MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting

Date: June 24, 2021

Location: MS Teams (virtual)

Minutes by: Justin Mills – SP Chair

Actions:

1. Justin/Alan to incorporate feedback received on draft revision to ASTM D7528 recirculated among the Surveillance Panel.
2. Tom Schofield to generate a mock-up and proposal “clean-up” the reference oil table in the LTMS. Only “active” reference oils will remain ROBO section and “obsolete” oils will be moved to Appendix A of the document.
3. Justin Mills to tentatively schedule the next ROBO SP meeting for July 22, 2021.

Membership and Attendance:

Ace Glass	Dave Lawrence, *Tom Petrocella
Afton	Shelia Thompson, Jeff Yang, *Todd Dvorak
ASTM TMC	*Tom Schofield
Chevron Oronite	Robert Stockwell
ExxonMobil	Dennis Gaal
Infineum	Andy Richie, Sapna Eticala
Intertek	*Joe Franklin, *Matt Schlaff, *Rachel Stone
Lubrizol	*Aimee Shinhearl, Jerimiah Westbrook
PetroChina	Li Shaohui , Sun Ruihua, Peng Wang, Xiaogang Li, Xu Li
Evonik Oil Additives	*Justin Mills, *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
Guests	

* Denotes attendance

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Summary:

- Meeting convened at 10:03EDT on June 24, 2021
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
    - Amy Ross (Valvoline) added to SP list.
    - Jerimiah Westbrook (Lubrizol) added to SP list
- Meeting minutes from April 15, 2021 SP meeting were accepted (motion made by Joe Franklin and seconded by Tom Petrocella)
- Actions from the April 15th meeting were reviewed.
    - Reference oil 436 (and corresponding limits) added into LTMS. Use of 438-2 has been suspended.
    - Dilute NO2 workgroup (Alan Flamberg, Matt Schlaff, Justin Mills, Tom Schofield) collected available feedback from SP and incorporated it into latest draft of ROBO method.
- Current status of ROBO
    - ROBO report from June D02.B0.07 meeting was shared. Report indicated that ROBO test was in overall good health with not immediate concerns for the method, parts availability, reference oils, test availability, or severity and precision.
    - Statistics for current semester not available on TMC website. Tom Schofield to investigate (may just require the system to refresh).
        - ***Update*** Issue resolved after our SP meeting. Precision is improving at expense of bias. Stats as posted are:

Period	N-size	Degrees of Freedom	Pooled s	Mean Δ/s
4/1/21 through 9/30/21	55	49	0.1912	-0.41

- TMC reference oils
    - At the April 15th meeting, SP approved limits for 436 as a replacement for 438-2. Since this time, acceptance bands for 436 have been entered into the LTMS document, and usage of 438-2 has been suspended.
        - Final status of 438-2 to be clarified at our next meeting.
- Dilute Nitrogen Dioxide
    - A draft revision of ASTM D7528 (version June 2021) was reviewed with the Surveillance Panel. Feedback on the revisions was collected during the meeting. Many were editorial in nature.
        - Update Footnote #2 to include latest information letter (IL20-1).
        - 6.7.2 "…valve to switch between the two ~~air~~ gas sources"
        - 6.8.2 "Second ~~air~~ gas supply…"
        - 7.1 should be updated to reflect both concentrated and dilute NO2 as reagents.
            - 7.1 Nitrogen Dioxide…7.1.1 Liquid Nitrogen Dioxide…7.1.2 Dilute Nitrogen Dioxide in Air
        - 10.3.1 "Start subsurface ~~pure-~~dry-air flow…"
        - 10.5.3.2 Should include a statement about dilute NO2 equivalent to 2.0 mL +/- 0.1 mL
        - 13.3.6.1.1 Should include a reference to X7
        - X7 Numbering needs to be updated
        - X7 Should also add statement or footnote to that indicates this calculation is not necessary when using a mass flow controller.
    - Once the draft method is accepted by the SP, a TMC information letter along with memo detailing equivalence of dilute NO2 to liquid NO2 will be issued.
        - Drafting this information letter may take longer than usual due to the large amount of changes to include.
    - The data dictionary will also require updating to introduce new items associated with dilute NO2. Updates to the data dictionary may take ~2 months due to need to 30-day beta.
- Updating reference oil table in LTMS
    - It was proposed to update the reference oil table in the LTMS to reflect the status of the reference oil – e.g. active or obsolete. This effort was supported by the SP. Based on current practice in the LTMS, it is likely that "active" oils would remain in ROBO section while "obsolete" oils would be moved to the Appendix A:

"HISTORY OF LTMS REFERENCE OIL MEANS AND STANDARD DEVIATIONS". One potential issue is that Appendix A does not include acceptance bands and instead just includes the average and standard deviation. SP would prefer to preserve the acceptance bands.
    - Tom Schofield agreed to generate a mock-up and proposal for our next meeting.
- Additional topics
    - Intertek has been experiencing issues with some of the newer Instatherm flasks. Flasks have been burning out after 1-5 runs. No other labs have reported this issue. Ace Glass is investigating.
        - Unfortunately, the SP was not able to discuss this topic any further due to time. This topic will be addressed at the next SP meeting.
- Next meeting tentatively scheduled on July 22, 2021. Date may be postponed if necessary.
- Meeting adjourned 11:39EDT

Meeting Outcome:

1. Reference oil 436 added to LTMS and is actively being assigned. Use of reference oil 438-2 has been suspended.
2. The draft revision to ASTM D7528 was reviewed. Feedback was gathered during the meeting and will be implemented into next draft and recirculated among the SP. If all goes according to plan, we will seek a SP vote at the next meeting.
3. SP agreed that it would be beneficial to "clean-up" the reference oil list for ROBO in the LTMS document. Proposal is to only include "active" reference oils in ROBO section and move "obsolete" oils to Appendix A of the document. Tom Schofield agreed to generate a mock-up and proposal for our next meeting.
4. Next meeting scheduled for July 22, 2021.

-End report-

ASTM D7528: Bench Oxidation of Engine Oils by ROBO Apparatus

ROBO Surveillance Panel Meeting

June 24, 2021

Justin Mills

Agenda

- Welcome, ASTM statement
- Review membership of surveillance panel
- Review and approve minutes from previous meetings (see attachment)
- Review and follow-up on actions from April 15th meeting
- Current status of ROBO including statistics
  - Report shared at D02.B0.07
- Reference oil update including
  - TMC 436 status
- Dilute nitrogen dioxide update
- Additional topics
  - Proposal to talk about adding additional field to LTMS to reflect test oil status e.g. “obsolete”, “active”
  - Data dictionary
- Set next meeting

ASTM Antitrust and Recording Policy

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Electronic recording of ASTM meetings is prohibited.

Membership

Ace Glass	Dave Lawrence, **Tom Petrocella,**
Afton	Shelia Thompson, Jeff Yang, **Todd Dvorak**
ASTM TMC	**Tom Schofield**
Chevron Oronite	Robert Stockwell
ExxonMobil	Dennis Gaal
Infineum	Andy Richie, Sapna Eticala
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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, **Amy Ross**
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	**Alan Flamberg**
Guests	

Summary of changes:

1. Amy Ross added to Valvoline.

2. Jerimiah Westbrook added Lubrizol.

Motion to accept April 15, 2021 meeting minutes

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting

Date: April 15, 2021

Location: MS Teams (virtual)

Minutes by: Justin Mills – SP Chair

Actions:

1. Tom Schofield to add reference oil 436 (and corresponding limits) in LTMS. Upon its adoption, use of 438-2 will be suspended.
1. ROBO SP members to review draft ASTM D7528 with dilute NO2 procedure. Any comments or recommendations should be provided to the dilute NO2 workgroup (Alan Flamberg, Matt Schlaff, Justin Mills, Tom Schofield) prior to the next SP meeting.
2. Justin Mills to tentatively schedule the next ROBO SP meeting for May 13, 2021.

