MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting

Date: October 24, 2019

Location: Skype meeting

Minutes by: Justin Mills - SP Chair

Actions:

- 1. Tom Schofield to incorporate new limits for TMC 438-2 and 434-3 into LTMS.
- 2. Justin Mills to revisit TMC 436 as a potential reference oil for ROBO.
- 3. Justin Mills, Matt Schlaff, Tom Schofield to update ASTM D7528 to include dilute NO2 (likely in early-2020).
- 4. Justin Mills to schedule the next ROBO SP meeting in Q1 2020.

Membership and Attendance:

Ace Glass	Dave Lawrence, *Tom Petrocella
Afton	*Shelia Thompson, Jeff Yang, Todd Dvorak
ASTM TMC	*Tom Schofield
BASF	
Chevron Oronite	Man Hon Tsang, Robert Stockwell
ExxonMobil	Dennis Gaal
Infineum	Andy Richie, Sapna Eticala
Intertek	Joe Franklin, *Matt Schlaff, *Rachel Stone (guest)
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, *Gabriel Walkup
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

Summary:

- Meeting convened at 10:04EST on October 24, 2019
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
 - o Gabriel Walkup to replace John Maxwell at Evonik
- Meeting minutes from August 8, 2019 SP meeting were accepted
 - Motion made by Bruce Zweitzig and seconded by Mike Faile.
- Actions from the August 8th meeting were reviewed
 - o Outstanding actions include:
 - Justin Mills and Matt Schlaff 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 method. (addressed in later notes)
 - Justin Mills to review request to change SCFM level from 0.6 to 1.0 in Section 10.3.2.1 (addressed in later notes)
- ROBO industry statistics
 - The 2019OCT (4/1/19 9/30/19) semester recently ended with a slight mild trend; Yi=-0.31. Precision was worse than target but better than previous semester; Pooled s = 0.2491.
 - There is not enough data to make an assessment on current semester (n=3).
- Reference Oil 438-2
 - At the February 11th 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. To date, there are 19 valid runs available - the SP agreed this was enough.
 - Upon review of the limits, there was concern voiced over the overlap of MRV ranges on the current reference oils. After some discussion the SP agreed to revisit this topic at a future SP meeting. In addition, Justin Mills took an action to revisit TMC 436 data to see if that could be a suitable alternative one day.
 - Proposals for new limits were discussed. The SP agreed that it would be appropriate to apply a bias correction factor to the new limits – just as we did previously for 434-2 and 438-2 (interim limits) limit setting.
 - Matt Schlaff made a motion to approve the limits as defined by Option #2 in Slide #11 with an effective date of November 1, 2019. The motion was seconded by Tom Schofield. No one opposed and the motion carried.

TMC 438-2			Natural Log Transformed Mean (In)	Mean in Original Units	<u>s.d.</u> (ln)	95% band in mPa*s, min	95% band in mPa*s, max	95% band (In), min	95% band (ln), max
Option #1	No bias correction	19	10.5158	36,894	0.0506	22,181	61,365	10.007	11.0246
Option #2	Average Yi from TMC statistics (Yi = -0.1316)		10.5404	37,813	0.2596	22,734	62,894	10.0316	11.0492

• Tom Schofield will update the LTMS accordingly and issue a technical memo next week.

- Reference Oil 434-3
 - To date, 13 ROBO runs with 434-3 have been completed. The SP agreed this was enough data to set interim limits.
 - 2 of the 13 runs exhibited yield stress, but the SP agreed that the yield stress was not a critical parameter for this reference oil.
 - Proposals for new limits were discussed. The SP agreed that it would be appropriate to apply a bias correction factor to the new limits – just as we did previously for 434-2 and 438-2 (interim limits) limit setting.
 - Justin Mills made a motion to approve the limits as defined by Option #2 in Slide #13 with an effective date of November 1, 2019; as well as to remove the EOT yield stress requirement for 434-3 in the LTMS. The motion was seconded by Mike Faile. No one opposed and the motion carried.

