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#### **ASTM Test Monitoring Center Semiannual Report**

ASTM D02.B07 Bench Reference Test Monitoring From April 1, 2012 through September 30, 2012

<u>D6417</u>, <u>D5800</u>, D6335 (<u>TEOST</u>), D7097 (<u>MTEOS</u>), D5133 (<u>GI</u>), <u>D6082</u>, <u>D874</u> and D7528 (<u>ROBO</u>)

#### **Executive Summary Page 1 of 2**

#### Calibrated Labs and Instruments as of 20120930

Test	Labs	Instruments
D6417	5	7
D5800	6	13
GI	4	7
TEOST	5	6
MTEOS	6	27
D6082	3	4
D874	3	
ROBO	7	18

#### **D5800 Evaporation Loss of Lubricating Oils by the Noack Method**

- Long-term severe trend with an unexplained increase in severity since 01JUL06
- Oil 52 continues to perform nearly 1 s severe (Attachment 1)
- Since April 1, 2009, 27 of 30 statistically failing tests were severe fails on oil 52
- Surveillance Panel is trying to address severity issue
  - o Operational survey issued to participating labs
  - Teleconferences held
  - Workshop proposed for first quarter 2013



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# ASTM D02.B07 Bench Reference Test Monitoring From April 1, 2012 through September 30, 2012

#### **Executive Summary Page 2 of 2**

#### **D5133: Gelation Index (GI)**

- Precision this period is slightly degraded compared to last period, but continues to be better than most prior periods, and better than target.
- Severity is quite mild compared to past periods (-0.89 s)

#### **D6335 TEOST-33C**

Rod Batch K Introduced

#### **D7097 MHT-4 TEOST**

Rod Batch K introduced

#### **D874 Sulfated Ash**

- One extremely mild result (-11.5 s) reported as operationally valid (included in statistics)
  - Unusual occurrence

#### **D7528: Bench Oxidation of Engine Oils by ROBO Apparatus**

 Precision (Pooled s) is degraded this period compared to target and last two periods



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#### **D6417: Estimation of Engine Oil Volatility by Capillary GC**

#### **MONITORED TESTING ACTIVITY**

TABLE 1
Reference Tests Reported to the TMC This Period (5 labs reporting)

	No. of Tests
Statistically Acceptable and Operationally Valid (AC)	15
Operationally Valid but Statistically Unacceptable (OC)	0
Operationally Invalid (initially reported as) (LC, XC)	0
Operationally Invalid (after informed of failing calibration) (RC)	0
Total	15

Fail Rate of Operationally Valid Tests: 0%

No operationally invalid tests reported this period.

TABLE 2 Statistically Unacceptable Tests (OC)

Reason for Fail	No. of Tests
Area % Volatized @ 371°C Severe	0
Area % Volatized @ 371°C Mild	0

#### TMC MEMORANDA

No D6417 TMC technical updates this period.

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#### **D6417 PRECISON AND SEVERITY**

TABLE 3
Period Precision and Severity Estimates

Area % Volatized @ 371°C	N	df	Pooled s	Mean ∆/s
Initial Round Robin Study	107	101	0.46	
4/1/09 through 9/30/09	15	12	0.34	0.23
10/1/09 through 3/31/10	13	10	0.33	80.0
4/1/10 through 9/30/10	16	13	0.30	0.41
10/1/10 through 3/31/11	20	17	0.38	0.06
4/1/11 through 9/30/11	16	13	0.37	0.21
10/1/11 through 3/31/12	14	11	0.24	0.17
4/1/12 through 9/30/12	15	12	0.28	-0.19

TABLE 4
Current Period Severity Estimates by Lab

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	Ν	Mean ∆/s
Lab A	4	0.77
Lab B	2	0.03
Lab D	4	-0.98
Lab G	3	-0.39
Lab S	2	-0.46

- Precision (Pooled s) is comparable to last period and more precise than prior periods
  - o More precise than the target precision
- Performance (Mean Δ/s) is slightly mild at -0.19 s
- <u>Figure 1</u> shows overall nearly on-target performance since the 01OCT11 timeline

#### **D5800:** Evaporation Loss of Lubricating Oils by the Noack Method

#### **MONITORED TESTING STATUS**

TABLE 5
Reference Tests Reported to the TMC This Period (7 labs reporting)

	No. of Tests
Statistically Acceptable and Operationally Valid (AC)	29
Operationally Valid but Statistically Unacceptable (OC)	4
Operationally Invalid (initially reported as) (LC, XC)	0
Operationally Invalid (after informed of failing calibration) (RC)	0
Total	33

Fail Rate of Operationally Valid Tests: 12%

• No operationally invalid tests reported this period.

TABLE 6
Statistically Unacceptable Tests (OC)

Reason for Fail	No. of Tests
Sample Evaporation Loss Severe	4
Sample Evaporation Loss Mild	0

- Four severe OC tests on oil 52
- Ten passing AC results on oil 52 this period

#### **TMC MEMORANDA**

No D5800 TMC technical updates this period.



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#### **D5800 PRECISION AND SEVERITY**

TABLE 7
Period Precision and Severity Estimates

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Sample Evaporation Loss, mass %	n	df	Pooled s	Mean ∆/s
New Targets Effective 7/21/2003	102	99	0.70	
10/1/07 through 3/31/08	34	31	0.50	0.75
4/1/08 through 9/30/08	36	33	0.54	0.82
10/1/08 through 3/31/09	36	33	0.84	0.51
4/1/09 through 9/30/09	36	33	0.56	0.88
10/1/09 through 3/31/10	35	32	0.69	0.56
4/1/10 through 9/30/10	34	31	0.67	0.64
10/1/10 through 3/31/11	34	31	0.76	0.49
4/1/11 through 9/30/11	39	36	0.59	0.77
10/1/11 through 3/31/12	32	29	0.78	0.54
4/1/12 through 9/30/12	33	30	0.67	0.56

TABLE 8
Current Period Precision and Severity Estimates by Method Procedure

Sample Evaporation Loss, mass %	n	df	Pooled s	Mean ∆/s
Procedure A	0	0		
Procedure B	28	25	0.60	0.68
Procedure C	5	2	0.97	-0.13

TABLE 9
Current Period Severity Estimates by Lab

	n	Mean ∆/s
Lab A	3	0.19
Lab B	10	0.01
Lab D	3	-0.71
Lab F	2	0.61
Lab G	7	0.93
Lab I	2	1.16
Lab J	6	1.64



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#### **D5800: Evaporation Loss of Lubricating Oils by the Noack Method**

- Precision (Pooled s) is more precise than the previous period
  - o Comparable to the target precision
- Performance (Mean  $\Delta$ /s) is severe at 0.56 s
- <u>Figure 2A</u> & 2B show a long-term severe trend with an unexplained increase in severity since the 01JUL06 timeline.
- Oil 52 performs less than 1 s severe (<u>Attachment 1</u>) for the first time in the past ten report periods (5 years)
  - All four statistically failing results this period were on oil 52
  - Also had ten passing results on oil 52
- Since April 1, 2009, 27 of 30 statistically failing tests were on oil 52
  - All failed severe of acceptance bands



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#### **D5133:** Gelation Index (GI)

#### **MONITORED TESTING STATUS**

TABLE 10
Reference Tests Reported to the TMC This Period (4 labs reporting)

	No. of Tests
Statistically Acceptable and Operationally Valid (AC)	21
Operationally Valid but Statistically Unacceptable (OC)	3
Operationally Invalid (initially reported as) (LC, XC)	0
Operationally Invalid (after informed of failing calibration) (RC)	0
Total	24

Fail Rate of Operationally Valid Tests: 13%

No operationally invalid tests reported this period.

