

### Test Monitoring Center

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MEMORANDUM: 10-041

DATE: November 3, 2010

TO: Messrs. Ted Selby and Mark Devlin, Co-Chairs ASTM D02.B0.07

FROM: Tom Schofield

SUBJECT: TMC Bench Reference Test Monitoring Semiannual Report

From April 1, 2010, through September 30, 2010, for Test Areas

D6417, D5800, D6335 (TEOST), D7097 (MTEOS), D5133 (GI), D6082,

D874 and D7528 (ROBO)

I respectfully submit the TMC's ASTM D02.B07 Bench Reference Test Monitoring Semiannual Report for Test Areas D6417, D5800, D6335 (TEOST), D7097 (MTEOS), D5133 (GI), D6082 D874 and D7528 (ROBO), with statistical summaries broken down by test area (Attachment 1).

Calibration testing precision and severity are monitored by comparing a recent period of reference test performance to "target" performance (as determined by the surveillance panels), and to performance over previous periods. The TMC monitors test precision by a pooled standard deviation (pooled s), and test severity by mean  $\Delta$ /s ("mean delta over s"), where:

Pooled s = Standard deviation pooled across labs and reference oils (i.e., The pooled precision of the test this period in standard deviations.)

 $\Delta$ /s = [(Single Test Result) - (Reference Oil's Target Mean Performance)] / (R.O.'s Target Precision) (i.e., "How many standard deviations from the target mean is this test?")

Mean  $\Delta/s = [\Sigma (\Delta/s)] / n$  (across reference oils and labs, and over a period of time) (i.e., "On average, how many standard deviations from the target mean are <u>all</u> the operationally valid calibration tests for each period?")

Note that the period severity estimates (mean  $\Delta$ /s) can be averaged across oils of different performance levels because the individual test results used to calculate mean  $\Delta$ /s have all been normalized into standard deviations ( $\Delta$ /s) for each corresponding reference oil. Using a pooled s for estimating precision simplifies the interpretation of precision across all reference oil performance levels. These two calculations (pooled s and mean  $\Delta$ /s) allow all calibration performance levels to be combined into overall period precision and severity estimates for each test type, providing a means to compare current test performance (precision and severity) to target performance and to prior periods. Individual oil targets, and current performance summaries by oil, are also reported (Attachments 2 and 3).

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The tables in Attachment 1, comparing current and previous period precision and severity, have become too large to conveniently show all prior report periods. Older period comparison periods have been eliminated to keep the information succinct and relevant.

The blind lab codes in this report are cross-referenced, as they were in previous reports. That is, in this report, Lab A represents the same lab in each section, which is the same as Lab A in previous reports, and should remain the same lab in future reports.

All operationally valid test data and severity plots are available on the TMC's website. Please contact the TMC if you require further information.

#### Attachments

c: D02.B07 Bench Test Mailing List

F. Farber, TMC

J. Clark, TMC

ftp://ftp.astmtmc.cmu.edu/docs/bench/bo7semiannualreports/mem10-041.pdf

Distribution: Email

### **ASTM Test Monitoring Center**

### **Semiannual Report**

ASTM D02.B07 Bench Reference Test Monitoring From April 1, 2010 through September 30, 2010

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

#### **D6417:** Estimation of Engine Oil Volatility by Capillary Gas Chromatography

#### MONITORED TESTING STATUS

Table 1 summarizes the reference tests reported to the TMC this period (6 labs reporting):

TABLE 1

	No. of Tests
Statistically Acceptable and Operationally Valid	16
Operationally Valid but Failed Acceptance Criteria	0
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	0
Total	16

Fail Rate of Operationally Valid Tests: 0.0%

Table 2 is a breakdown of the statistically unacceptable tests.

TABLE 2

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

#### PRECISION AND SEVERITY

Table 3 shows the current Industry precision and severity for the Sample Area % Volatized @ 371°C test parameter for all operationally valid tests for the report period. (First TMC calibration test completed 10/5/00.)

TABLE 3

Area % Volatized @ 371°C	n	df	Pooled s	Mean Δ/s
Initial Round Robin Study	107	101	0.46	
10/1/06 through 3/31/07	12	9	0.54	-0.17
4/1/07 through 9/30/07	12	9	0.31	0.22
10/1/07 through 3/31/08	14	11	0.29	0.84
4/1/08 through 9/30/08	14	11	0.34	0.54
10/1/08 through 3/31/09	14	11	0.23	-0.10
4/1/09 through 9/30/09	15	12	0.34	0.23
10/1/09 through 3/31/10	13	10	0.33	0.08
4/1/10 through 9/30/10	16	13	0.30	0.41

Table 4 shows the current severity for the Sample Area % Volatized @ 371°C parameter for each lab for all operationally valid tests for the report period.

TABLE 4

	n	Mean Δ/s
Lab A	6	0.99
Lab B	2	0.19
Lab D	2	0.53
Lab G	2	0.03
Lab H	2	0.00
Lab S	2	-0.46

#### D6417: Estimation of Engine Oil Volatility by Capillary Gas Chromatography, continued

#### **INDUSTRY PERFORMANCE**

D6417 reference testing precision, as measured by pooled s, is nearly the same as previous report periods and remains more precise than the target precision (Table 3). Overall performance is severe at 0.41 standard deviations (Table 3), influenced mostly by severe overall performance by lab A (Table 4). Two new instruments were added to the test monitoring system this period. Severity is represented graphically in Figure 1.

#### TMC MEMORANDA

There were no TMC technical updates issued this report period for the D6417 test method.

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

#### **MONITORED TESTING STATUS**

Table 5 summarizes the reference tests reported to the TMC this period (7 labs reporting):

TABLE 5

	No. of Tests
Statistically Acceptable and Operationally Valid	30
Operationally Valid but Failed Acceptance Criteria	4
Operationally Invalid (initially reported as)	1
Operationally Invalid (after informed of failing calibration)	1
Total	36

Fail Rate of Operationally Valid Tests: 11.8%

The explanations for the operationally invalid tests are incorrect orifice size (identified after notification of failing calibration result) and insufficient test sample (aborted test).

Table 6 is a breakdown of the statistically unacceptable tests.

TABLE 6

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

<sup>\*</sup>Severe results were all on oil 52

#### PRECISION AND SEVERITY

Table 7 shows the current Industry precision and severity for the Sample Evaporation Loss test parameter for all operationally valid tests for the report period. (First calibration test completed 5/1/96.)

TABLE 7

Sample Evaporation Loss, mass %	n	df	Pooled s	Mean Δ/s
New Targets Effective 7/21/2003	102	99	0.70	
4/1/07 through 9/30/07	36	33	0.50	0.92
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

Table 8 shows statistical comparisons by procedure for all operationally valid tests for the report period.

