

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting

Date: November 29, 2018

Location: Skype meeting

Minutes by: Justin Mills – SP Chair

Actions:

1. Justin Mills to follow up with Stats Group for an update
2. Tom Schofield to organize shipments of 438-2 to Intertek, Evonik, SWRI, and Lubrizol
3. Justin Mills took to calculate concentration limits for dilute NO₂
4. Tom Schofield to include 434-2 footnote in the TMC Calibration Requirement.
5. Justin Mills to schedule next SP meeting for Thursday, January 10th.

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 Zweitzig, *Joan Souchik, *John Maxwell
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 Miiller, Ted Selby
General Interest	*Alan Flamberg
Guests	Jessica Roach - Intertek

* Denotes attendance

Summary:

- Meeting convened at 10:03EST on November 29, 2018
- Agenda accepted by SP without any modifications
- ASTM Antitrust and Recording Policy reviewed

MEETING MINUTES: ROBO SURVEILLANCE PANEL

- Membership review and update
 - No changes to report
- Meeting minutes from October 25th SP meeting were accepted
 - Motion made by Matt Schlaff and seconded by Tom Schofield
- Actions from the October 25th meeting were reviewed
 - The only outstanding action is for Matt Schlaff or Justin Mills have not yet contacted ASTM's ILS group for advice on approving dilute nitrogen dioxide as an alternative to concentrated nitrogen dioxide in the ASTM D7528 method.
- Stats Group update
 - Nothing to report. Justin Mills will contact Stats Group for an update.
- Reference oil supply – TMC 438 supply is critically low
 - TMC has 16.4 gallons of 434-2 in their inventory, but Tom believes they can get more once the Sequence IIIG is offline.
 - TMC inventory of 438 is running critically low – there is only enough remaining for 7 ROBO samples.
 - Tom Scofield was hoping to get additional 438 from Sequence IIIG, but that has not happened yet
 - While inventory is low at TMC, most labs should have some 438 already in their inventory.
 - TMC 438-2 was proposed as an alternative to 438 (438-1 is not available in sufficient quantities).
 - TMC can obtain 20-55 gallons for ROBO
 - Intertek, SWRI, Evonik, and Lubrizol (through email) offered their support and are willing to donate 438-2 runs. Justin Mills asked that these donated runs only be ran on certified units.
 - Tom Schofield to organize shipment of 438-2 to labs that volunteered.
- Dilute nitrogen dioxide
 - Adding dilute nitrogen dioxide (1.13% NO₂ in air) at 185 ml/min over first 12 hours is equivalent to mixing 2ml of concentrated NO₂ with 185ml of dry air over 12 hours.
 - Details of Evonik's dilute NO₂ configuration were shared again.
 - Intertek shared details of their dilute NO₂ configuration as well as results from their TMC runs.
 - Intertek is using standard cylinder size – Matt believes one cylinder can support ~20 tests.
 - Utilize a 3-way purge valve on regulator
 - Incorporated a mass flow meter from Cole-Parmer to regulate dilute NO₂ flow
 - Intertek ran all three TMC reference oils using dilute NO₂ and compared those results to average results for concentrated NO₂. No substantial differences were observed.
 - Matt commented that the dilute NO₂ is much easier to run than concentrated NO₂.
 - Justin Mills took an action to calculate concentration limits for dilute NO₂
 - Several opinions were shared in regards to implementing dilute NO₂ as an alternative to concentrated NO₂.
 - Since an equivalent amount of NO₂ is being used overall, we can treat this as an addition to the method and handle the change within our SP then move it to ballot – likely path.
 - Once SP is confident in results, we can provisionally allow labs to reference using dilute NO₂ allowing our SP to quickly expand the dataset. Prior to taking this path we would need to draft a provisional method for dilute NO₂ addition.
 - Members noted concern about reduced capacity with dilute gas due to the large size of cylinders.
 - Set up of dilute nitrogen dioxide will require additional changes to ROBO setup for safety concerns.
 - Motion to add a field to data dictionary for NO₂ concentration was approved
 - Allowed values = concentrated or dilute
 - Motion made by Matt Schlaff and seconded by Tom Schofield
- Calibration requirements for new labs
 - Calibration requirements were discussed, but no clear consensus was reached. Agreed to keep as agenda topic for next SP meeting.
- Footnote for TMC 434-2
 - Matt Schlaff made a motion to approve the following footnote for TMC 434-2 in TMC Calibration Requirements: "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)."
 - Motion was seconded by Mike Birke and approved.
- Next meeting to be scheduled for January 10, 2018.

