### **MEETING MINUTES: ROBO SURVEILLANCE PANEL**

Meeting: ROBO SP Meeting

Date: September 30, 2021

Location: MS Teams (virtual)

Minutes by: Justin Mills - SP Chair

#### Actions:

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

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, Ron Hiza
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	

#### Membership and Attendance:

\* Denotes attendance

#### Summary:

- Meeting convened at 10:02EDT on September 30, 2021
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
  - Bruce Tonkel of Valvoline added
- Meeting minutes from July 22, 2021 SP meeting were accepted
- Actions from the July 22<sup>nd</sup> meeting were reviewed.
  - Completed Justin Mills to calculate and propose final limits for reference oil 436.
  - Completed 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.
  - Incomplete Tom Schofield to draft an information letter regarding the acceptance of the alternative ROBO method. To be effective 3 months from today (July 22)
  - Completed Tom Schofield to add the following items to the data dictionary: 1) Nitrogen dioxide option: (L or D) 2) Total NO2 delivered: (numeric, one decimal, 2 digits, units=mL). To be effective 3 months from today (July 22)
  - o Completed Justin Mills to tentatively schedule the next ROBO SP meeting for September 23 30, 2021.
- 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).
- TMC website will move to following URL on October 1: <u>www.astmtmc.org</u>
- Method revision
  - Motion to accept the following changes to Section 13 and Appendix X7 was accepted. (Motion made by Gabe Walkup, Seconded by Alan Flamberg. Verbal vote results: All affirmative, no negatives, no abstains.)
     Implementation date will be December 1, 2021.

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 dioxide delivered to the reactor. See Appendix X.7 for an example calculation.

13.3.6.1.1 The time-averaged subsurface dilute nitrogen dioxide in air flow rate to the nearest 1 ml/min.

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.

13.3.6.1.4 Report the concentration of nitrogen dioxide in air as certified by the supplier. If more than one nitrogen dioxide cylinder was used during the ROBO test, report a time-averaged concentration.

13.3.6.1.5 Calculate and report the total amount of nitrogen dioxide delivered to the reactor. See Appendix X.7 for an example calculation.

#### MEETING MINUTES: ROBO SURVEILLANCE PANEL

X7.1 If the flow rate never varies outside the range of  $185 \pm 2$  ml/min or if a mass flow meter is used, then the deviations are deemed not significant and the time-averaged value can be estimated as either 185 or whatever value within this range the operator estimates based on the observations.

X7.2 To calculate the time-averaged flow rate, assume any changes observed in flow rate are linear between observations. If the switch from dilute nitrogen dioxide in air to dry-air is done automatically with no opportunity to observe the flow rate at that time, then calculate a flow rate by assuming that the average change in flow rate (change/hour) continues for the period between the last observation and the switching time.

X7.3 In this example, the initial flow rate is set at 185 ml/min and it decreases at 1 ml/min until the switching time at about 12 hours. One of the observation times is not exactly on the hour. The observed readings have some small variations. The switching time is not exactly at 12.0 hours. The calculations are done in a spreadsheet shown in Table X7.1 An example spreadsheet for calculating dilute nitrogen dioxide flow rate and total nitrogen dioxide delivered is available on the TMC's<sup>2</sup> website.

→ Relocate spreadsheet table (Table X7.1) and corresponding description to the TMC website (see next slide)