TABLE 8

Sample Evaporation Loss, mass %	n	df	Pooled s	Mean ∆/s
Procedure A	0	0		
Procedure B	30	27	0.62	0.80
Procedure C	4	1	0.35	-0.53

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

Table 9 shows the current severity for the Sample Evaporation Loss parameter for each lab for all operationally valid tests for the report period.

TABLE 9

	n	Mean Δ/s
Lab A	4	0.74
Lab B	8	0.32
Lab D	2	-0.21
Lab F	4	0.71
Lab G	7	0.36
Lab I	3	1.44
Lab J	6	1.16

#### **INDUSTRY PERFORMANCE**

D5800 reference testing precision, as measured by pooled s, is similar to the previous period and to the target precision (Table 7). Overall performance remains severe this period with six of seven participating labs performing severe (Table 9). Severity is graphically represented in Figures 2A and 2B. Figure 2A shows a long-term severe trend with an unexplained increase in severity since the 01JUL06 timeline. Oil 52 continues to perform more than 1 s severe (Attachment 3) and all statistically failing test results this period were on oil 52. Since April 1, 2009, eleven of twelve statistically failing tests were on oil 52; all failed severe of acceptance bands.

Table 8 compares the procedures for the period. There were no Procedure A calibration tests reported and four Procedure C calibration tests reported this period.

#### **TMC MEMORANDA**

There was one TMC technical update issued this report period for the D5800 test method:

TMC Memo 10-038, September 10, 2010, Subject: Updated Test Method D5800-10

# <u>D5133:</u> Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature Scanning Technique (Gelation Index or GI)

#### **MONITORED TESTING STATUS**

Table 10 summarizes the reference tests reported to the TMC this period (7 labs reporting):

TABLE 10 Reference Tests

	No. of Tests
Statistically Acceptable and Operationally Valid	21
Operationally Valid but Failed Acceptance Criteria	3
Operationally Invalid (initially reported as)	2
Operationally Invalid (after informed of failing calibration)	0
Total	26

Fail Rate of Operationally Valid Tests: 12.5%

The explanations for the operationally invalid tests are power failure and data acquisition failure, both resulting in aborted runs.

Table 11 is a breakdown of the statistically unacceptable tests.

TABLE 11

Reason for Fail	No. of Tests
Gelation Index Mild	1
Gelation Index Severe	2

#### PRECISION AND SEVERITY

Table 12 shows the current Industry precision and severity for the Gelation Index test parameter for all operationally valid tests for the report period. (First calibration test completed 4/20/96.)

TABLE 12

<b>Gelation Index</b>	n	df	Pooled s	Mean Δ/s
Revised Targets Effective 20030715	68	65	2.86	
(Oils 58 & 62 targets unchanged, added oil				
1009, dropped oils 52 & 53)				
4/1/06 through 9/30/06	29	26	3.76	-0.46
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

## <u>D5133: Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature Scanning Technique (Gelation Index or GI), continued</u>

Table 13 shows the current severity for the Gelation Index for each lab for all operationally valid tests for the report period.

TABLE 13

	n	GI Mean Δ/s
Lab A	7	-0.20
Lab B	4	0.11
Lab D	2	-0.74
Lab G	4	0.32
Lab H	1	3.33
Lab I	3	0.54
Lab S	3	-0.34

#### **INDUSTRY PERFORMANCE**

D5133 reference testing is much less precise, as measured by pooled s, compared to last period and is less precise than the target precision (Table 12). Severity is graphically represented in Figures 3A and 3B (attached) showing nearly on-target performance.

Three tests this period were more than 1 s mild, and five tests were more than 1 s severe, with three tests falling outside the acceptance bands.

#### TMC MEMORANDA

There were no TMC technical updates issued this report period for the D5133 test method.

#### **D6335: High Temperature Deposits by Thermo-Oxidation Engine Oil Simulation Test (TEOST)**

#### **MONITORED TESTING STATUS**

Table 14 summarizes the reference tests reported to the TMC this period (5 labs reporting):

TABLE 14

	No. of Tests
Statistically Acceptable and Operationally Valid	15
Operationally Valid but Failed Acceptance Criteria	1
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	0
Total	16

Fail Rate of Operationally Valid Tests: 6.2%

There were no operationally invalid tests reported this report period.

Table 15 is a breakdown of the statistically unacceptable tests.

TABLE 15

Reason for Fail	No. of Tests
Total Deposits Mild	1
Total Deposits Severe	0

#### PRECISION AND SEVERITY

Table 16 shows the current Industry precision and severity for the Total Deposits test parameter for all operationally valid tests for the report period. (First calibration test completed 2/13/96.)

TABLE 16

		-		
Total Deposits Initial Round Robin Study	n 54	<b>df</b> 52	Pooled s 4.18	Mean Δ/s
10/1/06 through 3/31/07* 10/1/06 through 3/31/07*	12 11	10	8.66 5.67	0.14
4/1/07 through 9/30/07* 4/1/07 through 9/30/07*	10 9	8 7	9.59 8.08	0.43
10/1/07 through 3/31/08 4/1/08 through 9/30/08 10/1/08 through 3/31/09	22 15 18	20 13 16	9.65 6.99 4.90	0.92 0.20 0.98
4/1/09 through 9/30/09* 4/1/09 through 9/30/09*	14 13	10 9	8.24 3.71	0.32 0.68
10/1/09 through 3/31/10* 10/1/09 through 3/31/10*	12 11	8 7	14.36 6.46	0.85 0.18
4/1/10 through 9/30/10	16	12	4.70	0.16

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

#### **D6335: TEOST, continued**

Table 17 shows the current severity for the Total Deposits parameter for each lab for all operationally valid tests in the report period.

TABLE 17

	n	Mean Δ/s
Lab A	4	0.73
Lab B	6	0.24
Lab D	2	-0.34
Lab G	1	1.59
Lab V	3	-0.91

#### **INDUSTRY PERFORMANCE**

Reference testing precision, as measured by pooled s, is significantly improved compared the previous report period but remains less precise than the target precision (Table 16). Performance is only slightly severe at 0.16 s. Severity is graphically represented in Figure 4 (attached) showing an overall severe trend since the 01APR08 timeline, but with more recent leveling since the 01OCT09 timeline. There were no "extreme" test results reported this period, as has been seen in past periods and as indicated in Table 16. The largest deviation for a single result this period was -2.1 s mild of target (the single failing result this period).

Overall, we see a lower fail rate, improved precision, no extreme results and reasonably on-target severity performance for the report period. Last period, calibration testing was significantly worse in all those evaluations (fail rate 25%, precision at 14.6 mg total deposits, one extreme result at 8.2 s severe of target and overall severity performance at 0.85 s severe).

Rod batch J was introduced this period, with seven tests reported on rod batch H and nine tests using rod batch J.