MEETING MINUTES: ROBO SURVEILLANCE PANEL

- Meeting adjourned

ROBO Surveillance Panel Meeting

November 29, 2018

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 October 25th meeting
- Update on recent activity with ASTM Stats Group
- Reference oil supply – TMC 438 is running low and we need to begin 438-x round robin study
- Dilute nitrogen dioxide discussion – review available data and determine next steps
- Calibration requirements for new units
- Adding a footnote to TMC Calibration Requirements to reflect bias correction factor to 434-2 limits
- Additional topics, if any
- Set next meeting

ASTM Antitrust and Recording Policy

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Electronic recording of ASTM meetings is prohibited.

Membership – Updated 10/25/18

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
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 Miiller, Ted Selby
General Interest	Alan Flamberg

Summary of changes:

- Removed Elizabeth Wagoner from Evonik Oil Additives

Motion to accept October 25, 2018 meeting minutes

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting
Date: October 25, 2018
Location: Skype meeting
Minutes by: Justin Mills

Agenda:

- Matt Schloff or Justin Mills to contact ASTM's ILS group and determine appropriate course of action for approving dilute nitrogen dioxide as an alternative to concentrated nitrogen dioxide in the ASTM D7528 method.
- Justin Mills to provide link to TMC LTMS document that contains ROBO calibration requirements.
 - <http://www.astmtmc.cmu.edu/ftp/docs/ltms/>
- Justin Mills to include "calibration requirements for new units" as a topic in the next SP meeting.
- Justin Mills and Tom Schofield to develop wording for footnote regarding 434-2 bias correction in the TMC Calibration Requirement. Justin Mills to include this as a topic in next SP meeting.
- Justin Mills to schedule next SP meeting for Thursday, November 29th.

Membership and Attendance:


Ace Glass	Dave Lawrence
Alfon	Shelia Thompson, Jeff Yang, Todd Dvorak
ASTM TMC	Tom Schofield
BASF	Mary Dery, Bridget Rakastraw
Chevron Oronite	Man Hon Tsang, Robert Stockwell
Exxon-Mobil	Dennis Gasi
Infinium	Andy Rishka, Sagna Eboata
Intertek	Joe Franklin, Matt Schloff
Lubrizol	Mike Felle, Kamesh Chinnappa, Rick Hartman
Penn/Oronite	Li Shaojun, Sun Ruihua, Peng Wang, Xiangang Li, Ju Li
Evonik Oil Additives	Justin Mills, Thore Ziering, Jan Szwedlik, John Maxwell
Vandermil Chemicals	Jai Fife, Ron Hays
Shell	Betsy Grinstead, Joe De La Cruz, Mike Birke, Young Li, McFarland
Valvoline	Amal Savant, Kevin Figgitt, Steve Labarre
Koch/Instruments	Raj Shah, Vinod Chaitani
Tamco/Savant	Drew Miller, Ted Setty
General Interest	Alan Flomberg
Guests	

* Denotes attendance

Summary:

MEETING MINUTES: ROBO SURVEILLANCE PANEL

- Meeting convened at 10:05ET on October 25, 2018
- Agenda accepted by SP without any modifications
- ASTM Approval and Meeting Primary reviewed
- Membership review and update
 - Elizabeth Wagner of Evonik was removed from the membership list, because she is no longer with Evonik.
- Meeting minutes from June SP meeting were accepted
 - Motion made by Matt Schloff and seconded by Shelia Thompson
- Actions from the July SP meeting were reviewed - all actions from July SP meeting have been completed (see attached slides for further details)
 - Leen Fioot of Kuwait Petroleum was removed from ROBO SP membership list due to inactivity (likely retired from Kuwait Petroleum).
 - Justin Mills emailed API to provide an update on ROBO test issues at independent labs so that API may assess whether or not provisional licensing is still necessary. Justin reported that API decided to maintain provisional licensing despite the relatively low test queues.
 - The current queue at IRI is about 1 week and at Intertek the queue is about 2-3 weeks.
 - Tom Schofield has updated the TMC Calibration Requirements to reflect the new TMC 434-2 limits and specify yield stress as a pass/fail criteria for all current reference oils.
 - Tom reported that all calibration documents for TMC monitored tests will now be housed in a single LTMS document that can be accessed through the TMC website <http://www.astmtmc.cmu.edu/ftp/docs/ltms/>