Table and text to be removed from method and relocated to TMC website

ROBO dilute	NO2 flow rate	e				Shaded data is input, the rest of the values are calculated. Observations are made every hour for 6 hours (in this case) and
						the 12 hour observation is either calculated or observed at 12.1 hours. The time since last reading is the time between
Time from	Flow rate	Time since last	Average	Flow x Time	Change flow	observations. Two observations are recorded at each observation time, the value at the observation time and the adjusted
start (hr)	(ml/min)	reading (hr)	flow rate	(hr ml/min)	rate/time	value at that same time. Adjustments are optional. The average flow rate is the average for the time period between two
0	185	0				observations. The Flow x Time column is the product of the time since last reading and the average flow for that time
1	184	1	184.5	184.5	-1	paried. It is used to calculate the time averaged flow rate. The change flow rate/time is the difference between the flow at an
1	185	0				period. It is used to calculate the interaveraged now rate. The change now rate time is the difference between the now at an
2.1	184	1.1	184.5	202.95	-0.9090909	observation (before any adjustment) and the flow in the previous observation (after any adjustment) divided by the time
2.1	185	0			-	between observations. It is only calculated for time intervals that are not zero. It is used to calculate the flow rate at the
3	185	0.9	185	166.5	0	switching time if it not observed then.
3	185	0				-
4	183	1	184	184	-2	The Average change flow rate/time is the average in that column. This assumes the change in flow rate/time is constant and
4	103	0	1045	104 E		the reverse change for the measurement with the detail. This assumes the change in flow facture is constant and
5	104	1	104.5	104.5	-1	the variation observed is due to measuring variability, which in this case is a good assumption. If the change flow rate/time
5	193	1	1935	193.5	-1	systematically changes over time, then continued observations for the remaining time would be advised.
6	185	1	163.5	103.5	-1	
12.1	103	0				The calculated (flow rate) at switch time is the flow rate at the last observed time (before the switch time) plus average
Automatical share		(1)			0.0848485	change flow rate/time times the time between the switch time and the preceding observation. This can be used in cases where
Average chai	nge now rate/	time			-0.9646465	the formation of the media the server in a standard and the protecting observation. This can be used in cases where
Colsulated at						the now rate is not observed at the switch time. The time-averaged now rate is then the sum of the now x time values
calculated at	178 00 7/		6 1 181 996	2 1110 1769	-	divided by the sum of the time since last reading values (which should equal the switch time).
switch drife	1/0.5524		0.1 101.550	2 1110.170.	, ,	
Time-average	ed flow rate		183.15	1		If the flow rate at the switching time is observed, in this case 179 ml/min, then it does not need to be estimated and is used in
			2001201	-		the calculation. In this case, a very similar calculated result is obtained
If measured						the encounter. In this case, a very similar careful to contract to contract.
at switch						
time	179	1	6.1 182	2 1110.2	-0.9836066	The value for the true average flow rate for this example is the value calculated where the flow rate changes precisely at 1.0
						ml/min each hour, it is adjusted back to exactly 185 ml/min at each observation, and the switch occurs at 12.1 hours.
Time-average	ed flow rate		183.1529	9		
True average	flow cato (wi	ith accumptions)	193 214	c.		The flow rates will ultimately be reported to three significant figures, but more figures were used during the calculations to
inue average	Wiate (Wi	an essemptions)	165.214	-		avoid rounding errors.

#### Update on Data Dictionary / Report Forms

- Changes made to the data dictionary are in Beta phase. Tom will issue memo indicated changes. Rollout expected November 1 with implementation date to be December 1, 2021. Changes include:
  - Revision of ROBO version
  - Addition of "Total Nitrogen Dioxide Delivered"
  - Addition of "Nitrogen Dioxide Delivery Option"
- 436 Limits
  - Surveillance Panel accepted a motion to include Option #2 as the final limits for 436 (Motion made by Alan Flamberg, Seconded by Tom Schofield. Verbal vote results: All affirmative, no negatives, no abstains.)

TMC 436		n	Natural Log Transformed Mean (In)	Mean in Original Units	s.d. (In)	95% band in mPa*s, min	95% band in mPa*s, max	95% band (ln), min	95% band (In), max
For reference	With Outliers – No bias correction	39	10.2901	29,440	0.1660	21,262	40,762	9.9647	10.6155
Option #1	With outliers – Bias corrected.	39	10.3391	30,918	0.1660	22,330	42,809	10.0137	10.6645
For reference	Outliers (3) removed – No bias correction	36	10.2828	29,226	0.1290	22,697	37,632	10.03	10.5356
Option #2	Outliers (3) removed – Bias corrected	36	10.3319	30,696	0.1290	23,840	39,525	10.0791	10.5847
Current limits for TMC 436			10.3437	31,061	0.1605	22,677	42,544	10.0291	10.6583

#### MEETING MINUTES: ROBO SURVEILLANCE PANEL

#### 438 and 438-2

- Surveillance Panel accepted a motion to make reference oils 438 and 438-2 obsolete effective immediately (Motion made by Joe Franklin, Seconded by Alan Flamberg. Verbal vote results: All affirmative, no negatives, no abstains.)
- Tom will provide test keys to labs with remaining 438 and 438-2 samples. They can either dispose of them or use for internal purposes.
- LTMS reference oil table
  - Surveillance Panel accepted a motion modify the ROBO data tables in the LTMS as shown below and to move 438 to the historic reference oil table. (Motion made by Joe Franklin, Seconded by Alan Flamberg. Verbal vote results: All affirmative, no negatives, no abstains.)

