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

Meeting: ROBO SP Meeting

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

#### **Actions:**

- 1. Matt Schlaff 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.
- 2. Justin Mills to provide link to TMC LTMS document that contains ROBO calibration requirements.
  - a. http://www.astmtmc.cmu.edu/ftp/docs/ltms/
- 3. Justin Mills to include "calibration requirements for new units" as a topic in the next SP meeting.
- 4. 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.
- 5. Justin Mills to schedule next SP meeting for Thursday, November 29th.

# **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				

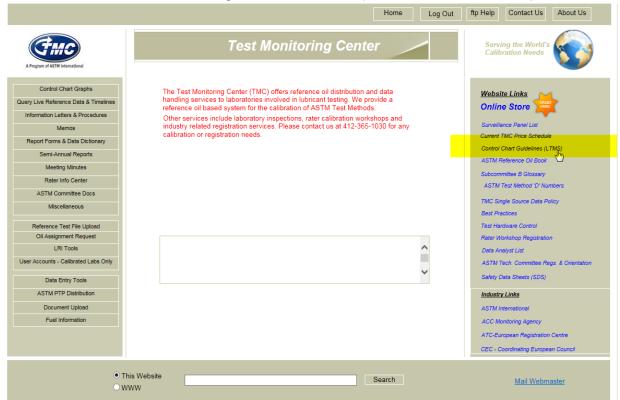
<sup>\*</sup> Denotes attendance

### **Summary:**

ASTM D 7528 ROBO SP Meeting October 25, 2018

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

- Meeting convened at 10:03EST on October 25, 2018
- Agenda accepted by SP without any modifications
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
  - Elizabeth Wagoner 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 Schlaff and seconded by Shelia Thompson
- Actions from the July 19<sup>th</sup> meeting were reviewed all actions from July 19<sup>th</sup> meeting have been completed (see attached slides for further details)
  - Leen Poot 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 queues 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 gueue at SWRI is about 1 week and at Intertek the gueue is about 2.5 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 <a href="http://www.astmtmc.cmu.edu/ftp/docs/ltms/">http://www.astmtmc.cmu.edu/ftp/docs/ltms/</a>



- Evonik has resumed work on the dilute nitrogen dioxide study/comparison.
- Stats Group update
  - Justin Mills and Tom Schofield provided the ASTM Stats Group with a ROBO timeline containing dates for critical ROBO events (see attached slides for timeline)
  - The Stats Group is currently very busy with supporting Sequence engine tests for GF-6
    - Justin Mills has expressed to the Stats Group that investigating severity factors or some alternative form of a correction factor has been on our SP's to-do list for a while but we consider it to be low priority.
- 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.

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

- Evonik has resumed work on the dilute nitrogen dioxide study/comparison
  - Evonik's configuration was shared (see attached slides)
  - Evonik compared results for standard NO₂ and dilute NO₂ ROBO runs on their internal 5W-20 QC oil, and observed comparable values for oxidation, pVis, and MRV.
  - Evonik plans to run the three TMC reference oils back-to-back using the dilute NO₂ to confirm equivalency.
    - At 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 Faile 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 ILS may be necessary.
   Matt Schlaff believes there may be less rigorous alternative for equivalency studies. Matt to contact ILS group or provide Justin with appropriate contact.
  - If implemented, labs will likely need to conduct a 2-test reference when switching from standard/concentrated nitrogen dioxide to dilute nitrogen dioxide
- o 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 NO<sub>2</sub> configuration.
- Additional topics
  - Unresolved/unclear 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 labs with multiple ROBO units should only be required to complete the standard 2 back-to-back blind reference runs. Agreed to address this topic at next SP meeting
  - Matt Schlaff 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

ASTM D 7528 ROBO SP Meeting October 25, 2018

# **ROBO Surveillance Panel Meeting**

October 25, 2018

Justin Mills

# Agenda

- Welcome, ASTM statement
- Review membership of SP
- Review and approve minutes from previous meetings (see attachment)
- Review actions from July 19<sup>th</sup> meeting
- Update on recent activity with ASTM Stats Group
- Dilute nitrogen dioxide discussion review available data
- Additional topics?
- Set next meeting

### **ASTM Antitrust and Recording Policy**

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.