Membership and Attendance:

Ace Glass	Dave Lawrence, *Tom Petrocella
Afton	Shelia Thompson, Jeff Yang, Todd Dvorak
ASTM TMC	*Tom Schofield
Chevron Oronite	Robert Stockwell
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General Interest	*Alan Flamberg
Guests	

* Denotes attendance

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Summary:

- Meeting convened at 10:03EDT on April 15, 2021
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
  - None were provided by during meeting, but afterward it was requested to add Amy Ross (Valvoline) to SP list.
- Meeting minutes from January 7, 2021 SP meeting were accepted (motion made by Tom Schofield and seconded by Alan Flamberg)
- Actions from the August 13th meeting were reviewed. Outstanding actions include:
  - Justin Mills, Matt Schlaff, Tom Schofield to update ASTM D7528 to include dilute NO2.
- ROBO industry statistics
  - The last semester, 2021APR (10/01/20 – 3/31/21) concluded with 112 runs reported. Yi= -0.10, indicating a slight mild bias. Pooled s = 0.3190 indicating the test is running less precise than target. Neither the severity or precision are cause for alarm.
- TMC reference oils
  - At the January 7th meeting, SP voted to finalize limits for 434-3 which became effective in February:

TMC 434-3	n	Natural Log Transformed Mean (ln)	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 #2	**22**	**10.8172**	**49,871**	**0.1389**	**37,987**	**65,473**	**10.5450**	**11.0894**

  - At the January 7th meeting, SP agreed 436 could replace 438-2 assuming more data was generated to set limits. Consensus was that limits for 438-2 are overly broad and the test would benefit from having a clean, passing reference oil like 436.
    - Limit setting for 436 was discussed in detail. Of the 17 donated runs, 2 were identified as outliers through Tukey Box methodology (any data points <Q1 - 1.5*IQR or >Q3 + 1.5*IQR are considered outliers). Both outliers fell just outside the acceptance criteria, so there some discussion on whether they should be removed or included for interim limit setting. The SP reviewed the following three options:

TMC 436		n	Natural Log Transformed Mean (ln)	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. Outliers removed.	15	10.3297	30,629	0.1087	24,750	37,904	10.1166	10.5428
Option #2	Average Yi from TMC statistics (Yi = -0.1000). Outliers removed.		10.3475	31,179		25,195	38,584	10.1344	10.5606
Option #3	Average Yi from TMC statistics (Yi = -0.1000). Outliers included	17	10.3437	31,061	0.1605	22,677	42,544	10.0291	10.6583

      - After some discussion it seemed Option #3 was most favorable, so a motion was made to Matt Schlaff to use Option #3 (as shown above) as the interim limits for 436 with the understanding that final limits would be set when a sufficient amount of data is available. The motion was seconded by Alan Flamberg.
        - A hand vote was taken and there was one negative – Gabriel Walkup (Evonik) preferred Option #2 because it was more consistent with our limit setting practices in the past.
        - Despite the negative, the majority ruled in favor, so the motion passed. Interim limits for 436 are as follows:
        -

TMC 436		n	Natural Log Transformed Mean (ln)	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 #3	Average Yi from TMC statistics (Yi = -0.1000). Outliers included	17	10.3437	31,061	0.1605	22,677	42,544	10.0291	10.6583

      - Following the acceptance of the 436 interim limits, a motion was made by Alan Flamberg to add 436 to the LTMS and include it as approved reference oil assignment. Upon its adoption, suspend the use of 438-2 as a reference oil. The motion was seconded by Gabriel Walkup.
        - A vote was taken, and all were in favor. The motion passed.
- Dilute Nitrogen Dioxide