Table 1 <u>Current Reference Oils</u> MRV VISCOSITY Unit of Measure 1 N(MRV)										
Unit of Measure: LN(MRV) D7528 (ROBO) Aged Oil MRV Acceptance Bands, <u>mPas</u> and ln( <u>mPas</u> )										
					95%	95%	0.597	0.58/		
		Natural Log	Mean in		band in	band in	95% Banda	95% Banda		
Oil	•	Mean (ln)	Unite	ed (In)	Minl	Max1	Min (ln)	Max (lp)		
434_1	13	10.6599	42.612	0.1672	30.706	59.136	10 3322	10.9876		
434-2	36	<sup>2</sup> 10.9284	<sup>2</sup> 55,737	0.1551	<sup>2</sup> 41.126	<sup>2</sup> 76.008	<sup>2</sup> 10.6244	<sup>2</sup> 11.2386		
434-3	22	<sup>2</sup> 10.8172	249,871	0.1389	237,987	<sup>2</sup> 65,473	<sup>2</sup> 10,5450	<sup>2</sup> 11.0894		
435	15	11.4895	97,685	0.2932	<sup>3</sup> 60,000	173,546	<sup>3</sup> <del>11.0021</del>	12.0642		
435-1	22	11.0416	62,420	0.20295	444570	92910	410.7048	11.4394		
436	17	210.3437	231,061	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	<sup>2</sup> 10.5404	<sup>2</sup> 37813	0.2596	<sup>2</sup> 22,734	<sup>2</sup> 62,894	<sup>2</sup> 10.0316	<sup>2</sup> 11.0492		



Natural Log Mean in band in band in band in Transformed Original mPas	0.594	059/
Transformed Original mPas mPas	059/-	
Oil a Man (la) Units ad (la) Mini Man	Bands	Bands
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	<u>10.3322</u>	10.9876

Appendix	A-53
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D7528 ROBO Test Reference Oil Targets									
		Effec	tive Dates	LN(EO	T MRV)				
Oil	Ν	From <sup>1</sup>	To <sup>2</sup>	X	s				
434-1	13	20080801	20200901***	10.6599	0.1672				
424.2	5	20170713	20180727	10.941	0.1672				
434-2	36	20180728	***	10.9284	0.1551				
434-3	13	20191101	***	10.8411	0.1342				
435	15	20080801	20110928	11.4895	0.2932				
435-1	22	20100408	***	11.0416	0.20295				
436	17	20210429	***	10.3437	0.1605				
438	14	20080801	***	10.2676	0.2037				
420.2	10	20190221	20191031	10.4421	0.2322				
438-2	19	20191101	***	10.5404	0.2596				

- ••
- LTMS will be updated in approximately 3 weeks
- Instatherm update
  - Intertek continues to work with ACE Glass to determine root cause of reactor "burn out" issue. Continue to monitor
- Next meeting tentatively scheduled on November 18, 2021. Date may be postponed if necessary.
- Meeting adjourned 11:17EDT

#### Meeting Outcome:

- 1. Surveillance Panel accepted to changes to Section 13 and Appendix X7 of D7528.
- 2. Surveillance Panel accepted final limits for reference oil 436
- 3. Surveillance Panel agreed to make reference oils 438 and 438-2 obsolete.
- 4. Surveillance Panel accepted changes to the reference oil tables in the LTMS will now include tables for current, historic, and all reference oils.

-End report-

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

September 30, 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 July 22<sup>nd</sup> meeting
- Current status of ROBO including statistics
- Dilute nitrogen dioxide
  - Approve additional revisions
  - Data dictionary update
- Reference oil list "clean-up" in LTMS
- Reference oil 436 limits
- Update on Instatherm flasks one lab has reported issues with newer flasks "burning out" after <5 runs.</p>
- Set next meeting
- 2 ROBO SP Meeting September 2021

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Electronic recording of ASTM meetings is prohibited.

# Membership

Ace Glass	Dave Lawrence, Tom Petrocella,
Afton	Shelia Thompson, Jeff Yang, <b>Todd Dvorak</b>
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, Ron Hiza
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. Bruce Tonkel of Valvoline added

### Motion to accept July 22, 2021 meeting minutes



MEETING MINUTES: ROBO SURVEILLANCE PANEL

1. There is enough data to set the final limits for Reference oil 436. Limit setting will be addressed at our next meeting.

3. The Surveillance Panel accepted a motion to add the following two items to the data dictionary: 1) Nitrogen dioxide

option; (L or D) 2) Total NO2 delivered; (numeric, one decimal, 2 digits, units=mL). The effective date will be 3

-End report-

ROBO SP Meeting

4. Two labs have reported similar "burn out" issues with the Instatherm reaction flask. Ace Glass is investigating.

Dilute NO2 alternative method has been approved by the Surveillance Panel. The effective date will be 3 months from

Next meeting tentatively scheduled on September 23, 2021. Date may be postponed if necessary.

take a lot of effort to demonstrate equivalence.

months from today, coinciding with the effective date of the revised method.

Meeting adjourned 11:52EDT

Meeting Outcome:

today

Approval of alternative reaction flasks was also briefly discussed; however, it was acknowledged that it may

#### MEETING MINUTES: ROBO SURVEILLANCE PANEL

#### Meeting: ROBO SP Meeting

Date: July 22, 2021

- Location: MS Teams (virtual)
- Minutes by: Justin Mills SP Chair

#### Actions:

- 1. Justin Mills to calculate and propose final limits for reference oil 436.
- 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.
- Tom Schofield to draft an information letter regarding the acceptance of the alternative ROBO method. To be effective 3 months from today (July 22)
- Tom Schofield to add the following items to the data dictionary: 1) Nitrogen dioxide option: (L or D) 2) Total NO2 delivered: (numeric, one decimal, 2 digits, units=mL). To be effective 3 months from today (July 22)
- 5. Justin Mills to tentatively schedule the next ROBO SP meeting for September 23, 2021.