Oils 71-1 and 72-1 were introduced two periods ago. Projected usage rates show those oils being depleted possibly within a year due to increased testing. The oil supplier has provided additional quantities of each oil to the TMC. However, discussions between the panel chair, the TMC and the TMC's suppliers are underway to find suitable replacement reference oils with current formulation technologies, as the existing reference oil technologies are at least 15 years old.

#### **TMC MEMORANDA**

There were no TMC technical updates issued this report period for the D6335 test method.

#### <u>D7097: Determination of Moderately High Temperature Piston Deposits by Thermo-oxidation</u> Engine Oil Simulation Test (MTEOS or MHT-4 TEOST)

#### **MONITORED TESTING STATUS**

Table 18 summarizes the reference tests reported to the TMC this period (7 labs reporting):

TABLE 18

	No. of Tests
Statistically Acceptable and Operationally Valid	52
Operationally Valid but Failed Acceptance Criteria	3
Operationally Invalid (initially reported as)	7
Operationally Invalid (after informed of failing calibration)	4
Operationally Valid but Never Passed Calibration on New	1
Instrument (held out of statistics)	
Total	67

Fail Rate of Operationally Valid Tests: 5.5%

The explanations for the eleven operationally invalid tests are sample leaks (3 tests), temperature control problems (3 tests), using incorrect catalyst weights (4 tests) and power failure (1 test).

Table 19 is a breakdown of the statistically unacceptable tests.

TABLE 19

Reason for Fail	No. of Tests
Total Deposits Mild	1
Total Deposits Severe	2

#### **PRECISION AND SEVERITY**

Table 20 shows the current Industry precision and severity for the Total Deposits test parameter for all operationally valid tests for the report period. (First calibration test completed 9/6/00.)

TABLE 20

<b>Total Deposits</b>	n	df	Pooled s	Mean Δ/s
Updated Targets Effective 7/31/06	90	87	5.62	
10/1/06 through 3/31/07	47	44	7.53	-0.17
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

#### <u>D7097: Determination of Moderately High Temperature Piston Deposits by Thermo-oxidation</u> Engine Oil Simulation Test (MTEOS or MHT-4 TEOST)

Table 21 shows the current severity for the Total Deposits parameter for each lab for all operationally valid tests in the report period.

TABLE 21

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	n	Mean ∆/s		
Lab A	15	-0.88		
Lab AK	2	1.45		
Lab B	18	0.00		
Lab D	7	0.23		
Lab G	11	0.07		
Lab J	1	0.29		
Lab V	1	1.08		

#### **INDUSTRY PERFORMANCE**

D7097 (MTEOS) reference testing overall precision, as measured by pooled s, is more precise compared to the prior report period and compared to the target precision (Table 20). Overall performance this period is only slightly mild of targets.

The D7097 severity is graphically represented in Figures 5A & 5B, with Figure 5B showing when the new performance targets were implemented, when the monitored test method was changed and when new rod and catalyst batches were introduced.

Rod batch J was introduced this period, with 21 operationally valid test reported using rod batch H and 34 using rod batch J. Eleven operationally valid tests were reported using catalyst batch 0902C (all on rod batch H) and the remaining forty-four tests were reported using catalyst batch 0911, which was introduced late in the previous report period (five tests).

By Email ballot, the surveillance panel agreed to phase out TMC reference oil 74. The TMC is permitted to assign any blind coded inventory already shipped for TMC calibration assignments, but the TMC has stopped shipping any additional samples of oil 74 as directed by the panel. There were 13 operationally valid tests reported on oil 74 this period and there are only a few samples of oil 74 remaining throughout the industry.

#### TMC MEMORANDA

There were no TMC technical updates issued this report period for the D7097 test method.

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

#### **MONITORED TESTING STATUS**

Table 22 summarizes the reference tests reported to the TMC this period (3 labs reporting):

TABLE 22

	No. of Tests
Statistically Acceptable and Operationally Valid	8
Operationally Valid but Failed Acceptance Criteria	0
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	1
Total	9

Fail Rate of Operationally Valid Tests: 0.0%

The reason for the operational fail was that the blending option A was not performed (test aborted). In addition to the calibration tests, there were three discrimination oil tests reported this period; all met the acceptance criteria for the discrimination oil.

#### TMC 1007 PRECISION AND SEVERITY

Tables 23 and 24 show the current industry precision and severity for the Foam Tendency and Foam Stability test parameters for all operationally valid tests on oil 1007 for the report period. (First calibration test on TMC 1007 completed 4/12/99.)

TABLE 23

1007 Foam Tendency, ml	n	Mean	S	Mean ∆/s
Initial Round Robin Study (targets)	28	65.71	19.28	
4/1/07 through 9/30/07	10	63	18	-0.16
10/1/07 through 3/31/08	10	64	16	-0.13
4/1/08 through 9/30/08	10	65	16	-0.05
10/1/08 through 3/31/09*	11	72	34	0.31
10/1/08 through 3/31/09*	10	62	10	-0.21
4/1/09 through 9/30/09	10	61	10	-0.26
10/1/09 through 3/31/10	8	59	10	-0.38
4/1/10 through 9/30/10	8	65	16	-0.05

<sup>\*</sup>Period statistics with and without extreme results included.

#### D6082: High Temperature Foaming Characteristics of Lubricating Oils, continued

Note that TMC reference oil 1007 has a Foam Stability (one minute after disconnect) target mean performance of zero ml and a target precision (standard deviation) of zero ml. A negative (mild) result for this parameter is unlikely and a severity estimate for any positive result would be indeterminate in standard deviations ( $\Delta$ /s). Therefore, for Foam Stability, only a count of non-zero occurrences is noted to flag any severity trends.

TABLE 24

1007 Foam Stability @ 1 min., ml	n	Mean	S	
Initial Round Robin Study	28	0.00	0.00	
4/1/07 through 9/30/07	10	No non-zero	occurrences	
10/1/07 through 3/31/08	10	No non-zero	occurrences	
4/1/08 through 9/30/08	10	No non-zero	occurrences	
10/1/08 through 3/31/09	11	No non-zero	occurrences	
4/1/09 through 9/30/09	10	No non-zero	occurrences	
10/1/09 through 3/31/10	8	No non-zero	occurrences	
4/1/10 through 9/30/10	8	No non-zero	occurrences	

Table 25 shows the current 1007 severity for the monitored result parameter for each lab for all operationally valid tests reported for the report period.

TABLE 25 TMC 1007

		Foam Tendency
	n	Mean ∆/s
Lab A	2	1.26
Lab B	4	-0.58
Lab G	2	-0.32

#### **INDUSTRY PERFORMANCE**

The D6082 Foam Tendency precision, as measured by standard deviation (s) on TMC oil 1007, is less precise than last report period but more precise than the target precision (Table 23). Overall Foam Tendency performance is on target. There were no non-zero occurrences of Foam Stability on 1007 suggesting Foam Stability precision is as expected. Foam Tendency severity is graphically represented in Figure 6.