- Evonik has resumed work on the dilute nitrogen dioxide study/compaction.
 - Justin Mills and Tom Schofield provided the ASTM State Group with a ROBO timeline outlining dates for critical ROBO events (see attached slides for timeline).
 - The State Group is currently very busy with supporting Cummins engine tests for SP-6.
 - Justin Mills has expressed to the State Group that investigating severity factors or some alternative form of a severity factor has been on our SP's to-do list for a while but we consider it to be low priority.
- Dilute nitrogen dioxide
 - Active dilute nitrogen dioxide (1.13% NO₂ in air) at 195 micron over the 12 hours is available to assist TMC

MEETING MINUTES: ROBO SURVEILLANCE PANEL

- Evonik has resumed work on the dilute nitrogen dioxide study/compaction
 - Evonik's configuration was shared (see attached slides)
 - Evonik compared results for standard NO₂ and dilute NO₂ ROBO runs on their internal 500-20 QC oil, and observed comparable values for oxidation, pVla, and MBV.
 - Evonik plans to run the three TMC reference oils back-to-back using the dilute NO₂ to confirm epistropy.
 - As the request of the SP, Evonik will run two back-to-back sets of TMC reference oils. If two back-to-back sets are completed with passing results, Mike Fioot of Lubrizol said he would feel confident to begin his comparison study.
- To implement dilute nitrogen dioxide as an alternative to pure nitrogen dioxide, an ILG may be necessary. Matt Schloff believes there may be a regional alternative for epistropy studies. Matt to contact ILS group or properly Justin with appropriate contacts.
 - If implemented, lots will likely need to conduct a 2 test reference when switching from standard concentrated nitrogen dioxide to dilute nitrogen dioxide.
 - Intertek plans to participate in the dilute nitrogen dioxide study, but their current backlog of samples is preventing them from converting an existing, calibrated ROBO unit to a dilute NO₂ configuration.
- Additional topics
 - Unclear/undiscuss whether or not all new ROBO units must complete 3 back-to-back runs with TMC reference oils followed by 2 back-to-back blind reference runs. Justin Mills and Tom Schofield believe that existing lots with multiple ROBO units should only be required to complete the standard 3 back-to-back blind reference runs. Agreed to address this topic at next SP meeting.
 - Matt Schloff suggested we add a footnote on the TMC Calibration Requirements for 434-2 limits to describe the bias correction. Justin Mills to work with Tom Schofield on wording for footnote. Agreed to address this topic and possibly vote on its inclusion into calibration requirements at the next meeting.
- Next meeting to be scheduled for November 29, 2018
- Meeting adjourned

Meeting minutes approved.

Actions from October 25th meeting

- Matt Schloff or Justin Mills to contact ASTM's ILS group and determine appropriate course of action for approving dilute nitrogen dioxide as an alternative to concentrated nitrogen dioxide in the ASTM D7528 method.
 - Open
- Justin Mills to provide link to TMC LTMS document that contains ROBO calibration requirements.
 - Complete. Included in minutes from previous meeting. Can also be found at <http://www.astmtmc.cmu.edu/ftp/docs/ltms/>
- Justin Mills to include "calibration requirements for new units" as a topic in the next SP meeting.
 - Complete. See agenda
- Justin Mills and Tom Schofield to develop wording for footnote regarding 434-2 bias correction in the TMC Calibration Requirement. Justin Mills to include this as a topic in next SP meeting.
 - Complete. Will discuss in later slides
- Justin Mills to schedule next SP meeting for Thursday, November 29th.
 - Complete.

ASTM Stats Group Support Update

- Nothing to report

Reference oil supply

TMC 438 supply is critically low

Oil	Year Rec'd By TMC	Tests	TMC Inventory, gallons	Gallons Shipped last 12 months
434-2	2014	ROBO	16.4	5.1
435-1	2008	ROBO	416.2	23.5
438***	2003	ROBO	0.6	3.0