TABLE 11
Statistically Unacceptable Tests (OC)

Reason for Fail	No. of Tests
Gelation Index Severe	0
Gelation Index Mild	3

#### **TMC MEMORANDA**

No D5133 (GI) TMC technical updates this period.



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#### **D5133 (GI) PRECISION AND SEVERITY**

TABLE 12
Period Precision and Severity Estimates

Gelation Index	n	df	Pooled s	Mean ∆/s
Revised Targets Effective 20030715	68	65	2.86	
10/1/06 through 3/31/07	29	26	3.23	-0.68
4/1/07 through 9/30/07	24	21	3.35	-0.28
10/1/07 through 3/31/08	26	23	4.13	-0.31
4/1/08 through 9/30/08	27	24	3.54	0.18
10/1/08 through 3/31/09	24	21	2.32	0.10
4/1/09 through 9/30/09	33	30	2.79	-0.10
10/1/09 through 3/31/10	31	28	2.37	-0.15
4/1/10 through 9/30/10	24	21	3.89	0.12
10/1/10 through 3/31/11	33	30	3.17	-0.53
4/1/11 through 9/30/11	23	20	1.70	-0.25
10/1/11 through 3/31/12	24	21	1.36	0.06
4/1/12 through 9/30/12	24	21	1.88	-0.89

TABLE 13
Current Period Severity Estimates by Lab

	<u> </u>	
		GI
	n	Mean ∆/s
Lab A	9	-1.26
Lab B	6	-1.09
Lab G	3	-0.19
Lab S	6	-0.48



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#### **D5133: Gelation Index (GI)**

- Precision (Pooled s) is less precise than the previous period
  - More precise than the target precision
  - Last three report periods show historically improved precision
- Severity (Mean Δ/s) is mild at -0.89 s
  - All labs mild to some extent
- Figure 3A & 3B show a shift to mild since the JAN11 timeline with short periods of leveling to on-target performance



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**D6335: TEOST-33C** 

#### **MONITORED TESTING STATUS**

TABLE 14
Reference Tests Reported to the TMC This Period (5 labs reporting)

	No. of Tests
Statistically Acceptable and Operationally Valid (AC)	15
Operationally Valid but Statistically Unacceptable (OC)	3
Operationally Invalid (initially reported as) (LC, XC)	1
Operationally Invalid (after informed of failing calibration) (RC)	0
Total	19

Fail Rate of Operationally Valid Tests: 17%

 One operationally invalid test (LC) due to incorrect pump speed setting

TABLE 15
Statistically Unacceptable Tests (OC)

Reason for Fail	No. of Tests
Total Deposits Severe	3
Total Deposits Mild	0

#### **TMC MEMORANDA**

No D6335 (TEOST-33C) TMC technical updates this period.

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#### **D6335 (TEOST-33C) PRECISION AND SEVERITY**

TABLE 16
Period Precision and Severity Estimates

<b>Total Deposits</b>	n	df	Pooled s	Mean ∆/s
Initial Round Robin Study	54	52	4.18	
4/1/08 through 9/30/08	15	13	6.99	0.20
10/1/08 through 3/31/09	18	16	4.90	0.98
4/1/09 through 9/30/09*	14	10	8.24	0.32
4/1/09 through 9/30/09*	13	9	3.71	0.68
10/1/09 through 3/31/10*	12	8	14.36	0.85
10/1/09 through 3/31/10*	11	7	6.46	0.18
4/1/10 through 9/30/10	16	12	4.70	0.16
10/1/10 through 3/31/11	14	10	6.25	0.14
4/1/11 through 9/30/11	19	15	6.52	-0.27
10/1/11 through 3/31/12	16	12	8.60	0.37
4/1/12 through 9/30/12	18	15	7.06	0.79

<sup>\*</sup>Period statistics with and without a single very severe result included

TABLE 17
Current Period Severity Estimates by Lab

	n	Mean ∆/s
Lab A	5	0.81
Lab B	5	-0.04
Lab D	2	1.02
Lab G	4	2.15
Lab V	2	-0.12



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#### **D6335: TEOST-33C**

- Precision (Pooled s) is more precise than last period, but still worse than other recent prior periods
  - o Remains less precise than the target precision
- Performance (Mean Δ/s) is severe at 0.79 s
  - o Two results over 3 s from target
    - 4.1 s severe, lab G
    - 3.5 s severe, lab G
- Figure 4A & 4B show severity CUSUM plots
- All tests this period reported using Rod Batch J or K



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**D7097: MHT-4 TEOST** 

#### **MONITORED TESTING STATUS**

TABLE 18
Reference Tests Reported to the TMC This Period (7 labs reporting)

	No. of Tests
Statistically Acceptable and Operationally Valid (AC)	57
Operationally Valid but Statistically Unacceptable (OC)	8
Operationally Invalid (initially reported as) (LC, XC)	0
Operationally Invalid (after informed of failing calibration) (RC)	2
Total	67

Fail Rate of Operationally Valid Tests: 12%

- One test invalid due to contaminated filter deposits (after advised of statistically failing calibration result, RC)
- One test invalid due to faulty mass flow controller (after advised of statistically failing calibration result, RC)

TABLE 19
Statistically Unacceptable Tests (OC)

Reason for Fail	No. of Tests
Total Deposits Severe	8
Total Deposits Mild	0

- All eight severe failing results on severe performing reference oil 432
  - Multiple labs/instruments

#### TMC MEMORANDA

• No D7097 (MHT-4 TEOST) TMC technical updates this period.