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#### Membership – Updated 7/24/18

Dave Lawrence
Shelia Thompson, Jeff Yang, Todd Dvorak
Tom Schofield
Mary Dery, Bridgett Rakestraw
Man Hon Tsang, Robert Stockwell
Dennis Gaal
Andy Richie, Sapna Eticala
Joe Franklin, Matt Schlaff
Mike Faile, Aimee Shinhearl, Rick Hartman
Li Shaohui , Sun Ruihua, Peng Wang, Xiaogang Li, Xu Li
Justin Mills, Bruce Zweitzig, Joan Souchik, John Maxwell, Elizabeth Wagoner
Al Filho, Ron Hiza
Becky Grinfield, Joe De La Cruz, Mike Birke, Yong-Li McFarland
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Raj Shah, Vincent Colantuini
Greg Miiller, Ted Selby
Alan Flamberg

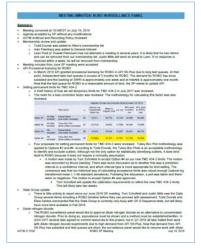
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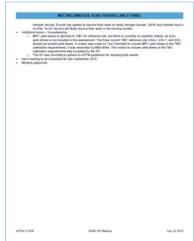
#### Summary of changes:

- Todd Dvorak added to Afton's membership list
- Alan Flamberg added to General Interest
- Leen Poot of Kuwait Petroluem removed due to inactivity

# Motion to accept July 19, 2018 meeting minutes







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# Actions from July 19th meeting

- ✓ Justin Mills to email Leen Poot of Kuwait Petroleum to see if he still wishes to be a ROBO SP member. If no response is received within a week, he will be removed from membership list.
  - Complete. Email sent to Leen, but no response was received. Leen has been removed from SP membership.
- ✓ Justin Mills to email API (Kevin Ferrick) with an update on test queue at independent labs.
  - Complete. API chose not remove ROBO from provisional licensing.
- ▼ Tom Schofield to update the TMC Calibration Requirements to reflect the new TMC 434-2 limits.
  - Complete. TMC Calibration Requirements updated with version 20180720.
- Evonik to resume work on dilute nitrogen dioxide project and present initial finding at next SP meeting.
  - Complete. Will discuss in later slides
- Tom Schofield to update TMC Calibration Requirements to include pass/fail requirements for MRV yield stress. Current TMC reference oils (434-2, 435-1, 438) to have <35 Pa yield stress.
  - Complete. TMC Calibration Requirements updated with version 20180720.
- Justin Mills to schedule next SP meeting for late September.
  - Complete. Meeting was scheduled for September but postponed to October 25<sup>th</sup> due to lack of updates.
- ROBO SP Meeting October 2018

# **TMC Calibration Requirements update**

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

D7528 (ROBO) Aged Oil MRV Acceptance Bands, mPa·s and In(mPa·s)								
		Natural Log	Mean in		95% band in	95% band in	95%	95%
		Transformed	Original		mPa·s	mPa·s	Bands	Bands
Oil	n	Mean (In)	Units	s.d. (ln)	Min <sup>2</sup>	Max <sup>2</sup>	Min (In)	Max (In)
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	160,000	173,546	<sup>1</sup> 11.0021	12.0642
435-1	22	11.0416	62,420	0.20295	344570	92910	310.7048	11.4394
438	14	10.2676	28,785	0.2037	19,308	42,912	9.8683	10.6669

 EOT yield stress (MRVYSEOT) for the reference oils, in a properly run test, should always be <35 Pa. Tests with EOT yield stress measured or reported at anything other than <35 will be declared operationally invalid.</li>

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#### **Email sent to API regarding provisional licensing**