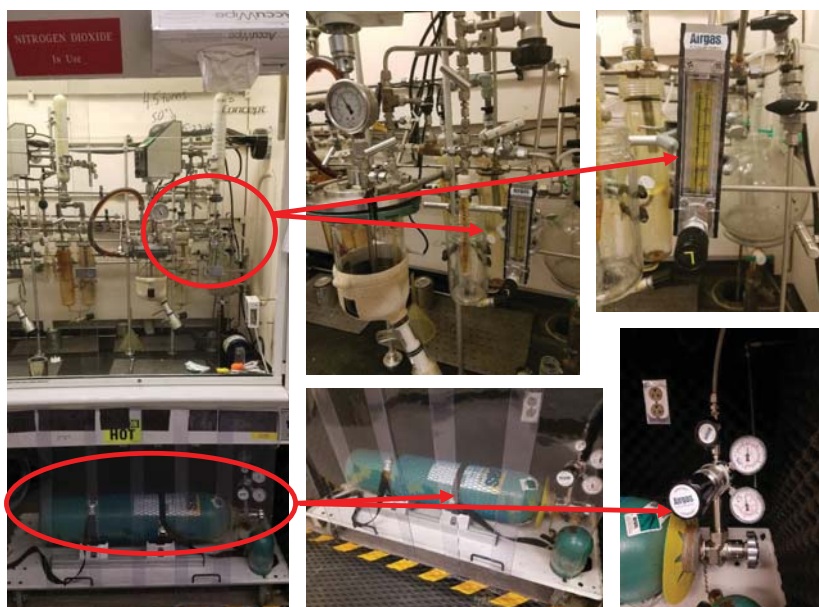
- TMC 438 is running critically low – there is only enough remaining for 7 ROBO samples.
 - Tom Scofield was hoping to get additional 438 from Sequence IIIIG, but that has not happened yet
- Propose that we introduce 438-2 as a replacement – TMC can obtain 20-55 gallons for ROBO
- **Discussion and next steps**
 - Donate runs or use as calibration oil?

Dilute nitrogen dioxide: Current setting vs proposed alternative

Ingredient	ROBO Setting	Proposed dilute NO ₂ setting
Test fluid	200 grams	200 grams
Iron ferrocene	15 PPM	15 PPM
Nitrogen dioxide	2 ml "pure" NO ₂ fed over first 12 hours	1.13% NO ₂ in air fed at 185 ml/min over first 12 hours
Dry air	185 ml / minute (entire test)	185 ml / minute (12 hours – EOT)
Agitation	200 RPM	200 RPM
Vacuum	0.61 Bar 56.6 L/min	0.61 Bar 56.6 L/min
Temperature	170°C	170°C
Time	40 Hours	40 Hours

Only difference is nitrogen dioxide and dry air.

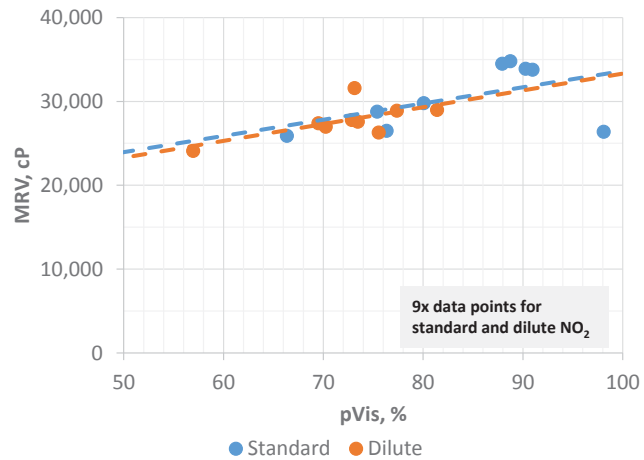
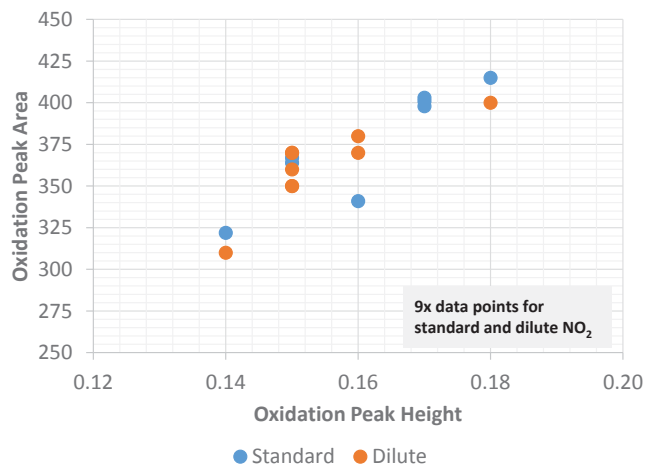
Evonik's configuration for dilute NO₂