#### **D7097 (MHT-4 TEOST) PRECISION AND SEVERITY**

TABLE 20 Period Precision and Severity Estimates

Total Deposits	n	df	Pooled s	Mean ∆/s
Updated Targets Effective 7/31/06	90	87	5.62	
4/1/07 through 9/30/07	48	45	7.68	0.32
10/1/07 through 3/31/08	46	43	7.41	-0.21
4/1/08 through 9/30/08	46	43	6.09	0.01
10/1/08 through 3/31/09	53	50	5.25	0.73
4/1/09 through 9/30/09	48	45	4.35	-0.08
10/1/09 through 3/31/10	43	40	5.46	-0.19
4/1/10 through 9/30/10	55	52	4.45	-0.12
10/1/10 through 3/31/11	55	52	7.59	0.27
4/1/11 through 9/30/11	46	43	6.00	0.03
10/1/11 through 3/31/12	56	54	5.88	0.09
4/1/12 through 9/30/12	65	62	5.63	0.26

TABLE 21 Current Period Severity Estimates by Lab

	n	Mean ∆/s
Lab A	19	0.32
Lab AK	1	1.64
Lab B	16	-0.31
Lab D	10	0.27
Lab G	14	0.53
Lab J	3	1.61
Lab V	2	-0.46



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#### **D7097 MHT-4 TEOST**

- Precision (Pooled s) is more precise than the previous period
  - o Comparable to the target precision
- Performance (Mean Δ/s) is slightly severe at 0.26 s
  - <u>Figure 5A</u> & 5B show severity CUSUM plots with slight severe trend
- All tests this period reported using Rod Batch J or K
- All tests this period reported using catalyst batch 1201



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#### **D6082: High Temperature Foaming Characteristics of Oils**

#### **MONITORED TESTING STATUS**

TABLE 22
Reference Tests Reported to the TMC This Period (3 labs reporting)

	No. of Tests
Statistically Acceptable and Operationally Valid (AC)	9
Acceptable Discrimination (AS)	4
Operationally Valid but Statistically Unacceptable (OC)	0
Operationally Invalid (initially reported as) (LC, XC)	0
Operationally Invalid (after informed of failing calibration) (RC)	0
Total	13

Fail Rate of Operationally Valid Tests: 0%

No operationally invalid tests reported this period

TABLE 23 Statistically Unacceptable Tests (OC)

Reason for Fail	No. of Tests
Foam Tendency Severe	0
Foam Tendency Mild	0

#### **TMC MEMORANDA**

No D6082 TMC technical updates this period



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#### **D6082 PRECISION AND SEVERITY**

TABLE 24
Period Precision and Severity Estimates

1007 Foam Tendency, ml	n	Mean	S	Mean ∆/s
Initial Round Robin Study (targets)	28	65.71	19.28	
10/1/09 through 3/31/10	8	59	10	-0.38
4/1/10 through 9/30/10	8	65	16	-0.05
10/1/10 through 3/31/11	8	61	10	-0.25
4/1/11 through 9/30/11	9	80	26	0.74
10/1/11 through 3/31/12	8	65	13	-0.05
4/1/12 through 9/30/12	9	63	13	-0.14

TABLE 25
Period Precision and Severity Estimates

r onder roddom and Coverty Edimates				
1007 Foam Stability @ 1 min., ml	n	Mean	S	
Initial Round Robin Study	28	0.00	0.00	
10/1/09 through 3/31/10	8	No non-ze	ero occurrences	
4/1/10 through 9/30/10	8	No non-ze	ero occurrences	
10/1/10 through 3/31/11	8	No non-ze	ero occurrences	
4/1/11 through 9/30/11	9	No non-ze	ero occurrences	
10/1/11 through 3/31/12	8	No non-ze	ero occurrences	
4/1/12 through 9/30/12	9	No non-ze	ero occurrences	

TABLE 26 Current Period Severity Estimates by Lab TMC 1007

	n	Foam Tendency Mean ∆/s
Lab A	2	1.00
Lab B	4	-0.45
Lab G	3	-0.49



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#### **D6082: High Temperature Foaming Characteristics of Oils**

- Foam Tendency Precision (Pooled s) is the same as last period
   More precise than target precision
- Foam Tendency performance (Mean  $\Delta$ /s) is nearly on-target (-0.14 s)
  - Figure 6 shows a severity CUSUM plot for Foam Tendency
- There were no non-zero occurrences of Foam Stability on 1007
  - Suggests Foam Stability precision is as expected
- All operationally valid discrimination tests reported this period meet the acceptance criteria (that is, all reporting labs could discriminate oil 66 as a GF-5/SN failing oil for Foam Tendency).



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#### **D874:** Sulfated Ash from Lubricating Oils and Additives

#### **MONITORED TESTING STATUS**

TABLE 27
Reference Tests Reported to the TMC This Period (3 labs reporting)

	No. of Tests
Statistically Acceptable and Operationally Valid (AC)	6
Operationally Valid but Statistically Unacceptable (OC)	1
Operationally Invalid (initially reported as) (LC, XC)	0
Operationally Invalid (after informed of failing calibration) (RC)	0
Total	7

Fail Rate of Operationally Valid Tests: 14%

No operationally invalid tests reported this period

TABLE 28 Statistically Unacceptable Tests (OC)

Reason for Fail	No. of Tests
Sulfated Ash Severe	0
Sulfated Ash Mild	1

#### **TMC MEMORANDA**

No D874 TMC technical updates this period.



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#### **D874 PRECISION AND SEVERITY**

TABLE 29
Period Precision and Severity Estimates

Gelation Index	n	df	Pooled s	Mean ∆/s
Initial Round Robin Targets	81	79	0.07	
4/1/08 through 9/30/08	6	3	0.04	-0.62
10/1/08 through 3/31/09	6	3	0.07	-1.23
4/1/09 through 9/30/09	7	4	0.03	-0.41
10/1/09 through 3/31/10	7	4	0.04	-0.23
4/1/10 through 9/30/10	5	2	0.03	0.11
10/1/10 through 3/31/11	6	3	0.05	0.11
4/1/11 through 9/30/11	6	3	0.01	-0.28
10/1/11 through 3/31/12	6	4	0.02	0.25
4/1/12 through 9/30/12*	7	4	0.37	-1.64
4/1/12 through 9/30/12*	6	3	0.04	0.01

<sup>\*</sup>Period stats with and without extreme result included

TABLE 30 Current Period Severity Estimates by Lab

	n	Mean ∆/s
Lab A	3	-3.96**
Lab B	2	-0.16
Lab G	2	-0.38

<sup>\*\*</sup>Lab A severity is -0.19 with single extreme result excluded

#### **D874: Sulfated Ash from Lubricating Oils and Additives**

- Lab A reported a result as operationally valid that was -11.5 s mild of target performance on oil 820-2
  - Lab A has no objective operational explanation for the unusually mild result
  - Statistics are shown with the result included and excluded for comparison
- With single very extreme result excluded, the precision (Pooled s) is more precise than the target precision
- With single very extreme result excluded, the performance (Mean  $\Delta$ /s) is on-target at 0.01 s
- Figure 7 shows a CUSUM severity plot with the period's unusually mild result obvious



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#### **D7528:** Bench Oxidation of Engine Oils by ROBO Apparatus

#### **MONITORED TESTING STATUS**

TABLE 31
Reference Tests Reported to the TMC This Period (9 labs reporting)

