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Standards Worldwide

COMMITTEE D02 ON PETROLEUM PRODUCTS, LIQUID FUELS, AND LUBRICANTS

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> Issued: 09.13.2019 Reply to: Patrick Lang Southwest Research Institute 6220 Culebra Rd. San Antonio. TX 78238 Phone: 210.522.2820 Email: plang@swri.org

These are the unapproved minutes of the 09.13.2019 Sequence VI Conference Call.

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The meeting was called to order at 9:00 AM Central Time by Chair Andrew Stevens.

- 1.0 Roll Call: The Attendance list is Attachment 1.
 - 1.1 There were no membership changes.
 - 1.2 The Agenda is Attachment 2.

2.0 Old Business

- 2.1 The minutes for the 03.22.2019 and 04.17.2019 calls were approved unanimously.
- 3.0 New Business
 - 3.1 Intermediate precision and repeatability estimates incorrect in procedure.
 - 3.1.1 Rich Grundza brought to the attention of the group that the intermediate precision for the 109 h and reproducibility at 16 h in table 8 of the D8114-18a appear to be reversed (attachment 3).

Discussion:

The group came to the conclusion that some of the numbers are actually incorrect as they don't match the numbers that were agreed to in March.

<u>Action</u>: An information letter will be issued to update the numbers in the precision statement of the procedure to match the values approved in March.

Rich informed the group that several labs are low on 542-3. The QA on batch 542-4 matches the previous oil. 5th run results for 542-4 looked similar to 5th run results on 542-3. This oil will now be assigned for reference testing using the current targets. This will be a rolling change.

- 3.2 What specific tests need to be run on the final drain
 - 3.2.1 Test procedure states to take oil samples but does not give any specifics on what analyticals should be run on those samples. The group needs to define what is required.

Discussion:

Todd Dvorak commented that the BOI/VGRA matrix suggested that the HTHS at 150 deg C is of interest. Ron Romano commented that he felt the used oil HTHS wouldn't tell you much and that HTHS is typically run on the new oil and is not the responsibility of the test labs.

Motion: Charlie Leverett/Adrian Alfonso

The Sequence VIE/F test procedures will be modified to require that D445 be performed on the candidate/reference oil sample taken prior to the BLA flush.

Motion passed unanimously.

- <u>Action</u>: An information letter will be issued once Rich adds the information to both VIE and VIF procedures.
 - 3.3 Alternate fuel supplier protocol.
 - 3.3.1 The recommendation from the Sequence VI Alternate Fuel Supplier Task Force was presented to the group (see attachment 4). Note that attachments 5 and 6 also support this discussion.

Discussion:

Lengthy discussion took place regarding the details of this proposal. Bob Campbell commented that this proposal doesn't address all of the items that are significant. An example was that the proposal did not address the required volume of fuel that an alternate supplier must blend for each batch. Ron Romano commented about the pilot batch method that was previously utilized with the Sequence VH fuel. Adrian commented that we see regular batch changes with the current supplier and have not quantified a difference. He further stated that if the fuel has a shift it will shift both the candidate and the baseline.

Bob Campbell stated that the 1 sigma criteria for acceptance is too large and he would have to see it lower to approve the recommendation.

Concerns were also raised regarding the allowance of multiple suppliers being used at one time. If a fuel is perceived to have a severity bias, labs may pick and choose fuels.

Action:

The group agreed that the Surveillance Panel would need to determine if there are going to be any batch code size criteria added to the requirements.

As a result of running out scheduled time for this meeting, the chairman called for adjournment. In his closing comments he stated that there are still several items that needs to be discussed further relative to the alternate fuel supplier protocol. The next meeting will be scheduled in a couple of weeks to continue this discussion.

The meeting adjourned at 10:30 AM Central Time

Attachment 1

Attendance List

After dance List

9/13/19 Conf Call

ASTM SEQUENCE VI Email/Phone Company Attend Name ELEN STA Adrian Alfonso Present **Voting Member** Jason Bowden Presont **Voting Member** Kevin Brodwater **Voting Member** Aleise Gauer Present **Voting Member** Rich Grundza Present **Voting Member** Jeff Hsu Present Voting Member Teri Kowalski **Voting Member** Dan Lanctot Present Present **Voting Member** Ben Maddock **Voting Member Brianne Pentz** Voting Member Mike Raney **Voting Member** Andy Ritchie **Voting Member** Ron Romano Present **Voting Member** Clifford Salvesen **Voting Member** Amol Savant Voting Member Andrew Stevens Present **Voting Member** Haiying Tang Voting Member Present Prasad Tumati **Voting Member** Dan Worcester Voting Member

