

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

**Meeting:** ROBO SP Meeting

**Date:** August 8, 2019

**Location:** Skype meeting

**Minutes by:** Justin Mills – SP Chair

### Actions:

1. Justin Mills to draft letter asking TMC 434-3 supplier for permission to conduct low temperature screening on it (MRV, Scanning Brookfield), so we can better understand why it is exhibiting yield stress after ROBO.
2. Justin Mills, Matt Schlaff, Tom Schofield to draft procedure for introducing dilute NO2. Additionally, SP members were encouraged to review the current D7528 method and provide feedback on best approach to drafting the alternative procedure.
3. Justin Mills to review request to change SCFM level from 0.6 to 1.0 in Section 10.3.2.1.
4. Justin Mills to review minutes from previous ROBO workshops for resolution on NO2 addition.
5. Justin Mills to schedule next SP meeting for Thursday, October 3<sup>rd</sup>.

### Membership and Attendance:

Ace Glass	Dave Lawrence, *Tom Petrocella
Afton	Shelia Thompson, Jeff Yang, Todd Dvorak
ASTM TMC	*Tom Schofield
BASF	<del>Mary Dery, Bridgett Rakestraw</del>
Chevron Oronite	Man Hon Tsang, Robert Stockwell
ExxonMobil	*Dennis Gaal
Infineum	Andy Richie, Sapna Eticala
Intertek	Joe Franklin, Matt Schlaff,
Lubrizol	*Mike Faile, Aimee Shinhearl, Rick Hartman
PetroChina	Li Shaohui , Sun Ruihua, Peng Wang, Xiaogang Li, Xu Li
Evonik Oil Additives	*Justin Mills, *Bruce Zweitzig, *Joan Souchik, *John Maxwell, *Justin Kontra
Vanderbilt Chemicals	Al Filho, Ron Hiza
SwRI	Becky Grinfield, Joe De La Cruz, *Mike Birke, *Young-Li McFarland
Valvoline	Amol Savant, Kevin Figgatt, *Steve Lazzara
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	*Alan Flamberg

\* Denotes attendance

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

### Summary:

- Meeting convened at 10:04EST on August 8, 2019
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
  - Tom Petrocella added for Ace Glass
  - Mary Dery and Bridgett Rakestraw removed for BASF (Mary requested removal and Bridgett's email no longer active)
- Meeting minutes from June 20<sup>th</sup> SP meeting were accepted
  - Motion made by Mike Faile and seconded by Alan Flamberg.
- Actions from the June 20<sup>th</sup> meeting were reviewed
  - Outstanding actions include:
    - Justin Mills and Tom Schofield to track the number of 438-2 runs. Once there are >20 runs, the limits will be recalculated and shared with the SP.
    - Tom Schofield to ask TMC 434-3 supplier if it is okay to conduct low temperature screening on it (MRV, Scanning Brookfield), so we can better understand why it is exhibiting yield stress.
    - Justin Mills and Matt Schlaff to draft procedure for introducing dilute NO<sub>2</sub>. Additionally, SP members were encouraged to review the current D7528 method and provide feedback on best approach to drafting the alternative method.
- ROBO industry statistics
  - The current 2019OCT semester (4/1/19 – 9/30/19) is running slightly severe with an average  $Y_i$  of -0.12. The precision has improved over last semester with a pooled  $s$  of 0.2326.
- Reference Oil 438-2
  - At the February 11<sup>th</sup> SP meeting we agreed to track # of 438-2 runs in ROBO LTMS. Once >20 runs are reached, new limits will be calculated and proposed to SP
  - As of 8/6/19, there were only 3 additional data points generated for 438-2 since February, bringing the total to 13.
  - Justin and Tom will continue to monitor # of runs. Once >20 runs on 438-2 are recorded, Justin will re-evaluate 438-2 limits.
- Reference Oil 434-3
  - To date, 10 ROBO runs with 434-3 have been completed.
  - 2 of the 10 runs exhibited yield stress. This requires further investigation because previous batches (434-1 and 434-2) typically did not exhibit any yield stress. Justin Mills to draft a letter requesting permission from supplier to run additional low temperature screening on fresh oil (SBT and MRV)
  - The MRV viscosity of 434-3 appears to be more comparable to 434-1 results.
- Dilute nitrogen dioxide
  - Editing the D7528 method has proven to be more challenging than originally expected. Nitrogen dioxide is referenced >40 times throughout the method, so a significant revision to the D7528 method may be required.
  - Justin Mills requested a facilitator from B.10 (Standards Acceleration) and Tom Schofield was assigned. Justin and Tom will continue to work on revision. A request to the SP was made for additional support.
  - SP members were encouraged to review the current D7528 method and provide feedback on best approach to drafting the alternative method.
- Method housekeeping
  - All SP members were encouraged to review the ASTM D7528-17a and recommend any additional changes that may be necessary.
  - Prior to meeting Mike Faile and Aimee Shinhearl sent the following: "For method housekeeping, we do have a proposed change regarding the acrylic block air flow meter readings. We would like to have the level in 10.3.2.1 changed to 1.0 SCFM. We realize that 0.6 is ideal, but this is difficult to always achieve. Even with the Edwards pump trials that we ran in the past and now are currently trialing again, we have not always achieved the 0.6 level but did achieve passing results on some of those runs."
    - Justin Mills took an action to further review.
- Additional topics