All operationally valid discrimination tests reported this period meet the acceptance criteria (that is, all reporting labs could discriminate oil 66 as a GF-4/SM failing oil for Foam Tendency).

#### TMC MEMORANDA

There were no TMC technical updates issued this report period for the D6082 test method.

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

#### **MONITORED TESTING STATUS**

Table 26 summarizes the reference tests reported to the TMC this period (3 labs reporting):

TABLE 26 Reference Tests

	No. of Tests
Statistically Acceptable and Operationally Valid	5
Operationally Valid but Failed Acceptance Criteria	0
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	0
Total	5

Fail Rate of Operationally Valid Tests: 0.0%

Table 27 is a breakdown of the statistically unacceptable tests.

TABLE 27

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

#### PRECISION AND SEVERITY

Table 28 shows the current Industry precision and severity for the Sulfated Ash Mass % test parameter for all operationally valid tests for the report period. (First calibration test completed 7/27/07.)

TABLE 28

<b>Gelation Index</b>	n	df	Pooled s	Mean Δ/s
Initial Round Robin Targets	81	79	0.07	
4/1/07 through 9/30/07	2	1	0.01	-0.50
10/1/07 through 3/31/08	5	2	0.11	-0.41
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

Table 29 shows the current severity for Sulfated Ash Mass % for each lab for all operationally valid tests for the report period.

**TABLE 29** 

	n	Mean Δ/s
Lab A	2	0.38
Lab B	1	0.12
Lab G	2	-0.16

#### D874 Sulfated Ash from Lubricating Oils and Additives, continued

#### **INDUSTRY PERFORMANCE**

D874 precision, as measured by pooled s, is more precise than the target precision and performance is slightly severe of targets. Severity is graphically represented in Figure 7.

#### TMC MEMORANDA

There were no TMC technical updates issued this report period for the D874 test method.

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

#### **MONITORED TESTING STATUS**

Table 30 summarizes the reference tests reported to the TMC this period (7 labs reporting):

TABLE 30 Reference Tests

	No. of Tests
Statistically Acceptable and Operationally Valid	78
Operationally Valid but Failed Acceptance Criteria	36
Operationally Invalid (initially reported as)	17
Operationally Invalid (after informed of failing calibration)	1
Operationally Valid but Never Passed Calibration on New	10
Instrument (held out of statistics)	
Total	142

Fail Rate of Operationally Valid Tests: 31.6%

The explanations for the eighteen operationally invalid tests are:

- Vacuum system leaks or failures (six tests)
- Airflow not in specification or restricted airflow (four tests)
- Reactor temperature control problems (four tests)
- Problems with NO<sub>2</sub> flow (one test)
- Sample stirrer failure (two tests)
- Power failure (one test)

Table 31 is a breakdown of the statistically unacceptable calibration tests.

TABLE 31

TIBLE 31				
Reason for Fail	No. of Tests			
MRV Viscosity Mild	25			
MRV Viscosity Severe	11			

#### D7528 Bench Oxidation of Engine Oils by ROBO Apparatus, continued

#### PRECISION AND SEVERITY

Table 32 shows the current Industry precision and severity for the transformed MRV viscosity test parameter for all operationally valid tests for the report period.

TABLE 32

Natural Log (MRV Viscosity)	n	df	Pooled s	Mean Δ/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

Table 33 shows the current severity for the transformed MRV Viscosity for each lab for all operationally valid tests for the report period.

TABLE 33

	n	Mean Δ/s
Lab A	30	-0.28
Lab AM	11	-1.34
Lab AN	5	1.38
Lab B	17	-0.21
Lab D	8	-2.86
Lab G	37	0.24
Lab Q	6	0.84

#### **INDUSTRY PERFORMANCE**

ROBO precision, as measured by pooled s, is much less precise than the target precision and performance is slightly mild of targets at -0.26 s (Table 32). Severity is graphically represented in Figure 8. Results on tests reported as operatiaonally valid were highly variable this period. A breakdown of Mean  $\Delta$ /s values more than three standard deviations from target for tests reported as operationally valid is as follows:

3-4 s from target (mild and severe): 8 tests
4-5 s from target (severe): 2 tests
5-6 s from target (mild and severe): 4 tests
10-11 s from target (severe) 1 test
12-13 s from target (severe) : 1 test

#### D7528 Bench Oxidation of Engine Oils by ROBO Apparatus, continued

On April 8, 2010 the panel agreed to replace reference oil 435 (nearly depleted) with a reblend, 435-1, with new targets and acceptance bands. However, the 435-1 aged oil MRV performance is generally considered to be milder than is optimal. Because of this, the TMC has been asked to pursue another reblend, preferably with somewhat more severe mean performance on the aged oil MRV. The reblend, 435-2, has been received by the TMC and is presently undergoing quality assurance processing.

#### **TMC MEMORANDA**

There were no TMC technical updates issued this report period for the D7528 test method.

#### **D6922 Determination of Homogeneity and Miscibility in Automotive 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.

#### **REFERENCE OIL SUPPLIES**

There is adequate supply of B0.07 Bench Test reference oils on hand at the TMC. Tables 34A - 34C list the bench test reference oils currently on hand at the TMC.

Table 34A Current Reference Oils

	Current Reference Ons				
Oil	For Tests	Quantity Left (gallons)	Quantity Used Last 12 Months (gallons)		
52	D6417, D5800	63.9	0.7		
55	D6417, D5800	69.0	0.7		
58	D6417, D5800, GI	120.1	0.8		
62	GI	1.8	0.2		
66	D6082 (Discrimination)	94.4	1.1		
71-1	TEOST	1.0	0.6		
71-2	TEOST	3.0	0.0		
72-1	TEOST	0.5	0.7		
72-1	TEOST	3.0	0.0		
90	D874 & D874 Daily Check	38.9	5.0		
91	D874	4.6	0.1		
**432	MTEOS	Adequate			
**434	MTEOS	Adequate			
**820-2	D874	Adequate			
*1007	D6082	Est. 20			
**1009	GI	Adequate			
*434-1	ROBO	Adequate			
*435-1	ROBO	Adequate			
*435-2	ROBO	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>Five gallon aliquot set aside for bench testing; hard to get an inventory reading on amount set aside.

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

Table 34B
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	
72	Obsolete TEOST	2 Samples	
74	Obsolete MTEOS	0.2	0.1
^83	Obsolete ROBO (RR)	47.3	0.0
^84	Obsolete ROBO (RR)	3.3	0.0
^85	Obsolete ROBO (RR)	3.3	0.0
^**433	Obsolete MTEOS	Adequate	
435	Obsolete ROBO	13 Samples	

<sup>^</sup>Not selected as reference oil; TMC holding for further instructions from Surveillance Panel.