TMC 434-3		n	Natural Log Transformed Mean (In)	Mean in Original Units	<u>s.d.</u> (ln)	95% band in mPa*s, min	95% band in mPa*s, max	95% band (In), min	95% band (ln), max
Option #1	No bias correction	13	10.7833	48,209	0.4040	37,060	62,711	10.5203	11.0463
Option #2	Average Yi from TMC statistics (Yi = -0.3091)		10.8411	51,078	0.1342	39,265	66,443	10.5781	11.1041

- Tom Schofield will update the LTMS accordingly and issue a technical memo next week.
- Transition from 434-2 to 434-3 in TMC's reference oil assignments will occur when inventory of 434-2 is depleted.

MEETING MINUTES: ROBO SURVEILLANCE PANEL

- Permanent limits will be reviewed once >20 runs with 434-3 are complete.
- Dilute nitrogen dioxide
 - Justin Mills, Matt Schlaff, and Tom Schofield will revise the method in early-2020. Revision will likely need to be face-to-face.
- Method housekeeping.
 - In the next revision of the method the following changes are recommended:
 - Update Footnote 8 in Section 9.1.6 to reference new LTMS document: <u>http://www.astmtmc.cmu.edu/ftp/docs/bench/robo/procedure_and_ils/20170713_ROBO_TMC_Calibr_ation_Requirements.pdf</u>"
 - Remove Note 8 in Section 10.8.2
 - A request to change flow rate limit from 0.6 SCFM to 1.0 SCFM in 10.3.2.1 was reviewed. After some discussion, it was suggested that labs check their system for leaks because <0.6 SCFM should be achievable. If <0.6 SCFM is still not achievable, we can revisit at a later SP meeting.
- Additional topics
 - Intertek is evaluating alternative flow meters to King Instrument Co.,7520 Series, Order number 2C-17. The method specifies an air flow of 2.0 SCFM +/- 0.1 SCFM in Section 6.11, but this meter only has increments of 0.2 SCFM. Matt Schlaff will share his findings at the next SP meeting.
- Next meeting will be scheduled in Q1 2020. Actual date will be determined when method revision is complete or enough data is available to set final limits for 434-3.
- Meeting adjourned

-End report-

ROBO Surveillance Panel Meeting

October 24, 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 August 8th meeting
- TMC 438-2 If enough data is available (>20 runs) we will vote on permanent limits
- TMC 434-3 If enough data is available we will review data and compare to 434-2 (current reference oil)
- Dilute nitrogen dioxide update
- Method housekeeping
- Additional topics, if any
- Set next meeting

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Membership

Ace Glass	Dave Lawrence, Tom Petrocella
Afton	Shelia Thompson, Jeff Yang, Todd Dvorak
ASTM TMC	Tom Schofield
BASF	
Chevron Oronite	Man Hon Tsang, Robert Stockwell
ExxonMobil	Dennis Gaal
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Intertek	Joe Franklin, Matt Schlaff
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Evonik Oil Additives	Justin Mills, Bruce Zweitzig, Joan Souchik, Gabe Walkup, 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:

- 1. Tom Petrocella added for Ace Glass
- 2. Mary Dery and Bridgett Rakestraw removed for BASF (Mary requested removal and Bridgett's email no longer active)
- 3. Gabe Walkup to replace John Maxwell at Evonik.

Motion to accept August 8, 2019 meeting minutes

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting

Date: August 8, 2019

Location: Skype meeting

Minutes by: Justin Mills - SP Chair

Actions:

- 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.
- 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 3rd.

Membership and Attendance:

Ace Glass Afton ASTM TMC BASF Chevron Oronite ExxonMobil Infineum Intertek Lubrizol	Dave Lawrence, *Tom Petrocella Shelia Thompson, Jeff Yang, Todd Dvorak *Tom Schofield Mary Dery, Bridgett Raksetraw Man Hon Tsang, Robert Stockwell *Dennis Gaal Andy Richie, Sapna Eticala Joe Franklin, Matt Schlaff,
Afton ASTM TMC BASF Chevron Oronite ExxonMobil Infineum Intertek Lubrizol	Shelia Thompson, Jeff Yang, Todd Dvorak "Tom Schofield Mary Dery, Bridgett Raksetraw Man Hon Tsang, Robert Stockwell "Dennis Gaal Andy Richie, Sapna Eticala Joe Franklin, Matt Schlaff,
ASTM TMC BASE Chevron Oronite ExxonMobil Infineum Intertek Lubrizol	*Tom Schofield Mary Dery, Bridgett Rakestraw: Man Hon Tsang, Robert Stockwell *Dennis Gaal Andy Richie, Sapna Eticala Joe Franklin, Matt Schlaff,
BASE Chevron Oronite ExxonMobil Infineum Intertek Lubrizol	Mary Dery, Bridgett Rakestraw Man Hon Tsang, Robert Stockwell *Dennis Gaal Andy Richie, Sapna Eticala Jee Franklin, Matt Schlaff,
Chevron Oronite ExxonMobil Infineum Intertek Lubrizol	Man Hon Tsang, Robert Stockwell *Dennis Gaal Andy Richie, Sapna Eticala Joe Franklin, Matt Schlaff,
ExxonMobil Infineum Intertek Lubrizol	*Dennis Gaal Andy Richie, Sapna Eticala Joe Franklin, Matt Schlaff,
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Lubrizol	
	*Mike Faile, Aimee Shinhearl, Rick Hartman
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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 20th SP meeting were accepted
 - Motion made by Mike Faile and seconded by Alan Flamberg.
 - Actions from the June 20th 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 are 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.
 - (mrt), Seaming brokinstal), as we can proceed indication in the advantage for a decise Justin Mills and Matt Schaft 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 method.
 - ROBO industry statistics
 - The current 2019OCT semester (4/1/19 9/30/19) is running slightly severe with an average Yi of -0.12. The
 precision has improved over last semester with a pooled s of 0.2326.
 - Reference Oil 438-2
 - At the February 11th 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 reevaluate 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
 - ASTM D7528 ROBO SP Meeting August 8, 2019

MEETING MINUTES: ROBO SURVEILLANCE PANEL

- The NO2 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 NO2.
 - 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 NO2 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

ASTM D7528

-End report-

ROBO SP Meeting

August 8, 2019

Actions from August 8th meeting

- 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.
- 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.

- Addressed in later slides

- Justin Mills to review request to change SCFM level from 0.6 to 1.0 in Section 10.3.2.1.
 - Addressed in later slides
- Justin Mills to review minutes from previous ROBO workshops for resolution on NO2 addition.
 - Addressed in later slides
- Justin Mills to schedule next SP meeting for Thursday, October 3rd.

ROBO Industry Statistics

Period	N-size	Degrees of Freedom	Pooled s	Mean ∆/s	Comments
Current Targets	49	46	0.1945		
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
10/1/17 through 3/31/18**	83	79	0.2076	-0.74	results from two rigs
4/1/18 through 9/30/18	126	122	0.2184	-0.49	Period statistics with and without one extreme
4/1/18 through 9/30/18	125	121	0.1958	-0.53	result included
10/1/18 through 3/31/19	100	96	0.2738	0.04	
4/1/19 through 9/30/19	94	90	0.2491	-0.31	
10/1/19 through 3/31/20	3	2	0.1430	-1.80	

Source: http://www.astmtmc.cmu.edu/ftp/refdata/bench/robo/data/statistics.txt (10/17/19)

CUSUM severity analysis





ROBO SP Meeting - October 2019 8

TMC 438-2 Setting final limits

	D7528 (ROBO) Aged Oil MRV Acceptance Bands, mPas and ln(mPas)												
		Natural Log	Mean in Original		95% band in	95% band in	95% Bands	95% Bands					
Oil	n	Mean (ln)	Units	s.d. (ln)	Min ¹	Max ¹	Min (ln)	Max (ln)					
434-1	13	10.6599	42,612	0.1672	30,706	59,136	10.3322	10.9876					
434-2	36	² 10.9284	² 55,737	0.1551	² 41,126	² 76,008	² 10.6244	² 11.2386					
435	15	11.4895	97,685	0.2932	³ 60,000	173,546	³ 11.0021	12.0642					
435-1	22	11.0416	62,420	0.20295	444570	92910	410.7048	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	² 21,742	54,025	9.9870	10.8972					

¹ 95% bands in mPas are listed for information purposes only, the transformed values will be used to judge acceptance in all cases.

