MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting

Date: November 18, 2021

Location: MS Teams (virtual)

Minutes by: Justin Mills - SP Chair

Actions:

- 1. Tom Schofield to update LTMS to reflect latest limits for 436 and incorporate the revised reference oil tables into the LTMS (approved at September 30, 2021 SP meeting).
- 2. Tom Schofield to incorporate editorial changes to IL21-01 proposed by Terry Bates.
- 3. Justin Mills to tentatively schedule the next ROBO SP meeting for February 3, 2022.

Membership and Attendance:

Ace Glass	Dave Lawrence, Tom Petrocella
Afton	Shelia Thompson, Jeff Yang, Todd Dvorak
ASTM TMC	*Tom Schofield
BG Products	*Madeleine Dellinger
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, *Christine Katrenya
SwRI	Becky Grinfield, Joe De La Cruz, *Mike Birke, *Young-Li McFarland
Valvoline	Amol Savant, *Amy Ross, Bruce Tonkel
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	*Alan Flamberg
Guests	

* Denotes attendance

Summary:

 \circ

- Meeting convened at 10:01EDT on November 18, 2021
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
 - o Ron Hiza of Vanderbilt has retired, and Christine Katrenya has replaced him.
 - Kris Flectcher represented in Oronite in place of Robert Stockwell at this meeting.
- Meeting minutes from September 30, 2021 SP meeting were accepted
- Actions from the July 22nd meeting were reviewed.
 - Tom Schofield to incorporate accepted changes to Section 13 and Appendix X7 into information letter.

 Status= Complete
 - Tom Schofield to update limits for reference oil 436.
 - Status= New 436 are active/effective, but LTMS still needs to be updated.
 - Tom Schofield to make reference oil 438 and 438-2 obsolete. Tom also to send test keys to labs with remaining 438/438-2 inventory so they may either dispose of or use internally.
 - Status= Complete
 - o Tom Schofield to incorporate the revised reference oil tables into the LTMS
 - Status= Awaiting next update to LTMS
 - Justin Mills to tentatively schedule the next ROBO SP meeting for November 18, 2021. Current status of ROBO
 - Status= Complete
 - Last semester (4/1/2021 through 9/30/21) finished slightly mild (-0.37), but precision in line with target (0.1992).
 - New precision limits have been set based on current reference oils \rightarrow 0.1551
 - Stats can be found here <u>https://www.astmtmc.org/ftp/refdata/bench/robo</u> on the recently moved TMC website.
- Dilute NO2

- ROBO Information Letter 21-01 was issued November 3, 2021 by the TMC with an effective date of December 1, 2021. Upon review of the IL, there were several editorial changes recommended by Terry Bates. SP agreed that all changes could be adopted without issue. Note, adopting these editorial changes does not impact the December 1, 2021 effective date. Summary of changes is below:
 - Add color code to IL to explain what each color means (e.g. red vs blue)
 - Change "% volume" and "concentration" to "volume fraction" to comply with ASTM standards. Many
 of the SP member felt "% volume" and "concentration" better described what we were trying to
 convey, but in the end agreed that change could be implemented to comply with ASTM standards.
 - Changes to format temperature is reported
- Reporting NO2 volume delivered during test was discussed at length. In the revised method it states "13.3.6.1 If the dilute nitrogen dioxide option was used, calculate and report the total amount of nitrogen dioxide delivered to the reactor. See Appendix X.7 for an example calculation."; however, the data dictionary (and report forms) will include an entry for "TOTAL NITROGEN DIOXIDE DELIVERED". As a result, there was some debate whether volume of concentrated NO2 delivered should be reported. Specifically, concern was raised that if this field was reported on the report form / certificate shared with customers, then there would be confusion/concern for it is blank. In the end, we agreed that reporting volume of dilute NO2 delivered is mandatory and reporting volume of liquid/concentrated volume is non-mandatory (can either be reported or left blank at the discretion of each lab).
- Summary prepared for upcoming D02.B0.07 meeting was shared with the SP.

