

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

**Meeting:** ROBO SP Meeting

**Date:** January 10, 2109

**Location:** Skype meeting

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

**Actions:**

1. Justin Mills to add D7528 housekeeping as topic for next SP meeting.
  - a. SP members are encouraged to review method and bring forward any changes that may be necessary.
2. Justin Mills to schedule next SP meeting for Thursday, January 10<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 Shinheartl, 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 Miller, Ted Selby
General Interest	Alan Flamberg
Guests	None

\* Denotes attendance

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

### Summary:

- Meeting convened at 10:02EST on January 10, 2019
- Agenda accepted by SP without any modifications
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
  - No changes to report
- Meeting minutes from November 29<sup>th</sup> SP meeting were accepted
  - Motion made by Shelia Thompson and seconded by Greg Miller
- Actions from the November 29<sup>th</sup> meeting were reviewed
  - Tom Schofield has shipped 438-2 to Intertek, Evonik, SWRI, and Lubrizol
  - Justin Mills calculated concentration limits for dilute NO<sub>2</sub> and presented limits on subsequent slides
  - Tom Schofield has started the workflow to include a footnote for 434-2 in the LTMS. The change will be reflected when the updated version of the LTMS is posted on TMC's website
- Stats Group update
  - Nothing to report.
- Reference oil 438-2
  - TMC has secured a full drum (55 gallons) of 438-2 for ROBO
  - At the last SP meeting, Intertek, SWRI, Evonik, and Lubrizol agreed to donate 438-2 runs for the round robin. TMC has shipped 438-2 samples to each lab.
    - Evonik has completed two runs; however one was declared operationally invalid due to vacuum line failure. Data from the valid run was shared. The MRV was high compared average 438 value, but it is still premature to make any judgements.
    - Lubrizol is running 438-2 this week
    - SwRI and Intertek will start their runs next week
- Dilute nitrogen dioxide limits
  - Justin shared his calculation for concentration limits of NO<sub>2</sub> in air: 1.07% - 1.19%
- Implementation of dilute nitrogen dioxide
  - The likely path forward to implement dilute NO<sub>2</sub> as an alternative to pure NO<sub>2</sub> is the following:
    - Demonstrate equivalence to the SP – Thus far, 2+ runs on each reference oils have been conducted between Intertek and Evonik. Intertek has either completed (or will complete in near future) an additional set of runs on TMC reference oils. Evonik will also generate more data. At this point in time, no other lab is able to provide additional support.
    - Develop a procedure for dilute NO<sub>2</sub>
    - Approve by SP – Vote at SP meeting
    - Issue information letter allowing use of dilute NO<sub>2</sub> as an alternative
    - Ballot the recommended changes at ASTM
  - Lubrizol is currently assessing whether or not they want to implement dilute NO<sub>2</sub> – may diminish capacity
  - SwRI reported they are not interested in the dilute alternative due to space and safety constraints.
  - Tom Schofield will set aside (3) 438 at Evonik and Intertek for dilute NO<sub>2</sub> equivalency testing
- Calibration requirements
  - Calibration requirements for ROBO test are listed in three places: the D7528 method in Section 9, the ROBO TMC Calibration Requirements, and most recently Section 47 of the LTMS document (The ROBO TMC Calibration previously served as a stand-alone document but it has been incorporated into the LTMS document)
  - Justin Mills shared a proposal to update the TMC calibration requirements to specify three categories for acceptance criteria – New Laboratory/New Test Stand(s), Existing Laboratory/New Test Stand(s), and Existing Laboratory/Existing Test Stand(s). Under the proposed changes, New Laboratory/New Test Stand(s) would be required to demonstrate their stand can successfully run all three (3) current TMC calibration oils within the TMC acceptance bands prior to requesting the double-blind calibration tests. Existing Laboratory/New Test Stand(s) would only be required to run the double-blind calibration. (see attachment for further details)
    - If this change is made to the calibration requirements or LTMS, Section 9 of D7528 would also need to be updated. Regardless of whether or not the calibration requirements change, most labs agreed

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that they would run all three (3) current TMC calibration oils prior to pursuing certification as part of their internal due diligence.

