

Test Monitoring Center

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MEMORANDUM: 11-043

DATE: November 3, 2011

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, 2011 through September 30, 2011, 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 Δ /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.)

 Δ /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 Δ /s) can be averaged across oils of different performance levels because the individual test results used to calculate mean Δ /s have all been normalized into standard deviations (Δ /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 Δ /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/b07semiannualreports/semiannualreports/mem11-043.pdf

Distribution: Email

ASTM Test Monitoring Center

Semiannual Report

ASTM D02.B07 Bench Reference Test Monitoring From April 1, 2011 through September 30, 2011

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%

There were no operationally invalid tests reported this period.

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	
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
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

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	4	0.90
Lab B	2	-0.02
Lab D	4	-0.54
Lab G	2	-0.31
Lab H	2	0.17
Lab S	2	1.13

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

INDUSTRY PERFORMANCE

D6417 reference testing precision, as measured by pooled s, is comparable to the previous report period and remains more precise than the target precision (Table 3). Overall performance is severe at 0.21 standard deviations (Table 3). Severity is graphically represented in Figure 1 showing a slight severe bias, overall, since at least the OCT08 timeline.

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 (8 labs reporting):

TABLE 5

	No. of Tests
Statistically Acceptable and Operationally Valid	33
Operationally Valid but Failed Acceptance Criteria	6
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	0
Total	39

Fail Rate of Operationally Valid Tests: 15.4%

There were no operationally invalid tests reported this period.

Table 6 is a breakdown of the statistically unacceptable tests.

TABLE 6

Reason for Fail	No. of Tests
Sample Evaporation Loss Severe	6
Sample Evaporation Loss Mild	0

^{*}All severe failing results were on TMC 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
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

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 Δ/s
Procedure A	0	0		
Procedure B	35	32	0.52	0.90
Procedure C	4	1	0.21	-0.31

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/ A/
	n	Mean Δ/s
Lab A	5	0.55
Lab B	11	0.94
Lab D	2	-0.56
Lab F	4	0.39
Lab G	8	1.20
Lab H	2	0.01
Lab I	3	0.65
Lab J	4	1.27

INDUSTRY PERFORMANCE

D5800 reference testing precision, as measured by pooled s, is more precise than the previous period and more precise than the target precision (Table 7). Overall performance is more severe this period with six of eight participating labs performing severe at some level (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). All six statistically failing results this period were severe, and all were on oil 52 (but also with eleven passing results on oil 52; three of those were mild of targets, and two of those mild results were procedure C runs). Since April 1, 2009, nineteen of twenty-one 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 were no TMC technical updates issued this report period for the D5800 test method.

D5133: 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 (6 labs reporting):

TABLE 10 Reference Tests

	No. of Tests
Statistically Acceptable and Operationally Valid	21
Operationally Valid but Failed Acceptance Criteria	2
Operationally Invalid (initially reported as)	1
Operationally Invalid (after informed of failing calibration)	0
Total	24

Fail Rate of Operationally Valid Tests: 8.7%

The explanation for the one operationally invalid test is the assigned reference oil was run on the wrong instrument.

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	1

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

Gelation Index	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)				
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

<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	-0.44
Lab B	4	-0.90
Lab G	2	-1.18
Lab H	1	0.51
Lab I	3	-0.10
Lab S	6	0.51

INDUSTRY PERFORMANCE

D5133 reference testing is significantly more precise than last period, as measured by pooled s, and more precise than the target precision (Table 12). In fact, precision this period is more precise than any prior report period since the TMC began monitoring this test in 1996. Severity is mild at -0.25 s. Severity is graphically represented in Figures 3A and 3B (attached) showing a shift to mild since the JAN11 timeline.

Last period had a fail rate on operationally valid tests of 21%, a pooled s precision of 3.17 (GI units), and a mean severity of -0.53 s mild. This period shows marked improvements in all of those markers. There were 10 fewer tests (29%) this period. There were no GI tests reported from lab D this period, and one from Lab H, which did not report any last period.

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	13
Operationally Valid but Failed Acceptance Criteria	6
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	1
Total	20

Fail Rate of Operationally Valid Tests: 31.6%

There were an additional 32 donated tests reported for a round robin on new reference oils 75 and 435-2, and one non-blind instrument shakedown run. None of these 33 tests are included in the period calibration statistics in Tables 16 and 17, and also not in the CUSUM severity plot in Figure 4.

Table 15 is a breakdown of the statistically unacceptable tests.

TABLE 15

Reason for Fail	No. of Tests
Total Deposits Mild	4
Total Deposits Severe	2

The reason for the operationally invalid result is an incorrect PID setting.

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	n	df	Pooled s	Mean ∆/s
Initial Round Robin Study	54	52	4.18	
10/1/07 through 3/31/08	22	20	9.65	0.92
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

^{*}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	8	0.27
Lab B	4	-0.01
Lab D	3	-1.36
Lab G	2	-0.98
Lab V	2	-0.65

INDUSTRY PERFORMANCE

Reference testing precision, as measured by pooled s, is less precise compared the previous report period and remains less precise than the target precision (Table 16). Performance is mild at -0.27 s. Severity is graphically represented in Figure 4 (attached). While there were no "extreme" test results reported again this period, as have been reported in some prior periods, there was one result reported as operationally valid that was nearly -4 s severe of target. Of particular note is the unusually high (31.6%) fail rate of the operationally valid tests.

