LDEOC/EOEC SURVEILLANCE PANEL

A LDEOC/EOEC conference call was held on 1-18-23, at 9 am Central Standard Time. The following esteemed members were on the call:

Mike Birke – SwRI
Vince Donndelinger - Lubrizol
Robert Stockwell – Oronite
Laura Birnbaumer - Oronite
Becky Grinfield – SwRI
Mike Lopez - Intertek
Kimberly Gutierrez - Intertek
Olivia Schmitz - SwRI
Dennis Gaal – ExxonMobil
Jason Bowden – OHT
Charles Nystrom – SwRI
Maggie Smerdon – Savant
Julie Suhadolnik - Lubrizol
John Loop – TMC
Gefei Wu – Valvoline
Conika Own-Robinson – Savant

The purpose of the call was to discuss updating the standard deviations used in the calculation of the fixed and variable limits using the data obtained from the new SL107 reference oil (presentation attached). Currently TMC has approximately 300 points which can be used. John Loop estimates these data can be calculated in two weeks. Robert Stockwell proposed using all data acquired up to Dec 31st 2022, and then updating the numbers annually on February 1st. The future deviations will be based on a 2-year rolling average. Laura Birnbaumer will provide Mike Birke with the new equations (presentation attached) used to calculate the adjusted limits. Another LDEOC/EOEC teleconference will be scheduled to look at the new standard deviations and how they compare to the old ones. Additionally, the report forms will be updated to remove any reference to TMC-1006. Robert Stockwell made a motion that TMC will no longer assign TMC-1006. The motion was unanimously approved. John Loop will initiate the process to change the report forms, however, it is unclear how long it will take. Mike Birke and John Loop will begin working on some housekeeping updates on D7216 to reflect these changes. The topic of establishing a volume change correction factor for ACM-1 batch 26 was discussed. At the last meeting, Vince Donndelinger suggested using an average ICF based on the mean of the previous 6 batches. Robert Stockwell noted batches 20 and 22 were inconsistent with the other 4 batches and that batch 26 should be screened just like the previous batches. Vince Donndelinger made a motion to accept -2.52% for batch 26 and all future ACM-1 batches. Dennis Gaal and Jason Bowden abstained, Robert Stockwell voted negative, and all others voted affirmative. The motion did not carry. The participating labs will be instructed to screen batch 26 in the same manner as the previous batches. Jason Bowden has been informed there is a strong possibility SAE J2643 will revert to the original ACM-1 curing schedule, and if implemented, should bring back the numbers to the original values. When the new "old" formulation is official, the participating labs will be running round robins to confirm. There was no new business, and the meeting adjourned at 9:45 am.

EOEC/LDEOC CONFERENCE CALL 1-18-23

AGENDA

1) Update/replace the standard deviations used in the EOEC fixed and adjusted limit calculations with the SL107 data acquired to date.
2) Determine frequency of updating standard deviations.
3) Updating the report forms to reflect new reference oil.
4) Discuss using an average CF for ACM-1 instead of screening each batch.

Background

- ASTM D 4485 ANNEX A4
- Procedure for Deriving Adjusted Specification Limits for Elastomer Compatibility
  - Method to account for the inherent test variability in the elastomer compatibility method which arises in part because batch-to-batch, sheet-to-sheet and within-sheet variations in the properties of the reference elastomers can be sufficiently large that they complicate making a decision as to whether or not a candidate oil has passed the elastomer compatibility requirements.

TABLE A4.1 Unadjusted Specification Limits for the Elastomer Test Method as Part of the CI-4 Engine Oil Category

Elastomer	Volume Change, %	Hardness Change, Points	Tensile Strength Change, MPa	Elongation at Break Change, %
Nitrile (NBR)	(+5, -3)	(+7, -5)	(+10, -TMC 1006)	(+10, -TMC 1006)
Silicone (VMQ)	(+TMC 1006, -3)	(+5, -TMC 1006)	(+10, -45)	(+20, -30)
Polyacrylate (ACM)	(+5, -3)	(+8, -5)	(+18, -15)	(+10, -35)
Fluoroelastomer (FKM)	(+5, -2)	(+7, -5)	(+10, -TMC 1006)	(+10, -TMC 1006)

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A5. PROCEDURE FOR DERIVING ADJUSTED SPECIFICATION LIMITS FOR ELASTOMER COMPATIBILITY

A5.1 Background

A5.1.1 This annex describes a statistical method to account for the inherent test variability in the elastomer compatibility test method. The need to take account of the inherent test variability arises in part because batch-to-batch, sheet-to-sheet and within-sheet variations in the properties of the reference elastomers (the four elastomers listed for the CI-4 category in Table 3) can be sufficiently large that they complicate making a decision as to whether or not a candidate oil has passed the elastomer compatibility requirements.

