MEMORANDUM: 07-053

DATE: October 12, 2007

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, 2007, through September 30, 2007, for Test Areas

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

D874 and 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 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.)

 $\Delta$ /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?")

(i.e., 110 w 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).

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

Memorandum 07-053 October 12, 2007

Messrs. Ted Selby & Mark Devlin

Page 2

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. (The initial TMC PCEOCP Bench Test Report, of November 8, 1996, did not cross reference the labs.)

Prior to April 1, 2001, period precision and severity estimates were based on 12-months of data for some tests and six-months of data for other tests. Beginning with the report period April 1, 2001 through September 30, 2001, all test areas are analyzed over consecutive six-month intervals (a TMC report period). For more information on this decision, please refer to the TMC's web page:

ftp://ftp.astmtmc.cmu.edu/docs/bench/bo7semiannualreports/mem01-143.pdf

TMC semiannual monitoring reports for D6557 (BRT), D6795 (EOFT) and D6794 (EOWT) are being reported separately based on the division of assigned responsibilities within the TMC. (EEOC, CBT & HTCBT have always been reported separately.)

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

J. Zalar (TMC)

ftp://ftp.astmtmc.cmu.edu/docs/bench/bo7semiannualreports/mem07-053.pdf

Distribution: Email

### **ASTM Test Monitoring Center**

### **Semiannual Report**

ASTM D02.B07 Bench Reference Test Monitoring From April 1, 2007 through September 30, 2007

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

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

### **STATUS**

Table 1 summarizes the reference tests reported to the TMC this period (5 labs reporting):

TABLE 1

	No. of Tests
Statistically Acceptable and Operationally Valid	12
Operationally Valid but Failed Acceptance Criteria	0
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	0
Total	12

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

### **INDUSTRY PERFORMANCE**

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 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	
4/1/03 through 9/30/03	14	11	0.36	-0.45
10/1/03 through 3/31/04	15	12	0.50	-0.42
4/1/04 through 9/30/04	15	12	0.40	0.28
10/1/04 through 3/31/05	16	13	0.46	-0.04
4/1/05 through 9/30/05	17	14	0.61	-0.21
10/1/05 through 3/31/06	11	8	0.23	-0.58
4/1/06 through 9/30/06	12	9	0.45	0.36
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

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	0.11
Lab B	2	1.30
Lab G	2	-0.63
Lab H	2	0.68
Lab S	2	-0.26

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

### PRECISION AND SEVERITY

D6417 reference testing is more precise, as measured by Pooled s, compared to the previous period and to target precision (Table 3). Overall performance is severe of targets. Severity is represented graphically in Figure 1. Table 4 shows three labs trending severe while two labs are trending mild of targets. No test results were outside of the corresponding reference oil acceptance ranges and there were no operationally invalid tests reported this period.

Lab B's two passing quarterly calibrations this period, on the same instrument, were both more than 1 s severe of targets, suggesting a possible bias.

### **TMC MEMORANDA**

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

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

### **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	34
Operationally Valid but Failed Acceptance Criteria	2
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	1
Total	37

Fail Rate of Operationally Valid Tests: 5.6%

Table 6 is a breakdown of the statistically unacceptable tests.

TABLE 6

Reason for Fail	No. of Tests
Sample Evaporation Loss Severe	2
Sample Evaporation Loss Mild	0

### **INDUSTRY PERFORMANCE**

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	
4/1/04 through 9/30/04	30	27	0.64	0.24
10/1/04 through 3/31/05	35	32	0.69	0.11
4/1/05 through 9/30/05	34	31	0.55	0.23
10/1/05 through 3/31/06	34	31	0.74	0.07
4/1/06 through 9/30/06	35	32	0.62	0.54
10/1/06 through 3/31/07*	39	36	0.99	0.36
10/1/06 through 3/31/07*	38	35	0.61	0.51
4/1/07 through 9/30/07	36	33	0.50	0.92

<sup>\*</sup>Period statistics with and without a single unusually mild result (-5.51 s) included

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

Sample Evaporation Loss, mass %	n	df	Pooled s	Mean Δ/s
Procedure A	0	0		
Procedure B	33	30	0.46	0.95
Procedure C	3	0		0.68

### **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	0.72
Lab B	8	0.41
Lab D	3	0.68
Lab F	6	1.12
Lab G	4	0.75
Lab H	2	1.07
Lab I	4	1.58
Lab J	5	1.36

### **PRECISION AND SEVERITY**

Effective September 26, 2000, the TMC began monitoring the three Noack procedures under the revised D5800 test method. Revised reference oil targets and acceptance bands for all three current reference oils (52, 55 and 58), based on 18-months of TMC reference data, became effective July 21, 2003.