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

- The NO<sub>2</sub> vial – with the increments being 0.1 mL and the goal to dose at 0.167 per hour, it makes it difficult. We were wondering if anyone ever investigated a different vial that had smaller increments to better help track the flow with pure NO<sub>2</sub>.
  - Currently the method specs the following “*Graduated Tube* (Ace Glass, Inc., part number D120677), 12 mL capacity, with 0.1 mL graduations and having appropriate provisions for connection to the reaction vessel’s subsurface gas delivery system—see [Annex A10](#) for more details.”
  - Given the method specifies Ace Glass part # D120677 it would be difficult to allow use of a different graduated tube without revising the method.
- For historical understanding, why was it decided to start the NO<sub>2</sub> flow right away rather than waiting for the reactor to reach the 170 °C test temperature? Would results be more severe if it was introduced after test temperature was reached?
  - Unclear why this was decided. Justin agreed to review minutes from previous SP workshops to see if this was discussed.
- Next meeting is tentatively scheduled for October 3, 2019. The meeting may be postponed if there is not enough 434-3 data or enough 438-2 data to further discuss limits or if there is not significant progress on the revision of the method.
- Meeting adjourned

-End report-

# ROBO Surveillance Panel Meeting

August 8, 2019

Justin Mills

# Agenda

---

- Welcome, ASTM statement
- Review membership of SP
- Review and approve minutes from previous meetings (see attachment)
- Review and follow-up on actions from June 20<sup>th</sup> meeting
- TMC 438-2
- TMC 434-3
- Dilute nitrogen dioxide – Drafting alternative procedure
- Method housekeeping
- Additional topics, if any
- Set next meeting

# ASTM Antitrust and Recording Policy

---

ASTM International is a not-for-profit organization and developer of voluntary consensus standards. ASTM's leadership in international standards development is driven by the contributions of its members: more than 30,000 technical experts and business professionals representing 135 countries.

The purpose of antitrust laws is to preserve economic competition in the marketplace by prohibiting, among other things, unreasonable restraints of trade. In ASTM activities, it is important to recognize that participants often represent competitive interests. Antitrust laws require that all competition be open and unrestricted.

It is ASTM's policy, and the policy of each of its committees and subcommittees, to conduct all business and activity in full compliance with international, federal and state antitrust and competition laws. The ASTM Board of Directors has adopted an antitrust policy which is found in Section 19 of ASTM Regulations Governing Technical Committees. All members need to be aware of and compliant with this policy. The Regulations are accessible on the ASTM website (<http://www.astm.org/COMMIT/Regs.pdf>).

Electronic recording of ASTM meetings is prohibited.

# Membership

---

Ace Glass	Dave Lawrence
Afton	Shelia Thompson, Jeff Yang, Todd Dvorak
ASTM TMC	Tom Schofield
BASF	Mary Dery, Bridgett Rakestraw
Chevron Oronite	Man Hon Tsang, Robert Stockwell
ExxonMobil	Dennis Gaal
Infineum	Andy Richie, Sapna Eticala
Intertek	Joe Franklin, Matt Schlaff
Lubrizol	Mike Faile, Aimee Shinhearl, Rick Hartman
PetroChina	Li Shaohui , Sun Ruihua, Peng Wang, Xiaogang Li, Xu Li
Evonik Oil Additives	Justin Mills, Bruce Zweitzig, Joan Souchik, John Maxwell, Justin Kontra
Vanderbilt Chemicals	Al Filho, Ron Hiza
SwRI	Becky Grinfield, Joe De La Cruz, Mike Birke, Yong-Li McFarland
Valvoline	Amol Savant, Kevin Figgatt, Steve Lazzara
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	Alan Flamberg

