHILLED AIR -21°C
AIR refers to Chilled Air cooling via external chiller
AIR HR3-3 @ -30°C
AIR HR3-4 @ -30°C
PELTIER COOLED @ -30°C
AIR HR3-3 @ -27°C
AIR HR3-3 @ -24°C
AIR HR3-3 @ -21°C



1.00E+09
1.00E+08
MODULUS, Pa
AIR -24°C
PEL -27°C
AIR -21°C
PEL -24°C
AIR -18°C, 2 tests, 2 DSR
AIR refers to Chilled Air cooling via external chiller
0.1
1
10
100
FREQUENCY, RADIANS/SEC
CHILLED AIR HR3-3 @ -24°C
CHILLED AIR HR3-3 @ -21°C
CHILLED AIR HR3-4 @ -24°C
PELTIER COOLED @ -24°C
PELTIER COOLED @ -27°C
CHILLED AIR HR3-3 @ -18°C
CHILLED AIR HR3-4 @ -18°C



7.00E+08
7.00E+07
MODULUS, Pa
0.1
1
10
100
FREQUENCY, RADIANS/SEC
PEL -27°C
PEL -24°C
AIR -18°C, 2 tests, 2 DSR
PEL -18°C, 2 tests, 1 DSR
AIR -12°C, 2 tests, 2 DSR
PEL -12°C, 2 test, 1 DSR
AIR refers to Chilled Air cooling via external chiller
AIR HR3-3 @ -18°C
AIR HR3-4 @ -18°C
PELTIER COOLED @ -24°C
PELTIER COOLED @ -27°C
PELTIER COOLED @ -18°C
AIR HR3-3 @ -12°C
AIR HR3-4 @ -12°C
PELTIER COOLED @ -12°C
PELTIER COOLED -18°C 1 mm gap
PELTIER COOLED -12°C 1 mm gap