From: Mills, Justin

**Sent:** Monday, July 23, 2018 1:33 PM

o: 'Dennis Bachelder'; 'Ferrick@API.Org'; 'doug\_anderson@americanchemistry.com'; 'JRFrederick@Valvoline.com'

Subject: RE: API SN Plus and ROBO Queue at Independent Labs

All,

I am writing this email to you as the ROBO Surveillance Panel Chair. On March 12<sup>th</sup> I sent an email (see below) informing you of the long test queue for the ROBO test at the independent test labs. As a result, provisional licensing was granted for the ROBO test. Since this time the long test queues have subsided. This is likely due to two reasons: demand for API SN Plus testing is winding down and some labs have added additional ROBO test capacity. At our ROBO Surveillance Panel meeting on July 19<sup>th</sup>, I was informed that one independent lab has a test queue of approximately one week and the other independent lab has a test queue of approximately one month.

Please use this information as you see fit. My intention is only to inform you of the test queue.

Best Regards,

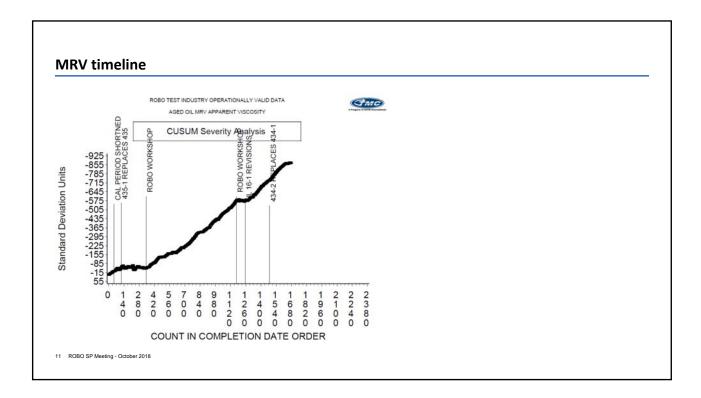
Justin Mills - Chair of ROBO SP

#### API chose to maintain provisional licensing for ROBO.

# Statistics ROBO SP Meeting - October 2018

# **ASTM Stats Group Support Update**

- ASTM Stats Group has been provided with ROBO timeline
- Stats Group is very busy with Sequence tests for GF-6
  - I expressed that investigating severity factors or some alternative form of a correction factor has been on our SP's to-do list for a while but we consider it to be low priority.
- No additional updates to report



#### Critical dates for ROBO timeline

#### Slide 1 of 2

- 2008 First RR to set preliminary targets, participating rigs retroactively given calibrated status. Not all RR data is included in TMC data base! Data was collected outside of TMC database system, only retroactive tests that calibrate rig were added to TMC database.
- 20081113 First calibration requirements issued, includes oil targets and acceptance bands (based on natural log transformed results, Ti), oils 434-1, 435 & 438.
- 20081220 DTCOMP of first non-RR calibration test (1 of 2 test calibration)
- 20081113 First calibration requirements document issued (approximate date)
- 20090115 TMC sent notice we are open for ROBO business, report packet issued (some test reported retroactively).
- 20090122 Labs informed of critical update to MRV test method (D4684), making update to new MRV method mandatory.
- 20090708 Test method update from 'Draft 5' to D7528-09
- 20091012 Calibration period shortened from '25 tests or 100 days' to '15 tests or 50 days'. Retroactively implemented, but labs ready to expire were given a 'three-test lead' to recalibrate.
- 20100408 Replaced 435 w/ 435-1 (some overlap as labs use up 435)
- 20110423 Calibration requirements update effective (v 20110420), includes new two-test requirement following two successive OC fails, and VOLEOT > = 60 % fails a test operationally. 20110816 Report packet update (fields added to track changes to vacuum pumps, reactor vessels, heater voltage, all now considered 'significant changes' that would trigger a required two-test calibration).
- 20110406 ROBO Workshop