9/13/19 Conference Call

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Attachment 2

Sequence VI Surveillance Panel Call Meeting Agenda

Sequence VI Surveillance Panel Call Meeting Agenda September 13, 2019 @ 10:00-11:30 EST

Webex Meeting Details Below Agenda

1. Roll Call (start 10:05 EST)

1.1. SP Membership changes and additions

2. Old Business

2.1	Approve meeting minutes from 3/22 and 4/17 calls	Andrew Stevens
	earlier this year	

3. New Business

3.1	Intermediate precision and repeatability estimates incorrect in procedure	Rich Grundza
3.2	Discussion regarding specifics of tests to be run on drains (procedure is not very specific and not all labs run exactly the same tests)	Panel
3.3	Discussion about standards for introducing a new fuel supplier	Panel

4. Next Meeting

4.1. SP Meeting: TBD

5. Meeting Adjourned

Meeting Information

Meeting link:

https://lubrizol-1586.my.webex.com/lubrizol-1586.my/j.php?MTID=maeb90d16aae14953d4327c009df91093

Meeting number: 624 030 625 Password: CscRwe3D Host key: 561012 *More ways to join* Join by video system Dial 624030625@lubrizol-1586.my.webex.com You can also dial 173.243.2.68 and enter your meeting number. Join by phone +1-510-338-9438 USA Toll

Access code: 624 030 625 Global call-in numbers

Appendix

A1

Andrew:

Recently it was noted the intermediate precision estimates for 109 h (FEI2) and repeatability estimates for 16 h (FEI1) have been switched.

I've attached a word document which I think better illustrates the issue.

A2

Documents regarding alternative fuel supplier discussion are attached to meeting

Attachment 3

Error in Table 8 of Test Method D8114-18a

Error in Table 8 of Test Method D8114-18a

Recently, it was noted that there was an error in the precision statement contained in Table 8 of the Test Method. Current table 8 is shown below:

				on otat
Variable Fu Economy	el Intermediate	e Precision	Reproducibility	
Improvemer %	nt,	i.p.	Sr ^B	R
at 16 h	0.29	0.81	0.12	0.34
at 109 h	0.29	0.81	0.25	0.70

TABLE 8 Sequence VIE Reference Oil Precision Statistics^A

The intermediate precision for 109 h and reproducibility at 16 h appear to be reversed.

Below is Table 8 with the values as they should appear, highlighted in red.

Variable Fuel Economy	Intermediate Precision		Reproducibility	
Improvement, %	S <i>i.p.</i> [₿]	i.p.	S _R [₿]	R
at 16 h	0.29	0.81	0.29	0.81
at 109 h	0.12	0.34	0.25	0.70

TABLE 8 Sequence VIE Reference Oil Precision Statistics^A

Attachment 4

Approval Requirements for Alternate Fuel Supplier

Approval Requirements for Alternate Fuel Supplier, Sequence VI

For an alternate supplier to obtain approval for Sequence VI tests, the supplier must demonstrate, through chemical analysis of the fuel candidate and engine testing, that the potential fuel will provide acceptable results when used for Sequence VI registered testing. The supplier will provide a C of A documenting that the fuel meets the current Sequence VI fuel specification, as well as conducting a prove-out program.

The prove-out program will be completed using the Sequence VIE test, and is to be performed in one test stand, using a minimum of two engines and a single reference oil, 1010-1 (or subsequent approved reblends). Testing will utilize the first four runs of the engines' life and will be alternated between the current fuel and the alternate fuel candidate, as shown in Table 1 (below).

Engine	Break-in Fuel	Run #1	Run #2	Run #3	Run #4
Engines 1, 3,	Current Fuel	Current Fuel	Alternate Fuel	Current Fuel	Alternate Fuel
Engines 2, 4,	Alternate Fuel	Alternate Fuel	Current Fuel	Alternate	Current Fuel

Table 1: Engine Tests Run Order

At the completion of each engine after Engine #2, two ANOVA models will be constructed using the engine hour corrected results. The response variables will be FEI1 Yi and FEI2 Yi, which are the standardized results, and factors to include are "Engine", with levels Engine1, Engine2, ..., EngineN, and "Fuel", with two levels. For the alternate fuel to be qualified, the following must be true of the ANOVA model results for both the FEI1 Yi model and the FEI2 Yi model:

- 1. The absolute difference in the least squares mean for Fuel A and the least squares mean for Fuel B is less than one.
- 2. When forming a 95% confidence interval on the least squares mean difference between fuels, the upper and lower limits are both less than 2.5 in absolute value.