D5800 reference testing precision has improved for the report period and is more precise than the target precision. Overall performance is unusually severe this period, with 4 of 17 instruments (at 3 of 8 labs) performing more than 1 s severe on all operationally valid calibration tests during the report period. Severity is graphically represented in Figures 2A and 2B. Figure 2A shows an unexplained increase in severity since the 01JUL06 timeline, with an additional increase in severity since the 01JAN07 timeline. It was noted last period that Lab G had a strong severe instrument bias, that bias is not indicated this period. However, it appears that many instruments, across a number of labs, are performing severe. That is, the unusually strong severe trend this period (0.92 s) seems to be a general trend, and not wholly attributable to any particular instrument or lab, or to any particularly anomalous test results. The test is drifting severe, with only 4 of the 36 operationally valid test results being mild (and all four less than 1 s mild), while 17 of the 36 test results, nearly half, are more than 1 s severe. Table 9 shows all eight labs performing severe, with half of them at more than 1 s severe for the report period. (Last period the concern was that only 2 of eight labs were performing near target, with 6 of 8 labs performing greater than 0.9 s mild or severe.)

With improved precision for the report period, but a widely observed severe trend, D5800 Procedure B calibration testing seems to be trending significantly severe since at least January 2007.

Table 8 compares the procedures for the period. There were no Procedure A calibration tests reported and only three Procedure C calibration tests reported this period. With zero degrees of freedom, there is insufficient data to make any precision evaluation on Procedure C testing this period.

### **TMC MEMORANDA**

There were no TMC technical memos issued this report period for the D5800 test method.

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

### **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	20
Operationally Valid but Failed Acceptance Criteria	4
Operationally Invalid (initially reported as)	2
Operationally Invalid (after informed of failing calibration)	3
Total	29

Fail Rate of Operationally Valid Tests: 16.7%

Table 11 is a breakdown of the statistically unacceptable tests.

TABLE 11

Reason for Fail	No. of Tests
Gelation Index Mild	4
Gelation Index Severe	0

### **INDUSTRY PERFORMANCE**

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)				
4/1/03 through 9/30/03	27	22	2.30	0.06
10/1/03 through 3/31/04	37	34	5.86	1.73
4/1/04 through 9/30/04	27	24	3.05	0.40
10/1/04 through 3/31/05	34	31	2.51	0.40
4/1/05 through 9/30/05	22	19	3.44	-0.17
10/1/05 through 3/31/06	22	19	3.09	-0.16
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

### <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	6	0.37
Lab B	7	-1.32
Lab G	4	-0.94
Lab H	1	-0.59
Lab I	4	0.69
Lab S	2	0.94

### **PRECISION AND SEVERITY**

Effective July 15, 2003, new D5133 reference oils, targets and acceptance bands were implemented for TMC calibration monitoring. The current GI reference oils are 58, 62 & 1009.

Effective March 8, 2006, TMC instrument calibration periods changed from 90-days to 60-days and a 480-day head calibration period was introduced for all successful calibrations completed March 8, 2006, or later (see TMC Technical Memo 06-004).

D5133 reference testing is slightly less precise compared to last period and continues to be less precise than the target precision. Overall performance is mild of targets. Severity is graphically represented in Figures 3A and 3B with a mild trend for the better part of the most recent two report periods and some signs of leveling during the present report period.

Last period reported Lab G having a strong instrument bias (mild) contributing to the overall period severity. The mild bias on that instrument is not as pronounced this period. However, Table 13 shows Lab B averaging -1.32 s mild on seven operationally valid calibration tests for this period. Five of those seven test are reported at more than 1 s mild of target (on two instruments), and one result more than 1 s severe. So, Lab B is contributing substantially mild calibration results this period as reflected in the severity estimate for lab B in Table 13, and reported only one calibration result that was less than 1 s from target.