	No. of Tests
Statistically Acceptable and Operationally Valid (AC)	76
Operationally Valid but Statistically Unacceptable (OC)	10
Operationally Invalid (initially reported as) (LC, XC)	11
Operationally Invalid (after informed of failing calibration) (RC)	3
Non-reference Rig Shakedown, excluded from statistics	2
Total	102

Fail Rate of Operationally Valid Tests: 12%

- 4 tests invalid due to NO<sub>2</sub> flow problems or leaks (LC)
- 1 test invalid because of incorrect vacuum control valve setting (LC)
- 2 tests invalid or aborted due to vacuum pump failure (XC & LC)
- 1 test invalid because of stirrer failure (LC)
- 1 test aborted by power failure (XC)
- 1 test aborted because of a loose thermocouple connection (XC)
- 2 tests invalid due to a restricted vacuum lines (RC)
- 2 tests invalid due to faulty reactors (RC & LC)

TABLE 32
Statistically Unacceptable Tests (OC)

	(00)
Reason for Fail	No. of Tests
Natural Log MRV Viscosity Severe	2
Natural Log MRV Viscosity Mild	8

#### TMC MEMORANDA

• No D7528 ROBO TMC technical updates this period.

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#### **D7528 ROBO PRECISION AND SEVERITY**

TABLE 33
Period Precision and Severity Estimates

Natural Log (MRV Viscosity)	n	df	Pooled s	Mean ∆/s
Initial Round Robin Targets	42	39	0.2309	
8/31/08 through 3/31/09	22	19	0.2302	-0.47
4/1/09 through 9/30/09	26	23	0.1872	-0.58
10/1/09 through 3/31/10	59	56	0.3989	-0.24
4/1/10 through 9/30/10	114	110	0.5134	-0.26
10/1/10 through 3/31/11*	121	118	0.7092	0.29
10/1/10 through 3/31/11*	120	117	0.4628	0.05
4/1/11through 9/30/11	96	92	0.2593	-0.69
10/1/11 through 3/31/12	93	90	0.2068	-0.39
4/1/12 through 9/30/12	86	83	0.2975	-0.29

<sup>\*</sup>Period results with a result of more than 29 s severe included and excluded.

TABLE 34
Current Period Severity Estimates by Lab

	n	Mean ∆/s
Lab A	30	0.09
Lab AM	12	0.23
Lab AN	7	-0.64
Lab AQ	3	-0.18
Lab B	9	-1.23
Lab D	6	-0.93
Lab G	19	-0.48

Labs AO and AP also reported data, but not included in statistics (Operationally invalid or shakedown runs only)



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#### **D7528: Bench Oxidation of Engine Oils by ROBO Apparatus**

- Precision (Pooled s) is less precise than the target precision
  - Less precise than the prior two report periods
- Performance (Mean  $\Delta$ /s) is mild at -0.29 s
  - o Five of seven calibrating labs performing mild to some extent
  - Lab A nearly on target with 30 tests for the report period
  - o All three reference oils continue performing mild (Attachment 1)
- Severity is graphically represented in <u>Figure 8A</u> & 8B



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#### <u>D6922: Determination of Homogeneity and Miscibility in Automotive</u> Engine Oils

The TMC distributes six reference oils for D6922 testing. The TMC does not collect data or monitor any test results for this test at this time.

# <u>D7563: Evaluation of the Ability of Engine Oil to Emulsify Water and Simulated Ed85 Fuel</u>

The TMC distributes two reference oils for D7563 testing. The TMC does not collect data or monitor any test results for this test at this time.

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#### **REFERENCE OIL SUPPLIES**

There is adequate supply of B0.07 Bench Test reference oils at the TMC.

Table 35A Current Reference Oils

Oil	For Tests	Quantity Left (gallons)	Quantity Used Last 12 Months (gallons)
52	D6417, D5800	62.6	0.6
55	D6417, D5800	68.1	0.3
58	D6417, D5800, GI	118.8	0.5
62	GI	1.6	0.1
66	D6082	92.6	0.8
75	MTEOS	7.3	0.8
90	D874 & D874 Daily	35.1	1.9
91	D874	4.5	0.0
**432	MTEOS	Adequate	
434	MTEOS	5.0	0.4
820-2	D874	10.5	0.1
**1007	D6082	15.2	
**1009	GI	Adequate	
*434-1	ROBO	Adequate	
*435-1	ROBO	Adequate	
*435-2	ROBO/MTEOS	Adequate	
*438	ROBO	Adequate	

<sup>\*</sup>One drum of oil is set aside for bench calibration testing; the TMC has a larger supply of this oil.

<sup>\*\*</sup>Multi-Test Oil, estimated aliquot set aside for bench testing.



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#### REFERENCE OIL SUPPLIES, continued

Table 35B Obsolete or Test Development Reference Oils

Oil	For Tests	Quantity Left (gallons)	Quantity Used Last 12 Months (gallons)		
^51	Obsolete Vol. & GI	94.6	0.0		
^53	Obsolete Vol. & GI	96.8	0.0		
^54	Obsolete Volatility	97.8	0.0		
71	Obsolete TEOST	4 Samples	0.0		
71-1	Obsolete TEOST	12 samples	0.0		
72	Obsolete TEOST	2 Samples	0.0		
72-1	Obsolete TEOST	4 samples	0.0		
74	Obsolete MTEOS	0.2	0.1		
^83	Obsolete ROBO	47.3	0.0		
^84	Obsolete ROBO	3.3	0.0		
^85	Obsolete ROBO	3.3	0.0		
^433	Obsolete MTEOS	Adequate			
435	Obsolete ROBO	7 Samples			

<sup>^</sup>Test development oil; holding for instructions from Surveillance Panel.

Table 35C Homogeneity and Miscibility Reference Oils

Oil	For Tests	Quantity Left (gallons)				
HMA	H&M (D6922)	158.7	7.7			
HMB	H&M (D6922)	162.7	7.7			
HMC	H&M (D6922)	148.7	7.7			
HMD	H&M (D6922)	156.4	7.7			
HME	H&M (D6922)	142.4	7.7			
HMF	H&M (D6922)	164.9	7.7			



# Test Monitoring Center http://astmtmc.cmu.edu 412-365-1000

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#### **REFERENCE OIL SUPPLIES, continued**

Table 35D **Emulsion Retention Reference Oils** 

Oil	For Tests		Quantity Used Last 12 Months (gallons)			
EM2	Emulsion Retention	8.7	0.0			
EM2-1	Emulsion Retention	25.0	0.0			
EM5	Emulsion Retention	8.7	0.0			
EM5-1	Emulsion Retention	25.0	0.0			