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Status	Test Aspect	Comments
	Method	 Test method is in good standing. IL 21-01 effective December 1, allowing usage of dilute NO2 as alternative to concentrated NO2.
	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 effective December 1.
	Reference Oils	 All current reference oils are in good supply at TMC: multiyear supply of each oil Final limits set for 436 Surveillance panel replaced 438-2 with 436
	Test Availability	 Test is available with no significant queues to report. Less activity than prior semesters Available at 5 labs with 22 calibrated stands
	Severity and Precision	 In last semester (Apr 2021 – Sep 2021) precision was on target and test ran with a slight mild bias: N = 106, Pooled s = 0.1992 and Mean ∆/s = -0.37

- 0
- During the meeting it was also confirmed by Denny Gaal (Chair of B.07) that additional time (up to 10 minutes) will be available for SP to present their reports since the session was extended from one hour to two hours.
- Flow meters
 - The method describes the use of two flowmeters/rotameters: "6.10.1 Acrylic Block Airflow Meter" and "6.10.2 Airflow Meter". One of the SP members inquired if there was described way or best practices to calibrate these meters. The outcome of this discussion is below:
 - Rotameters have no means of adjustment so if out of tolerance rotameters should first be cleaned. If still out of tolerance, then rotameter may need to be discarded.
 - Also recommended to allow time for meter to acclimate/stabilize.
 - Validated/verified vs. calibrated. Performance of rotameters can be compared against a known standard to validate/verify its performance (measurements). It within the tolerance range meter can be considered calibrated; however, if not, there is no way to adjust it.
 - Tolerances of the flow meters are not included in the method, so it was recommended to use tolerances provided by manufacturer when validating performance.
 - There also the option to send rotameters for calibration. Maddie Dellinger recommended the company Applied Technical Services.
 - Will keep a placeholder at next meeting to discuss best practices for equipment calibration
- Instatherm update
 - Rachel Stone provided a short update on their reactors experiencing "burn out" after a limited number of runs. Ace Glass was able to find an electrical short in the affected reactors, but unable to determine a root cause. At this time, it appears this issue may be isolated to just small number of reactors produced earlier this year. SP will continue to monitor.
- Other topics
 - ISO 17025: Maddie Dellinger inquired if any of the SP members had or were seeking ISO 17025 on their ROBO units. Some labs reported interest, but it appears that no labs have ISO 17025 for their ROBO units.
 - J-Chem controllers: Maddie inquired if anyone had experience troubleshooting J-Chem controllers. No immediate feedback from any SP members.
- Next meeting tentatively scheduled on February 3, 2022. Date may be postponed if necessary.
- Meeting adjourned 11:17EDT

Meeting Outcome:

- 1. Surveillance panel agreed to accept editorial changes proposed by Terry Bates in ROBO Information Letter 21-01.
- 2. Surveillance panel agreed that reporting volume of liquid/concentrated NO2 delivered is non-mandatory and can therefore be included or left blank on the report forms.

-End report-

ASTM D7528: Bench Oxidation of Engine Oils by ROBO Apparatus ROBO Surveillance Panel Meeting

November 18, 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 September 30th meeting
- Current status of ROBO including statistics
- Dilute nitrogen dioxide update
 - Approve additional revisions
 - Data dictionary update
- ASTM in Anaheim
- Update on Instatherm flasks one lab has reported issues with newer flasks "burning out" after <5 runs.</p>
- Set next meeting

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

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

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

Electronic recording of ASTM meetings is prohibited.

Membership

Ace Glass	Dave Lawrence, Tom Petrocella,
Afton	Shelia Thompson, Jeff Yang, Todd Dvorak
BG Products	Madeleine Dellinger
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, Gabe Walkup, Justin Kontra
Vanderbilt Chemicals	Al Filho, Christine Katrenya
SwRI	Becky Grinfield, Joe De La Cruz, Mike Birke, Yong-Li McFarland
Valvoline	Amol Savant, Amy Ross, Bruce Tonkel
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	Alan Flamberg
Guests	

Summary of changes:

1. Ron Hiza from Vanderbilt has retired.

Motion to accept September 30, 2021 meeting minutes

PDF September 2020 utes and Presenta

	MEETING MINUTES: ROBO SURVEILLANCE PANEL	MEETING MINUTES: ROBO SURVEILLANCE PANEL	MEETING MINUTES: ROBO SURVEILLANCE PANEL MEETING MINUTES: ROBO SURVEILLANCE PANEL
 Tom Schofield to update Tom Schofield to make re 438/438-2 inventory so th Tom Schofield to incorpo Justin Mills to tentatively :) C har Trate accepted changes to Section 13 and Appendix X7 into information letter. Immits for reference of 436. Terrence of 434 and 438 obsolvets. Tom also to send test keys to labs with remaining ray may effert dispose of or use informally. Test the revised reference of tables into the TLMS schedule the next ROBO SP meeting for November 18, 2021.	Summary: Summary: Summary: Summary: Summary: Summary: Summary: Summary: Summary: Summary: Su	 1.1.1 If the flow rate near outs contribute the range of 185.2.2 million and participants in the intervention is used, then the deviations are deemed not significant and the time-averaged value on the estimated as either 1150 or whichever value within this range the period restinct the same and the intervention of t
Membership and Attendance	Dave Lawrence. *Tom Petrocella	(July 22) o Completed - Justin Mills to tentatively schedule the next ROBO SP meeting for September 23 30, 2021.	Table and text to be removed from method and relocated to TMC website
Ace Glass Afton	Dave Lawrence, *Tom Petrocella Shelia Thompson, Jeff Yang, *Todd Dvorak	 Current status of ROBO Current semester (4/1/2021 through 9/30/21) is running mild (-0.35), but precision in line with target (0.1999). 	Table and text to be removed from method and relocated to TMC website Image: Control in the state in the sta
ASTM TMC	*Tom Schofield	 TMC website will move to following URL on October 1: <u>www.astmtmc.org</u> 	
BG Products	*Madeleine Dellinger	 Method revision Motion to accept the following changes to Section 13 and Appendix X7 was accepted. (Motion made by Gabe 	1 m 1 m m m m m m m m m m m m m m m m m
Chevron Oronite	*Bobert Stockwell	Walkup, Seconded by Alan Flamberg. Verbal vote results: All affirmative, no negatives, no abstains.) Implementation date will be December 1, 2021.	1 1
ExxonMobil	Dennis Gaal		
Infineum	Andy Richie. Sapna Eticala	13.3.6. The option used to add nitrogen dioxide. Liquid nitrogen dioxide or dilute nitrogen dioxide. 13.3.6.1 If the dilute nitrogen dioxide option was used, also report calculate and report the total amount of nitrogen	and any and a set of the set of t
Intertek	*Joe Franklin, *Matt Schlaff, Rachel Stone	dioxide delivered to the reactor. See Appendix X.7 for an example calculate and report the total and out of motigen	
Lubrizol	*Aimee Shinhearl. Jerimiah Westbrook	13.3.6.1.1 The time-averaged subsurface dilute nitrogen dioxide in air flow rate to the nearest 1-mi/min.	transportions 2020 2016/or min and providing two indexed at them of Palatia, then indexed at them and to be estimated and in ordin
PetroChina	Li Shaohui , Sun Ruihua, Peng Wang, Xiaogang Li, Xu Li	13.3.6.1.2 Report the actual time the subsurface air was changed from dilute nitrogen dioxide in air to dry-air. Report to the nearest 0.1 hour.	and the second s
Evonik Oil Additives	*Justin Mills. *Justin Kontra. *Gabriel Walkup	13.3.6.1.4 Report the concentration of nitrogen dioxide in air as certified by the supplies-if more than one nitrogen	Incompression 31.0 The for an all addult to provide the adjust of plan. Mana Figure was adding for addition and the second state and th
Vanderbilt Chemicals	Al Filho. Ron Hiza	diaxide cylinder was used during the ROBO test; report a time-averaged concentration.	Meeting adjourned 11:17EDT
SwRI	*Becky Grinfield, Joe De La Cruz, *Mike Birke, *Young-Li McFarland	13.3.6.1.5 Calculate and report the total amount of nitrogen disside delivered to the reactor. See Appendix X.7 for an example calculation.	Update on Data Dictionary / Report Forms Changes made to the dat dictionary are in Beta phase. Tom will issue memo indicated changes. Rollout
Valvoline	Amol Savant, *Amy Ross, *Bruce Tonkel		expected November 1 with implementation date to be December 1, 2021. Changes include: Meeting Outcome: Meeting Outcome:
Koehler Instruments	Rai Shah. Vincent Colantuini		Addition of "Total Nitrogen Dioxide Delivered" Surveillance Panel accepted to changes to Section 13 and Appendix X7 of D7528.
Tannas/Savant	*Greg Miller, Ted Selby		Addition of "Nitrogen Dioxide Delivery Option" Surveillance Panel accepted final Imilia for reference oil 33 Surveillance Panel accepted final areadore to make reference oil 43 and 432- obsolete.
General Interest	*Alan Flamberg		 Surveillance Panel accepted a motion to include Option #2 as the final limits for 436 (Motion made by Alan Surveillance Panel accepted changes to the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables in the LTMS – will now include tables for the reference oil tables for tables for the reference oil tables for the reference oil t
Guests	-		Flamberg, Seconded by Tom Schofield. Verbal vote results: All affirmative, no negatives, no abstains.) historic, and all reference oils.
	* Denotes attendance		NCAT Normality Nor
ASTM D7628	ROBO SP Meeting September 30, 2021	ASTM D7528 ROBO SP Meeting September 30, 2021	ASTM 07528 ROBO SP Meeting September 30, 2021 ASTM 07528 ROBO SP Meeting Sep