#### Membership and Attendance:

Ace Glass	Dave Lawrence, Tom Petrocella	
Afton	*Shelia Thompson, Jeff Yang, *Todd Dvorak	
ASTM TMC	Tom Schofield	
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Intertek	*Joe Franklin, Matt Schlaff, *Rachel Stone	
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General Interest	*Alan Flamberg	
Guests		
		* Denotes attendance

#### MEETING MINUTES: ROBO SURVEILLANCE PANEL

#### Summary:

- Meeting convened at 10:02EDT on July 22, 2021
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
  - Madeleine Dellinger of BG Products added
     Steve Lazzara of Valvoline to retire and to be removed from the SP Many thanks for his contributions to the SP over the years!
- Meeting minutes from June 24, 2021 SP meeting were accepted (motion made by Joe Franklin and seconded by Alan Flamberg)
- Actions from the June 24<sup>th</sup> meeting were reviewed.
  - Completed Justin/Alan to incorporate feedback received on draft revision to ASTM D7528 recirculated among the Surveillance Panel.
  - To be completed by next meeting 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. (Tom Schofield on vacation. To be revisited next meeting.)
     Completed Justin Mills to tentatively schedule the next ROBO SP meeting for July 22, 2021.
- Complete Guard many to containing to containing to containing to complete and the next Nobio Shi meeting for only 22, 2021.
- Current semester (4/1/2021 through 9/30/21) is running mild (-0.39), but precision in line with target (0.1965).
   TMC reference oils
- As of July 19, 2021, there are 24 acceptable datapoints for 436. SP agreed with the recommendation to set
- final limits at our next meeting.
- Dilute Nitrogen Dioxide
  - SP reviewed the latest (and final) draft of the D7528 revision. Alan Flamberg walked the SP through each of the changes that had been made since the prior draft – most of these changes were editorial in nature and the overall "spirit" of the draft remained unchanged.
    - Two numbering errors were identified and corrected during this meeting in the Summary of Changes we corrected the following "addition of Appendix X5 and Appendix A6" to "addition of Appendix X6 and Appendix X7".
  - Following the walkthrough of the changes, Alan Flamberg made a motion "to accept the (ASTM D7528) method as edited during today's meeting and put it into an information letter. Changes to be effective
    - 3 months from today tentatively"
    - The motion was seconded by Joe Franklin.
    - A verbal vote was carried out with all participating parties voting affirmative. There were no negatives or abstains.
    - The motion carried and has been accepted.
  - Updating the data dictionary to reflect the addition of dilute NO2 as an alternative was also reviewed. After

some discussion, general alignment was reached within the SP-A motion was made by Joe Franklin to "Update the data dictionary to include the following: 1) Nitrogen dioxide option: (L or D) 2) Total NO2 delivered: (numeric, one decimal, 2 digits, units=mL) → example 2.0. To be effective in 3 months from

- today to coincide with the effective date of the revised method."
- The motion was seconded by Alan Flamberg
   A verbal vote was carried out with all participating parties voting affirmative. There were no negatives
- or abstains.
- The motion carried and has been accepted.
- Instatherm reactors
  - At our last SP meeting (June 24), Intertek reported issues with newer flasks "burning out" after ~5 runs. All
    affected reactors were purchased by Intertek in 2021.
    - Madeleine of BG Products reported a similar issue with one reactor purchased in June 2020. She also reported the reactor was severe and had a lot of deposits before it burned out. ACE Glass replaced it at no cost.
  - Rachel Stone of Intertek is scheduled to have a meeting with ACE Glass in the coming weeks to discuss the root cause. Rachel agreed to share any relevant information at our next SP meeting.
- ASTM D7528 ROBO SP Meeting July 22, 2021

ASTM D7528

July 22, 2021

# Actions from July 22<sup>nd</sup> meeting

- 1) Justin Mills to calculate and propose final limits for reference oil 436.
- 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) Tom Schofield to draft an information letter regarding the acceptance of the alternative ROBO method. To be effective 3 months from today (July 22)
  - 7 4) Tom Schofield to add the following items to the data dictionary: 1) Nitrogen dioxide option: (L or D)
     2) Total NO2 delivered: (numeric, one decimal, 2 digits, units=mL). To be effective 3 months from today (July 22)
- $\bigcirc$
- 5) Justin Mills to tentatively schedule the next ROBO SP meeting for September 23 30, 2021.