Air cylinder	Air Gas
Regulator	Air Gas
Flow meter	Air Gas

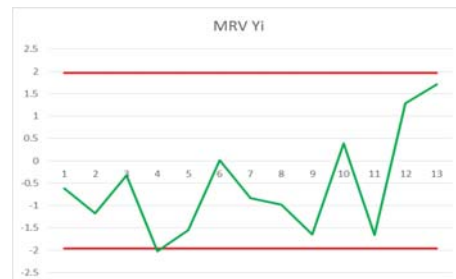
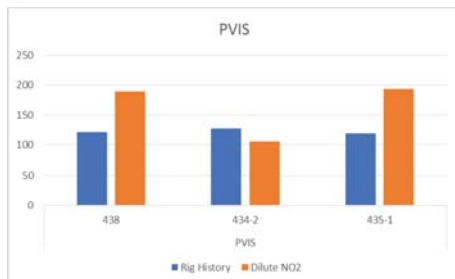
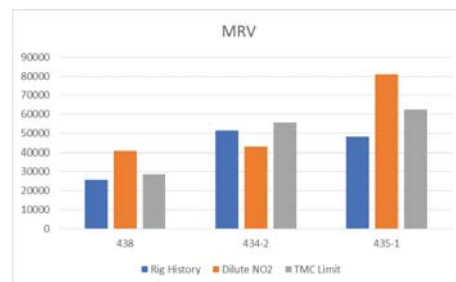
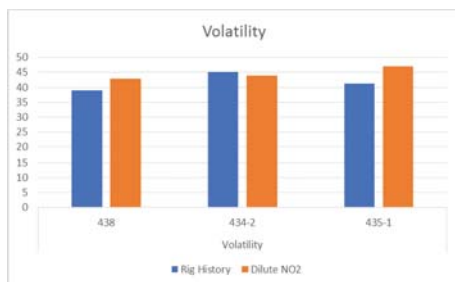
- Temporary configuration
- Permanent configurations will likely include:
 - Permanent cabinet for storing larger, upright NO₂ bottles
 - Digital flow meter/controller
 - On/Off solenoid or switch to convert from dilute NO₂ to dry air

Dilute nitrogen dioxide: Evonik's data on internal reference oil



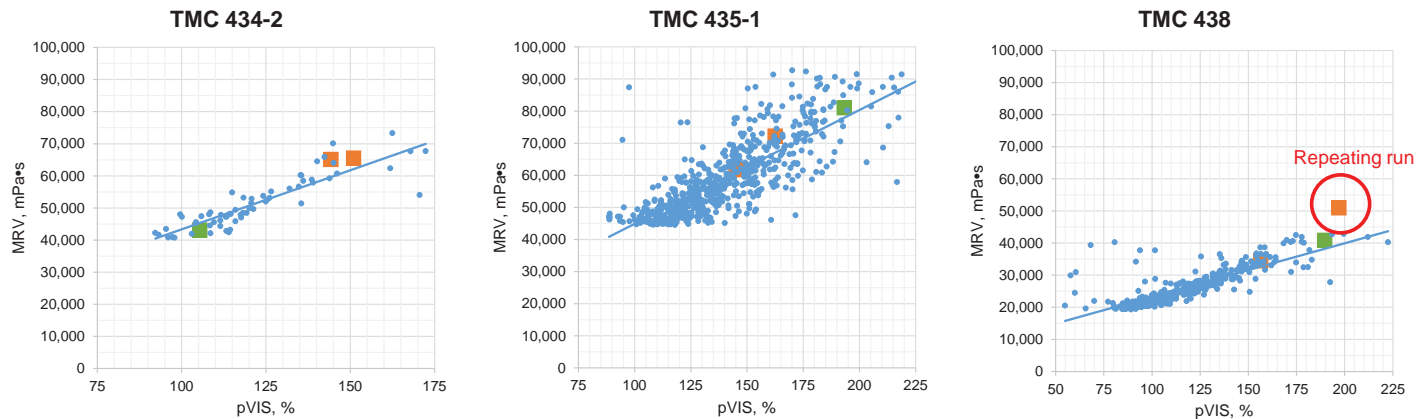
Comparable levels of oxidation achieved. Similar pVis and MRV.

Intertek's configuration for dilute NO₂



Dilute nitrogen dioxide: Results with TMC calibration oils

As requested by SP at October 25th meeting, Evonik has completed two back-to-back sets of runs with the TMC reference oils.



- Comparable pVIS and MRV values.
- Similar MRV vs pVIS relationship observed.