### **TMC MEMORANDA**

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

### <u>D6335</u>: Determination of High Temperature Deposits by Thermo-Oxidation Engine Oil Simulation Test (TEOST)

### **STATUS**

Table 14 summarizes the reference tests reported to the TMC this period (3 labs reporting):

TABLE 14

	No. of Tests
Statistically Acceptable and Operationally Valid	7
Operationally Valid but Failed Acceptance Criteria	3
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	4
Total	14

Fail Rate of Operationally Valid Tests: 30.0%

Table 15 is a breakdown of the statistically unacceptable tests.

TABLE 15

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

### **INDUSTRY PERFORMANCE**

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 Δ/s
Initial Round Robin Study	54	52	4.18	
10/1/04 through 3/31/05	10	8	6.30	-0.32
4/1/05 through 9/30/05	11	9	4.13	-0.73
10/1/05 through 3/31/06	14	12	4.96	-0.29
4/1/06 through 9/30/06	10	8	5.11	-0.16
10/1/06 through 3/31/07*	12	10	8.66	0.14
10/1/06 through 3/31/07*	11	9	5.67	-0.45
4/1/07 through 9/30/07*	10	8	9.59	0.43
4/1/07 through 9/30/07*	9	7	8.08	-0.11

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

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	3	-0.35
Lab B	4	-0.75
Lab G	3	2.79

### <u>D6335: Determination of High Temperature Deposits by Thermo-Oxidation Engine Oil Simulation Test (TEOST), continued</u>

### **PRECISION AND SEVERITY**

Reference testing precision has degraded substantially and performance is severe of targets for the report period. Last period Lab G reported a single test result that was 6.55 s severe of target, and has maintained the status of the test as operationally valid. This period Lab G reported a test as operationally valid at 5.28 s severe. Table 16 shows that even with this extreme result excluded, precision has degraded substantially for the current period, while performance becomes slightly mild. Only two of the ten operationally valid tests for the period are within 1 s of target (both mild), with three of ten results, one from each lab, at more than 2 s from target (two mild and one severe). Additionally, the overall fail rate of tests reported as operationally valid is unusually high at 30% (5% would be expected based on the acceptance bands). And, finally, Lab A reported four consecutive failing tests on the same instrument as operationally valid before uncovering a specific instrument problem and the lab subsequently changed those four tests to operationally invalid.

With that summary of testing problems this period, the test has had worsening precision estimates for the last four report periods (two years). A review of the rod batch indicates that all labs have been reporting batch G rods on all TMC calibrations since April 2006. Additionally, all labs report using either 192 or 193 microlitres of 6% ferric napthenate catalyst (the procedure specifies the addition of 193 microlitres, but no tolerance is specified). So, a specific cause of the worsening precision has not yet been identified.

Severity is graphically represented in Figure 4 (attached) with a recent overall severe trend (mostly biased by the two extreme test results previously noted), but also an increasing scatter of the data points, particularly noticeable since the 01OCT06 timeline, indicating degraded precision.

### **TMC MEMORANDA**

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

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

### **STATUS**

Table 18 summarizes the reference tests reported to the TMC this period (5 labs reporting):

TABLE 18

	No. of Tests
Statistically Acceptable and Operationally Valid	43
Operationally Valid but Failed Acceptance Criteria	5
Operationally Invalid (initially reported as)	1
Operationally Invalid (after informed of failing calibration)	0
Total	49

Fail Rate of Operationally Valid Tests: 10.4%

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	4

### **INDUSTRY PERFORMANCE**

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 ∆/s
Updated Targets Effective 2/18/04	50	46	4.92	
10/1/03 through 3/31/04	35	31	9.40	-0.69*
4/1/04 through 9/30/04	40	36	7.29	-0.55
Updated Targets Effective 1/12/05	30	27	3.42	
10/1/04 through 3/31/05	36	31	5.15	-0.11**
Updated Targets Effective 6/30/05	42	39	4.60	
4/1/05 through 9/30/05	39	36	6.36	-0.17**
10/1/05 through 3/31/06	40	37	6.68	-0.26
Updated Targets Effective 7/31/06	90	87	5.62	
4/1/06 through 9/30/06	43	40	5.99	-0.09**
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

<sup>\*</sup>New oil performance targets and acceptance bands were implemented twice during the period; severity is estimated using the targets that were in effect at the time each test was reported.