Actions from September 30th meeting

- Tom Schofield to incorporate accepted changes to Section 13 and Appendix X7 into information letter.
- Tom Schofield to update limits for reference oil 436.
- Tom Schofield to make reference oil 438 and 438-2 obsolete. Tom also to send test keys to labs with remaining 438/438-2 inventory so they may either dispose of or use internally.
- Tom Schofield to incorporate the revised reference oil tables into the LTMS
- Justin Mills to tentatively schedule the next ROBO SP meeting for November 18, 2021.

Current status of ROBO

ROBO Industry Statistics

Period	N-size	Degrees of Freedom	Pooled s	Mean Δ/s
Current Targets	80	77	0.1551	
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	116	110	0.1992	-0.37
10/1/21 through 3/31/22	22	19	0.1885	-0.12

Source https://www.astmtmc.org/ftp/refdata/bench/robo/data/statistics.txt (Nov15-2021)

CUSUM severity analysis



ROBO TEST INDUSTRY OPERATIONALLY VALID DATA

(Ŧmc

Source: https://www.astmtmc.org/ftp/refdata/bench/robo/plots/mrv%20INDUSTRY.pdf (Nov15-2021)

Dilute NO₂

ROBO Information Letter Issued November 3 → Effective December 1

A Program of AST	'M International	203 Armstrong Drive, Freeport, PA 16229, USA	www.astmtmc.org 412-365-1000
			ROBO Information Letter 21-01
			Sequence No. 3 November 3, 2021
issuea în oraei	r to achieve su	ich consensus.	
issuea în oraei	r to achteve su	ich consensus.	
	r to active su ROBO Maili		
TO:	ROBO Maili		
TO: SUBJECT:	ROBO Maili Revisions to The ROBO S are attached. T	ing List ROBO Test Method D7528 Surveillance Panel has approved revisio he changes add an option to use dilute r	
TO: SUBJECT: The revisions a	ROBO Maili Revisions to The ROBO S are attached. The ecember 1, 202	ing List ROBO Test Method D7528 Surveillance Panel has approved revision he changes add an option to use dilute n 21.	nitrogen dioxide in air. These changes
TO: SUBJECT: The revisions a are effective D	ROBO Maili Revisions to The ROBO S are attached. The ecember 1, 202	ing List ROBO Test Method D7528 Surveillance Panel has approved revision he changes add an option to use dilute n 21.	nitrogen dioxide in air. These changes M Fauber

ROBO IL 21-01

13.Report

••••

13.3.6. The option used to add nitrogen dioxide. Liquid nitrogen dioxide or dilute nitrogen dioxide.

13.3.6.1 If the dilute nitrogen dioxide option was used, calculate and report the total amount of nitrogen dioxide delivered to the reactor. See Appendix X.7 for an example calculation.