Dilute nitrogen dioxide: Next steps

- What other data is necessary to approve dilute NO₂ as an alternative to concentrated NO₂?
- When can other labs begin testing?
 - At the last SP meeting, Matt Schlaff (Intertek) indicated that they could participate in dilute NO₂ study as soon as their backlog lessened. Mike Faile (Lubrizol) indicated that he would be willing to participate after the two back-to-back sets were completed with passing results

Motion to add field to Data Dictionary for NO2 concentration

- Add field to Data Dictionary for NO2 concentration
 - Allowed values = Concentrated or dilute

Motion accepted.

Calibration requirements for new units

Current requirements for “B. Acceptance Criteria”

1. New Laboratory/Test Stand(s)

- a. The TMC calibration auditing system calibrates individual test stands at individual laboratories. There are no requirements to bring a lab into TMC calibrated status, there are only requirements to bring individual test stands into TMC calibrated status, as follows:
- b. A minimum of two (2) operationally valid calibration tests which fall within the acceptance bands for the oils assigned are required to calibrate a stand for the first time. These must be back-to-back consecutive runs on the same test stand, though exceptions can be made at the sole discretion of the TMC for operational fails for reasons that would be considered to have had no bearing on the operational performance of the test stand for subsequent tests (for example, a power failure)
- c. Passing two back-to-back consecutive TMC calibrations places the new test stand in TMC calibrated status. Both tests must pass on operational and statistical criteria.
- d. TMC calibrated status of a test stand is valid for no more than 50 days from date completed of a valid TMC calibration (that is, the end of the test’s 40-hour oil oxidation heating cycle), or no more than 15 subsequent test starts on the stand (as counted sequentially by run number; see Item 3), whichever comes first. To renew the calibration at the end of the calibration period, see Item 2 for Existing Laboratory/Test Stand(s).

Calibration requirements for new units

Proposed requirements for “B. Acceptance Criteria”

1. New Laboratory/New Test Stand(s)
 - a. The TMC calibration auditing system calibrates individual test stands at individual laboratories. There are no requirements to bring a lab into TMC calibrated status, there are only requirements to bring individual test stands into TMC calibrated status, as follows:
 - b. Prior to obtaining calibration test oils from the TMC, new laboratories introducing a test stand must demonstrate their stand can successfully run all three (3) current TMC calibration oils (unblind) within the TMC acceptance bands. Upon acceptance of these results by the TMC, the lab may request the two test calibration.
 - c. A minimum of two (2) operationally valid calibration tests which fall within the acceptance bands for the oils assigned are required to calibrate a stand for the first time. These must be back-to-back consecutive runs on the same test stand, though exceptions can be made at the sole discretion of the TMC for operational fails for reasons that would be considered to have had no bearing on the operational performance of the test stand for subsequent tests (for example, a power failure)
 - d. Passing two back-to-back consecutive TMC calibrations places the new test stand in TMC calibrated status. Both tests must pass on operational and statistical criteria.
 - e. TMC calibrated status of a test stand is valid for no more than 50 days from date completed of a valid TMC calibration (that is, the end of the test’s 40-hour oil oxidation heating cycle), or no more than 15 subsequent test starts on the stand (as counted sequentially by run number; see Item 3), whichever comes first. To renew the calibration at the end of the calibration period, see Item 2 for Existing Laboratory/Test Stand(s).
2. Existing Laboratory/New Test Stand(s)
 - a. Use current wording from “1. New Laboratory/Test Stand(s)” in 20180720 ROBO TMC Calibration Requirements.

Adding a footnote to TMC Calibration Requirements to reflect bias correction factor to 434-2 limits

Table 1
MRV VISCOSITY
Unit of Measure: LN(MRV)

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 ²	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	10.9284	55,737	0.1551	41,126	76,008	10.6244	11.2386
435	15	11.4895	97,685	0.2932	60,000	173,546	11.0021	12.0642
435-1	22	11.0416	62,420	0.20295	344570	92910	10.7048	11.4394
438	14	10.2676	28,785	0.2037	19,308	42,912	9.8683	10.6669

Proposed wording for footnote:

⁴ 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).

Any proposed changes?

¹ 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.

² 95% bands in mPa s are listed for information purposes only, the transformed values will be used to judge acceptance in all cases.

³ The minimum value for reference oil 435-1 is based on -1.66 standard deviations from the target (60,000 mPa s) (11.0021 in transformed units).

mean.

Proposed footnote approved.

Any Additional Topics?

Next Meeting

- Suggestions for next SP meeting?
 - Need adequate time to generate 438-2 data so that we can set limits at meeting.
 - ~~Thursday, December 20th?~~ **Thursday, January 10th?**