<sup>\*\*</sup> New oil performance targets and acceptance bands were implemented during the period; severity is estimated using the targets that were in effect at the time each test was reported.

### <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

	n	Mean Δ/s
Lab A	14	-0.57
Lab B	10	0.36
Lab D	6	-0.58
Lab G	16	1.45
Lab V	2	0.03

### PRECISION AND SEVERITY

D7097 reference testing precision has degraded slightly compared to last report period and remains less precise than the updated target precision. Attachment 3 shows degraded precision across all three reference oils compared to the target precision of each oil. Overall performance is severe with Lab G performing particularly severe compared to the other testing labs. Lab G reported ten of sixteen operationally valid tests on multiple instruments at more than 1 s severe of targets, four of those at more than 2 s severe, and one at nearly 4 s severe. So there seems to be a comparatively strong severe bias at Lab G that does not appear to be attributable to any single instrument. All labs are reporting using the same catalyst batch for the period (batch 0511), and a mix of rod batches F & G, with Lab G reporting three tests using rod batch D-1 (but nothing particularly alarming is noted on the severity of those results, see tables 21A and 21B below).

Table 21A is an overall precision and severity analysis of the period calibration data by rod batch.

Table 21A
Total Deposits Precision and Severity Analysis by Rod Batch
TMC Calibration Data 4/1/04 – 9/30/04

<b>Rod Batch</b>	n	df	Pooled s	Mean Δ/s
D-1*	3	1	1.20	1.31
F	12	11	9.30	0.56
G	33	30	7.08	0.14

<sup>\*</sup>All rod batch D-1 tests were run by Lab G, which is generally performing severe on all three rod batches for the period (see Table 21B).

Based on 12 tests on rod batch F, versus 33 on rod batch G, rod batch F performance seems to be somewhat more severe, and less precise, than rod batch G for the six-month report period.

Table 21B is a breakdown of severity performance by lab and rod batch to show the distribution and performance by rod batch among the labs for the report period.

Table 21B
Total Deposits Severity Analysis by Lab and Rod Batch
TMC Calibration Data 4/1/04 – 9/30/04

	Rod Batch		
		Mean ∆	/s
LAB	D-1	F	G
Lab A		-1.14	-0.34
		n=4	n=10
Lab B		1.66	0.22
		n=1	n=9
Lab D		-0.68	-0.57
		n=1	n=5
Lab G	1.31	1.71	1.28
	n=3	n=6	n=7
Lab V			0.03
			n=2

Table 21B suggests a range of severity performance, from severe to mild, across the labs on rod batches F and G.

The MTEOS severity trend 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 batches are introduced. Figure 5A shows the period severity with a modest overall severe bias for the report period. Worsening precision continues to be a concern this period, as it was last period.

### **TMC MEMORANDA**

There were no TMC technical memos issued this report period for the MTEOS test method.

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

D6082 Monitoring Historical and Statistical References Affecting the Statistical Estimates in This Report

In June 2000, the High Temperature Foam Surveillance Panel had given approval for the TMC to stop collecting data for Total Volume Increase.

On June 18, 2001, the section agreed to suspend the use of the severe performing TMC oil 1002 as a D6082 reference oil due to ongoing calibration precision and severity problems with that oil and on June 17, 2002 the section voted to discontinue the use of 1002 altogether.

On July 21, 2003 a severe performing "discrimination oil", TMC oil 66, was introduced to the monitoring system to be run by each participating lab once every six-months to show that each lab can discriminate a GF-3/SL passing oil (foam tendency) from a failing oil in the D6082 test method. The first discrimination test using oil 66 was completed on August 13, 2003. Because of apparent poor reproducibility of the D6082 test method on severe performing oils (greater than 100 ml foam tendency) in general, it was agreed that oil 66 discrimination results would not be statistically summarized by the TMC other than a count of the tests that do and don't meet the acceptance criteria.