- It was suggested that we reserve time at the next SP meeting to discuss method housekeeping. It was also suggested that for Section 9 of D7528 we reference the LTMS document for calibration requirements to ensure we don't misalign the two documents.
- Reactor clamps from ACE Glass
  - The design ACE Glass's reactor clamps has changed slightly. Most notably the inner diameter is smaller and the pitch angle may have changed. As a result, the clamp may need to be seated at a lower position on the reaction vessel. Operators may need to adjust the clamp position (height) on their lattice structure. It was also noted that the new clamps may have considerable "give" or flex where the two parts of the clamp are fastened/riveted together. After investigation, Justin Mills believes the clamp design change will not impact test operation.
- Next meeting to be scheduled for February 21, 2019.
- Meeting adjourned

# ROBO Surveillance Panel Meeting

January 10, 2019

Justin Mills

# Agenda

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- Welcome, ASTM statement
- Review membership of SP
- Review and approve minutes from previous meetings (see attachment)
- Review and follow-up on actions from November 29<sup>th</sup> meeting
- TMC 438-2 update
- Dilute nitrogen dioxide
  - Dilute nitrogen dioxide concentration limits
  - Path forward
- Calibration requirements for new units
- 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

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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:

- None to report

# Motion to accept November 29, 2018 meeting minutes

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting  
 Date: November 29, 2018  
 Location: Skype meeting  
 Minutes by: Justin Mills – SP Chair

### Actions:

- Justin Mills to follow up with Stats Group for an update
- Tom Schofield to organize shipments of 438-2 to Intertek, Evonik, SWRI, and Lubrizol
- Justin Mills took to calculate concentration limits for dilute NO<sub>2</sub>
- Tom Schofield to include 434-2 footnote in the TMC Calibration Requirement.
- Justin Mills to schedule next SP meeting for Thursday, January 10<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
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Valvoline	Amol Savant, Kevin Figgatt, *Steve Lazzara
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	*Alan Flamberg
Guests	Jessica Roach - Intertek

\* Denotes attendance

### Summary:

ASTM D7528 ROBO SP Meeting November 29, 2018

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

- Meeting convened at 10:03EST on November 29, 2018
- Agenda accepted by SP without any modifications
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
  - No changes to report
- Meeting minutes from October 25<sup>th</sup> SP meeting were accepted
  - Motion made by Matt Schlaff and seconded by Tom Schofield
- Actions from the October 25<sup>th</sup> 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 IIIIG is offline.
  - TMC inventory of 438 is running critically low – there is only enough remaining for 7 ROBO samples.
    - Tom Schofield was hoping to get additional 438 from Sequence IIIIG, 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% NO<sub>2</sub> in air) at 185 ml/min over first 12 hours is equivalent to mixing 2ml of concentrated NO<sub>2</sub> with 185ml of dry air over 12 hours.
  - Details of Evonik's dilute NO<sub>2</sub> configuration were shared again.
  - Intertek shared details of their dilute NO<sub>2</sub> 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 NO<sub>2</sub> flow
    - Intertek ran all three TMC reference oils using dilute NO<sub>2</sub> and compared those results to average results for concentrated NO<sub>2</sub>. No substantial differences were observed.
    - Matt commented that the dilute NO<sub>2</sub> is much easier to run than concentrated NO<sub>2</sub>
  - Justin Mills took an action to calculate concentration limits for dilute NO<sub>2</sub>
    - Several opinions were shared in regards to implementing dilute NO<sub>2</sub> as an alternative to concentrated NO<sub>2</sub>.
      - Since an equivalent amount of NO<sub>2</sub> 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 NO<sub>2</sub> allowing our SP to quickly expand the dataset. Prior to taking this path we would need to draft a provisional method for dilute NO<sub>2</sub> 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 NO<sub>2</sub> 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

ASTM D7528 ROBO SP Meeting November 29, 2018

## MEETING MINUTES: ROBO SURVEILLANCE PANEL

- 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 adjourned

ASTM D7528 ROBO SP Meeting November 29, 2018



## Actions from November 29<sup>th</sup> meeting

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- Justin Mills to follow up with Stats Group for an update
- Tom Schofield to organize shipments of 438-2 to Intertek, Evonik, SWRI, and Lubrizol
  - Complete.
- Justin Mills took to calculate concentration limits for dilute NO<sub>2</sub>
  - Complete.
- Tom Schofield to include 434-2 footnote in the TMC Calibration Requirement.
  - Pending.
- Justin Mills to schedule next SP meeting for Thursday, January 10th.
  - Complete.

# ASTM Stats Group Support Update

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- Nothing to report

# Reference oil supply

## TMC 438 supply is critically low

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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

- Will introduce 438-2 as a replacement – 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.
  - TMC has sent 438-2 to labs

## 438-2 results

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IND	DTCOMP	APPARATS	TESTKEY	MRVTEMP	MRVYSEOT	MRV	MRVti	VOLEOT	PVIS
438-2	20190104	AM 4	142341-ROBO	-30	<35	41,100	10.6238	52	148
Average of all charted 438 results				-30	<35	29,061	10.18633	44	121

## Concentration limits for dilute nitrogen dioxide

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- Current limits

- 10.5.3 *Charging Nitrogen Dioxide*—Transfer 2.0 mL ± 0.1 mL of liquid nitrogen dioxide (see Section 8 and warning in 7.1) into the graduated tube.