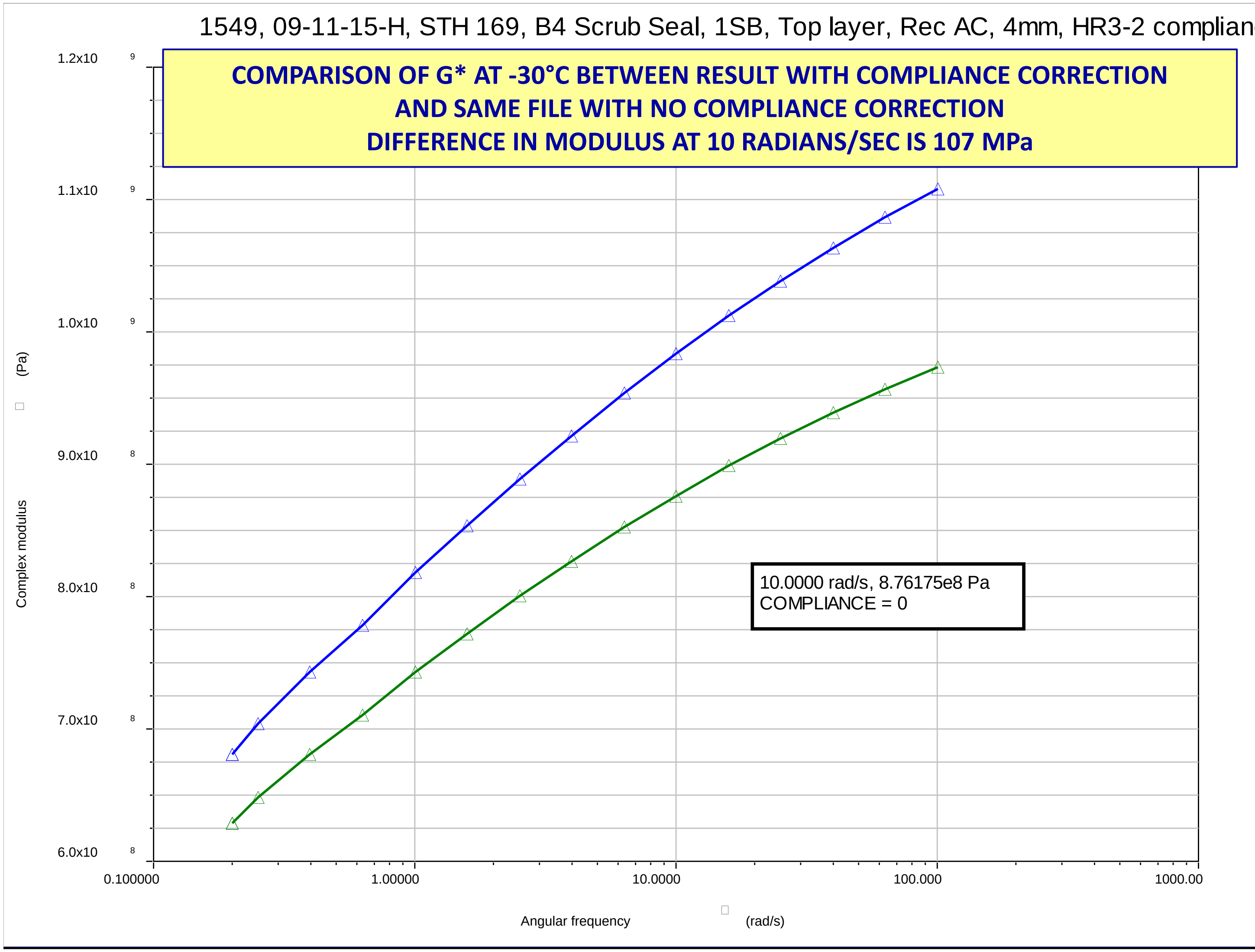


MODULUS, Pa
1.00E+08
1.00E+07
0.1
1
10
100
FREQUENCY, RADIANS/SEC
AIR -6°C, 2 tests, 2 DSR
PEL -6°C, 2 tests. 1 DSR
AIR 0°C, 2 tests, 2 DSR
PEL 0°C
AIR refers to Chilled Air cooling via external chiller
AIR HR3-3 @ -6℃
AIR HR3-4 @ -6℃
PELTIER COOLED @ -6℃
AIR HR3-3 @ 0℃
AIR HR3-4 @ 0℃
PELTIER COOLED @ 0°C
PELTIER COOLED -6°C 1 mm gap

# COMPLIANCE CORRECTION

- Compliance correction is essential for obtaining accurate results at low temperatures
- If the temperature is wrong the compliance correction can't fix that issue
- Depending on the machine compliance correction importance diminishes at around 0°C for the 4 mm test