If the above two criteria are not satisfied for both FEI1 and FEI2, then an additional four tests must be conducted on another engine, followed by another ANOVA model. This process will continue until both criteria have been satisfied for both parameters.

If approved, the alternate fuel may be used for registered testing in both the Sequence VIE and the Sequence VIF, provided acceptable reference testing has been completed on the stand/engine combination. All test run for registration purposes must be run on the same fuel (current or alternate) that the stand engine was calibrated on. When changing fuels, add fuel from a new batch to a laboratory's fuel tank when the current fuel level is below 10 % of the final fuel (new and previous) mixture's total volume.

Attachment 5

Sequence VI Alternate Fuel Supplier Testing Stopping Criteria 080119

Sequence VI Alternate Fuel Supplier Testing Stopping Criteria

Statistics Sub-Group

August 01, 2019

Statistics Sub-Group

- Jo Martinez, Chevron Oronite
- Richard Grundza, TMC
- Todd Dvorak, Afton
- Travis Kostan, SwRI

Recap – Test Design Requirements

The following test design requirements have been agreed upon by the group:

Test Design Requirements:

Test using reference oil 1010-1 on a minimum of two engines, using the first four runs of each engine's valid test life per the following procedure:

- Current Fuel = "Fuel A"
- Potential Alternative Supplier Fuel = "Fuel B"
- Run Order #1, Engine #1 Break in with Fuel A, then test Fuel A Fuel B Fuel A Fuel B
- Run Order #2, Engine #2 Break in with Fuel B, then test Fuel B Fuel A Fuel B Fuel A
- If the statistical stopping criteria has not been met after Engine #2, continue testing on additional engines, alternating between run order #1, and run order #2, until the stopping criteria has been met.
- All testing shall be conducted in a single lab and on a single stand.

Recap – Stopping Criteria Questions

To help determine what the statistical stopping criteria should be, the group should consider:

- 1. What does the group want the potential fuel to show? Is it...
 - a) that the potential fuel results are within a specified tolerance of the current fuel results?
 - b) that the potential fuel results are within a specified tolerance of the reference oil target?
 Selected, but the group was
 - c) a combination of a) and b) ?

d) other criteria?

Selected, but the group was reconsidering the need for criteria b.

2. What are the acceptable tolerances for the differences in #1 that are deemed important?

Criteria A Tolerances

a) that the potential fuel results are within a specified tolerance of the current fuel results?

What are the acceptable tolerances?

The statistics group recommends as a staring place for discussion the following tolerances for Criteria A. These tolerances would be judged based on an ANOVA model using the Yi values as the response variable (these are already in standard deviation units)

- 1. The estimate of the difference between fuels is less than 1 standard deviation.
- 2. No part of a 95% confidence interval of the difference between fuels exceeds 2.5 sigma.



Visualization of Stopping Criteria

- 1. The estimate of the difference between fuels is less than 1 standard deviation.
 - The black dot is the estimate of the fuel difference and cannot exceed the black dashed lines.
- 2. No part of a 95% confidence interval of the difference between fuels exceeds 2.5 sigma.
 - The confidence interval has a 95% probability of capturing the true difference between fuels. We should have at least 95% confidence the fuel difference is less than 2.5 sigma (the red dashed lines).



Distribution of 1010-1 Yi Results

FEI 1 Yi

FEI 2 Yi

- Data includes 2 years of 1010-1 • results from stands which had at least 2 tests after filtering for TMC validity codes AC and OC.
- The across lab Yi standard ٠ deviation is expected to be near one, since this is a normalized value. This across lab data does appear close to this value. The data also shows that the 1010-1 Yi results within a single stand are showing considerably less deviation than across labs, about 0.25 sigma less.