All tests reported this period used Rod Batch J.

Oils 71-1 and 72-1 were introduced four periods ago with a warning last period of pending depletion. The TEOST Surveillance Panel and participating labs completed a round robin this period on replacement oils 75 and 435-2. The consensus of the panel was that 75 and 435-2 showed acceptable performance to replace 71-1 and 72-1, and oils 71-1 and 72-1 should be phased out as the labs use up remaining inventory (the TMC has stopped shipping 71-1 and 72-1 for calibrations).

TMC MEMORANDA

There was one TMC technical updates issued this report period for the D6335 test method:

Memo 11-034, August 3, 2011, New Reference Oils 75 & 435-2

<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 (8 labs reporting):

TABLE 18

	No. of Tests
Statistically Acceptable and Operationally Valid	45
Operationally Valid but Failed Acceptance Criteria	1
Operationally Invalid (initially reported as)	3
Operationally Invalid (after informed of failing calibration)	3
Total	52

Fail Rate of Operationally Valid Tests: 2.2%

The reason for the operationally invalid tests are:

- Catalyst weight was off-specification (one test)
- Temperature control failed (three test)
- New rig, failed sequential calibration attempts, held out of statistics (two tests)

Table 19 is a breakdown of the statistically unacceptable tests.

TABLE 19

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

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

Total Deposits	n	df	Pooled s	Mean ∆/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
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

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

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

	n	Mean Δ/s
Lab A	13	0.21
Lab AK	2	1.10
Lab B	12	-0.74
Lab D	6	0.24
Lab G	10	0.04
Lab J	1	1.79
Lab V	2	0.90

Lab Q reported only two failing calibration attempts on a new instrument this period; those results are held out of the period statistics because the lab could not demonstrate a passing calibration on a new instrument.

INDUSTRY PERFORMANCE

D7097 (MTEOS) reference testing overall precision, as measured by pooled s, is more precise than the prior report period and less precise than the target precision (Table 20). Overall performance this period is on target.

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.

One test was reported using Rod Batch H, while all remaining tests this period were reported on Rod Batch J. Two tests were reported using Catalyst Batch 0911 and all others on Catalyst Batch 1011.

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 6 operationally valid tests reported on oil 74 this period and, at this writing, there is only 1 sample of oil 74 remaining throughout the industry D7097 oil inventories of TMC blind reference samples.

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	1
Operationally Invalid (initially reported as)	1
Operationally Invalid (after informed of failing calibration)	1
Total	11

Fail Rate of Operationally Valid Tests: 11.1%

The reason for the two operational fails was that one test was aborted after discovering the wrong test method was being followed, and one test was rejected by the TMC because a discrimination run was due but not run simultaneous with the calibration sample.

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 Δ/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
10/1/10 through 3/31/11	8	61	10	-0.25
4/1/11 through 9/30/11	9	80	26	0.74

^{*}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 (Δ /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
10/1/10 through 3/31/11	8	No non-zero	occurrences
4/1/11 through 9/30/11	9	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

1110 1007				
	n	Foam Tendency Mean Δ/s		
Lab A	3	1.61		
Lab B	4	-0.45		
Lab G	2	1.79		

INDUSTRY PERFORMANCE

The D6082 Foam Tendency precision, as measured by standard deviation (s) on TMC oil 1007, is considerably less precise than last period (26 ml foam versus 10 ml foam) and less precise than target precision (Table 23). Overall Foam Tendency performance this period is 0.74 s severe. 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-5/SN failing oil for Foam Tendency).

One lab reported an operationally valid test result that failed more than 3 s severe on foam tendency. With that single result excluded, the standard deviation "improves" from 26 ml foam to 19 ml foam, and the severity changes from 0.74 s severe to 0.41 s severe. Still considerably imprecise and severe compared to the prior report period. So, it appears that the poor precision and shift to severe

D6082: High Temperature Foaming Characteristics of Lubricating Oils, continued

performance is not entirely due to the one severe failing test result this period. The TMC has no explanation for the markedly worse D6082 test precision estimate this report period.

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	6
Operationally Valid but Failed Acceptance Criteria	0
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	0
Total	6

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

There were no operationally invalid tests reported this period.