## II. FACTORS AFFECTING 4 mm DSR TEST and RESULTS--COMPLIANCE



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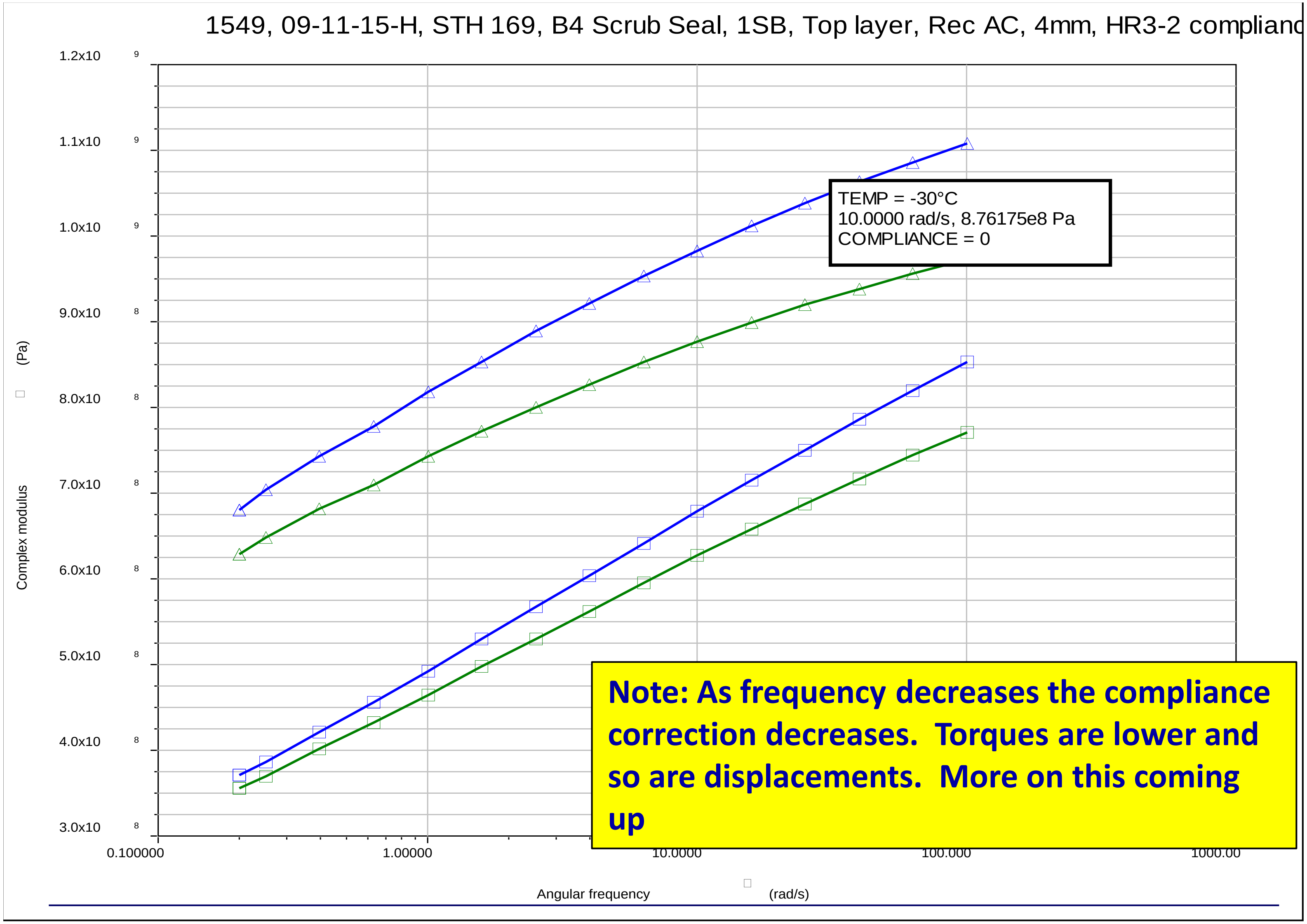
## II. FACTORS AFFECTING 4 mm DSR TEST and RESULTS--COMPLIANCE




**Note: As frequency decreases the compliance correction decreases. Torques are lower and so are displacements. More on this coming up**