² 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).

³ 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.

⁴The 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

- 20 runs available (19 valid)

TMC 438-2 Setting final limits

METHOD	IND	TESTKEY	DTCOMP	APPARATS	MRVTEMP	MRVYSEOT	MRV	MRVti	VAL	VOLEOT	PVIS
ROBO	438-2	-142342-ROBO	20190104	-AM-5	-30	-<35	20900	9.9475	LG	38	88.7
ROBO	438-2	142341-ROBO	20190104	AM 4	-30	<35	41100	10.6238	AG	52	147.6
ROBO	438-2	142343-ROBO	20190111	AM 3	-30	<35	52900	10.8762	AG	47	173.1
ROBO	438-2	142127-ROBO	20190111	В З	-30	<35	35100	10.466	AG	40	145.6
ROBO	438-2	142128-ROBO	20190111	B 4A	-30	<35	31800	10.3672	AG	42	133.8
ROBO	438-2	142005-ROBO	20190112	A 11	-30	<35	39700	10.5891	AG	46	156.8
ROBO	438-2	142129-ROBO	20190113	B 2	-30	<35	44600	10.7055	AG	34	177.7
ROBO	438-2	142004-ROBO	20190113	A 1	-30	<35	26900	10.1999	AG	44	105
ROBO	438-2	142082-ROBO	20190116	G 10	-30	<35	28000	10.24	AG	42	124
ROBO	438-2	142083-ROBO	20190116	G 3	-30	<35	31600	10.3609	AG	39	141
ROBO	438-2	142344-ROBO	20190201	AM 5	-30	<35	26600	10.1887	AG	42	101.7
ROBO	438-2	142006-ROBO	20190716	A 6	-30	<35	48300	10.7852	AC	48	193.7
ROBO	438-2	142130-ROBO	20190719	B 2	-30	<35	43500	10.6805	AC	46	170.5
ROBO	438-2	145016-ROBO	20190728	AM 5	-30	<35	66400	11.1035	OC	54	247.7
ROBO	438-2	145017-ROBO	20190804	AM 4	-30	<35	46600	10.7494	AC	52	190.3
ROBO	438-2	142084-ROBO	20190807	G 3	-30	<35	37900	10.5427	AC	45	159.4
ROBO	438-2	142007-ROBO	20190808	A 3	-30	<35	37900	10.5427	AC	45	160.8
ROBO	438-2	142085-ROBO	20190906	G 7	-30	<35	28100	10.2435	AC	40	118.8
ROBO	438-2	146110-ROBO	20190910	A 9	-30	<35	29300	10.2853	AC	43	126.7
ROBO	438-2	143856-ROBO	20190913	В З	-30	<35	28300	10.2506	AC	39	109.8

TMC 438-2 Statistics with and without correction factor applied to account for bias

TMC 438-2		n	Natural Log Transformed Mean (In)	Mean in Original Units	s.d. (ln)	95% band in mPa*s, min	95% band in mPa*s, max	95% band (ln), min	95% band (ln), max
Option #1	No bias correction	19	10.5158	36,894	0.2596	22,181	61,365	10.007	11.0246
Option #2	Average Yi from TMC statistics (Yi = -0.1316)		10.5404	37,813		22,734	62,894	10.0316	11.0492
	Limits for TMC 438	14	10.2676	28,785	0.2037	19,308	42,912	9.8683	10.6669
Current limits for TMC 438-2			10.4421	34,273	0.2322	21,742	54,025	9.9870	10.8972

TMC 438-2 runs were conducted over last two semesters. On average the ROBO test was running slightly mild (Pooled Yi = -0.1316)

- Compared to current limits for TMC 438-2 (n=10), the reference oil is running more severe and less precise.
- Applying correction factor has minor impact to overall range; nevertheless we should apply it if we wish to remain consistent with previous reference oil limit setting.
- Should we set limits today or wait for more data?