# **Critical dates for ROBO timeline**

#### Slide 2 of 2

- 20130814 Update test method from -09 to -13
- 20151014 ROBO Workshop
- 20160311 IL 16-1 Test Method Revisions implemented, many clarifications, TMC monitoring required.
- 20170606 Update test method from -13 to -17 (includes all IL items except TMC monitoring participation, unintended omission)
- 20170728 Updated calibration requirements, replace 434-1 w/ 434-2 (overlap as labs use up 434-1). Preliminary targets based on only 5 tests!
- 20171101 Updated test method from -17 to -17a to include making TMC monitoring mandatory (already effective through IL)
- 20180525 Updated report packet effective to expand CCSVEOT to allow for '>' symbol.
- 20180720 Updated calibration requirements to reflect new limits for 434-2 (permanent limits replaced temporary limits)

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# **Dilute Nitrogen Dioxide**

# **Dilute Nitrogen Dioxide**

Slide from July 19th meeting

- Past Activity
- Dilute NO<sub>2</sub> workgroup met in September 2017
  - Agreed that an equivalent concentration would be 1.13%  $NO_2$  in air fed at 185ml/min for 12hours.
- Lab AM completed 4 runs with dilute  $NO_2$  no change in severity with dilute  $NO_2$  was observed
- Work at all labs halted because of high ROBO demand at all labs to support API SN Plus.
- Now that demand from API SN Plus has subsided, it is time to start resuming work on the dilute NO<sub>2</sub> alternative.
- Evonik is in process of re-converting a unit to use dilute NO<sub>2</sub>
- Do any other labs have plans to resume their work on dilute  $\mathrm{NO}_2$  alternative? If so, when?

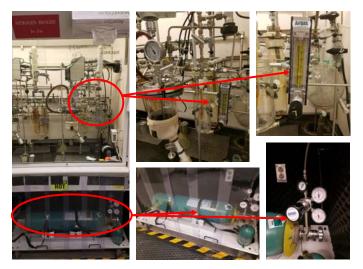
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# **Current setting vs proposed alternative**

Ingredient	ROBO Setting	Proposed dilute NO2 setting		
Test fluid	200 grams	200 grams		
Iron ferrocene	15 PPM	15 PPM		
Nitrogen dioxide	2 ml "pure" NO <sub>2</sub> fed over first 12 hours	1.13% NO <sub>2</sub> 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	0.61 Bar		
	56.6 L/min	56.6 L/min		
Temperature	170°C	170°C		
Time	40 Hours	40 Hours		

# Only difference is nitrogen dioxide and dry air.

# Evonik's setup



Air cylinder	Air Gas
Regulator	Air Gas
Flow meter	Air Gas

- Temporary configuration
- Permanent configurations will likely include:
  - Permanent cabinet for storing larger, upright NO<sub>2</sub> bottles
  - Digital flow meter/controller
  - On/Off solenoid or switch to convert from dilute NO<sub>2</sub> to dry air

#### Evonik's data on internal reference oil SAE 5W-20, API SN 450 40,000 425 Oxidation Beak Area 375 325 300 30,000 MRV, cP 20,000 10,000 9x data points for standard and dilute NO<sub>2</sub> 275 standard and dilute NO<sub>2</sub> 250 0 0.12 0.20 50 100 Oxidation Peak Height pVis, % StandardDilute StandardDilute Comparable levels of oxidation achieved. Similar pVis and MRV. 18 ROBO SP Meeting - October 2018

# **Next steps**

- Evonik will run TMC calibration oils
- Run all three test back-to-back to ensure calibration is possible
- Compare results with historic values
- What other data is necessary to approve dilute NO<sub>2</sub> as an alternative to concentrated NO<sub>2</sub>?
- When can other labs begin testing?

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# **Any Additional Topics?**

# **Next Meeting**

- Suggestions for next SP meeting?
- Suggest we have meeting before December ASTM
- November 29?, December 6?