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

Gelation Index	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
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

D874: Sulfated Ash from Lubricating Oils and Additives, continued

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	0.02
Lab B	2	-0.31
Lab G	2	-0.55

INDUSTRY PERFORMANCE

D874 precision, as measured by pooled s, is more precise than the target precision and performance has shifted to mild 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	79
Operationally Valid but Failed Acceptance Criteria	17
Operationally Invalid (initially reported as)	13
Operationally Invalid (after informed of failing calibration)	10
Operationally Valid but Never Passed Calibration on New	0
Instrument (held out of statistics)	
QC Evaluation of New Rig (held out of statistics)	4
Donated RR Test to Evaluate New Reference Oil (435-2)	22
Total	145

Fail Rate of Operationally Valid Tests: 17.7%

The explanations for the 23 operationally invalid tests are:

- Airflow or vacuum system leaks or failures (four tests)
- Reactor temperature control problem (one test)
- Problems with NO₂ flow (four tests)
- Power or data acquisition failure (two tests)
- MRV temperature incorrect and CCS result too mild (one test)
- MRV instrument found to be faulty long after failing results were reported (six tests)
- VCV Set Position incorrect (two tests)
- Stirrer failure (one test)
- EOT Volatiles >60 mass% (two tests)

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

TABLE 31

Reason for Fail	No. of Tests
MRV Viscosity Mild	15
MRV Viscosity Severe	2

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
		uı		Mean A/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

^{*}Period results with one result of more than 29 s severe included and excluded for comparison.

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	26	-0.51
Lab AM	13	-0.35
Lab AO	2	-0.10
Lab B	16	-1.12
Lab D	4	-0.75
Lab G	35	-0.78

(Lab AN reported only operationally invalid tests this period)

INDUSTRY PERFORMANCE

ROBO precision, as measured by pooled s, is somewhat less precise than the target precision but much more precise than the prior three report periods (Table 32). Performance is -0.69 s mild of targets, with, notably, all six calibrating labs performing mild to some extent, and all four reference oils performing mild (Attachment 3). Severity is graphically represented in Figure 8 showing a somewhat consistent mild trend for the report period.

One reason for the notably improved precision is because of calibration requirement changes this period that exclude very severe test results as operationally invalid (typically because of excessive EOT volatiles loss). Recent changes adopted by the surveillance panel to report MRV results above 400,000 cP as >400,000, as the MRV test method instructs, and to evaluate EOT volatiles that are equal to, or greater than, 60 mass% as operationally invalid, serve to limit the inclusion of extreme MRV results as operationally valid tests. Through last period, severe results as high as 29 s severe were being included in the period statistics. With the calibration review changes, these extreme results will be filtered as operationally invalid (again, typically due to excessive volatiles loss).

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

For example, if an EOT MRV result of 400,000 cP were to be reported on a ROBO test on TMC oil 435, that result would be 4.8 s severe of the current target mean for oil 435. Therefore, oil 435 results greater than 4.8 s severe will now likely be flagged as operationally invalid under the new calibration requirements. (It is of note that all such results to date also have EOT volatiles equal to, or greater than, 60 mass%, making them operationally invalid under the new requirements because of excessive volatiles loss.) Similar exclusions of very severe results will apply to test results on the other TMC reference oils as well.

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. The TMC acquired a reblend, 435-2, and a round robin comparing oil 435 and 435-2 was recently completed. However, the round robin oil 435 results were also used to calibrate ROBO rigs, so this period's statistics do include a number of oil 435 calibration runs. Unfortunately, oil 435-2 was found to perform only marginally more severe than oil the 435-1 target mean, and about 10,000 cP (-0.6923 s) milder than 435 by direct comparison.

TMC MEMORANDA

There was one TMC technical update issued this report period for the D7528 test method:

Email from Tom Schofield on 20110420, Updated ROBO TMC Calibration Guidelines (v 20110420)

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.

D7563: Evaluation of the Ability of Engine Oil to Emulsify Water and Simulated Ed85 Fuel

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.

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	63.2	0.8			
55	D6417, D5800	68.4	0.6			
58	D6417, D5800, GI	119.2	0.9			
62	GI	1.7	0.1			
66	D6082 (Discrimination)	93.4	1.0			
71-1	TEOST	12 samples	0.0			
72-1	TEOST	4 samples	0.0			
75	MTEOS	8.1	1.4			
90	D874 & D874 Daily Check	37.0	1.8			
91	D874	4.6	0.0			
**432	MTEOS	Adequate				
434	MTEOS	5.4	0.4			
820-2	D874	10.6	0.0			
**1007	D6082	15.2				
**1009	GI	Adequate				
*434-1	ROBO	Adequate				
*435-1	ROBO	Adequate				
*435-2	ROBO/MTEOS	Adequate				
*438	ROBO	Adequate				

^{*}One drum of oil is set aside for bench calibration testing; the TMC has a larger supply of this oil.

^{**}Multi-Test Oil, estimated aliquot set aside for bench testing.

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 Supply	
435	Obsolete ROBO	7 Samples	

[^]Not selected as reference oil; TMC holding for further instructions from Surveillance Panel.

Table 34C Homogeneity and Miscibility Reference Oils

Homogeneity and wisciomity Reference ons					
Oil	For Tests	Quantity Left (gallons)	Quantity Used Last 12 Months (gallons)		
HMA	H&M (D6922)	166.9	9.6		
HMB	H&M (D6922)	170.9	9.6		
НМС	H&M (D6922)	156.9	9.6		
HMD	H&M (D6922)	164.6	9.6		
HME	H&M (D6922)	150.6	9.6		
HMF	H&M (D6922)	173.1	9.6		

^{**}Multi-Test Oil, estimated aliquot set aside for bench testing.