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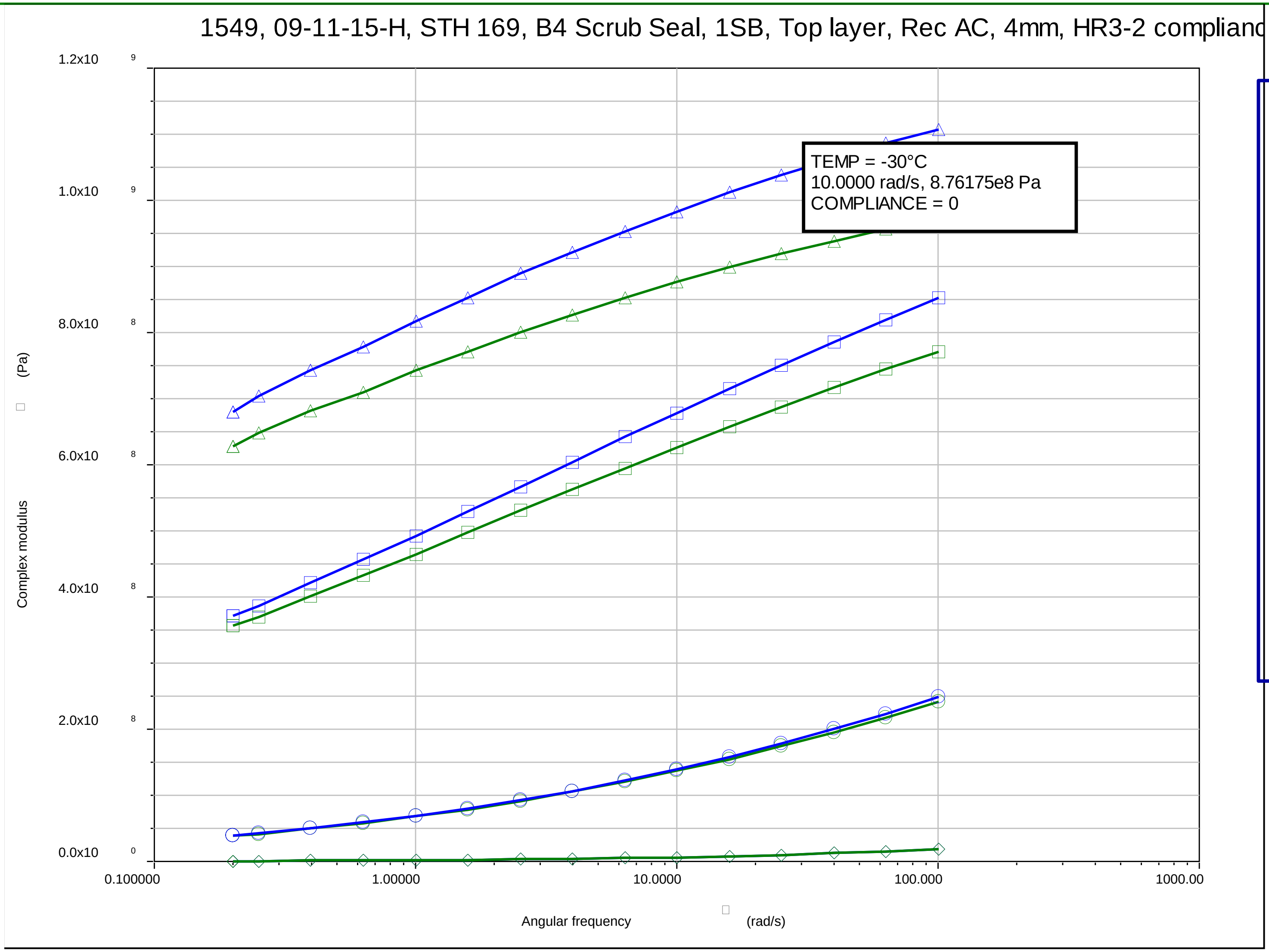
## II. FACTORS AFFECTING 4 mm DSR TEST and RESULTS--COMPLIANCE




When the test temp reaches 0°C the compliance correction is negligible even at 100 rad/sec. Why is this when the binder stiffness is still quite high? It really comes down to the relationship between torque and angular displacement