# **Current status of ROBO**

# **ROBO Industry Statistics**

Period	N-size	Degrees of Freedom	Pooled s	Mean ∆/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	107	101	0.1999	-0.35

Precision has improved; however, bias is has shifted mild

Source: http://www.astmtmc.cmu.edu/ftp/refdata/bench/robo/data/statistics.txt (Sept22-2021)

8 ROBO SP Meeting - September 2021

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Precision has improved; however, bias is has shifted mild

Source: http://www.astmtmc.cmu.edu/ftp/refdata/bench/robo/data/statistics.txt (Sept22-2021)

9 ROBO SP Meeting - September 2021

### **CUSUM severity analysis**



ROBO TEST INDUSTRY OPERATIONALLY VALID DATA

Source: http://www.astmtmc.cmu.edu/ftp/refdata/bench/robo/plots/mrv%20INDUSTRY.pdf (Sept22-2021)

Standard Deviation Units



# Dilute NO<sub>2</sub>

## Method revision – 13. Report Justification $\rightarrow$ align with reported values on report forms

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 dioxide delivered to the reactor. See Appendix X.7 for an example calculation.

13.3.6.1.1 The time-averaged subsurface dilute nitrogen dioxide in air flow rate to the nearest 1 ml/min.

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.

13.3.6.1.4 Report the concentration of nitrogen dioxide in air as certified by the supplier. If more than one nitrogen dioxide cylinder was used during the ROBO test, report a time-averaged concentration.

13.3.6.1.5 Calculate and report the total amount of nitrogen dioxide delivered to the reactor. See Appendix X.7 for an example calculation.

X7.1 If the flow rate never varies outside the range of  $185 \pm 2$  ml/min or if a mass flow meter is used, then the deviations are deemed not significant and the time-averaged value can be estimated as either 185 or whatever value within this range the operator estimates based on the observations.

X7.2 To calculate the time-averaged flow rate, assume any changes observed in flow rate are linear between observations. If the switch from dilute nitrogen dioxide in air to dry-air is done automatically with no opportunity to observe the flow rate at that time, then calculate a flow rate by assuming that the average change in flow rate (change/hour) continues for the period between the last observation and the switching time.

X7.3 In this example, the initial flow rate is set at 185 ml/min and it decreases at 1 ml/min until the switching time at about 12 hours. One of the observation times is not exactly on the hour. The observed readings have some small variations. The switching time is not exactly at 12.0 hours. The calculations are done in a spreadsheet shown in Table X7.1 An example spreadsheet for calculating dilute nitrogen dioxide flow rate and total nitrogen dioxide delivered is available on the TMC's<sup>2</sup> website.

 $\rightarrow$  Relocate spreadsheet table (Table X7.1) and corresponding description to the TMC website (see next slide)

## Table and text to be removed from method and relocated to TMC website

#### ROBO dilute NO2 flow rate

Time from	Flow rate	Time since last		Average	Flow x Time	Change flow
start (hr)	(ml/min)	reading (hr)		flow rate	(hr ml/min)	rate/time
0	185	0				
1	184	1		184.5	184.5	-1
1	185	0				
2.1	184	1.1		184.5	202.95	-0.9090909
2.1	185	0				
3	185	0.9		185	166.5	0
3	185	0				
4	183	1		184	184	-2
4	185	0				
5	184	1		184.5	184.5	-1
5	184	0				
6	183	1		183.5	183.5	-1
6	185	0				
12.1						
Average chan	ge flow rate/	time				-0.9848485
Calculated at						
switch time	178.9924		6.1	181.9962	1110.1769	
Time-average	d flow rate			183.151		
If measured						
at switch	170		<b>C</b> 1	102	1110.2	0.0000000
time	1/9		6.1	182	1110.2	-0.9836066
Time-average	d flow rate			183.1529	ı.	
True average	flow rate (wi	th assumptions)		183.2145	i	

Shaded data is input, the rest of the values are calculated. Observations are made every hour for 6 hours (in this case) and the 12 hour observation is either calculated or observed at 12.1 hours. The time since last reading is the time between observations. Two observations are recorded at each observation time, the value at the observation time and the adjusted value at that same time. Adjustments are optional. The average flow rate is the average for the time period between two observations. The Flow x Time column is the product of the time since last reading and the average flow for that time period. It is used to calculate the time-averaged flow rate. The change flow rate/time is the difference between the flow at an observation (before any adjustment) and the flow in the previous observation (after any adjustment) divided by the time between observations. It is only calculated for time intervals that are not zero. It is used to calculate the flow rate at the switching time if it not observed then.

The Average change flow rate/time is the average in that column. This assumes the change in flow rate/time is constant and the variation observed is due to measuring variability, which in this case is a good assumption. If the change flow rate/time systematically changes over time, then continued observations for the remaining time would be advised.