ASTM D7528 ROBO SP Meeting April 15, 2021

MEETING MINUTES: ROBO SURVEILLANCE PANEL

  - A draft revision of ASTM D7528 was prepared and issued to the surveillance panel prior to this meeting. In this meeting Alan Flamberg provided an overview of changes made to the method.
    - Alan discussed the challenges with specifying limits/tolerances for dilute NO2. He shared the proposed limits/tolerances along with an example of how to calculate total NO2 introduced during the test.
    - The surveillance panel was encouraged to review the draft method and provide any comments or recommendations to the dilute NO2 workgroup (Alan Flamberg, Matt Schlaff, Justin Mills, Tom Schofield) prior to the next SP meeting. If all goes according to plan, we will seek a SP vote at the next meeting.
  - It was also brought to our attention that the data dictionary and report forms will need to be updated before implementing dilute NO2. This process may take several weeks.
- No additional topics
- Next meeting tentatively scheduled on May 13, 2021. Date may be postponed if necessary.
- Meeting adjourned 11:28EDT

Meeting Outcome:

1. The following limits for reference oil 436 were accepted as interim limits:

TMC 436		n	Natural Log Transformed Mean (ln)	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 #3	Average Yi from TMC statistics (Yi = -0.1000). Outliers included	17	10.3437	31,061	0.1605	22,677	42,544	10.0291	10.6583

2. Reference oil 436 will be added to LTMS and is approved for reference oil assignment. Upon its adoption, use of 438-2 will be suspended.
3. The draft revision to ASTM D7528 was reviewed. The SP was encouraged to provide any comments or recommendations to the dilute NO2 workgroup (Alan Flamberg, Matt Schlaff, Justin Mills, Tom Schofield) prior to the next SP meeting. If all goes according to plan, we will seek a SP vote at the next meeting.

-End report-

ASTM D7528 ROBO SP Meeting April 15, 2021

Actions from April 15th meeting

- ☒ Tom Schofield to add reference oil 436 (and corresponding limits) in LTMS. Upon its adoption, use of 438-2 will be suspended.
- ☐ ROBO SP members to review draft ASTM D7528 with dilute NO2 procedure. Any comments or recommendations should be provided to the dilute NO2 workgroup (Alan Flamberg, Matt Schlaff, Justin Mills, Tom Schofield) prior to the next SP meeting.

Current status of ROBO

ASTM D7528: ROBO
Summary for D02.B0.07 – June 7, 2021

Status	Test Aspect	Comments
	Method	**Test method is in good standing**: ASTM D7528-17a was published in October 2017. ▪ Expect revision in 2021 to incorporate procedure for dilute NO2 alternative.
	Parts Availability	**All ROBO hardware and test materials are available** ▪ Nitrogen dioxide, the primary catalyst for ROBO, is available from multiple suppliers ▪ Alternative procedure with dilute nitrogen dioxide expected to be approved in 2021.
	Reference Oils	**All current reference oils are in good supply at TMC**: multiyear supply of each oil ▪ Final limits set for 434-3 ▪ Interim limits set for 436. Will be used to replace 438-2.
	Test Availability	**Test is available with no significant queues to report.** ▪ Less activity than prior semesters – reduced demand coincides with launch of GF-6 ▪ ROBO test is available at 6 labs (19 stands) – as of 3/31/2021
	Severity and Precision	**In last semester (Oct 2020 – Mar 2021) precision was worse than target and test ran with a slight mild bias:** ▪ N = 113, Pooled s = 0.3188 and Mean Δ/s = -0.11