## II. FACTORS AFFECTING 4 mm DSR TEST and RESULTS--COMPLIANCE

### IMPACT OF TORQUE & ANGULAR DISPLACEMENT AT DIFFERENT TEMPERATURES ON THE LEVEL OF COMPLIANCE CORRECTION

| Temperature, °C | Torque @ 10 rad/sec, µN·m | Angular Displacement, θ, radians | Compliance Correction, mrad/N·m | Displacement correction due to torque, radians | Percent of displacement due to correction |
|---|---|---|---|---|---|
| -30 | 563 | 5.56E-05 | 10.82 | 6.09E-06 | 10.9% |
| -21 | 820 | 1.14E-04 | 10.82 | 8.87E-06 | 7.8% |
| 0 | 1842 | 1.1699 | 10.82 | 1.99E-05 | 0.002% |
| 25 | 1566 | 0.0212 | 10.82 | 1.69441E-05 | 0.080% |

**To paraphrase the ads for stock market investments**
**“Individual results may vary”**
**Compliance Correction is machine and geometry dependent**

# Summary Comments

- The solution to the issue of uniform and accurate Peltier temperatures must be addressed by the DSR manufacturers
  - Not an asphalt issue, but an equipment issue
- A reliable method of temperature calibration needed down to -40°C
- Peltier appears to have unique issues with respect to uniformity of temperature between lower plate and upper geometry
- Chilled air or LN2 provides uniformity of cooling (calibration still important)
- The BBR result is still the basis of comparison

# Summary Comments

- An uniform method of data analysis needs to be developed and used
  - WRI and MTE have used Abatech RHEA software as have a few others
  - The relationship of 300 MPa BBR S(t) = 143 MPa G(t) and -0.300 BBR slope value = -0.28 or -0.275 4 mm slope has been used successfully
  - Uniformity of data analysis method is needed, there can't be multiple relationships between BBR and 4 mm