The calculated (flow rate) at switch time is the flow rate at the last observed time (before the switch time) plus average change flow rate/time times the time between the switch time and the preceding observation. This can be used in cases where the flow rate is not observed at the switch time. The time-averaged flow rate is then the sum of the flow x time values divided by the sum of the time since last reading values (which should equal the switch time).

If the flow rate at the switching time is observed, in this case 179 ml/min, then it does not need to be estimated and is used in the calculation. In this case, a very similar calculated result is obtained.

The value for the true average flow rate for this example is the value calculated where the flow rate changes precisely at 1.0 ml/min each hour, it is adjusted back to exactly 185 ml/min at each observation, and the switch occurs at 12.1 hours.

The flow rates will ultimately be reported to three significant figures, but more figures were used during the calculations to avoid rounding errors.

# Beta for data dictionary

10	1 ROBO	VERSION	8	0 C	YYYYMMDD	ROBO VERSION 20210819 Beta
20	1 ROBO	TSTSPON1	40	0 C		CONDUCTED FOR, FIRST LINE
30	1 ROBO	TSTSPON2	40	0 C		CONDUCTED FOR, SECOND LINE
40	1 ROBO	LABVALID	1	0 C	V or I	TEST LAB VALIDATION
50	1 ROBO	TSTOIL	2	0 C	NR or RO	OIL TEST TYPE
60	1 ROBO	INSTRUID	20	0 C		INSTRUMENT ID
70	1 ROBO	RUNNUM	10	0 C		TEST RUN NUMBER
80	1 ROBO	DTCOMP	8	0 C	YYYYMMDD	DATE COMPLETED
90	1 ROBO	EOTTIME	5	0 C	HH:MM	TIME COMPLETED
100	1 ROBO	OILCODE	45	0 C		OIL CODE
110	1 ROBO	ALTCODE1	15	0 C		ALTERNATE OIL CODE 1
120	1 ROBO	ALTCODE2	15	0 C		ALTERNATE OIL CODE 2
130	1 ROBO	ALTCODE3	15	0 C		ALTERNATE OIL CODE 3
140	1 ROBO	OPVALID	8	0 C	HAS or HAS NOT	OPERATIONAL VALIDITY - HAS/HAS NOT
150	1 ROBO	SUBLAB	40	0 C		SUBMITTED BY: TESTING LABORATORY
160	1 ROBO	SUBSIGIM	70	0 C		SUBMITTED BY: SIGNATURE IMAGE
170	1 ROBO	SUBNAME	40	0 C		SUBMITTED BY: SIGNATURE TYPED NAME
180	1 ROBO	SUBTITLE	40	0 C		SUBMITTED BY: TITLE
190	2 ROBO	LABOCODE	20	0 C		LABORATORY INTERNAL OIL CODE
200	2 ROBO	LAB	2	0 C		TESTING LABORATORY
210	2 ROBO	IND	15	0 C		TMC OIL CODE
220	2 ROBO	RNLSTCAL	10	0 C		RUN NUMBER OF LAST TMC CALIBRATION
230	2 ROBO	DTLSTCAL	8	0 C	YYYYMMDD	DATE OF LAST TMC CALIBRATION
240	2 ROBO	DTCALEXP	8	0 C	YYYYMMDD	TMC CALIBRATION EXPIRATION DATE
250	2 ROBO	METHVER	10	0 C		TEST METHOD - VERSION
260	2 ROBO	NO2OPT	1	0 C		NITROGEN DIOXIDE DELIVERY OPTION - LIQUID/DILUTE [L,D]
270	2 ROBO	NO2DEL	5	1 N	ml	TOTAL NITROGEN DIOXIDE DELIVERED
280	2 ROBO	VACPMPID	15	0 C		VACUUM PUMP SERIAL NUMBER
290	2 ROBO	VPMPLST	15	0 C		VACUUM PUMP SERIAL NUMBER AT LAST TMC CALIBRATION
300	2 ROBO	RVID	3	0 C		REACTOR VESSEL ID
310	2 ROBO	RVIDLST	3	0 C		REACTOR VESSEL ID ATLAST TMC CALIBRATION
320	2 ROBO	RVHV	3	0 C	Volts	REACTOR VESSEL HEATER VOLTAGE
330	2 ROBO	RVHVLST	3	0 N	Volts	REACTOR VESSEL HEATER VOLTAGE AT LAST TMC CALIBRATION
340	2 ROBO	VCVTOT	6	2 N	revolutions	VACUUM CONTROL VALVE TOTAL FULL TURNS FROM OPEN TO CLOSE
350	2 ROBO	VCVATCAL	6	2 N	revolutions	VACUUM CONTROL VALVE SET POINT AT LAST TMC CALIBRATION
360	2 ROBO	VCVSET	6	2 N	revolutions	VACUUM CONTROL VALVE SET POINT FOR THIS TEST
370	2 ROBO	SAEVISC	5	0 C		SAE J300 ENGINE OIL VISCOSITY CLASSIFICATION
380	2 ROBO	VOLEOTG	6	1 N	Grams	NET VOLATILES COLLECTED AT END OF TEST
390	2 ROBO	VOLEOT	5	0 N	mass %	VOLATILES AT END OF TEST
400	2 ROBO	VPCSOT	6	1 N	kPa	VACUUM PRESSURE CHECK ON CLOSED SYSTEM AT START OF TEST
410	2 ROBO	VPCEOT	6	1 N	kPa	VACUUM PRESSURE CHECK ON CLOSED SYSTEM AT END OF TEST
420	2 ROBO	KV40NEW	8	2 N	mm2/s	NEW OIL D445 KINEMATIC VISCOSITY @ 40 C
430	2 ROBO	KV40EOT	8	2 N	mm2/s	AGED OIL D445 KINEMATIC VISCOSITY @ 40 C
440	2 ROBO	KV40PVIS	7	1 N	%	PERCENT INCREASE KINEMATIC VISCOSITY @ 40 C AFTER AGING
450	2 ROBO	CCSTEMP	3	0 N	deg C	D5293 COLD CRANK SIMULATOR TEST TEMPERATURE
460	2 ROBO	CCSVEOT	8	0 A	mPa-s	AGED OIL D5293 COLD CRANK SIMULATOR APPARENT VISCOSITY [>, TVTM]
470	2 ROBO	MRVTEMP	3	0 N	deg C	D4684 MRV TEST TEMPERATURE
480	2 ROBO	MRVVEOT	8	0 A	mPa-s	AGED OIL D4684 MRV APPARENT VISCOSITY [<,>]
490	2 ROBO	MRVYSEOT	4	0 A	Pa	AGED OIL D4684 YIELD STRESS [<,>]
500	3 ROBO	TOTCOM	2	0 Z		TOTAL LINES OF COMMENTS & OUTLIERS
510	3 ROBO	OCOMRxxx	70	0 C		OTHER DOWNTIME COMMENT XXX
L						