- Proposed limits for concentration of dilute nitrogen dioxide

- Recommended (equivalent to 2.0 mL of liquid NO<sub>2</sub>) = 1.13% NO<sub>2</sub> in air by volume
- Minimum (equivalent to 1.9 mL of liquid NO<sub>2</sub>) = 1.07% NO<sub>2</sub> in air by volume
- Maximum (equivalent to 2.1 mL of liquid NO<sub>2</sub>) = 1.19% NO<sub>2</sub> in air by volume

# Dilute nitrogen dioxide:

## Next steps

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### Recommended path forward:

- 1) Demonstrate equivalence to Surveillance Panel. Complete?
  - As expected, data from Evonik and Intertek indicates equivalence.
- 2) Draft procedure for delivering dilute NO<sub>2</sub>.
- 3) Provisionally allow labs to reference using dilute NO<sub>2</sub> allowing our SP to quickly expand the dataset
- 4) Ballot the recommended changes

### Additional recommendations?

# Calibration requirements for new units

## Proposed requirements for “B. Acceptance Criteria”

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- Current version of acceptance criteria only has two classifications: “New Laboratory/Test Stand(s)” and “Existing Laboratory/Test Stand(s)”
- Beneficial to add a third classification for New Laboratory/New Stand(s) with more rigorous criteria.
  - Require new labs to demonstrate that their stand can successfully run all 3 reference oils within TMC acceptance bands prior to requesting calibration tests.
  
- See attachment for additional information.

**Any Additional Topics?**



## Next Meeting

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- Suggestions for next SP meeting?
  - **Thursday, February 21<sup>st</sup>?**

# Proposed changes to the LTMS calibration criteria

## Current Version

### 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).

#### 2. Existing Laboratory/Test Stand(s)

- a. An existing TMC calibrated test stand, or one where the TMC calibrated status had expired within the past 150 days, can renew its TMC calibrated status by demonstrating a successful calibration on another single TMC blind calibration audit. The test must pass on both operational and statistical criteria.
- b. TMC calibrated status of an existing 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 (as counted sequentially by run number) on the stand, whichever comes first. Test stands that exceed these time/run specifications are considered to be out of calibration for TMC monitoring purposes.
- c. A stand that has been out of TMC calibration for more than 150 days from the prior TMC calibration expiration date will require New Test Stand calibration as listed in B.1.b through B.1.d. of this document.
- d. A stand must pass the TMC calibration within two operationally valid test runs. If a stand cannot produce a calibration test that falls into the acceptance bands for the assigned oil within two operationally valid runs. Renewing calibration on that stand will require the two test calibration as listed in B.1.b through B.1.d.
- e. Changing the vacuum control valve set point, exchanging the reactor vessel or the vacuum pump, or changing the heating voltage setting by more than  $\pm 1$  volt on a stand for any reason voids any current TMC calibrated status. Renewing calibration on that stand will require the two test calibration as listed in B.1.b through B.1.d.

## Proposed Version

### 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 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).

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- 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)
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#### 3. Existing Laboratory/**Existing** Test Stand(s)

- a. An existing TMC calibrated test stand, or one where the TMC calibrated status had expired within the past 150 days, can renew its TMC calibrated status by demonstrating a successful calibration on another single TMC blind calibration audit. The test must pass on both operational and statistical criteria.
- b. TMC calibrated status of an existing 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 (as counted sequentially by run

- number) on the stand, whichever comes first. Test stands that exceed these time/run specifications are considered to be out of calibration for TMC monitoring purposes.
- c. A stand that has been out of TMC calibration for more than 150 days from the prior TMC calibration expiration date will require New Test Stand calibration as listed in B.2.b through B.2.d. of this document.
  - d. A stand must pass the TMC calibration within two operationally valid test runs. If a stand cannot produce a calibration test that falls into the acceptance bands for the assigned oil within two operationally valid runs. Renewing calibration on that stand will require the two test calibration as listed in B.2.b through B.2.d.
  - e. Changing the vacuum control valve set point, exchanging the reactor vessel or the vacuum pump, or changing the heating voltage setting by more than  $\pm 1$  volt on a stand for any reason voids any current TMC calibrated status. Renewing calibration on that stand will require the two test calibration as listed in B.2.b through B.2.d.