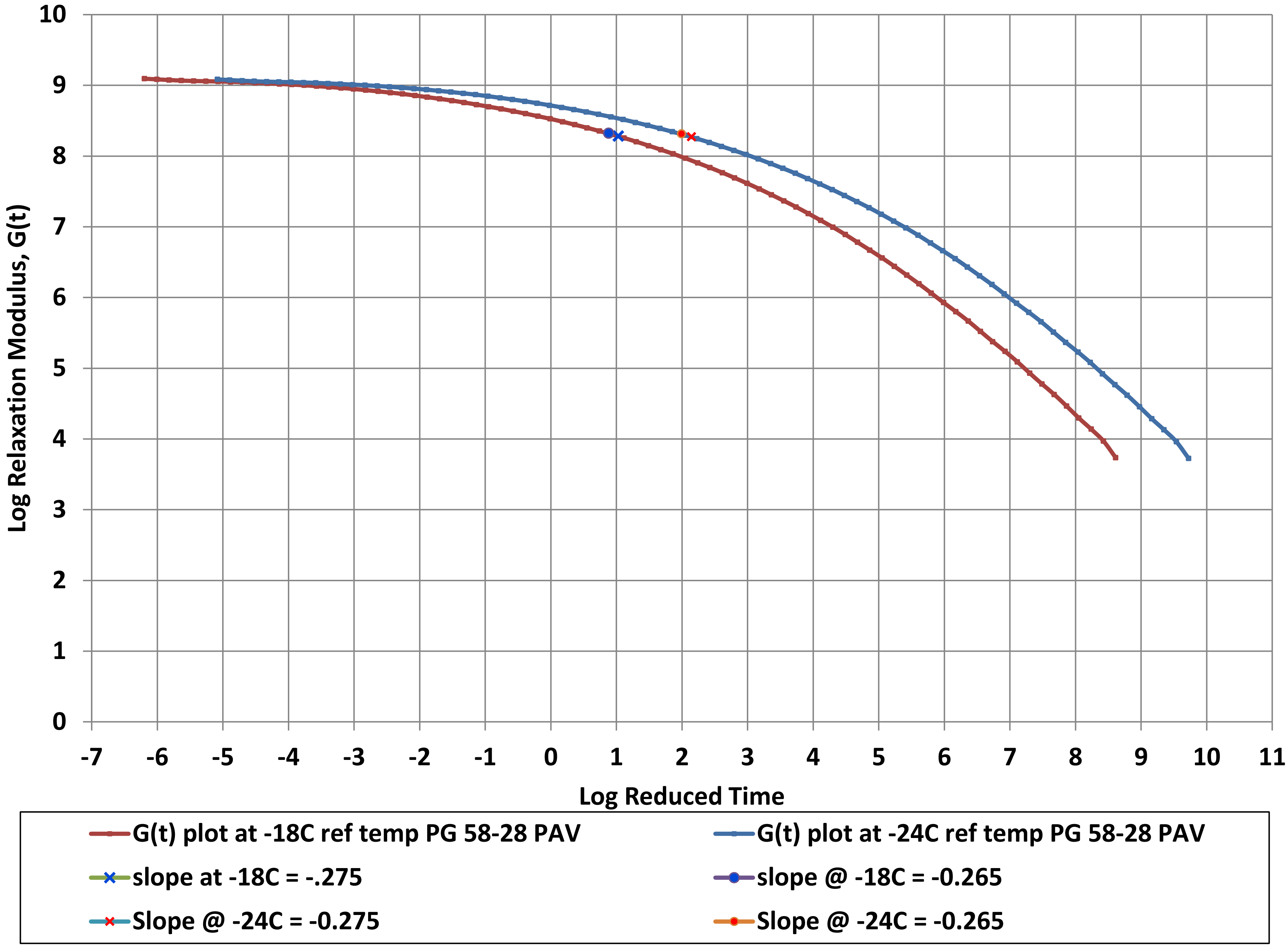
# IMPACT OF MINOR CHANGES IN THE SLOPE CRITICAL VALUE

| When | BBR m-value = -0.300 and 4 mm m-value = -0.275 | | | | |
|---|---|---|---|---|---|
| Test Device | S Critical Temp | m Critical temp | ΔTc | Δ(ΔTc) | LT PG Grade |
| BBR | -29.91 | -31.02 | **1.11** | **1.43** | -29.9 |
| 4 mm HR3-3 | -29.90 | -29.59 | **-0.32** | | -29.6 |