Summary of changes:

# Motion to accept June 20, 2019 meeting minutes

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

**Meeting:** ROBO SP Meeting  
**Date:** June 20, 2019  
**Location:** Skype meeting  
**Minutes by:** Justin Mills – SP Chair

### Actions:

- Justin Mills and Tom Schofield to track the number of 438-2 runs. Once there are >20 runs, the limits will be recalculated and shared with the SP.
- Tom Schofield to ask TMC 434-3 supplier if it is okay to conduct low temperature screening on it (MRV, Scanning Brookfield), so we can better understand why it is exhibiting yield stress.
- Justin Mills and Matt Schlaff to draft procedure for introducing dilute NO<sub>2</sub>. Additionally, SP members were encouraged to review the current D7528 method and provide feedback on best approach to drafting the alternative method.
- Justin Mills to schedule next SP meeting for Thursday, August 6<sup>th</sup>.

### Membership and Attendance:

Ace Glass	Dave Lawrence
Afton	Shelia Thompson, Jeff Yang, *Todd Dvorak
ASTM TMC	*Tom Schofield
BASF	Mary Dery, Bridgett Rakestraw
Chevron Oronite	Man Hon Tsang, Robert Stockwell
ExxonMobil	Dennis Gaal
Infineum	Andy Richie, Sapna Eticala
Intertek	Joe Franklin, *Matt Schlaff,
Lubrizol	*Mike Faile, *Aimee Shinhearl, Rick Hartman
PetroChina	Li Shaohui, Sun Ruihua, Peng Wang, Xiaogang Li, Xu Li
Evonik Oil Additives	*Justin Mills, Bruce Zwertzig, Joan Souchik, *John Maxwell, Justin Kontra
Vanderbilt Chemicals	*Al Filho, Ron Hiza
SwRI	*Becky Grinfield, Joe De La Cruz, *Mike Birke, *Young-Li McFarland
Valvoline	Amol Savant, Kevin Figgatt, *Steve Lazzara
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	*Alan Flamberg

\* Denotes attendance

ASTM D7528 ROBO SP Meeting June 20, 2019

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

### Summary:

- Meeting convened at 10:03EST on June 20, 2019
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update – no changes to report.
- Meeting minutes from April 11<sup>th</sup> SP meeting were accepted
  - Motion made by John Maxwell and seconded by Mike Faile
- Actions from the April 11<sup>th</sup> meeting were reviewed
  - Outstanding actions include:
    - Generating >20 runs of 438-2 so permanent limits can be set.
    - Edit method to include dilute NO<sub>2</sub> alternative procedure.
    - SP to bring forth any changes that need to be made to method (mostly editorial)
- ROBO industry statistics
  - The 2019APR semester (10/1/18 – 3/31/19) ended without a severity bias (Average Yi=0.04); however the precision is slightly worse than target (Pooled s = 0.27). The current semester (4/1/19 – 9/30/19) has an average Yi=0.20 (slightly severe) and pooled s=0.25.
  - It is not entirely clear how or why the test is running more severe at the moment, as there was no concerted effort to make the test more severe.
    - Evonik commented that they have been trying to tune their units toward the middle/target.
    - Lubrizol commented that they had a unit shift severe, but is not back on target.
    - Intertek commented that they had previously tuned their units 435-1 and are now using 434-2.
  - CUSUM plots were generated for each reference fluid. Observed the same trend in all reference oils.
- Reference Oil 438-2
  - At the February 11<sup>th</sup> SP meeting we agreed to track # of 438-2 runs in ROBO LTMS. Once >20 runs are reached, new limits will be calculated and proposed to SP
  - As of 6/19/19, there were no additional data points for 438-2 generated since February. Tom Schofield commented that labs are still using existing inventory of 438, so it may take a while before >20 results with 438-2 are generated.
  - Justin and Tom will continue to monitor # of runs. Once >20 runs on 438-2 are recorded, Justin will re-evaluate 438-2 limits.
- Reference Oil 434-3
  - 3-4 samples of 434-3 were dispatched to SwRI, Intertek, Evonik, and Lubrizol for evaluation.
  - To date, 7 runs have been completed. MRV viscosities seem more comparable to 434-1 than 434-2, but two of the runs exhibited yield stress. More data is needed before we can make an assessment. Labs are encouraged to run their 434-3 samples if they have not already done so.
    - Joan Souchik commented that it may be beneficial to run some low temperature screening on the lubricant to better understand the yield stress.
    - Alan Flamberg commented that it may be borderline yield stress (~30Pa), so within the repeatability/reproducibility of the MRV test it is possible to see <35 and <70Pa values for yield stress.
- Dilute nitrogen dioxide
  - To implement dilute nitrogen dioxide as an approved alternative, the SP still needs to develop and approve an alternative procedure for dilute nitrogen dioxide.
  - Editing the D7528 method has proven to be more challenging than originally expected. Nitrogen dioxide is referenced >40 times throughout the method, so a significant revision to the D7528 method may be required. In other words, we cannot just update the procedure portion of the method.
  - SP members were encouraged to review the current D7528 method and provide feedback on best approach to drafting the alternative method.
- Method housekeeping
  - All SP members were encouraged to review the ASTM D7528-17a and recommend any additional changes that may be necessary.
- Next meeting is tentatively scheduled for August 8, 2019.
- Meeting adjourned