Table 34C Homogeneity and Miscibility Reference Oils

Oil	For Tests	Quantity Left (gallons)	Quantity Used Last 12 Months (gallons)
HMA	H&M (D6922)	176.5	6.5
HMB	H&M (D6922)	180.5	6.5
НМС	H&M (D6922)	166.5	6.5
HMD	H&M (D6922)	174.3	6.7
HME	H&M (D6922)	160.0	6.0
HMF	H&M (D6922)	182.8	6.0

<sup>\*\*</sup>Five gallon aliquot set aside for bench testing; hard to get an inventory reading on amount set aside.

#### Shipping aliquots are:

D6417	1 ml
D6417QC	118 ml
D5800	100 ml
GI	25 ml
MTEOS	17 ml
TEOST	125 ml
D6082	525 ml
D874	32 ml
ROBO	300 ml
ROBOQC	950 ml
H&M	950 ml

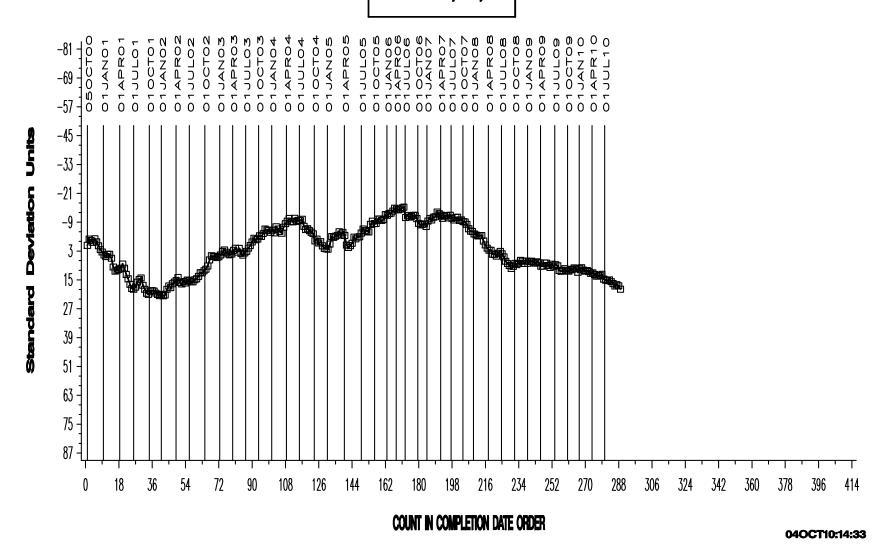
#### **MISCELLANEOUS**

The TMC posts monitored bench test calibration data on the TMC web sit. Lab identifications are coded on the TMC's web site as they are on the previous pages of this report. Also posted are statistics, CUSUM plots, reporting forms, flatfile templates, data dictionaries and data from various round-robin matrix programs. The TMC encourages all interested parties to access and download the data, statistics and plots for individual studies and analyses. Likewise, you are encouraged to access the web site to download the most recent test reporting formats and data dictionaries. The TMC's web site address is <a href="https://www.astmtmc.cmu.edu">www.astmtmc.cmu.edu</a>.

All currently monitored bench test data dictionaries and report form packages have been beta tested by the ASTM Data Communications Committee (DCC) and approved for electronic data transfer. Please contact Tom Schofield at (412) 365-1011 for more information.

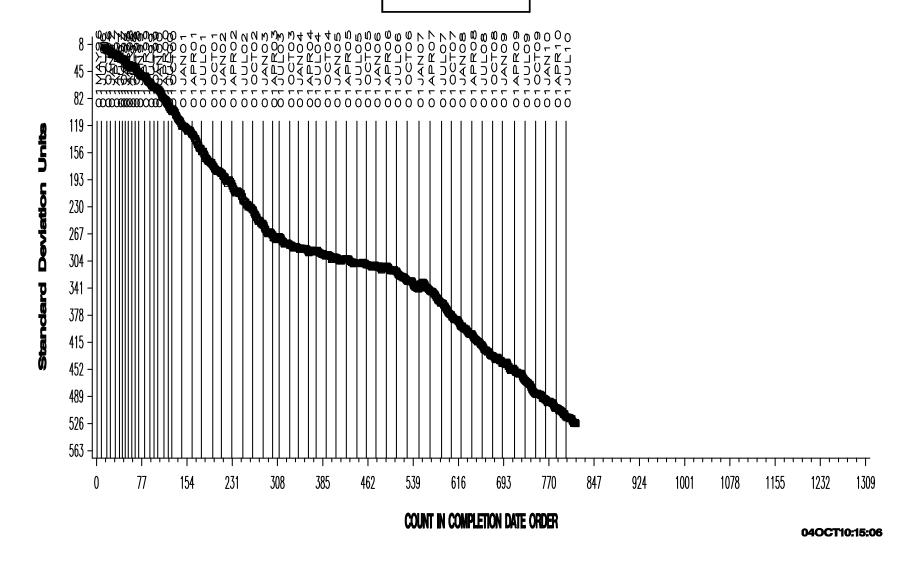
# D6417 VOLATILITY BY GC INDUSTRY OPERATIONALLY VALID DATA SAMPLE AREA % VOLATIZED

**CUSUM Severity Analysis** 



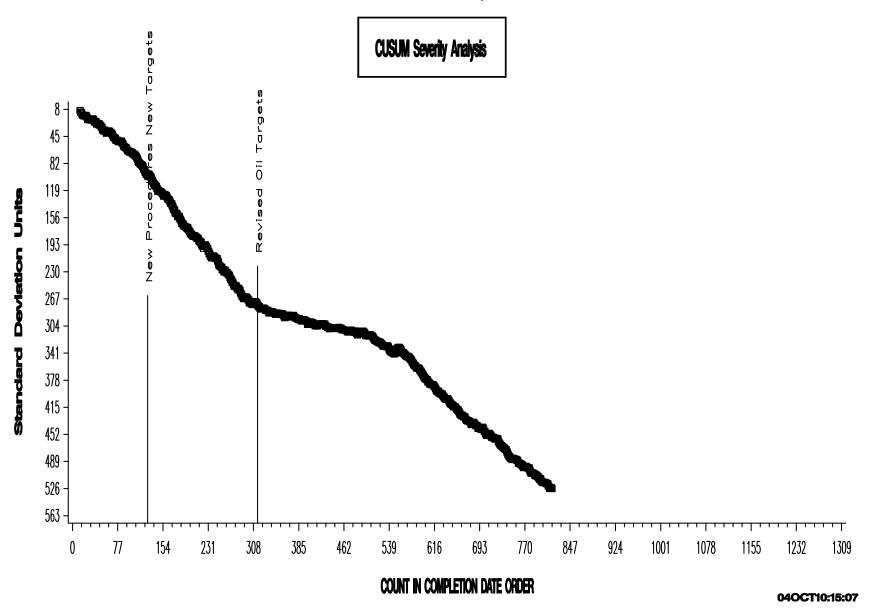
# D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA EVAPORATION LOSS, MASS%