Data dictionary:

OLD	VERSION	1 C	8	0 YYYYMMDD	ROBO VERSION 20180323
NEW	VERSION	1 C	8	0 YYYYMMDD	ROBO VERSION 20210819 Beta
ADD	NO2DEL	2 N	5	1 ml	TOTAL NITROGEN DIOXIDE DELIVERED
ADD	N020PT	2 C	1	0	NITROGEN DIOXIDE DELIVERY OPTION - LIQUID/DILUTE [L,D]

Seeking consensus from SP - Is there a requirement to report NO2 volume for both concentrated and dilute NO2? Or just for dilute NO2?

No plans to track or use NO2 volume from concentrated NO2.

ASTM in Anaheim

ASTM D7528: ROBO Summary for D02.B0.07 – December 6, 2021

Status	Test Aspect	Comments
	Method	 Test method is in good standing. IL 21-01 effective December 1, allowing usage of dilute NO2 as alternative to concentrated NO2.
	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 effective December 1.
	Reference Oils	 All current reference oils are in good supply at TMC: multiyear supply of each oil Final limits set for 436 Surveillance panel replaced 438-2 with 436
	Test Availability	 Test is available with no significant queues to report. Less activity than prior semesters Available at 5 labs with 22 calibrated stands
	Severity and Precision	In last semester (Apr 2021 – Sep 2021) precision was on target and test ran with a slight mild bias: • N = 106, Pooled s = 0.1992 and Mean Δ /s = -0.37

Any Additional Topics?

ROBO test makes use of two flow meters

- 6.10.1 Acrylic Block Airflow Meter (King Instrument Co., 7520 Series, Order number 2C-17), ⁷ having a scale of 0.4 to 4 Standard Cubic Feet per Minute (SCFM), with $1/_4$ in. NPT threaded female pipe end. It is used for measuring air flow in 10.3.2. The machined fitting for the top of the flow meter shall accommodate the vacuum line from the condenser to the reactor with a $3/_8$ in. inside diameter or larger. The machined fitting for the bottom of the flow meter shall accommodate the $1/_4$ in. vacuum control valve.
- 6.10.2 Airflow Meter, with a scale calibrated in mL/min for measuring subsurface airflow of 185 mL/min in 10.3.1 and 10.3.2. Two air flow meters may be used in the dilute nitrogen dioxide configuration depending on the location of the switching valve.
 6.10.2.1 A digital mass flow controller may also be used to measure and control the flow rate. This type of flow controller is recommended, but not required, for the dilute nitrogen dioxide in air option.

Any guidance from SP on calibration?

- In general, common practice is to compare (calibrate) against a known value.
- Flow meters
 - Calibrate against a controlled meters at various setpoints (0, 25, 50, 75, 100% of range) allowing enough time for stabilization.
 - If out of tolerance, then will reset according to factory procedure and recalibrate
- Rotameters
 - Compare against known value after allowing enough to time to acclimate/stabilize.
 - There is no way to adjust, so if out of tolerance the first measure is to clean it.

Next Meeting

- No immediate need for next SP meeting.
 - Suggest either late January or February

Janua	ary 2022	2		^	\sim	
Su	Мо	Tu	We	Th	Fr	Sa
26	27	28	29	30	31	1
2	3	4	5	6	7	8
9	10	11	12	13	14	15
16	17	18	19	20	21	22
23	24	25	26	27	28	29
30	31	1	2	3	4	5
Febru	ary 202	2			^	\sim
Su	Mo	Tu	We	Th	Fr	Sa
30	31	1	2	3	4	5
6	7	8	9	10	11	12
13	14	15	16	17	18	19
20	21	22	23	24	25	26
27	28					5