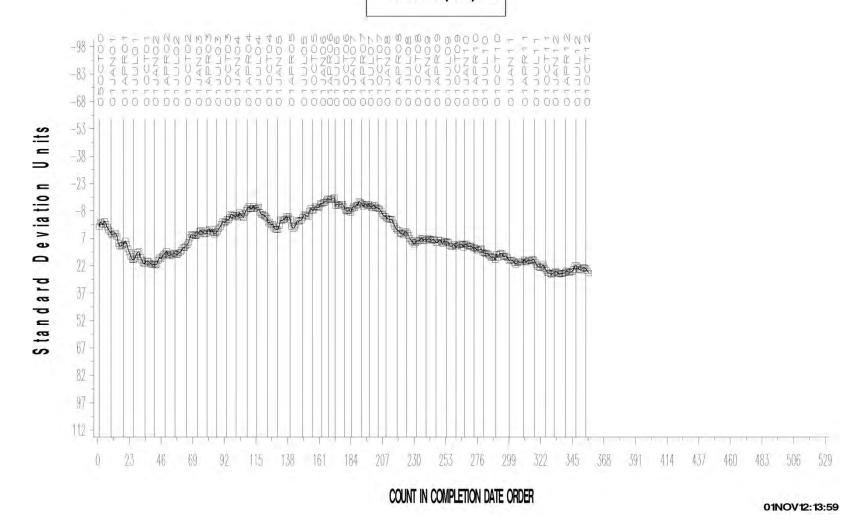
#### **SHIPPING ALIQUOTS**

D6417	1 ml
D6417QC	118 ml
D5800	100 ml
GI	25 ml
MTEOS	17 ml
TEOST	125 ml
D6082	525 ml
D874	32 ml
D874QC	1000 ml
ROBO	300 ml
<b>ROBOQC</b>	1000 ml
H&M	1000 ml
D7563	1000 ml