Table 34D Homogeneity and Miscibility Reference Oils

Oil	For Tests	Quantity Left (gallons)	Quantity Used Last 12 Months (gallons)
EM2	Emulsion Retention D7563	8.7	0.3
EM2-1	Emulsion Retention D7563	25.0	0.0
EM5	Emulsion Retention D7563	8.7	0.3
EM5-1	Emulsion Retention D7563	25.0	0.0

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
D874QC	1000 ml
ROBO	300 ml
ROBOQC	1000 ml
H&M	1000 ml
D7563	1000 ml

MISCELLANEOUS

The TMC posts monitored bench test calibration data on the TMC web site. 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 www.astmtmc.cmu.edu.

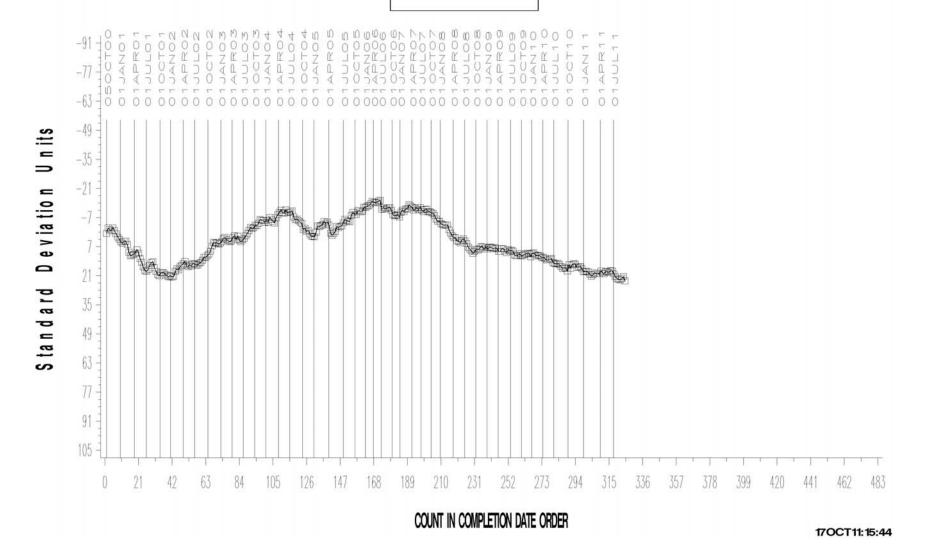
All currently monitored bench test data dictionaries and report form packages have been beta tested by the ASTM Data Communications Committee (DCC) and approved for electronic data transfer. Please contact Tom Schofield at (412) 365-1011 for more information.