A5.1.2 Applying this statistical method to the unadjusted specification limits noted in Tables 2-6 produces the adjusted specification limits. *Passing* candidate-oil results shall lie within the range defined by the adjusted specification limits.

A5.1.3 The statistical method for determining the adjusted specification limits uses updated information about the industry test variability relevant to the time frame in which the candidate oil is tested. The TMC provides the updated information based on test results obtained by different test laboratories with different batches of reference elastomers on the same TMC 1006 reference oil.

A5.2 Unadjusted Specification Limits

A5.2.1 The unadjusted specification limits are shown for the CI-4 category in Table 3. (These are reproduced in Table A5.1 for comparison purposes.) The test method involves sixteen criteria. These criteria are the unadjusted specified limits for the four elastomer types (nitrile, silicone, polyacrylate and fluoroelastomer), with changes in four properties (volume, hardness, tensile strength and elongation at break).

A5.3 Adjusted Specification Limits

A5.3.1 The adjusted specification limits are calculated by adjusting the numerical limits in Tables 2-6 (referred to as *fixed limits*), and the TMC 1006 limit in Tables 2-6 (referred to as a *variable limit*). The reference oil TMC 1006 is run in parallel with the candidate oil as a control for each experiment.

A5.3.2 The adjusted specification limits are determined as the unadjusted specification limits plus (in absolute terms) an amount to account for test variability.

A5.4 Inherent Test Variability

A5.4.1 Table A5.2 shows examples of the standard deviation estimates of the four reference elastomers and the four performance parameters, as reported by the TMC. The standard deviation estimates, applicable at the time a test oil is evaluated, are obtained from the TMC website (ftp://ftp.astmtmc.cmu.edu/refdata/bench/elastomer_pc9/PC-9_Elastomer_1006.xls).

A5.5 Adjusted Specification Limits—Calculations

A5.5.1 *Calculation of Fixed Limits:*

A5.5.1.1 Calculate the standard error of the test-oil mean by dividing the appropriate *total standard deviation* estimate by the square root of the number of observations in the sample. The number of observations in the sample, in the absence of outliers, is six.

A5.5.1.2 Multiply the standard error of the test-oil mean by 2.0.

A5.5.1.3 Add or subtract the resulting number to or from the respective upper or lower unadjusted specification limits to obtain the *fixed* adjusted specification limit(s).

A5.5.2 *Calculation of Variable Limits:*

A5.5.2.1 Calculate the standard error of the test-oil mean by dividing the appropriate *within-lab standard deviation* estimate by the square root of the number of observations in the sample. The number of observations in the sample, in the absence of outliers, is six.

A5.5.2.2 Multiply the standard error of the test-oil mean by 2.8.

A5.5.2.3 Add or subtract the resulting number to or from the mean result obtained with TMC 1006 (run in parallel with the test oil) to obtain either the upper or lower *variable* adjusted specification limit.

A5.5.3 Table A5.3 shows an example of the calculated adjusted specification limits.

A5.6 Comparison of Unadjusted and Adjusted Specification Limits

A5.6.1 Table A5.1 reproduces the unadjusted specification limits for comparison with the above adjusted specification limits.