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Fuel	Engine	FEI 1	FEI 1 Yi
Fuel A	Engine #1	1.89	-0.03
Fuel A	Engine #1	1.70	-1.01
Fuel A	Engine #2	2.10	1.03
Fuel A	Engine #2	1.95	0.27
Fuel B	Engine #1	1.67	-1.14
Fuel B	Engine #1	1.90	0.00
Fuel B	Engine #2	1.51	-1.96
Fuel B	Engine #2	1.69	-1.04



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Example FEI 1 After 3 Engines





Fuel	Engine	FEI 1	FEI 1 Yi
Fuel A	Engine #1	1.89	-0.03
Fuel A	Engine #1	1.70	-1.01
Fuel A	Engine #2	2.10	1.03
Fuel A	Engine #2	1.95	0.27
Fuel A	Engine #3	1.69	-1.04
Fuel A	Engine #3	1.84	-0.30
Fuel B	Engine #1	1.67	-1.14
Fuel B	Engine #1	1.90	0.00
Fuel B	Engine #2	1.51	-1.96
Fuel B	Engine #2	1.69	-1.04
Fuel B	Engine #3	1.87	-0.16
Fuel B	Engine #3	1.93	0.14



Probability of Meeting the Criteria Tolerances -Assuming Zero Fuel Difference and Yi Standard Deviation =1.0

P(Average Fuel Difference) < Various Sigma, Assuming Zero Actual Fuel Difference

# of Engines	P(Estimate < 0.5 Sigma) if Fuel Difference is Zero	P(Estimate < 0.75 Sigma) if Fuel Difference is Zero	P(Estimate < 1.0 Sigma) if Fuel Difference is Zero	P(Estimate < 1.25 Sigma) if Fuel Difference is Zero
2	0.52	0.71	0.84	0.92
3	0.61	0.81	0.92	0.97
4	0.68	0.87	0.95	0.99
5	0.74	0.91	0.97	0.99

P(CI Width within Various Sigma), Assuming Zero Actual Fuel Difference

# of Engines	P(CI Width within 1.5 Sigma) if Fuel Difference is Zero	P(CI Width within 2.0 Sigma) if Fuel Difference is Zero	P(CI Width within 2.5 Sigma) if Fuel Difference is Zero	P(CI Width within 3.0 Sigma) if Fuel Difference is Zero
2	-0.35	0.20	0.67	0.91
3	0.23	0.75	0.96	1.00
4	0.58	0.93	0.99	1.00
5	0.77	0.98	1.00	1.00
		A5 - 12		

Attachment 6

Sequence VI Alternate Fuel Supplier Testing Stopping Criteria (SwRI)

Sequence VI Alternate Fuel Supplier Testing Stopping Criteria

Southwest Research Institute®



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Test Design Requirements

The following test design requirements have been agreed upon by the group:

Test Design Requirements

Test using reference oil 1010-1 on a minimum of two engines, using the first four runs of each engine's valid test life per the following procedure:

- Current Fuel = "Fuel A"
- Potential Alternative Supplier Fuel = "Fuel B"
- Run Order #1, Engine #1 Break in with Fuel A, then test Fuel A Fuel B
 Fuel A Fuel B
- Run Order #2, Engine #2 Break in with Fuel B, then test Fuel B Fuel A
 Fuel B Fuel A
- If the statistical stopping criteria has not been met after Engine #2, continue testing on additional engines, alternating between run order #1, and run order #2, until the stopping criteria has been met.
- All testing shall be conducted in a single lab and on a single stand.



Statistical Stopping Criteria

To help determine what the statistical stopping criteria should be, the group should consider:

- I. What does the group want the potential fuel to show? Is it...
 - a) that the potential fuel results are within a specified tolerance of the current fuel results?
 - b) that the potential fuel results are within a specified tolerance of the reference oil target?
 - c) a combination of a) and b) ?
 - d) other criteria?
- 2. What are the acceptable tolerances for the differences in #1 that are deemed important?



Current Performance of Reference Oil 1010-1 – FEI I

Data is from June '18 through June '19 and shows that lab averages are different for FEI 1, and this should be taken into account when setting criteria.



1010-1 FEI 1	1010-1 FEI 1
Target	Std. Dev.
1.90	0.199

Lab	1010-1 FEI 1 Avg.	1010-1 Yi Avg.
А	1.620	-1.40
В	1.908	0.04
D	1.891	-0.05
G	1.783	-0.59



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Current Performance of Reference Oil 1010-1 – FEI 2

Data is from June '18 through June '19 and shows that labs are ¼ to ¾ standard deviations severe of target, and this should be taken into account when setting criteria.