D6417 VOLATILITY BY GC INDUSTRY OPERATIONALLY VALID DATA



SAMPLE AREA % VOLATIZED

CUSUM Severity Analysis

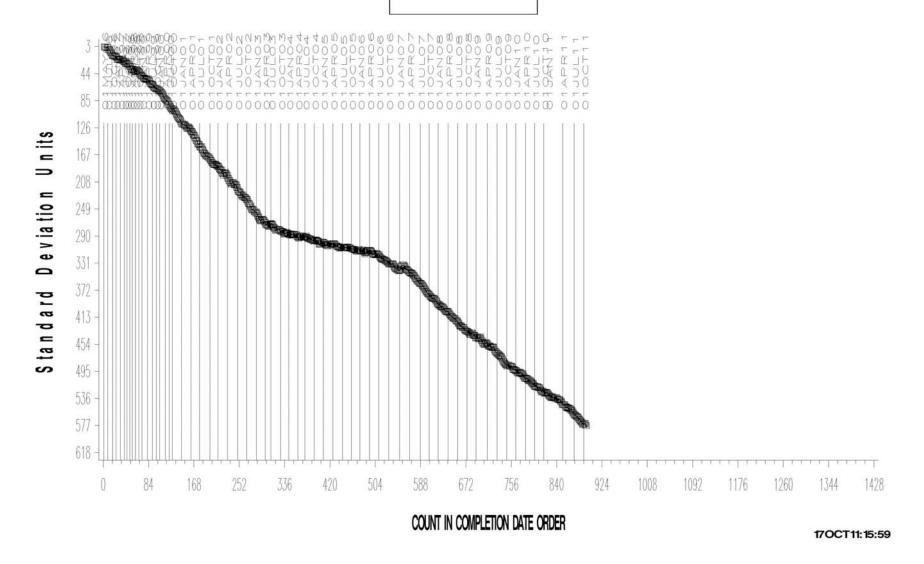


D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA



EVAPORATION LOSS, MASS%

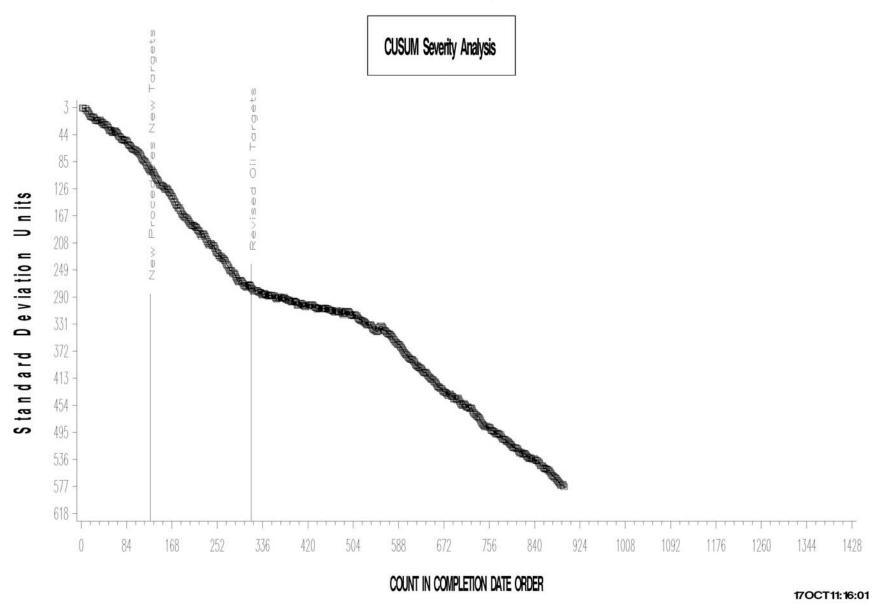
CUSUM Severity Analysis



D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA



EVAPORATION LOSS, MASS%

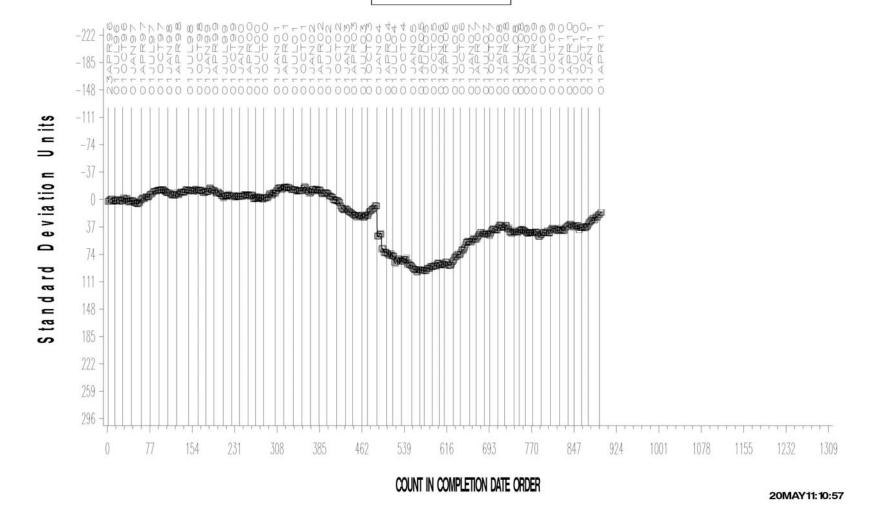


D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA



GELATION INDEX

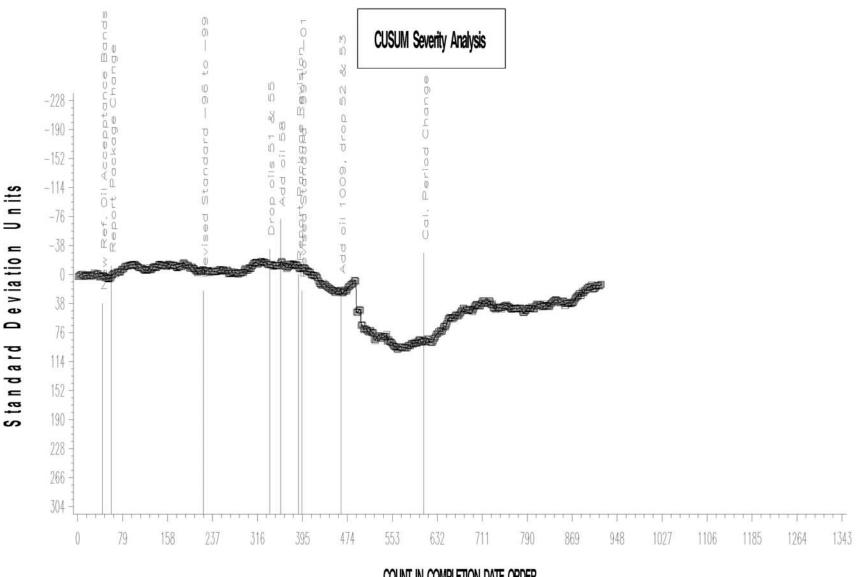
CUSUM Severity Analysis



D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA





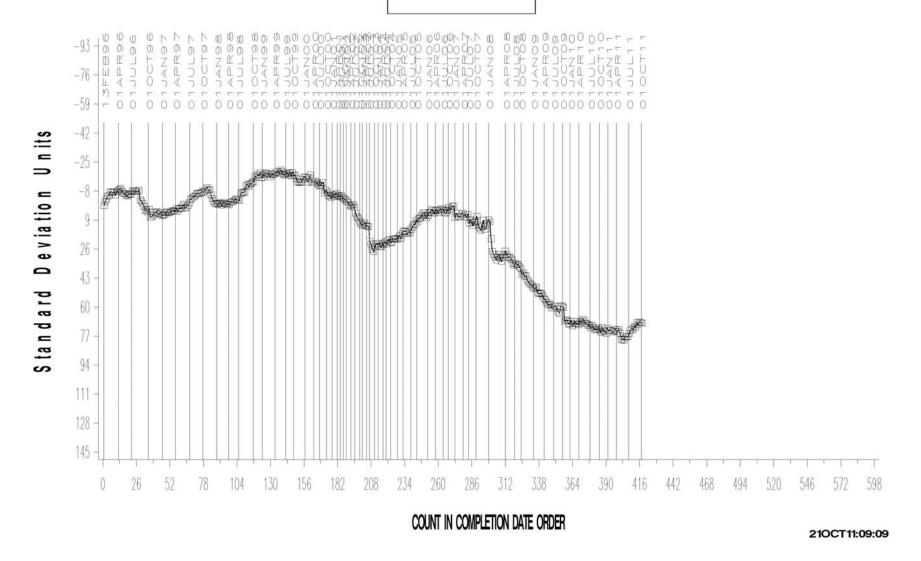


TEOST-33C INDUSTRY OPERATIONALLY VALID DATA



TOTAL DEPOSITS MG

CUSUM Severity Analysis

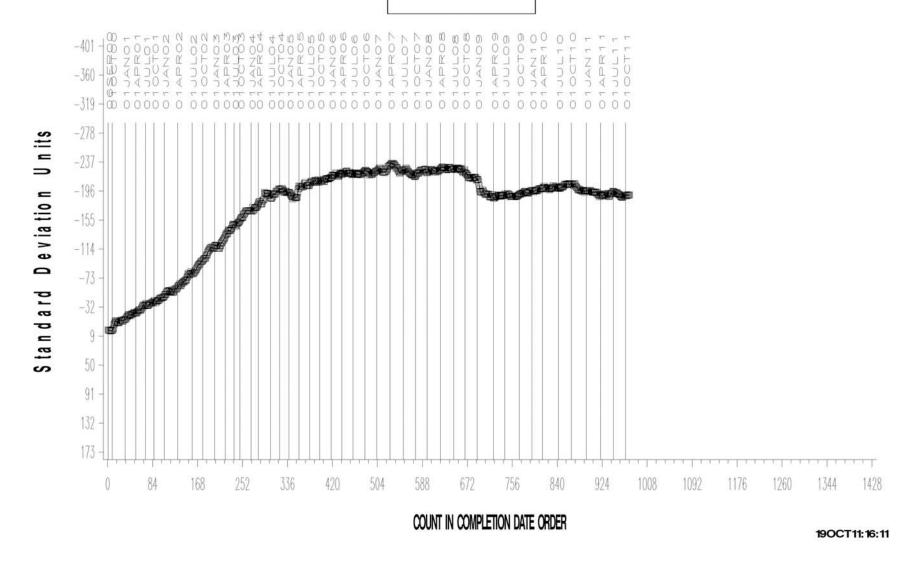


MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA



TOTAL DEPOSITS MG

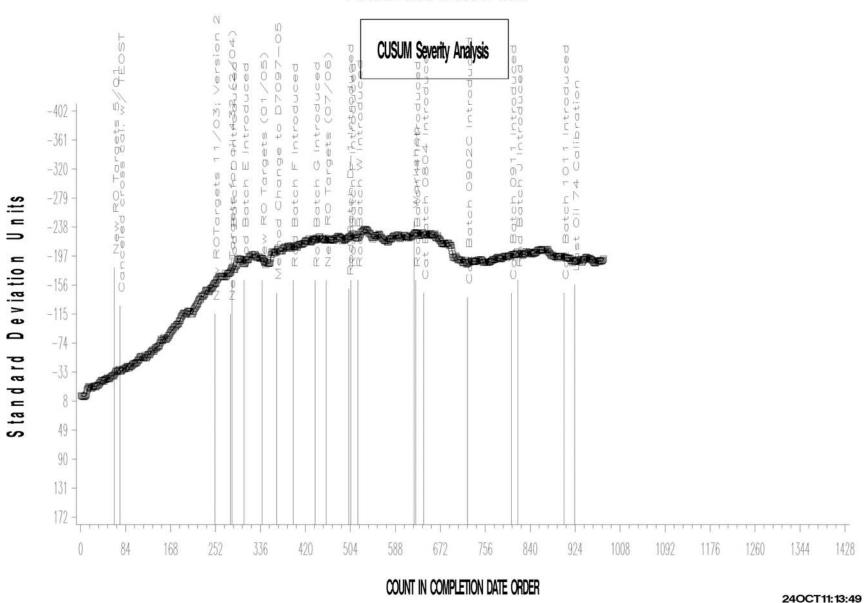
CUSUM Severity Analysis



MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA



TOTAL DEPOSITS MG

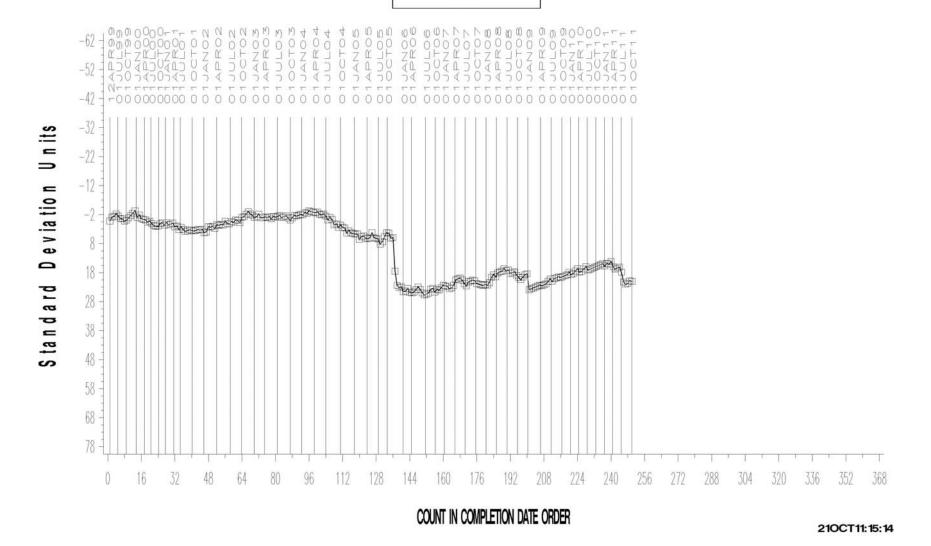