ROBO Industry Statistics

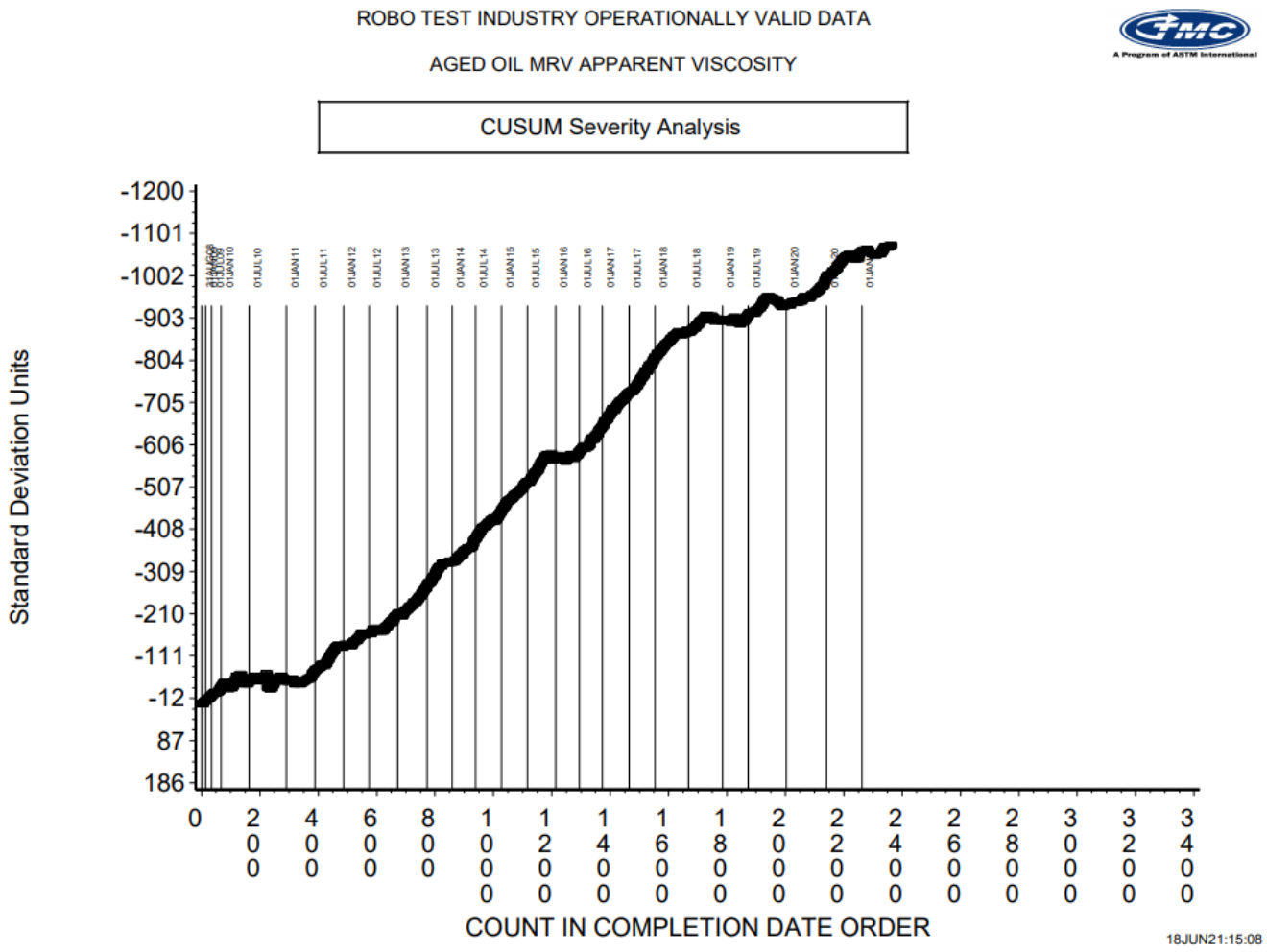
Period	N-size	Degrees of Freedom	Pooled s	Mean Δ/s
Current Targets	49	46	0.1945	------
4/1/17 through 9/30/17	99	95	0.2220	-0.76
10/1/17 through 3/31/18	91	87	0.2367	-0.91
4/1/18 through 9/30/18	126	122	0.2184	-0.49
10/1/18 through 3/31/19	100	96	0.2738	0.04
4/1/19 through 9/30/19	95	91	0.2492	-0.32
10/1/19 through 3/31/20	158	153	0.2723	-0.10
4/1/20 through 9/30/20	119	113	0.2264	-0.76
10/1/20 through 3/31/21	113	108	0.3188	-0.11
4/1/21 through 9/30/21	55	49	0.1912	-0.41

Added after our meeting.