Background (continued)

Adjusted Specification Limit Standard Deviations
Effective: March 1, 2008

Elastomer	Parameter	Within Lab STD	Overall STD	Total Individual Determinations
FLUOROELASTOMER	Volume	0.16	0.18	1719
FLUOROELASTOMER	Hardness	1.45	2.04	1665
FLUOROELASTOMER	Tension	4.77	5.31	1723
FLUOROELASTOMER	Elongation	7.75	10.22	1705
NITRILE	Volume	0.76	0.79	1748
NITRILE	Hardness	1.47	1.72	1696
NITRILE	Tension	8.96	9.3	1735
NITRILE	Elongation	6.99	7.07	1742
POLYACRYLATE	Volume	0.76	0.79	1768
POLYACRYLATE	Hardness	1.67	1.68	1718
POLYACRYLATE	Tension	10.09	10.12	1733
POLYACRYLATE	Elongation	11.2	11.28	1742
SILICONE	Volume	1.84	2.06	1733
SILICONE	Hardness	1.25	2.23	1661
SILICONE	Tension	6.99	7.04	1711
SILICONE	Elongation	9.87	10.02	1732
VAMAC	Volume	2.04	2.29	918
VAMAC	Hardness	1.17	1.17	898

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Background (continued)

- Initial Adjustments were based upon data from TMC 1006 Reference Oil
  - Between 898 and 1768 observations were analyzed for the various elastomer/parameter combinations to establish STD for the adjustment calculations
    - Reference Oil 1006 is no longer available from TMC
    - Some labs may have a few 1006 samples remaining
      - Total outstanding 1006 Reference Oil Samples at labs <10
- Elastomer Testing now uses Reference Oil SL107
- ASTM D 4485 Chair has requested that the Adjusted Limits for EOEC be reviewed. D7216 Elastomers Chair has agreed to hold a meeting in Q1 2023 to address this issue.
- TMC will provide support through an analysis of SL107 Reference Oil test runs.
  - Data on SL107 EOEC Elastomer Reference Oil runs up through 9DEC2022

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D7216 – Engine Oil Elastomer Compatibility
Form 2 – Candidate Data

Sample Code:	Lab:	EOT Date:
Test Method Version:		Test Length:

Elastomer Identification	TMC Identification	Parameter	Specification Limit	Acceptance Limits Updated on:	Reference Result	Candidate Result
Type:	Industry Oil:	Volume Change	+5% to -3%	to		
Nitrile		Hardness	+7 pts to -5 pts	to		
Batch:	CMIR:	Tensile Strength	+10% to –TMC1006	to		
		Elongation	+10% to –TMC1006	to		

Elastomer Identification	TMC Identification	Parameter	Specification Limit	Acceptance Limits Updated on:	Reference Result	Candidate Result
Type:	Industry Oil:	Volume Change	+5% to -3%	to		
Polyacrylate		Hardness	+8 pts to -5 pts	to		
Batch:	CMIR:	Tensile Strength	+18% to -15%	to		
		Elongation	+10 to -35%	to		

Elastomer Identification	TMC Identification	Parameter	Specification Limit	Acceptance Limits Updated on:	Reference Result	Candidate Result
Type:	Industry Oil:	Volume Change	+5% to -2%	to		
Fluoroelastomer		Hardness	+7 pts to -5 pts	to		
Batch:	CMIR:	Tensile Strength	+10% to –TMC1006	to		
		Elongation	+10% to –TMC1006	to		

Elastomer Identification	TMC Identification	Parameter	Specification Limit	Acceptance Limits Updated on:	Reference Result	Candidate Result
Type:	Industry Oil:	Volume Change	+TMC1006 to -3%	to		
Silicone		Hardness	+5 pts to –TMC1006	to		
Batch:	CMIR:	Tensile Strength	+10% to -45%	to		
		Elongation	+20% to -30%	to		

Elastomer Identification	TMC Identification	Parameter	Specification Limit	Acceptance Limits Updated on:	Reference Result	Candidate Result
Type:	Industry Oil:	Volume Change	+TMC1006 to -3%	to		
Vamac		Hardness	+5 pts –TMC1006	to		
Batch:	CMIR:	Tensile Strength	+10% to –TMC1006	to		
		Elongation	+10% to –TMC1006	to		

ACM-1 CORRECTION FACTORS

TABLE A2.2 Industry Correction Factor—Light Duty Polyacrylate Elastomer (ACM1)

Elastomer Batch	Volume Change Industry Correction Factor
Batches prior to 19	0.00
ACM1-19	–2.65
ACM1-20	–3.14
ACM1-21	–2.53
ACM1-22	–1.65
ACM1-23	–2.72
ACM1-24	–2.43
ACM1-25	–2.55

10-13-21 minutes Vince Donndelinger proposed an average ICF of -2.52% for all future batches.