D6082 HIGH TEMPERATURE FOAM INDUSTRY OPERATIONALLY VALID DATA



IND= 1007 FOAM TENDENCY

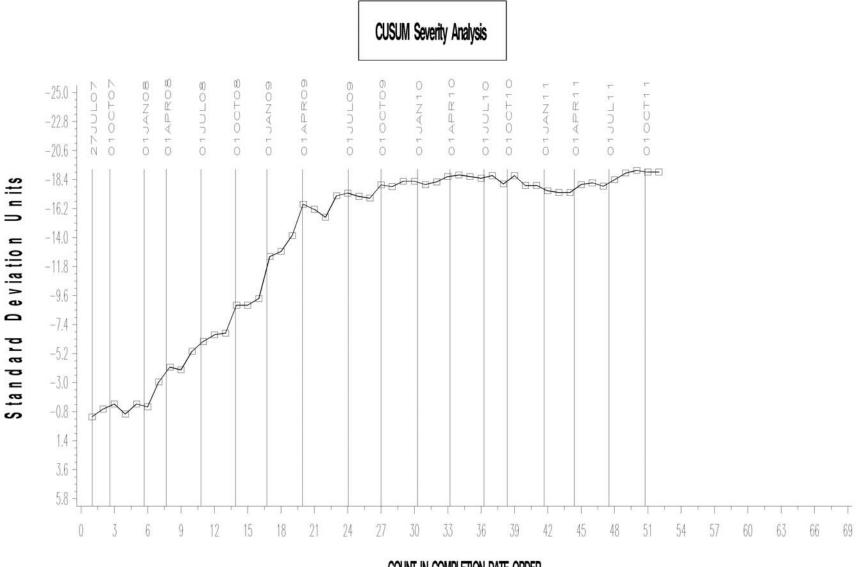
CUSUM Severity Analysis



D874 INDUSTRY OPERATIONALLY VALID DATA



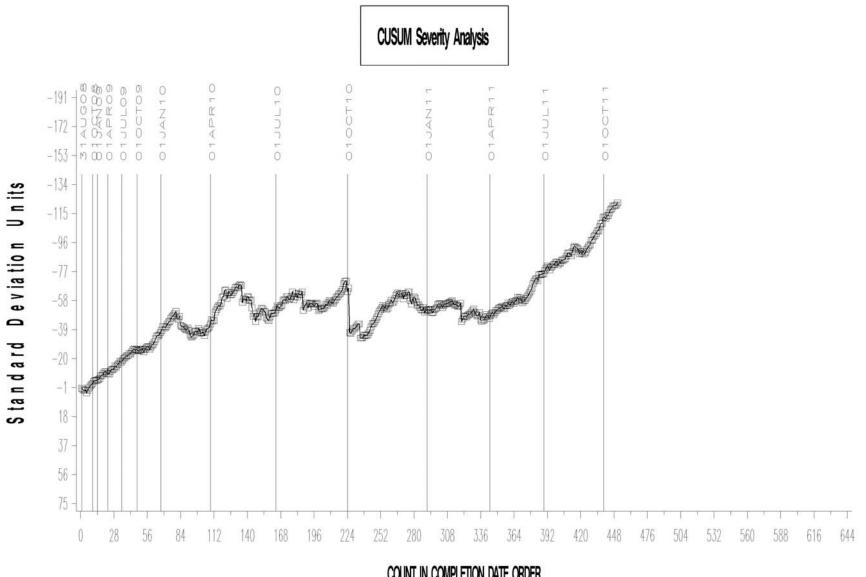
TEST SAMPLE PERCENT SULFATED ASH



ROBO TEST INDUSTRY OPERATIONALLY VALID DATA



AGED OIL MRV APPARENT VISCOSITY



TMC Monitored Bench Tests Reference Oil Test Targets and Acceptance Bands

Acceptance Bands *

			1	*					
						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	3 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	74	Total Deposit wt. (mg)	30	12.85	5.59	1.9	23.8		
D7097	432	Total Deposit wt. (mg)	30			38.2	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	3 0.68 6.		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	1.57 0.08		1.73		
ROBO	434-1	In MRV, In(mPa-s)	13	10.6599 (42612)	0.1672	10.3322 (30706)	10.9875 (59130)		
D7528			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)		

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

			Targets			4/1/10 - 9/30/10				10/1/10 - 3/31/11				4/1/11 - 9/30/11			