#### D6417 VOLATILITY BY GC INDUSTRY OPERATIONALLY VALID DATA



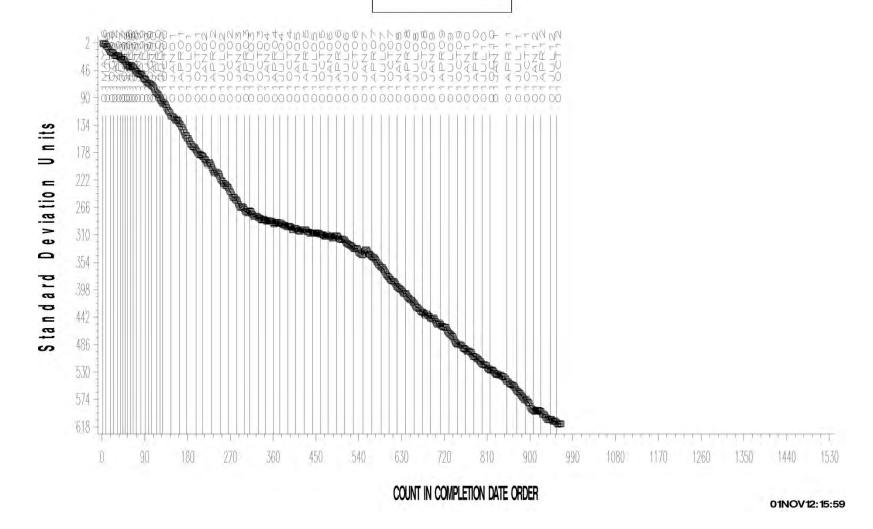
#### **SAMPLE AREA % VOLATIZED**



#### D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA



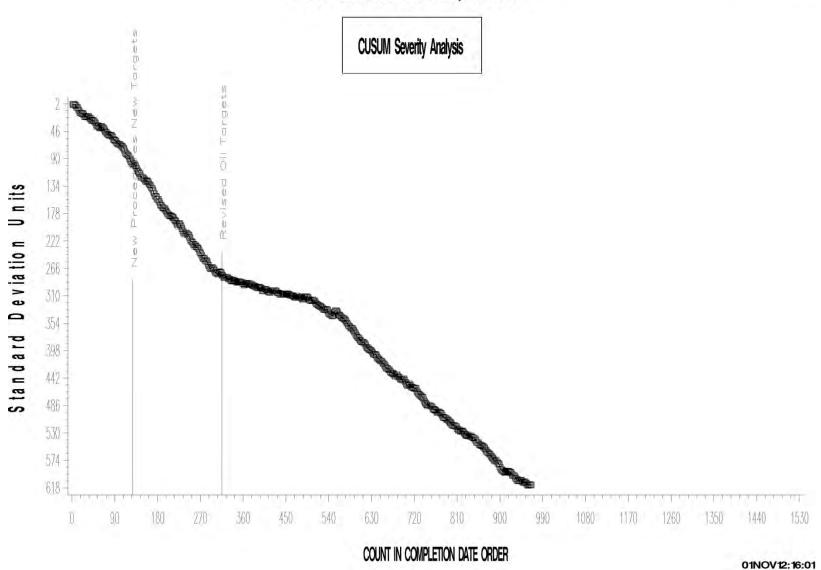
#### **EVAPORATION LOSS, MASS%**



#### D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA



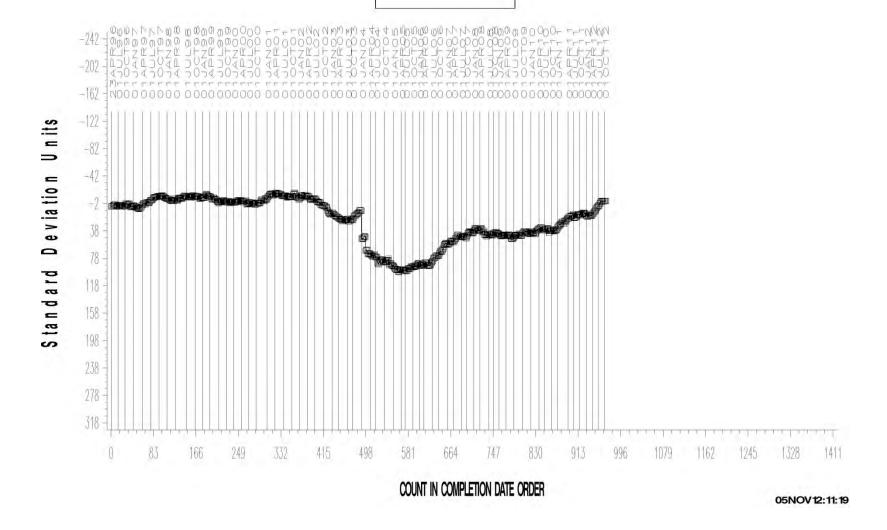




#### D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA



#### **GELATION INDEX**

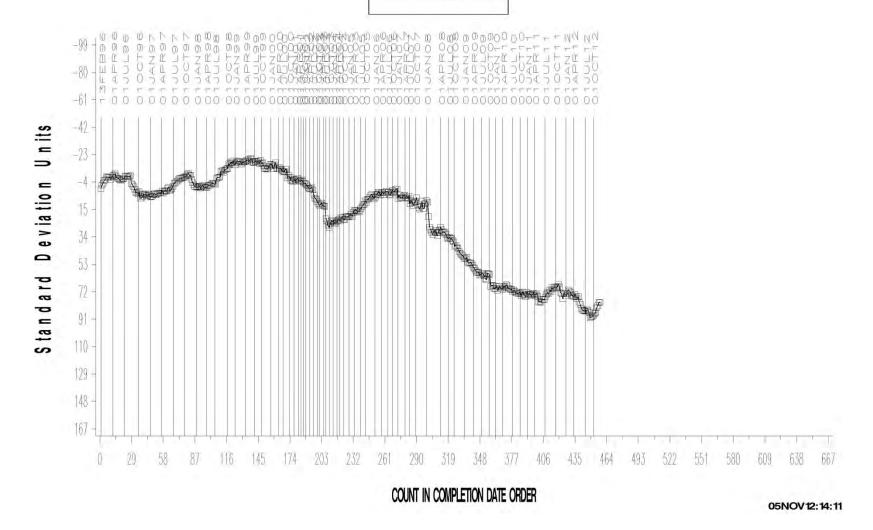


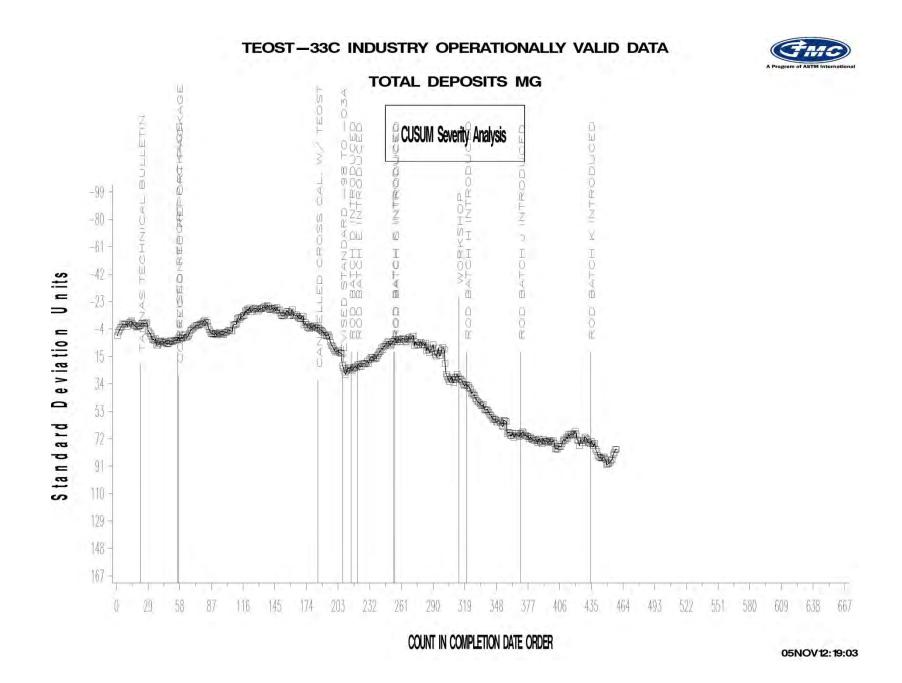
07MAY12:11:03

#### D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA **GELATION INDEX** CUSUM Severity Analysis 10 -236 --197-158-119-80 -41 e v ia tio n 37 -76 -Standard 115-154 -193 -232 271 310 162 243 324 405 567 648 972 1377 486 810 COUNT IN COMPLETION DATE ORDER