ASTM D7528 ROBO SP Meeting June 20, 2019



## Actions from June 20<sup>th</sup> meeting

---

- Justin Mills and Tom Schofield to track the number of 438-2 runs. Once there are >20 runs, the limits will be recalculated and shared with the SP.
- Tom Schofield to ask TMC 434-3 supplier if it is okay to conduct low temperature screening on it (MRV, Scanning Brookfield), so we can better understand why it is exhibiting yield stress.
- Justin Mills and Matt Schlaff to draft procedure for introducing dilute NO<sub>2</sub>. Additionally, SP members were encouraged to review the current D7528 method and provide feedback on best approach to drafting the alternative method.
- Justin Mills to schedule next SP meeting for Thursday, August 8th.

# ROBO Industry Statistics

Period	N-size	Degrees of Freedom	Pooled s	Mean $\Delta/s$	Comments
Current Targets	49	46	0.1945	-----	
10/1/15 through 3/31/16*	92	89	0.4115	-0.10	Period statistics with and without one extreme result included
10/1/15 through 3/31/16*	91	88	0.3661	-0.20	
4/1/16 through 9/30/16	74	71	0.3152	-0.53	
10/1/16 through 3/31/17	78	75	0.2771	-0.91	
4/1/17 through 9/30/17	99	95	0.2220	-0.76	
10/1/17 through 3/31/18**	90	86	0.2376	-0.91	Period statistics with and without seven suspect results from two rigs
10/1/17 through 3/31/18**	83	79	0.2076	-0.74	
4/1/18 through 9/30/18	126	122	0.2184	-0.49	Period statistics with and without one extreme result included
4/1/18 through 9/30/18	125	121	0.1958	-0.53	
10/1/18 through 3/31/19	100	96	0.2738	0.04	
4/1/19 through 9/30/19	58	54	0.2326	-0.12	