### Summary of changes:

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	NO20PT	2 C	1	0	NITROGEN DIOXIDE DELIVERY OPTION - LIQUID/DILUTE [L,D]

# LTMS Update

## LTMS update



At June 24<sup>th</sup> SP meeting it was recommended that reference oil table in LTMS to be "cleaned-up" with only "active" reference oils in the ROBO section and "obsolete" oils will be moved to Appendix A of the document.

	Current Reference Oils MRV VISCOSITY Unit of Measure: LN(MRV)												
	D7528 (ROBO) Aged Oil MRV Acceptance Bands, mPas and ln(mPas)												
					95%	95%							
		Natural Log	Mean in		band in	band in	95%	95%					
		Transformed	Original		mPas	mPas	Bands	Bands					
Oil	n	Mean (ln)	Units	<u>s.d.</u> (ln)	Min <sup>1</sup>	Max <sup>1</sup>	Min (ln)	Max (ln)					
4 <del>34-1</del>	13	10.6599	4 <del>2,612</del>	0.1672	<del>30,706</del>	<del>59,136</del>	10.3322	10.9876					
434-2	36	<sup>2</sup> 10.9284	<sup>2</sup> 55,737	0.1551	<sup>2</sup> 41,126	<sup>2</sup> 76,008	<sup>2</sup> 10.6244	<sup>2</sup> 11.2386					
434-3	22	<sup>2</sup> 10.8172	<sup>2</sup> 49,871	0.1389	<sup>2</sup> 37,987	<sup>2</sup> 65,473	<sup>2</sup> 10.5450	<sup>2</sup> 11.0894					
435	15	11.4895	<del>97,685</del>	0.2932	<sup>3</sup> 60,000	173,546	<sup>3</sup> 11.0021	12.0642					
435-1	22	11.0416	62,420	0.20295	444570	92910	410.7048	11.4394					
436	17	<sup>2</sup> 10.3437	<sup>2</sup> 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	<sup>2</sup> 10.5404	<sup>2</sup> 37813	0.2596	<sup>2</sup> 22,734	<sup>2</sup> 62,894	<sup>2</sup> 10.0316	<sup>2</sup> 11.0492					

Table 1

*Table 2
Historic Reference Oils (information only)
MRV VISCOSITY
Unit of Measure: LN(MRV)