# TEOST-33C INDUSTRY OPERATIONALLY VALID DATA TOTAL DEPOSITS MG

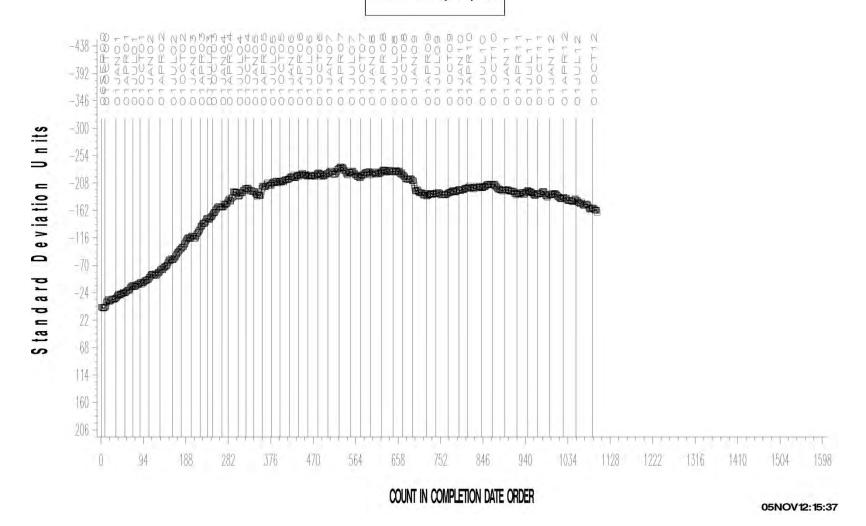


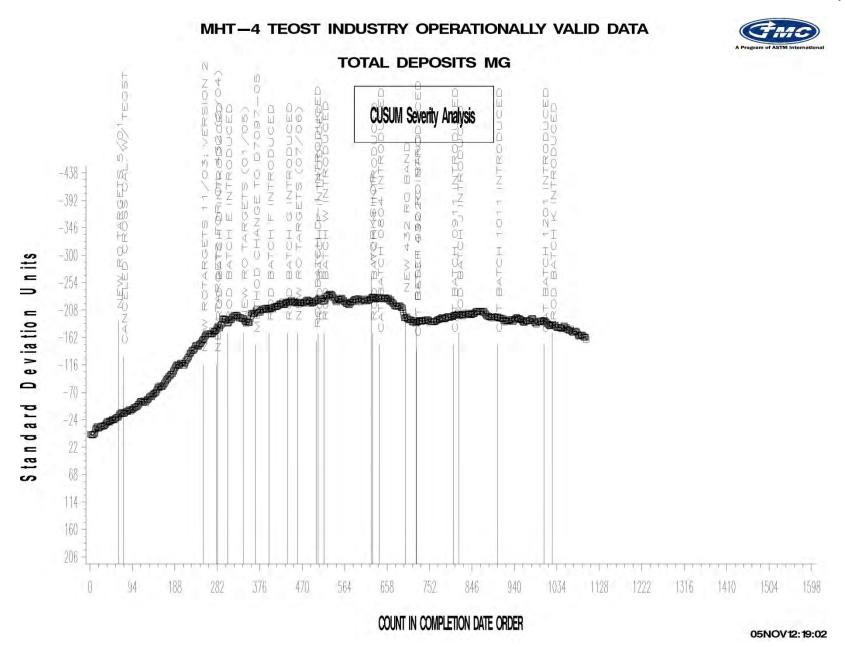




# MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA TOTAL DEPOSITS MG





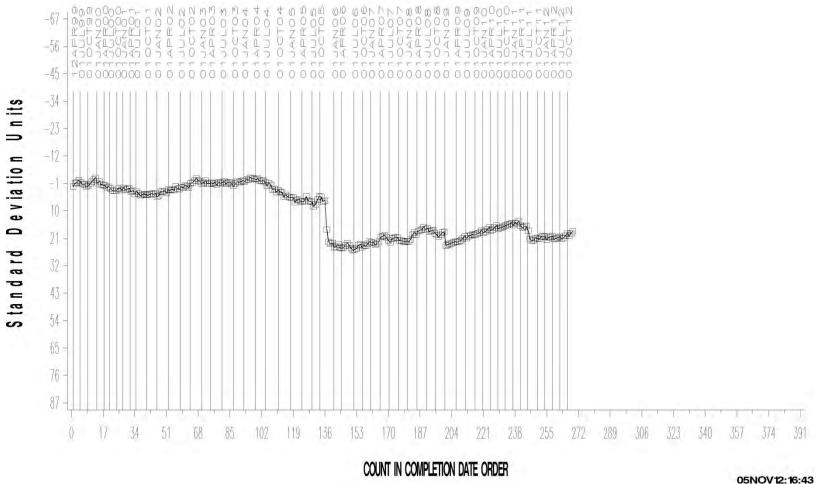


#### D6082 HIGH TEMPERATURE FOAM INDUSTRY OPERATIONALLY VALID DATA IND= 1007



#### Figure 6

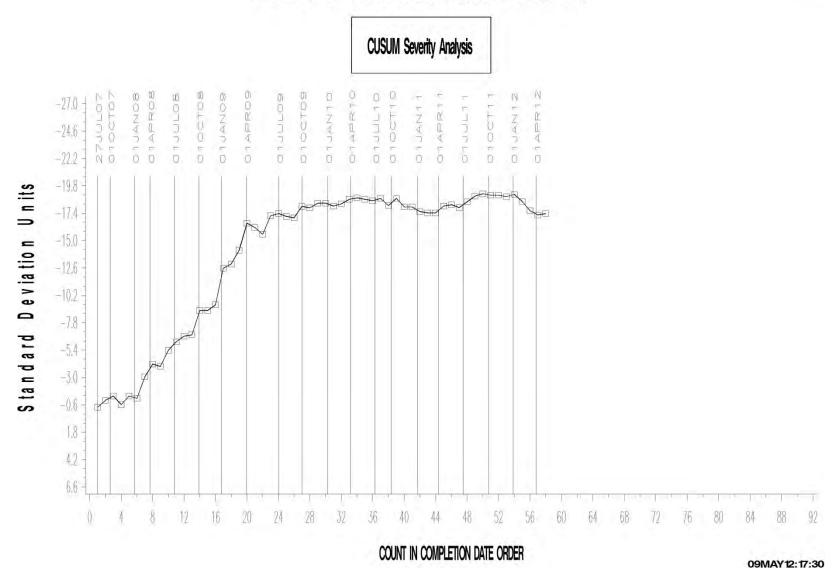
**FOAM TENDENCY** 



#### D874 INDUSTRY OPERATIONALLY VALID DATA



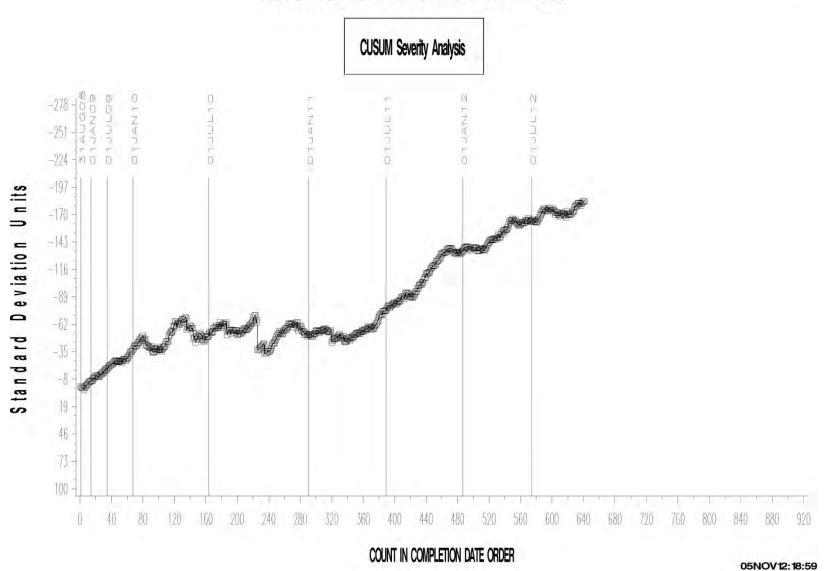
#### TEST SAMPLE PERCENT SULFATED ASH



### ROBO TEST INDUSTRY OPERATIONALLY VALID DATA

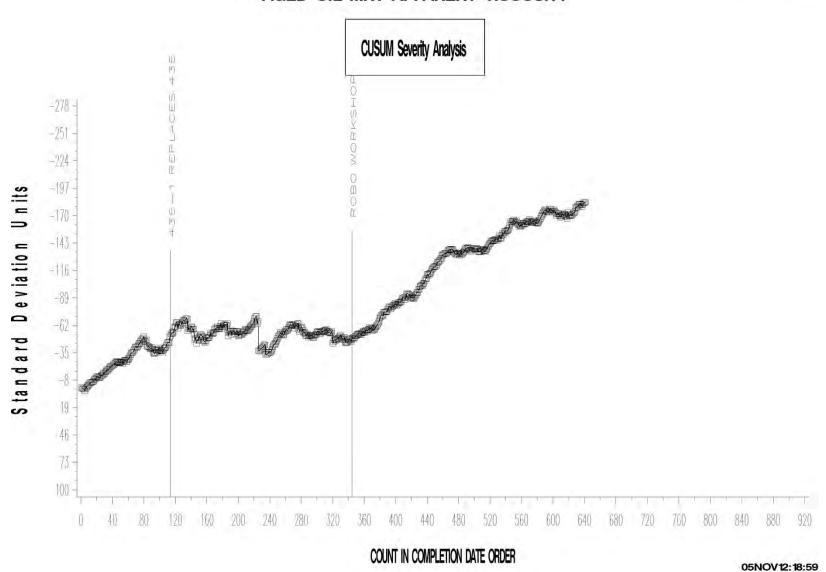


#### AGED OIL MRV APPARENT VISCOSITY









### TMC Monitored Bench Tests – Individual Reference Oil Statistics (Operationally Valid Tests Only)

			Targets 4/1/11 - 9/30/11			10/1/11 - 3/31/12				4/1/12 - 9/30/12							
Test	Oil Code	Parameter	n	Mean	sR	n	Mean	sR	Mean ∆/s	n	Mean	sR	Mean ∆/s	n	Mean	sR	Mean ∆/s
D6417	52	Area % Volatized	18	6.97	0.31	6	7.1	0.37	0.37	3	7.1	0.17	0.42	6	6.9	0.24	-0.12
	55	Area % Volatized	18	11.68	0.51	5	11.7	0.42	0.08	6	11.8	0.27	0.17	4	11.6	0.13	-0.25
	58	Area % Volatized	18	5.61	0.30	5	5.7	0.30	0.17	5	5.6	0.24	0.03	5	5.5	0.38	-0.23
D5800	52	% volatility loss	33	13.75	0.61	17	14.7	0.68	1.52	9	14.7	0.84	1.52	14	14.3	0.73	0.98
	55	% volatility loss	32	17.09	0.76	10	17.4	0.54	0.42	9	17.7	0.78	0.76	12	17.6	0.57	0.66
	58	% volatility loss	37	15.20	0.72	12	15.2	0.50	0.01	14	15.0	0.74	-0.24	7	14.9	0.69	-0.46
TEOST	435-2	Deposit wt. (mg)	15	26.95	2.86					4	30.3	10.19	1.18	10	31.0	3.43	1.43
D6335	71-1	Deposit wt. (mg)		51.79	4.79	9	48.6	7.27	-0.68	1	45.0		-1.42	0			
	72-1	Deposit wt. (mg)		26.72	3.46	7	29.3	5.78	0.73	4	28.5	4.06	0.52	1	31.4		1.35
	75	Deposit wt. (mg)	14	55.16	5.68	1	47.9		-1.28	7	55.5	9.36	0.06	7	54.0	10.35	-0.21
MTEOS	432	Deposit wt. (mg)	30	47.04	4.50	23	49.2	4.96	0.49	29	49.8	5.19	0.61	35	50.9	5.22	0.86
D7097	434	Deposit wt. (mg)	30	27.37	6.57	21	24.0	6.89	-0.51	27	24.3	6.55	-0.46	29	24.4	6.09	-0.45
	74	Deposit wt. (mg)	30	12.85	5.59	2	15.8	7.35	0.53					1	11.9		-0.17
<u>GI</u>	58	Gelation Index	17	5.8	0.69	8	6.0	1.09	0.27	10	6.2	0.69	0.61	8	5.4	0.75	-0.60
D5133	62	Gelation Index	35	17.0	3.90	7	16.3	2.79	-0.17	7	15.4	2.33	-0.40	8	13.4	3.15	-0.92
	1009	Gelation Index	16	7.30	0.68	8	6.72	0.60	-0.85	7	7.1	0.55	-0.25	8	6.5	0.43	-1.14