On March 28, 2006 the performance targets for oil 1007 were adjusted slightly by rounding the targets from a precision of 0.01 ml to 1 ml; this adjustment slightly changed the acceptance bands on oil 1007 (see TMC technical memo 06-08).

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.

### **STATUS**

Table 22 summarizes the reference tests reported to the TMC this period (4 labs reporting):

TABLE 22

	No. of Tests
Statistically Acceptable and Operationally Valid	10
Operationally Valid but Failed Acceptance Criteria	0
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	0
Total	10

Fail Rate of Operationally Valid Tests: 0.0%

In addition to the calibration tests, there were four discrimination oil tests reported this period; all met the acceptance criteria for the discrimination oil.

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

### TMC 1007 INDUSTRY PERFORMANCE

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	
10/1/03 through 3/31/04	12	62.5	10.55	-0.17
4/1/04 through 9/30/04	13	72.3	15.89	0.34
10/1/04 through 3/31/05	12	72.9	16.30	0.37
4/1/05 through 9/30/05	10	62.0	25.30	-0.19
10/1/05 through 3/31/06*	11	102	70	1.87
10/1/05 through 3/31/06*	9	74	19	0.45
4/1/06 through 9/30/06	12	66	16	-0.01
10/1/06 through 3/31/07	9	61	12	-0.26
4/1/07 through 9/30/07	10	63	18	-0.16

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

TABLE 24

1007 Foam Stability @ 1 min., ml	n	Mean	S	
Initial Round Robin Study	28	0.00	0.00	
10/1/03 through 3/31/04	12	No non-zero	occurrences	
4/1/04 through 9/30/04	13	No non-zero	occurrences	
10/1/04 through 3/31/05	12	No non-zero	occurrences	
4/1/05 through 9/30/05	10	No non-zero	occurrences	
10/1/05 through 3/31/06	11	No non-zero	occurrences	
4/1/06 through 9/30/06	12	No non-zero	occurrences	
10/1/06 through 3/31/07	9	No non-zero	occurrences	
4/1/06 through 9/30/06	10	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

	n	Foam Tendency Mean ∆/s
Lab A	2	-0.05
Lab B	4	0.08
Lab G	2	-0.32
Lab I	2	-0.58

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

### **PRECISION AND SEVERITY**

D6082 Foam Tendency on TMC oil 1007 is less precise for this report period but is still more precise than the target precision. Overall performance is slightly mild. 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 memos issued this report period for the D6082 test method.

### **D874-00 Sulfated Ash from Lubricating Oils and Additives**

On June 18, 2007, D02.B0.07 gave the approval for TMC monitoring of D874 to commence per the request of the Heavy Duty Classification Panel. The current TMC reference oils are 90, 91 and 830-2.

Three labs requested TMC reference oils to participate. The first calibration test was completed on 7/27/2007.

Two tests were reported to the TMC this period as follows:

TABLE 26

	TMC Oil	Sulfated Ash Mean Δ/s
Lab A	91	-0.40
Lab G	91	-0.60

Both tests were passing calibration tests; both tests were mild of target.

### **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.

### ROBO (Romaszewski Oil Bench Oxidation Test; Sequence IIIGA Replacement Test)

The first surveillance panel teleconference was held on March 29, 2007 where the panel organization was conducted and improvements to the test method draft were discussed. Several panel teleconferences have followed with robust discussions concerning clarifying the test method and conducting a "mini" round robin with four, or possibly five labs, and using one oil (TMC 434) to get a baseline on how well the labs compare by testing the same TMC oil using the latest test method draft. As of this writing, four of five labs have reported data to the TMC and the panel plans to hold another teleconference on October 18, 2007. To my knowledge, meeting minutes have not yet been issued for any of the teleconferences to date. Continued panel teleconferences are expected with the goal of issuing a standardized test method and implement an as yet unspecified TMC monitoring program.

### **REFERENCE OIL SUPPLIES**

There is adequate supply of B0.07 Bench Test reference oils on hand at the TMC. Tables 27A and 27B list the PCEOCP bench test reference oils currently on hand at the TMC.