Updated 08/06/19

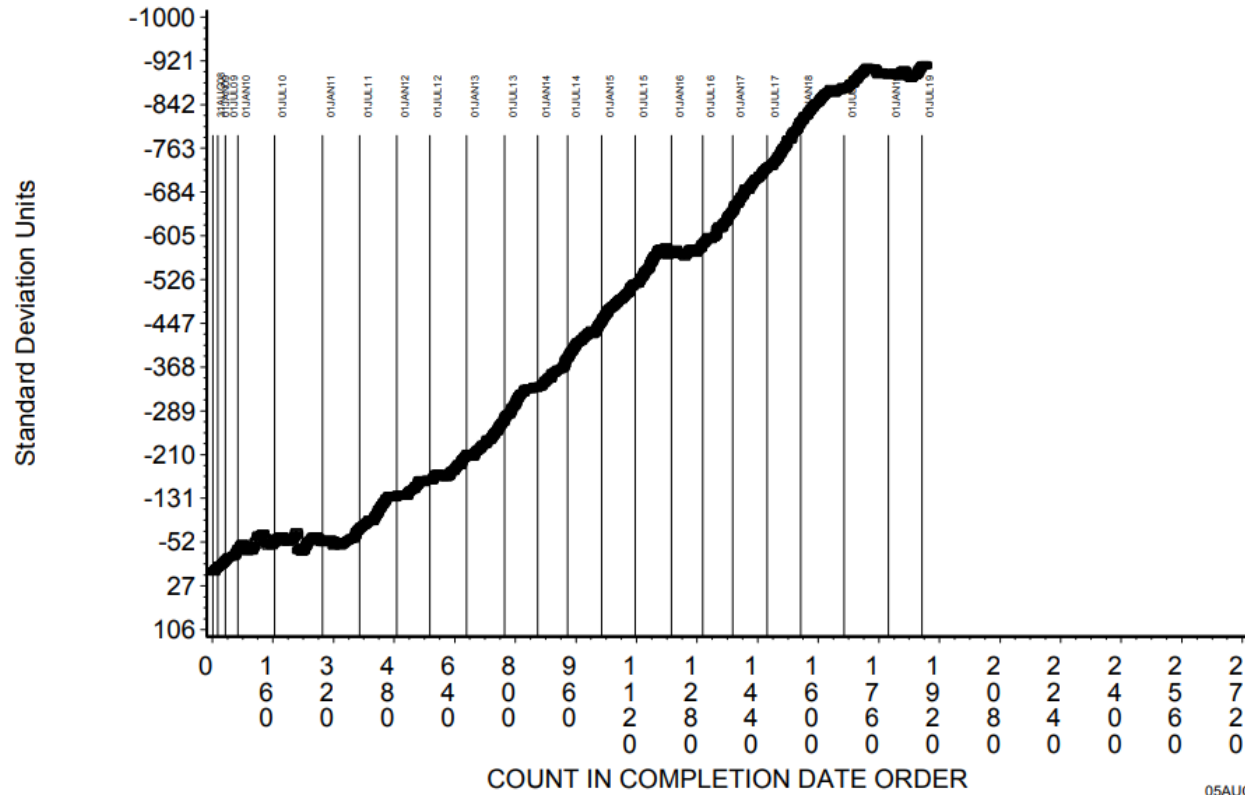
# CUSUM severity analysis

ROBO TEST INDUSTRY OPERATIONALLY VALID DATA

AGED OIL MRV APPARENT VISCOSITY



CUSUM Severity Analysis



05AUG19:09:57

# TMC 438-2

Oil	n	Natural Log Transformed Mean (ln)	Mean in Original Units	s.d. (ln)	95% band in mPa·s Min <sup>1</sup>	95% band in mPa·s Max <sup>1</sup>	95% Bands Min (ln)	95% Bands Max (ln)
434-1	13	10.6599	42,612	0.1672	30,706	59,136	10.3322	10.9876
434-2	36	<sup>2</sup> 10.9284	<sup>2</sup> 55,737	0.1551	<sup>2</sup> 41,126	<sup>2</sup> 76,008	<sup>2</sup> 10.6244	<sup>2</sup> 11.2386
435	15	11.4895	97,685	0.2932	<sup>3</sup> 60,000	173,546	<sup>3</sup> 11.0021	12.0642
435-1	22	11.0416	62,420	0.20295	<sup>4</sup> 44,570	92,910	<sup>4</sup> 10.7048	11.4394
438	14	10.2676	28,785	0.2037	19,308	42,912	9.8683	10.6669
438-2	10	10.4421	34,273	0.2322	<sup>2</sup> 21,742	54,025	9.9870	10.8972

<sup>1</sup> 95% bands in mPa·s are listed for information purposes only, the transformed values will be used to judge acceptance in all cases.

<sup>2</sup> A correction factor (severity adjustment) has been applied to the mean of reference oil 434-2 to account for the mild bias observed during the period this dataset was generated. The 95% confidence range reflects the inclusion of the correction factor (severity adjustment).

<sup>3</sup> The minimum value for Reference oil 435 is fixed at 60,000 (11.0021 in transformed units) and not a true 95% minimum as calculated from the statistics.

<sup>4</sup> The minimum value for reference oil 435-1 is based on -1.66 standard deviations from the target mean (to match the range previously approved for oil 435 min), so is not actually a 95% confidence range. A 95% confidence range would use 1.96 standard deviations from target mean.