	Oil	_			_			_	Mean			_	Mean			_	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	7.1	0.14	0.42	7	6.9	0.49	-0.32	6	7.1	0.37	0.37
	55	Area % Volatized	18	11.68	0.51	7	11.9	0.37	0.43	5	11.7	0.37	0.08	5	11.7	0.42	0.08
	58	Area % Volatized	18	5.61	0.30	5	5.7	0.29	0.37	8	5.7	0.28	0.38	5	5.7	0.30	0.17
D5800	52	% volatility loss	33	13.75	0.61	15	14.4	0.71	1.07	10	14.5	0.52	1.18	17	14.7	0.68	1.52
**	55	% volatility loss	32	17.09	0.76	9	17.4	0.63	0.45	15	17.5	0.91	0.49	10	17.4	0.54	0.42
	58	% volatility loss	37	15.20	0.72	10	15.3	0.66	0.17	9	15.0	0.70	-0.27	12	15.2	0.50	0.01
TEOST	71	Deposit wt. (mg)	27	51.79	4.79	3	53.8	6.91	0.43	2	54.2	5.87	0.49	2	44.7	3.82	-1.48
D6335	71-1	Deposit wt. (mg)		51.79	4.79	4	54.2	3.42	0.51	6	47.7	7.99	-0.85	9	48.6	7.27	-0.68
	72-1	Deposit wt. (mg)		26.72	3.46	8	26.2	4.39	-0.16	5	29.8	3.00	0.88	7	29.3	5.78	0.73
	75	Deposit wt. (mg)	14	55.16	5.68									1	47.9		-1.28
MTEOS	432	Deposit wt. (mg)	30	47.04	4.50	20	50.8	4.19	0.83	25	49.8	4.56	0.60	23	49.2	4.96	0.49
D7097	434	Deposit wt. (mg)	30	27.37	6.57	22	23.1	5.50	-0.65	24	26.9	9.90	-0.07	21	24.0	6.89	-0.51