**CUSUM Severity Analysis** 



### D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA

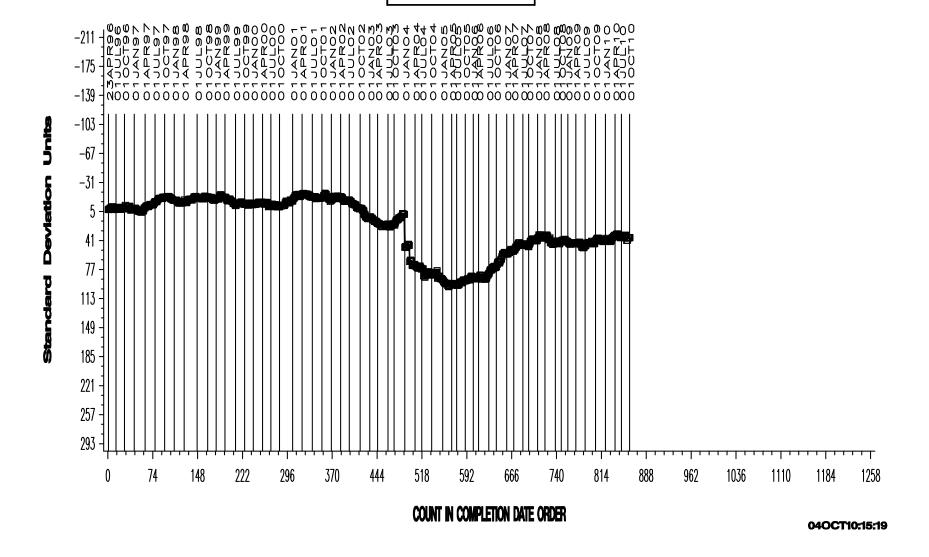
### **EVAPORATION LOSS, MASS%**



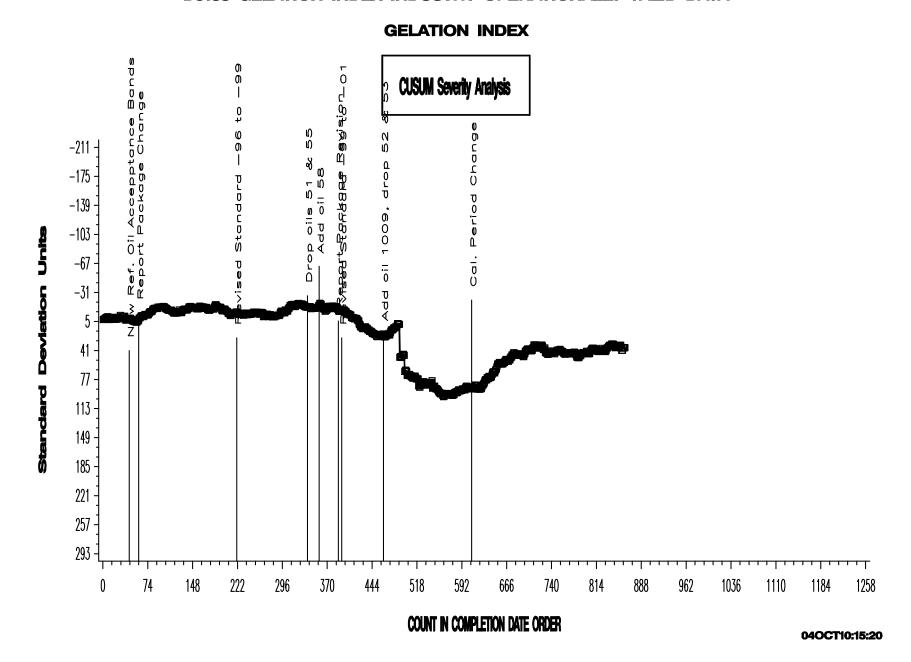
## D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA

#### **GELATION INDEX**

**CUSUM Severity Analysis** 

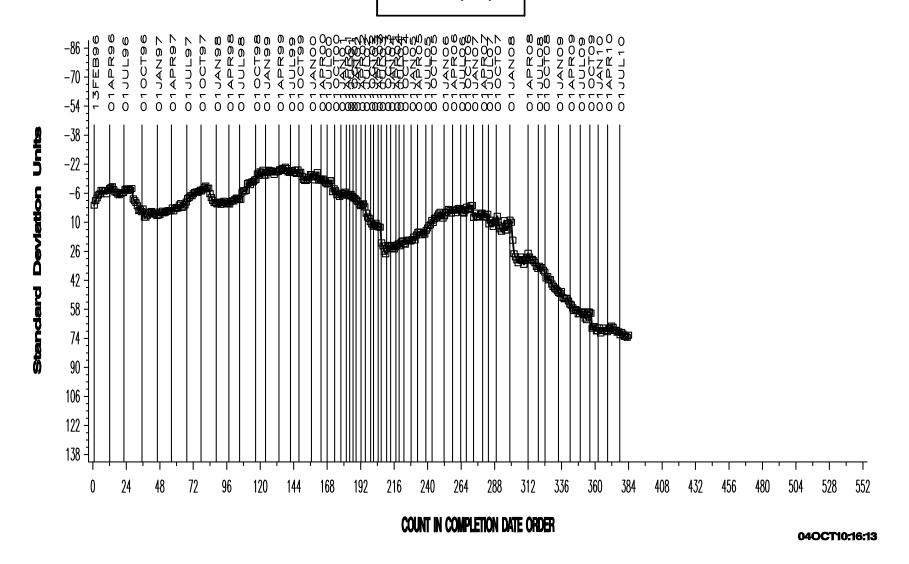


#### D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA



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

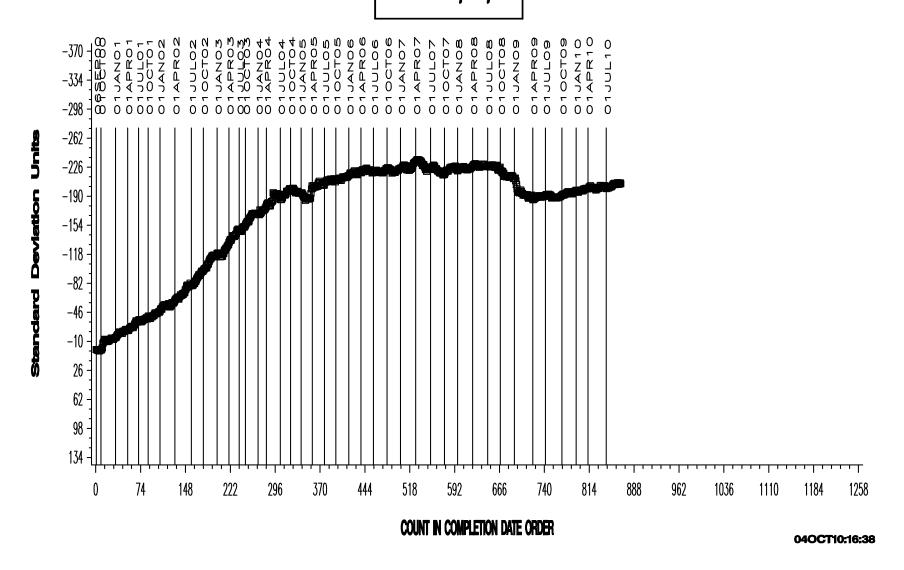
**CUSUM Severity Analysis** 



## MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA

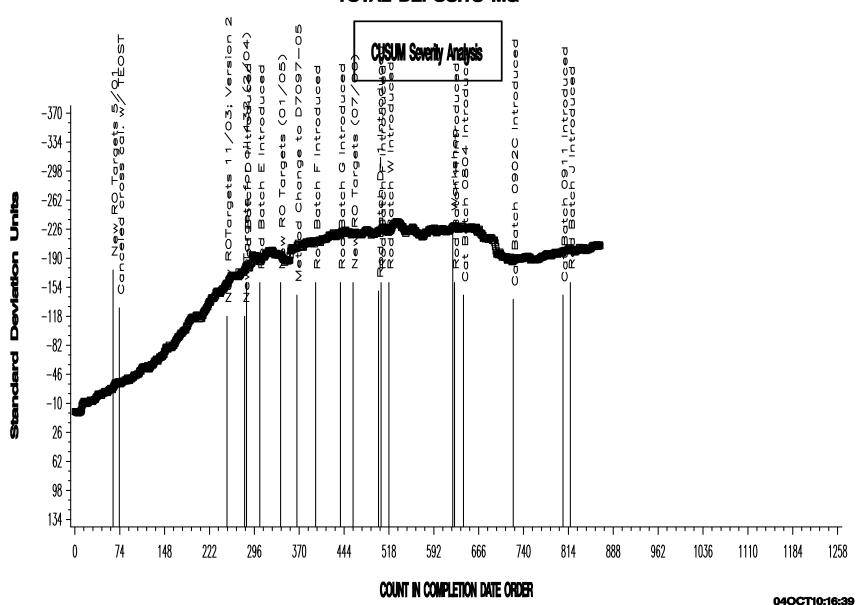
#### **TOTAL DEPOSITS MG**

## **CUSUM Severity Analysis**



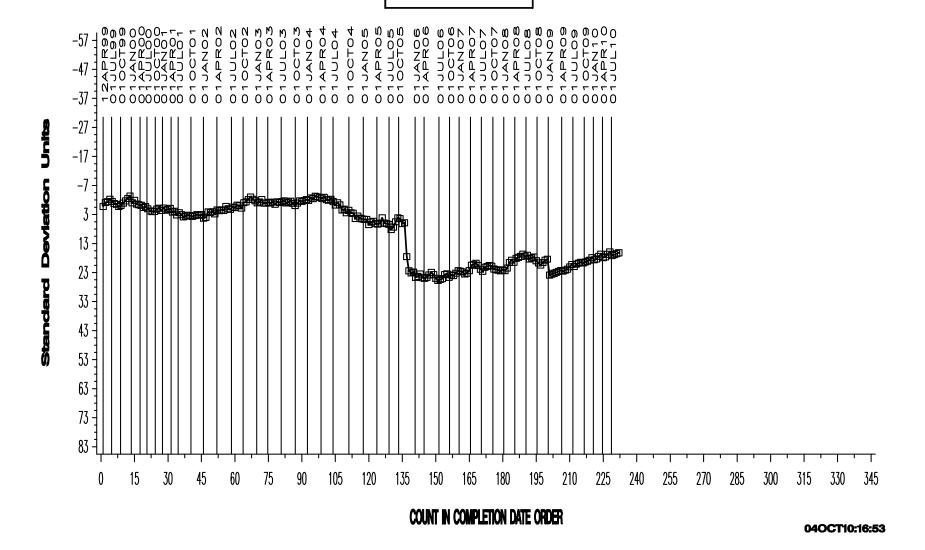
#### MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA

#### **TOTAL DEPOSITS MG**



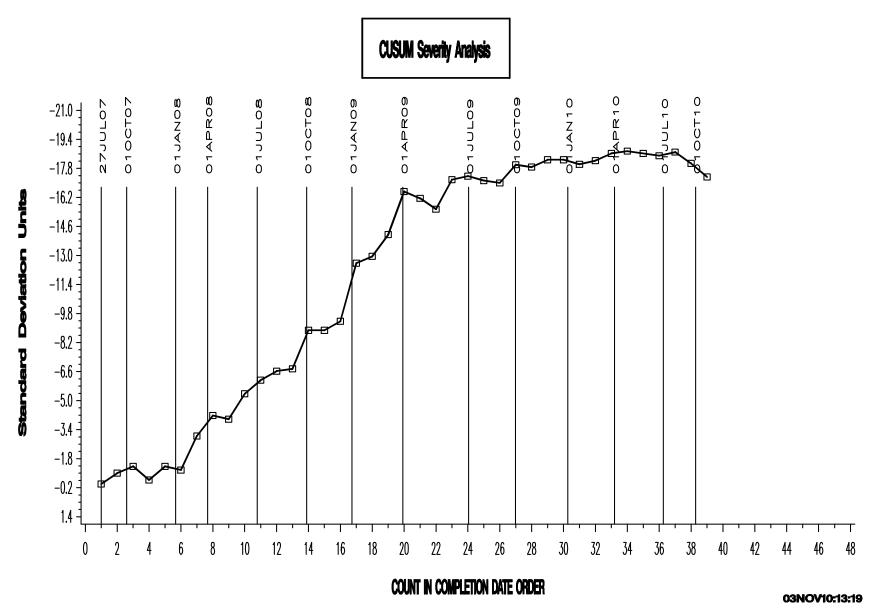
# D6082 HIGH TEMPERATURE FOAM INDUSTRY OPERATIONALLY VALID DATA IND=1007 FOAM TENDENCY