- At the April SP meeting we agreed to track # of 438-2 runs in ROBO LTMS. Once >20 runs are reached, new limits will be calculated and proposed to SP
  - Only 3 new data points for 438-2 since February 1, 2019 → Total of 13 runs
  - Will continue to monitor # of runs.

# TMC 434-3 approval

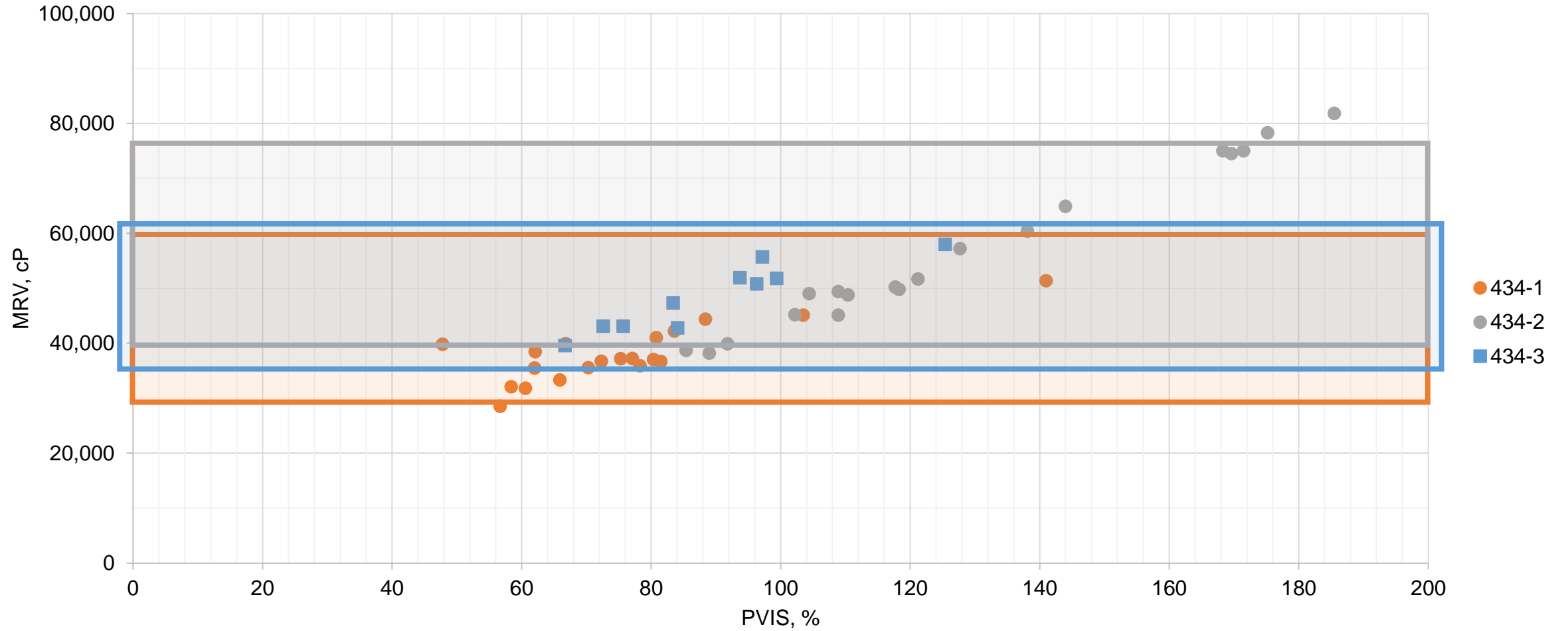
## TMC 434-2 levels are critically low

METHOD	IND	TESTKEY	DTCOMP	APPARATS	MRVTEMP	MRVYSEOT	MRV	MRVti	VAL	VOLEOT	PVIS
ROBO	434-3	145027-ROBO	20190502	A 11	-30	<70	47300	10.7643	RG	47	83.4
ROBO	434-3	145003-ROBO	20190503	B 2	-30	<35	51900	10.8571	AG	49	93.7
ROBO	434-3	145005-ROBO	20190505	B 2	-30	<35	50800	10.8357	AG	49	96.3
ROBO	434-3	145004-ROBO	20190508	B 4A	-30	<35	43100	10.6713	AG	46	75.7
ROBO	434-3	145006-ROBO	20190519	B 4A	-30	<35	39600	10.5866	AG	44	66.7
ROBO	434-3	145028-ROBO	20190531	A 5	-30	<70	43100	10.6713	AG	43	72.6
ROBO	434-3	145021-ROBO	20190607	AM 4	-30	<35	55700	10.9277	AG	48	97.2
ROBO	434-3	145029-ROBO	20190627	A 3	-30	<35	42800	10.6643	AG	47	84.1
ROBO	434-3	145051-ROBO	20190630	G 1	-30	<35	58000	10.9682	AG	49	125.4
ROBO	434-3	145053-ROBO	20190717	G 6	-30	<35	51800	10.8551	AG	44	99.4