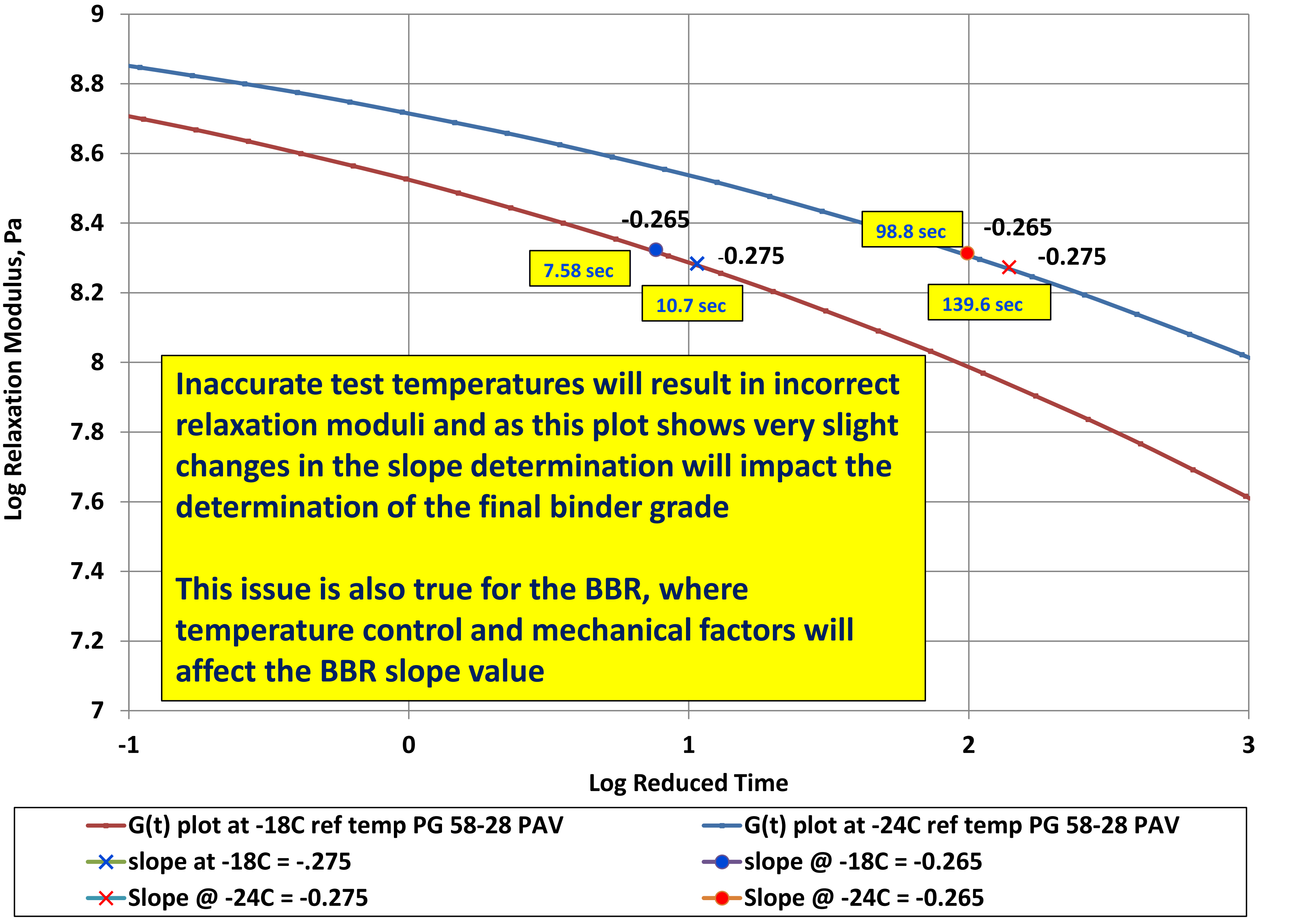
| WHAT IF | THE 4 mm m-value target slope is -0.265 rather than -0.275 | | | | |
|---|---|---|---|---|---|
| Test Device | S Critical Temp | m Critical temp | ΔTc | Δ(ΔTc) | LT PG Grade |
| BBR | -29.91 | -31.02 | **1.11** | **0.37** | -29.9 |
| 4 mm HR3-3 | -29.90 | -30.66 | **0.74** | | -29.9 |

| WHAT IF | The BBR m-value target slope is -0.310 rather than -0.300 | | | | |
|---|---|---|---|---|---|
| Test Device | S Critical Temp | m Critical temp | ΔTc | Δ(ΔTc) | LT PG Grade |
| BBR | -29.91 | -30.17 | **0.26** | **0.42** | -29.9 |
| 4 mm HR3-3 | -29.92 | -29.75 | **-0.16** | | -29.7 |

**These are good results, but they are not exceptional results especially for typical 20 hour PAV residues. However the sensitivity of the 4 mm to minor slope variations and the basic use of the BBR to determine whether or not a given binder meets a given low temperature grade on the basis of just one test temperature make it unlikely that the 4 mm test will replace the BBR for the standard grading of PG binders**



10
9
8
7
6
5
4
3
2
1
0
Log Relaxation Modulus, G(t)
-7
-6
-5
-4
-3
-2
-1
0
1
2
3
4
5
6
7
8
9
10
11
Log Reduced Time
G(t) plot at -18C ref temp PG 58-28 PAV
G(t) plot at -24C ref temp PG 58-28 PAV
slope at -18C = -.275
slope @ -18C = -0.265
Slope @ -24C = -0.275
Slope @ -24C = -0.265