**CUSUM Severity Analysis** 

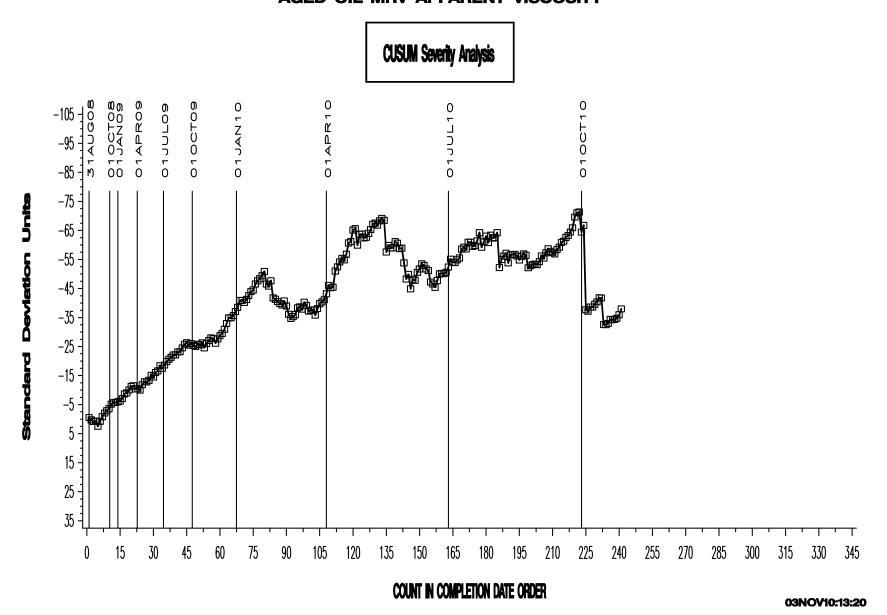


# D874 INDUSTRY OPERATIONALLY VALID DATA

#### TEST SAMPLE PERCENT SULFATED ASH



# ROBO TEST INDUSTRY OPERATIONALLY VALID DATA AGED OIL MRV APPARENT VISCOSITY



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

## Acceptance Bands \*

						95%			
Test	Oil Code	Parameter	n	Mean	sR	Lower	Upper		
D6417	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		
D5800	52	mass % volatility loss	33	13.75 0.61		12.6	14.9		
	55	mass % volatility loss	32	2 17.09 0.76		15.6	18.6		
	58	mass % volatility loss	37	15.20	0.72	13.8	16.6		
TEOST by	71	Total Deposit wt. (mg)	27	51.79	4.79	42.4	61.2		
D6335	71-1	Total Deposit wt. (mg)	27			42.4	61.2		
	72	Total Deposit wt. (mg)	27	26.72	3.46	19.9	33.5		
	72-1	Total Deposit wt. (mg)	27	26.72	3.46	19.9	33.5		
MTEOS by	74	Total Deposit wt. (mg)	30	12.85	5.59	1.9	23.8		
D7097	432	Total Deposit wt. (mg)	30	47.04	47.04 4.50		2 55.9		
	434	Total Deposit wt. (mg)	30	27.37	27.37 6.57		40.2		
GI by	58	Gelation Index	17	5.8	0.69	4.4	7.2		
D5133	62	Gelation Index	35	17.0	3.90 9.4		24.6		
	1009	Gelation Index	16	7.3	0.68 6.0		8.6		
D6082	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	35 In MRV, In(mPa-s)		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)		

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

			Targets			4/1/09 - 9/30/09				10/1/09 - 3/31/10				4/1/10 - 10/30/10			
	Oil								Mea n				Mea n				Mean
Test	Code	Parameter	n	Mean	sR	n	Mean	sR	∆/s	n	Mean	sR	∆/s	n	Mean	sR	∆/s
D6417	52	Area % Volatized	18	6.97	0.31	4	7.0	0.17	0.18	5	6.9	0.27	-0.16	4	7.1	0.14	0.42
	55	Area % Volatized	18	11.68	0.51	4	12.0	0.39	0.73	6	11.8	0.38	0.20	7	11.9	0.37	0.43
	58	Area % Volatized	18	5.61	0.30	7	5.6	0.37	-0.03	2	5.7	0.28	0.30	5	5.7	0.29	0.37
D5800	52	% volatility loss	33	13.75	0.61	16	14.6	0.50	1.32	12	14.4	0.79	1.13	15	14.4	0.71	1.07
**	55	% volatility loss	32	17.09	0.76	15	17.5	0.63	0.59	9	17.5	0.61	0.60	9	17.4	0.63	0.45
	58	% volatility loss	37	15.20	0.72	5	15.4	0.53	0.31	14	15.2	0.65	0.05	10	15.3	0.66	0.17
TEOST	71	Deposit wt. (mg)	27	51.79	4.79	6	52.4	11.51	0.13	4	55.9	9.40	0.86	3	53.8	6.91	0.43
D6335	71-1	Deposit wt. (mg)		51.79	4.79	1	46.4		-1.13	3	61.1	26.11	1.94	4	54.2	3.42	0.51
	72	Deposit wt. (mg)	27	26.72	3.46	4	29.8	2.22	0.89	2	24.8	3.61	-0.54	1	28.5		0.51
	72-1	Deposit wt. (mg)		26.72	3.46	3	28.2	0.85	0.42	3	29.1	2.15	0.68	8	26.2	4.39	-0.16
MTEOS	432	Deposit wt. (mg)	30	47.04	4.50	19	48.4	5.16	0.29	12	45.7	4.07	-0.29	20	50.8	4.19	0.83
D7097	434	Deposit wt. (mg)	30	27.37	6.57	13	26.1	4.05	-0.20	17	27.6	7.32	0.04	22	23.1	5.50	-0.65
***	74	Deposit wt. (mg)	30	12.85	5.59	16	10.5	3.42	-0.42	14	10.8	3.45	-0.37	13	9.0	2.23	-0.69
GI	58	Gelation Index	17	5.8	0.69	14	5.9	0.98	0.19	11	6.1	0.90	0.38	8	6.2	0.85	0.58
D5133	62	Gelation Index	35	17.0	3.90	11	16.6	4.68	-0.09	8	14.3	4.59	-0.70	9	16.5	6.23	-0.14
****	1009	Gelation Index	16	7.30	0.68	8	6.9	0.40	-0.62	12	7.1	0.49	-0.28	7	7.2	0.49	-0.08
D6082	1007	Tendency (ml)	28	65	19	10	61	10	-0.26	8	59	10	-0.38	8	65	16	-0.05
D874	820-2	Sulfated Ash m%	27	1.57	0.08	3	1.59	0.01	0.25	1	1.57		0.00	3	1.59	0.03	0.21
	90	Sulfated Ash m%	27	1.07	0.08	2	0.91	0.04	-2.00	3	1.05	0.06	-0.21	1	1.08		0.12
	91	Sulfated Ash m%	27	0.82	0.05	2	0.83	0.03	0.20	3	0.80	0.01	-0.33	1	0.81		-0.20
ROBO	434-1	In (MRV Vis)	13	10.6599	0.1672	7	10.5242	0.1288	-0.81	11	10.7927	0.2007	0.79	26	10.6193	0.3449	-0.24
	435	In (MRV Vis)	15	11.4895	0.2932	11	11.3302	0.1960	-0.54	43	11.3003	0.4272	-0.65	7	10.9061	0.6195	-1.99
	435-1	In (MRV Vis)	22	11.0416	0.20295									61	11.0385	0.5042	-0.02
	438	In (MRV Vis)	14	10.2676	0.2037	8	10.1778	0.2144	-0.44	5	10.4582	0.4591	0.94	20	10.1871	0.6677	-0.40