1010-1 FEI 2 Target		10 S	1010-1 FEI 2 Std. Dev.	
1.82		0.327		
Lab	1010-1 FEI 2 Avg.		1010-1 Yi Avg.	
А	1.58		-0.72	
В	1.71		-0.34	
D	1.73		-0.26	
G	1.68		-0.43	



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Statistical Stopping Criteria

Example Criteria:

At the completion of each engine beginning with the completion of Engine #2 tests, an ANOVA model will be constructed using the engine hour corrected results. Factors to include are "Engine", with levels Engine I, Engine2, ..., EngineN, and "Fuel", with levels Fuel A and Fuel B. To be qualified, the following must be true of the ANOVA model results:

- The absolute difference in the least squares mean for Fuel A and the least squares mean for Fuel B is less than 1 standard deviation, using the 1010-1 standard deviation (0.199 for FEI1 and 0.327 for FEI2).
 - The purpose of this criterion is to check if the qualifying fuel is reasonably close to the current fuel.
- 2. The least squares mean estimate for Fuel B are within 1.5 standard deviations from the 1010-1 target, and the upper and lower limit of a 95% confidence interval on the least squares mean for Fuel B are not outside of the +/- 2 standard deviation range of the 1010-1 target, using the 1010-1 standard deviation.
 - The purpose of these criteria are to check whether or not the qualifying fuel is close enough to the 1010-1 target, regardless of where the current fuel severity lies.



FEI I Visualization of Example Criteria #2

Below are 3 examples to help understand the example criteria #2.



SwRI

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FEI 2 Visualization of Example Criteria #2

Below are 3 examples to help understand the example criteria #2.





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Example Criteria Using Lab A Data -2 Engines

Below are 8 data points from Lab A on Oil 1010-1 that were randomly assigned to Fuel A and Fuel B to evaluate these criteria.



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Example Criteria I in Lab A – 2 Engines

ANOVA model is FEI X ~ Engine + Fuel

 The absolute difference in the least squares mean for Fuel A and the least squares mean for Fuel B is less than 1 standard deviation, using the 1010-1 standard deviation (0.199 for FEI1 and 0.327 for FEI2).

FEI 1

Least Squares Means Table Least

Level	Sq Mean	Std Error	Mean
Fuel A	1.7675000	0.10563203	1.76750
Fuel B	1.5100000	0.10563203	1.51000

LS Mean Fuel A – LS Mean Fuel B = 1.765 – 1.510 = 0.255 > 0.199 *Criteria Not Met

FEI 2

Least Squares Means Table			
Least			
Level	Sq Mean	Std Error	Mean
Fuel A	1.7450000	0.18592505	1.74500
Fuel B	1.4575000	0.18592505	1.45750

LS Mean Fuel A – LS Mean Fuel B = 1.745 – 1.457 = 0.288 < 0.327 **Criteria Met*



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Example Criteria 2 in Lab A – 2 Engines

 a) The least squares mean estimate for Fuel B are within 1.5 standard deviations from the 1010-1 target, and b) the upper and lower limit of a 95% confidence interval on the least squares mean for Fuel B are not outside of the +/- 2 standard deviation range of the 1010-1 target, using the 1010-1 standard deviation.





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Example Criteria Using Lab A Data -3 Engines

Below are 12 data points from Lab A on Oil 1010-1 that were randomly assigned to Fuel A and Fuel B to evaluate these criteria.



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Example Criteria I in Lab A – 3 Engines

ANOVA model is FEI X ~ Engine + Fuel

 The absolute difference in the least squares mean for Fuel A and the least squares mean for Fuel B is less than 1 standard deviation, using the 1010-1 standard deviation (0.199 for FEI1 and 0.327 for FEI2).

FEI 1

Least Squares Means Table				
	Least			
Level	Sq Mean	Std Error	Mean	
Fuel A	1.7150000	0.07301065	1.71500	
Fuel B	1.5266667	0.07301065	1.52667	

LS Mean Fuel A – LS Mean Fuel B = 1.715 – 1.527 = 0.188 < 0.199 *Criteria Met

FEI 2

Least Squares Means Table			
Least			
Level	Sq Mean	Std Error	Mean
Fuel A	1.6466667	0.13944956	1.64667
Fuel B	1.5200000	0.13944956	1.52000

LS Mean Fuel A – LS Mean Fuel B = 1.647 – 1.520 = 0.127 < 0.327 **Criteria Met*



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Example Criteria 2 in Lab A – 3 Engines

 a) The least squares mean estimate for Fuel B are within 1.5 standard deviations from the 1010-1 target, and b) the upper and lower limit of a 95% confidence interval on the least squares mean for Fuel B are not outside of the +/- 2 standard deviation range of the 1010-1 target, using the 1010-1 standard deviation.





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