- ~~No data available for current semester.~~

Source: http://www.astmtmc.cmu.edu/ftp/refdata/bench/robo/data/statistics.txt (Apr07-2021)

CUSUM severity analysis



Source: http://www.astmtmc.cmu.edu/ftp/refdata/bench/robo/plots/mrv%20INDUSTRY.pdf (June 18, 2021)

Reference oils

TMC reference oils
Current limits

D7528 (ROBO) Aged Oil MRV Acceptance Bands, mPa·s and ln(mPa·s)

Oil	n	Natural Log Transformed Mean (ln)	Mean in Original Units	s.d. (ln)	95% band in mPa·s Min[1]	95% band in mPa·s Max[1]	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	[2]10.9284	[2]55,737	0.1551	[2]41,126	[2]76,008	[2]10.6244	[2]11.2386
434-3	22	[2]10.8172	[2]49,871	0.1389	[2]37,987	[2]65,473	[2]10.5450	[2]11.0894
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	[4]44570	92910	[4]10.7048	11.4394
436	17	[2]10.3437	[2]31,061	0.1605	22,677	42,544	10.0291	10.6583
438	14	10.2676	28,785	0.2037	19,308	42,912	9.8683	10.6669
438-2	19	[2]10.5404	[2]37813	0.2596	[2]22,734	[2]62,894	[2]10.0316	[2]11.0492

[1] 95% bands in mPa·s are listed for information purposes only, the transformed values will be used to judge acceptance in all cases.
[2] A bias adjustment has been applied to the mean of reference oils 434-2, 434-3, 436 and 438-2 to account for biases observed in the TMC reference data during the periods that each oil target dataset was generated. The 95% confidence range reflects the inclusion of the bias adjustments.
[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.
[4] 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 15th meeting, SP approved limits for 436 and it could replace 438-2.
  - Only 3 new datapoints since preliminary limits were set. Will reevaluate limits when more data is available.

Source: http://www.astmtmc.cmu.edu/ftp/docs/ltms/ltms.pdf 05-2021 version

Dilute NO_2

Dilute nitrogen dioxide
Next steps

Microsoft Word Document

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

☒ **1) 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 → Draft completed, but now need input/review from SP.

☐ ▪ Revisions made from version shared at April 15th – mainly editorial in nature (e.g. correcting footnote numbering)

☐ 3) Approve by SP → 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

Proposal to include additional field on reference oil table

D7528 (ROBO) Aged Oil MRV Acceptance Bands, mPa·s and ln(mPa·s)

Oil	n	Natural Log Transformed Mean (ln)	Mean in Original Units	s.d. (ln)	95% band in mPa·s Min[1]	95% band in mPa·s Max[1]	95% Bands Min (ln)	95% Bands Max (ln)	Reference oil Status
434-1	13	10.6599	42,612	0.1672	30,706	59,136	10.3322	10.9876	Obsolete
434-2	36	[2]10.9284	[2]55,737	0.1551	[2]41,126	[2]76,008	[2]10.6244	[2]11.2386	Obsolete
434-3	22	[2]10.8172	[2]49,871	0.1389	[2]37,987	[2]65,473	[2]10.5450	[2]11.0894	Active
435	15	11.4895	97,685	0.2932	[3]60,000	173,546	[3]11.0021	12.0642	Obsolete
435-1	22	11.0416	62,420	0.20295	[4]44570	92910	[4]10.7048	11.4394	Active
436	17	[2]10.3437	[2]31,061	0.1605	22,677	42,544	10.0291	10.6583	Active
438	14	10.2676	28,785	0.2037	19,308	42,912	9.8683	10.6669	Obsolete
438-2	19	[2]10.5404	[2]37813	0.2596	[2]22,734	[2]62,894	[2]10.0316	[2]11.0492	Active

- Proposal to add additional column to LTMS's list of reference oils and acceptance bands to reflect the reference oil status.

Source: http://www.astmtmc.cmu.edu/ftp/docs/ltms/ltms.pdf 05-2021 version

Data dictionary update and report form update

- Need to add fields to reflect whether apparatus is configured for concentrated or dilute NO2.

Any Additional Topics?

Next Meeting

- Suggestions for next SP meeting?