TMC 434-3 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
ROBO	434-3	145052-ROBO	20190816	G 3	-30	<35	45900	10.7342	AG	41	124
ROBO	434-3	145030-ROBO	20190822	A 5	-30	<35	42000	10.6454	AG	44	72.4
ROBO	434-3	145022-ROBO	20190925	AM 2	-30	<35	60000	11.0021	AG	51	123.4

- MRV viscosities seem more comparable to 434-1 than 434-2, but issue of yield stress needs to be resolved.
- No statistical outliers identified
- See next slide for statistics and bias correction

TMC 434-3 Statistics with and without correction factor applied to account for bias

TMC 434-3		n	Natural Log Transformed Mean (In)	Mean in Original Units	s.d. (ln)	95% band in mPa*s, min	95% band in mPa*s, max	95% band (In), min	95% band (ln), max
Option #1	No bias correction	13	10.7833	48,209	0.1342	37,060	62,711	10.5203	11.0463
Option #2	Average Yi from TMC statistics (Yi = -0.3091)		10.8411	51,078		39,265	66,443	10.5781	11.1041
	Limits for TMC 434-1	13	10.6599	42,612	0.1672	30,706	59,136	10.3322	10.9876
Limits for TMC 434-2			10.9284	55,737	0.1551	41,126	76,008	10.6244	11.2386

- The ROBO test is running slightly mild this test period (2019OCT) Yi= -0.3091
- Applying correction factor has minor impact to overall range; nevertheless we should apply it if we wish to remain consistent with previous reference oil limit setting.
- How should we address yield stress observed at Lab A: Investigate further or just consider viscosity?
- Should we set limits today or wait for more data?

Dilute nitrogen dioxide Next steps

Path forward to implement dilute NO2 as an alternative to pure NO2 is the following:

- I) Demonstrate equivalence to the SP → Based on the available data, SP feels confident that dilute NO2 and concentrated NO2 yield comparable results?
 - 2) Develop a procedure for dilute NO2 → Justin Mills and Tom Schofield working on it, but likely won't have time to complete until early 2020.
 - 3) Approve by SP \rightarrow Seek approval after procedure is written.
 - 4) Issue information letter allowing use of dilute NO2 as an alternative
 - ☐ 5) Ballot the recommended changes at ASTM

Method housekeeping

- Section 9.1.6: If all the required test stand set-up runs meet the current, approved ROBO TMC calibration requirements¹¹ (both operationally and statistically), the TMC will notify the laboratory that it can proceed with calibrating the test stand per 9.2.
 - Footnote 11 needs to be updated: "11 The ROBO TMC Calibration Requirements document is available at: <u>http://www.astmtmc.cmu.edu/ftp/docs/bench/robo/procedure_and_ils/20170713_ROBO_TMC_Calibration_Requirements.pdf</u>"
 - Recommendation: Should reference TMC's LTMS instead.
- Section 10.8.2: Volatility calculation makes reference to Note 8.
 - NOTE 8—The significance of the % volatiles parameter is under investigation.
 - Recommendation: Remove note. No longer under investigation.
- Annex A.2.2.1: ...It is a laboratory's responsibility to keep the on-site reference oil inventory at or above the minimum level specified by the TMC test engineers.
 - Recommendation: Update or remove statement to reflect that TMC manages the inventory.

Method housekeeping Other

- "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."
 - Are any other labs experiencing this problem? My guess would be that this is an indication of "leaks" in shaft seal (packing) or vessel gasket.
- Should the procedure indicate when to turn on the water flow to the condenser?
- For historical understanding, why was it decided to start the NO2 flow right away rather than waiting for the reactor to reach the 170 °C test temperature?
 - Outcome of 2015 ROBO workshop intent was just to align all operators.
 - Would results be more severe if it was introduced after test temperature was reached? Possible, but differences would be negligible

Any Additional Topics?

Next Meeting

- Suggestions for next SP meeting?
 - November 21, 2019?
 - Or early 2020?