<u>D6082</u>	1007	Tendency (ml)	28	65	19	9	80	26	0.74	8	65	13	-0.05	9	63	13	-0.14
<u>D874</u>	820-2	Sulfated Ash m%	27	1.57	0.08	2	1.58	0.02	0.06	3	1.59	0.02	0.29	3	1.25	0.52	-3.96
	90	Sulfated Ash m%	27	1.07	0.08	2	1.03	0.00	-0.50					3	1.09	0.02	0.21
	91	Sulfated Ash m%	27	0.82	0.05	2	0.80	0.01	-0.40	3	0.83	0.03	0.20	1	0.81		-0.20
<u>ROBO</u>	434-1	In (MRV Vis)	13	10.6599	0.1672	29	10.5920	0.2124	-0.41	26	10.5899	0.2214	-0.42	26	10.6159	0.2416	-0.26
	435	In (MRV Vis)	15	11.4895	0.2932	17	11.0559	0.3189	-1.48								
	435-1	In (MRV Vis)	22	11.0416	0.20295	32	10.9091	0.2601	-0.65	42	10.6853	0.2191	-0.28	41	10.9835	0.3286	-0.29
	438	In (MRV Vis)	14	10.2676	0.2037	18	10.1724	0.2656	-0.47	25	10.1563	0.1654	-0.55	19	10.1964	0.2950	-0.35

#### **TMC Monitored Bench Tests Reference Oil Test Targets and Acceptance Bands**

## Acceptance Bands \*

						95%			
Test	Oil Code	Parameter	n	Mean	sR	Lower	Upper		
<u>D6417</u>	52	area % volatility loss	18	6.97	0.31	6.4	7.6		
	55	area % volatility loss	18	11.68	0.51	10.7	12.7		
	58	area % volatility loss	18	5.61	0.30	5.0	6.2		
<u>D5800</u>	52	mass % volatility loss	33	13.75	0.61	12.6	14.9		
	55	mass % volatility loss	32	17.09	0.76	15.6	18.6		
	58	mass % volatility loss	37	15.20	0.72	13.8	16.6		
TEOST by	71-1	Total Deposit wt. (mg)	27	51.79	4.79	42.4	61.2		
D6335	72-1	Total Deposit wt. (mg)	27	26.72	3.46	19.9	33.5		
	75	Total Deposit wt. (mg)	14	55.16	5.68	44.0	66.3		
	435-2	Total Deposit wt. (mg)	15	26.95	2.86	21.3	32.6		
MTEOS by	432	Total Deposit wt. (mg)	30	47.04	4.50	38.2	55.9		
D7097	434	Total Deposit wt. (mg)	30	27.37	6.57	14.5	40.2		
GI by	58	Gelation Index	17	5.8	0.69	4.4	7.2		
D5133			35	17.0	3.90	9.4	24.6		
	1009	Gelation Index	16	7.3	0.68	6.0	8.6		
<u>D6082</u>	1007	Tendency (ml)	28	66	19	29	103		
	1007	Stability (ml)	28	0	0	0	0		
D6082	66	Tendency (ml)				>100			
	66	Stability (ml)				0	0		
D874	90	mass % Sulfated Ash	27	1.07	0.08	0.91	1.23		
	91	mass % Sulfated Ash	27	0.82	0.05	0.72	0.92		
	820-2	mass % Sulfated Ash	27	1.57	0.08	1.40	1.73		
ROBO	434-1	In MRV, In(mPa-s)	13	10.6599 (42612)	0.1672	10.3322 (30706)	10.9875 (59130)		
D7528	435	In MRV, In(mPa-s)	15	11.4895 (97685)	0.2932	11.0021 (60000)	12.0642 (173546)		
	435-1	In MRV, In(mPa-s)	22	11.0416 (62420)	0.20295	10.7048 (44570)	11.4394 (92910)		
	438	In MRV, In(mPa-s)	14	10.2676 (28785)	0.2037	9.8683 (19308)	10.6669 (42912)		