	D7528 (ROBO) Aged Oil MRV Acceptance Bands, mPas and ln(mPas)											
<u>Oil</u> <u>434-1</u> <u>435</u>	<u>n</u> <u>13</u> <u>15</u>	<u>Natural Log</u> <u>Transformed</u> <u>Mean (ln)</u> <u>10.6599</u> <u>11.4895</u>	<u>Mean in</u> <u>Original</u> <u>Units</u> <u>42,612</u> 97,685	<u>s.d. (ln)</u> 0.1672 0.2932	95% band in mPas Min <sup>1</sup> 30,706 <sup>3</sup> 60,000	<u>95%</u> band in mPas <u>Max<sup>1</sup></u> 59,136 173,546	<u>95%</u> <u>Bands</u> <u>Min (ln)</u> <u>10.3322</u> <sup>3</sup> 11.0021	<u>95%</u> <u>Bands</u> <u>Max (ln)</u> <u>10.9876</u> 12.0642				

Appendix A-53

D7528 ROBO Test Reference Oil Targets									
		Effec	tive Dates	LN(EOT MRV)					
Oil	Ν	From <sup>1</sup>	To <sup>2</sup>	$\overline{\mathbf{X}}$	s				
434-1	13	20080801	20200901***	10.6599	0.1672				
424.2	5	20170713	20180727	10.941	0.1672				
434-2	36	20180728	***	10.9284	0.1551				
434-3	13	20191101	***	10.8411	0.1342				
435	15	20080801	20110928	11.4895	0.2932				
435-1	22	20100408	***	11.0416	0.20295				
436	17	20210429	***	10.3437	0.1605				
438	14	20080801	***	10.2676	0.2037				
128.2	10	20190221	20191031	10.4421	0.2322				
400-2	19	20191101	***	10.5404	0.2596				

# **Reference oils**

# **TMC reference oils** Current limits

	D7528 (ROBO) Aged Oil MRV Acceptance Bands, mPa's and ln(mPa's)												
		Natural Log	Mean in		95% band in	95% band in	95% Banda	95% Banda					
01		I ransformed	Unite	a = d = (1a)	mPa's Minl	mPa's	Bands	Bands Max (lm)					
On	n	Mean (In)	Units	s.d. (in)	Min	Max	Min (in)	Max (In)					
434-1	13	10.6599	42,612	0.1672	30,706	59,136	10.3322	10.9876					
434-2	36	<sup>2</sup> 10.9284	<sup>2</sup> 55,737	0.1551	<sup>2</sup> 41,126	<sup>2</sup> 76,008	<sup>2</sup> 10.6244	<sup>2</sup> 11.2386					
434-3	22	<sup>2</sup> 10.8172	<sup>2</sup> 49,871	0.1389	<sup>2</sup> 37,987	<sup>2</sup> 65,473	<sup>2</sup> 10.5450	<sup>2</sup> 11.0894					
435	15	11.4895	97,685	0.2932	<sup>3</sup> 60,000	173,546	<sup>3</sup> 11.0021	12.0642					
435-1	22	11.0416	62.420	0.20295	<sup>4</sup> 44570	92910	<sup>4</sup> 10.7048	11.4394					
436	17	<sup>2</sup> 10.3437	<sup>2</sup> 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	<sup>2</sup> 10.5404	<sup>2</sup> 37813	0.2596	<sup>2</sup> 22,734	<sup>2</sup> 62,894	<sup>2</sup> 10.0316	<sup>2</sup> 11.0492					

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

<sup>2</sup> 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.

<sup>3</sup> 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.

<sup>4</sup>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.

- Set interim limits for 436 in April 2021 with 17 datapoints with intent to revisit when more data is available.
- As of September 22, there are 39 datapoints (donated and charted). Recommend we set final limits today.



TMC 436			Natural Log Transformed Mean (In)	Mean in Original Units	s.d. (ln)	95% band in mPa*s, min	95% band in mPa*s, max	95% band (ln), min	95% band (ln), max
For reference	With Outliers – No bias correction	39	10.2901	29,440	0.1660	21,262	40,762	9.9647	10.6155
Option #1	With outliers – Bias corrected.	39	10.3391	30,918	0.1660	22,330	42,809	10.0137	10.6645
For reference	Outliers (3) removed – No bias correction	36	10.2828	29,226	0.1290	22,697	37,632	10.03	10.5356
Option #2	Outliers (3) removed – Bias corrected	36	10.3319	30,696	0.1290	23,840	39,525	10.0791	10.5847
	Current limits for TMC 436	17	10.3437	31,061	0.1605	22,677	42,544	10.0291	10.6583

- TMC 436 runs were conducted over **last three semesters**. ROBO test ran mild all periods.
- From the dataset (donated runs and charted runs), 3 outliers were identified (2 high, 1 low)
- Applying correction factor has minor impact to overall range; nevertheless we should apply it if we wish to remain consistent with previous reference oil limit setting.
- Recommend we proceed with Option #2 to be consistent with practices used for other ROs(434-2, 434-3, 438-2)

# Instatherm update, if any

# **Any Additional Topics?**

### **Next Meeting**

Next meeting is tentatively scheduled for November 18