Inaccurate test temperatures will result in incorrect relaxation moduli and as this plot shows very slight changes in the slope determination will impact the determination of the final binder grade
This issue is also true for the BBR, where temperature control and mechanical factors will affect the BBR slope value
Log Relaxation Modulus, Pa
Log Reduced Time
9
8.8
8.6
8.4
8.2
8
7.8
7.6
7.4
7.2
7
-1
0
1
2
3
-0.265
-0.275
-0.265
-0.275
7.58 sec
10.7 sec
98.8 sec
139.6 sec
G(t) plot at -18C ref temp PG 58-28 PAV
G(t) plot at -24C ref temp PG 58-28 PAV
slope at -18C = -.275
slope @ -18C = -0.265
Slope @ -24C = -0.275
Slope @ -24C = -0.265

# Summary Comments

- Compliance correction is needed for the lowest temperatures
  - At 0°C and warmer compliance correction appears negligible
- Validation of Peltier data against a chilled air or LN2 cooled system is recommended until such time as reliable Peltier temperatures can be obtained

# Summary Comments

- Important at low temperatures where displacements are low and torques can be high
- Becomes negligible at 0°C and above (at least for our machines)
- 4 mm is a valuable tool for forensic analysis, thin film emulsion residue testing, material screening
- May not be suitable for everyone
- I don’t see it as a replacement for BBR for routine PG QC testing

# Questions or Comments

| | | | | | | |
|---|---|---|---|---|---|---|
| **RHEA G(t) @-24°C 1649, 02-09-17-A, MIA PG 58-28 PAV, 02-08-17, pooled PAV residues, 4mm, HR3-3** | | | | | | |
| Critical temps based on 4 mm DSR test | | | | | | |
| TEMP | G)t) | m, creep | Log(G(t) | -24 | | |
| -24 | 233.70 | -0.2281 | 2.3686617 | -18 | | |
| -18 | 113.59 | -0.2944 | 2.055321 | -12 | | |
| -12 | | | | | | |
| -6 | | | | -6 | | |
| Critical values | 143 | -0.275 | 2.155336 | | | |
| slope | intercept | critical temps | | | | ΔTc |
| -0.052223451 | 1.115299 | -19.9151 | G(t) critical | **-29.915** | **-29.92** | **-0.16** |
| -0.011048225 | -0.49322 | -19.7518 | m critical | **-29.752** | **-29.75** | |
| | | | | Std Dev | | **ΔTc Std dev** |
| | | | | $T_{S\text{-Critical}}$ | 0.0163 | 0.2160 |
| | | | | $T_{m\text{-Critical}}$ | 0.2323 | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **RHEA G(t) @-24°C 1649, 02-09-17-A, MIA PG 58-28 PAV, 02-08-17, pooled PAV residues, 4mm, HR3-2** | | | | | | |
| Critical temps based on 4 mm DSR test | | | | | | |
| TEMP | G)t) | m, creep | Log(G(t) | -24 | 233.19 | -0.2260 |
| -24 | 233.19 | -0.2260 | 2.3677087 | -18 | 114.16 | -0.2902 |
| -18 | 114.16 | -0.2902 | 2.0575221 | -12 | | |
| -12 | | | | | | |
| -6 | | | | -6 | | |
| Critical values | 143 | -0.275 | 2.155336 | | | |
| slope | intercept | critical temps | | | | ΔTc |
| -0.051697765 | 1.1269623 | -19.8920341 | G(t) critical | **-29.892** | **-29.89** | **-0.47** |
| -0.010711017 | -0.4830434 | -19.4233103 | m critical | **-29.423** | **-29.42** | |
| COV | | **ΔTc COV** | | **Average** | | **ΔTc Avg** |
| $T_{S\text{-Critical}}$ | -0.1% | | | $T_{S\text{-Critical}}$ | **-29.90** | **-0.32** |
| $T_{m\text{-Critical}}$ | -0.8% | | | $T_{m\text{-Critical}}$ | **-29.59** | |