- MRV viscosities seem more comparable to 434-1 than 434-2, but issue of yield stress needs to be resolved.
- More data is still required for analysis

# TMC 434-3 approval

## Alignment with previous versions



# Dilute nitrogen dioxide

## Next steps

---

Path forward to implement dilute NO<sub>2</sub> as an alternative to pure NO<sub>2</sub> is the following:

- 1) **Demonstrate equivalence to the SP** → Based on the available data, SP feels confident that dilute NO<sub>2</sub> and concentrated NO<sub>2</sub> yield comparable results?
- 2) Develop a procedure for dilute NO<sub>2</sub> → Due to difficulty drafting an alternative procedure, an ASTM facilitator has been requested to provide support.
- 3) Approve by SP → Seek approval after procedure is written.
- 4) Issue information letter allowing use of dilute NO<sub>2</sub> as an alternative
- 5) Ballot the recommended changes at ASTM

# Method housekeeping

---

- 9. New and Existing Test Stand Calibration needs to be updated.
  - ROBO no longer has stand-alone calibration requirements and is now included in LTMS. As such, the reference in the method is no longer valid
  - <sup>11</sup> The ROBO TMC Calibration Requirements document is available at: [http://www.astmtmc.cmu.edu/ftp/docs/bench/robo/procedure\\_and\\_ils/20170713\\_ROBO\\_TMC\\_Calibration\\_Requirements.pdf](http://www.astmtmc.cmu.edu/ftp/docs/bench/robo/procedure_and_ils/20170713_ROBO_TMC_Calibration_Requirements.pdf)
  - Suggest we also update Section 9 calibration requirements and reference the LTMS for calibration requirements.

**[www.astmtmc.cmu.edu](http://www.astmtmc.cmu.edu) - /ftp/docs/ltms/**

---

[\[To Parent Directory\]](#)

1/31/2019	4:07 PM	1436421	<a href="#">ltms.pdf</a>
1/31/2019	4:09 PM	143	<a href="#">readme.txt</a>
1/31/2019	4:14 PM	<dir>	<a href="#">ReplacementPages</a>
1/31/2019	4:23 PM	<dir>	<a href="#">RevisionLetters</a>

---

**Any other changes?**



## Method housekeeping

### Feedback from Mike Faile and Aimee Shinhearl

---

“For method housekeeping, we do have a proposed change regarding the acrylic block air flow meter readings. We would like to have the level in 10.3.2.1 changed to 1.0 SCFM. We realize that 0.6 is ideal, but this is difficult to always achieve. Even with the Edwards pump trials that we ran in the past and now are currently trialing again, we have not always achieved the 0.6 level but did achieve passing results on some of those runs.”

10.3.2 On an assembled vessel, install the flow meter between the top connection of the vacuum control valve and the vacuum source. Apply vacuum to the vessel and block the vacuum relief orifice long enough to assure the system will attain 85 kPa with a subsurface airflow of 185 mL/min. The air flow meter shall read less than 0.6 SCFM.

**Any Additional Topics?**

## Additional topics from Mike Faile and Aimee Shinhearl

---

- The NO<sub>2</sub> vial – with the increments being 0.1 mL and the goal to dose at 0.167 per hour, it makes it difficult. We were wondering if anyone ever investigated a different vial that had smaller increments to better help track the flow with pure NO<sub>2</sub>.
  
- For historical understanding, why was it decided to start the NO<sub>2</sub> flow right away rather than waiting for the reactor to reach the 170 °C test temperature? Would results be more severe if it was introduced after test temperature was reached?

## Next Meeting

---

- Suggestions for next SP meeting?
  - **Thursday, September 26<sup>th</sup>?**
  - **Thursday, October 3<sup>rd</sup>?**