***	74	Deposit wt. (mg)	30	12.85	5.59	13	9.0	2.23	-0.69	6	14.2	6.93	0.24	2	15.8	7.35	0.53
GI	58	Gelation Index	17	5.8	0.69	8	6.2	0.85	0.58	12	5.9	1.27	0.10	8	6.0	1.09	0.27
D5133	62	Gelation Index	35	17.0	3.90	9	16.5	6.23	-0.14	12	13.1	5.06	-1.00	7	16.3	2.79	-0.17
****	1009	Gelation Index	16	7.30	0.68	7	7.2	0.49	-0.08	9	6.8	0.58	-0.72	8	6.72	0.60	-0.85
D6082	1007	Tendency (ml)	28	65	19	8	65	16	-0.05	8	61	10	-0.25	9	80	26	0.74
D874	820-2	Sulfated Ash m%	27	1.57	0.08	3	1.59	0.03	0.21	1	1.58		0.12	2	1.58	0.02	0.06
	90	Sulfated Ash m%	27	1.07	0.08	1	1.08		0.12	3	1.07	0.06	0.04	2	1.03	0.00	-0.50
	91	Sulfated Ash m%	27	0.82	0.05	1	0.81		-0.20	2	0.83	0.01	0.20	2	0.80	0.01	-0.40
ROBO	434-1	In (MRV Vis)	13	10.6599	0.1672	26	10.6193	0.3449	-0.24	34	10.5785	0.1904	-0.49	29	10.5920	0.2124	-0.41
	435	In (MRV Vis)	15	11.4895	0.2932	7	10.9061	0.6195	-1.99	0				17	11.0559	0.3189	-1.48
	435-1	In (MRV Vis)	22	11.0416	0.20295	61	11.0385	0.5042	-0.02	54	11.1361	0.9054	0.47	32	10.9091	0.2601	-0.65
	438	In (MRV Vis)	14	10.2676	0.2037	20	10.1871	0.6677	-0.40	33	10.4293	0.6779	0.79	18	10.1724	0.2656	-0.47