Table 27A

Oil	For Tests	Quantity Left (gallons)	Quantity Used Last 12 Months (gallons)
^51	Obsolete Vol. & GI	94.6	0.0
52	D6417, D5800	66.5	0.8
^53	Obsolete Vol. & GI	96.8	0.0
^54	Obsolete Volatility	97.8	0.0
55	D6417, D5800	71.4	0.8
^57	Old Volatility Candidate	51.2	0.0
58	D6417, D5800, GI	122.9	0.8
62	GI	1.4	0.1
66	D6082 (Discrimination)	98.2	1.2
71	TEOST	2.6	0.5
72	TEOST	2.7	0.3
74	MTEOS	0.8	0.2
90	D874 & D874 Daily Check	47.2	2.1
91	D874	4.7	0.1
**432	MTEOS	Adequate	
^**433	Obsolete MTEOS	Adequate	
**434	MTEOS	Adequate	
**820-2	D874	Adequate	
*1007	D6082	Est. 22	
**1009	GI	Adequate	

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

<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 27B

Oil	For Tests	Quantity Left (gallons)	Quantity Used Last 12 Months (gallons)
HMA	H&M (D6922)	191.0	2.75
HMB	H&M (D6922)	195.0	2.75
НМС	H&M (D6922)	181.0	2.75
HMD	H&M (D6922)	189.0	2.75
HME	H&M (D6922)	174.0	2.75
HMF	H&M (D6922)	196.7	2.75

### Shipping aliquots are:

D6417	1 ml
D5480	4 ml
D5800	100 ml
GI	25 ml
MTEOS	17 ml
TEOST	125 ml
D6082	525 ml
H&M	950 ml

### **MISCELLANEOUS**

The TMC posts monitored bench test calibration data on the Internet. Selected parameters from all operationally valid reference tests are posted on the TMC's World-Wide-Web page in real time. 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

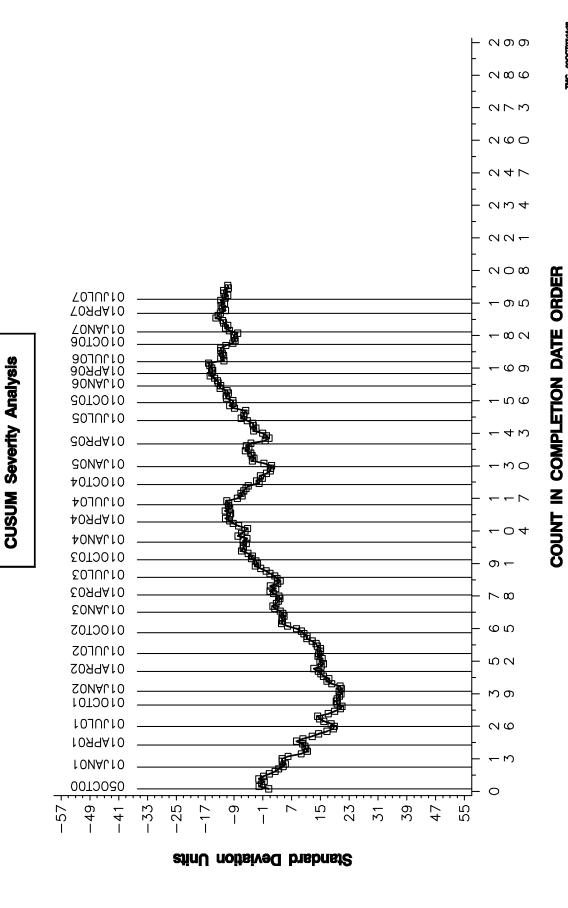
7007

:

371'C

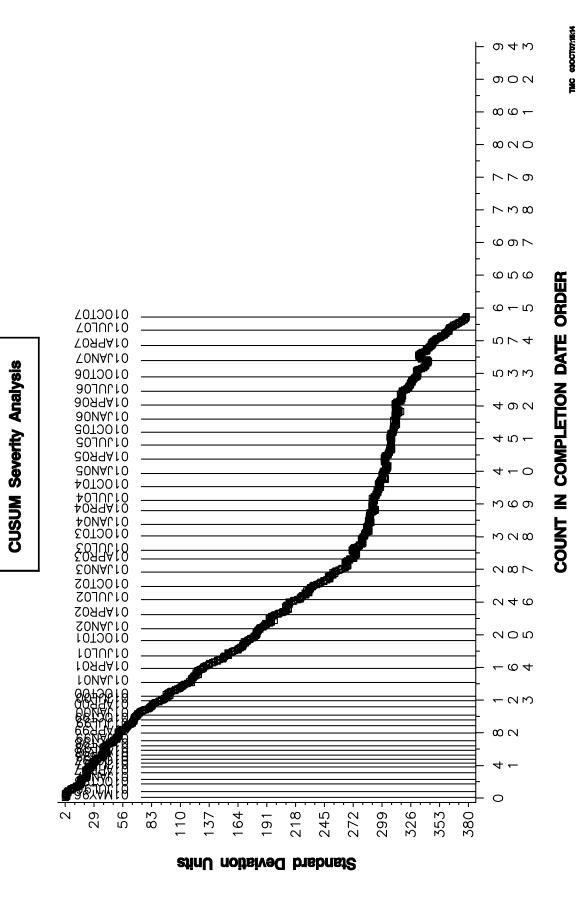
(3)

SAMPLE AREA % VOLATIZED



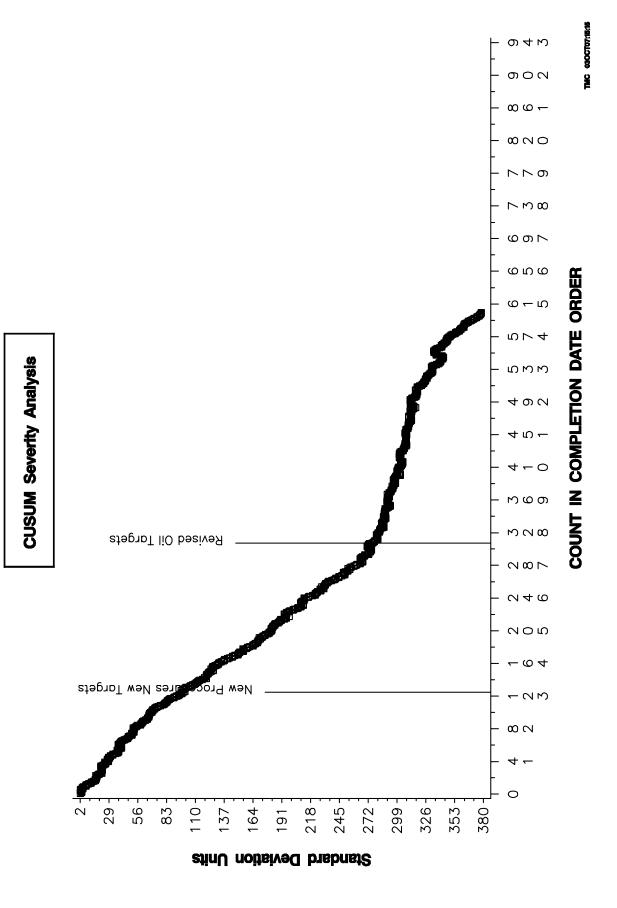
# D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA

TEST OIL SAMPLE EVAPORATION LOSS, MASS%

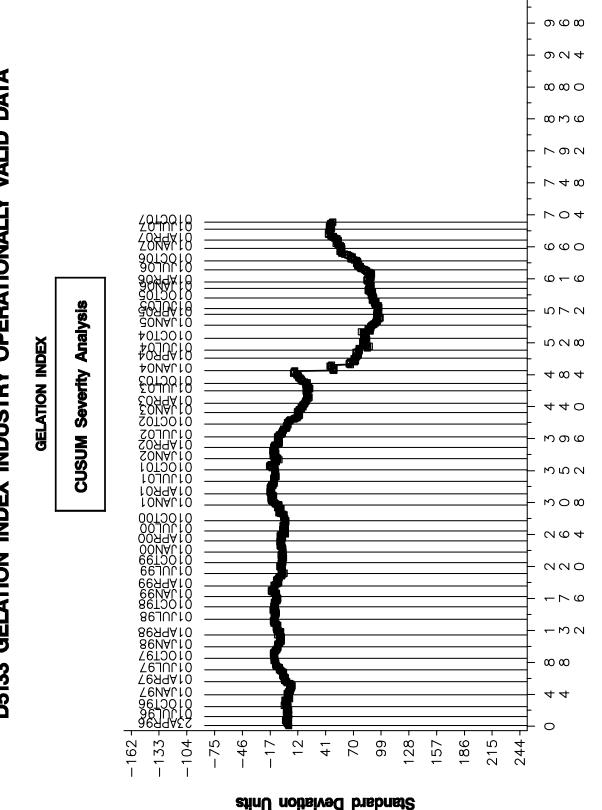


# D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA

TEST OIL SAMPLE EVAPORATION LOSS, MASS%



## D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA

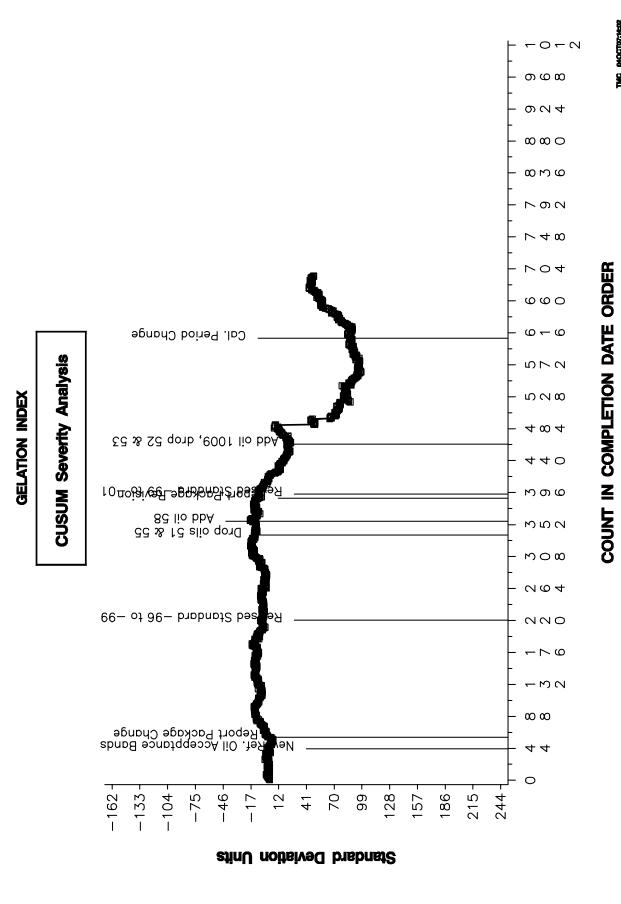


TIMC 080CT07:11:46

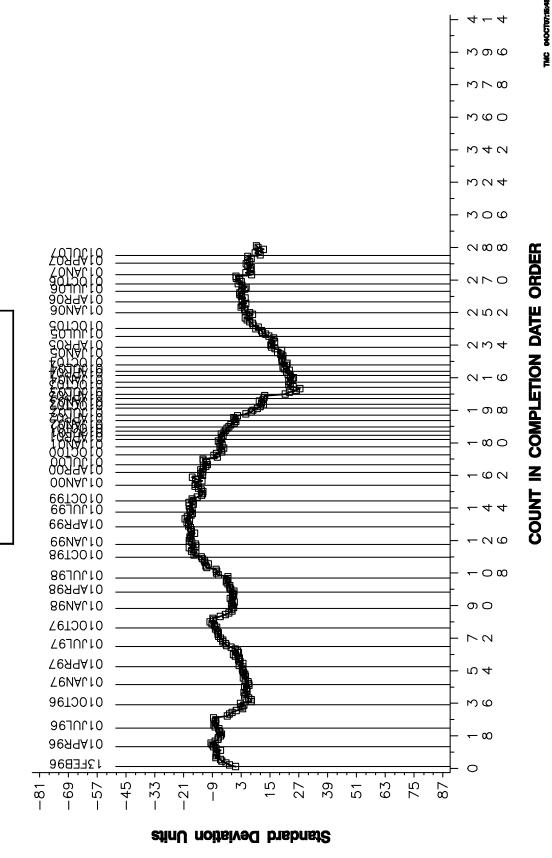
COUNT IN COMPLETION DATE ORDER

70-7

## D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA

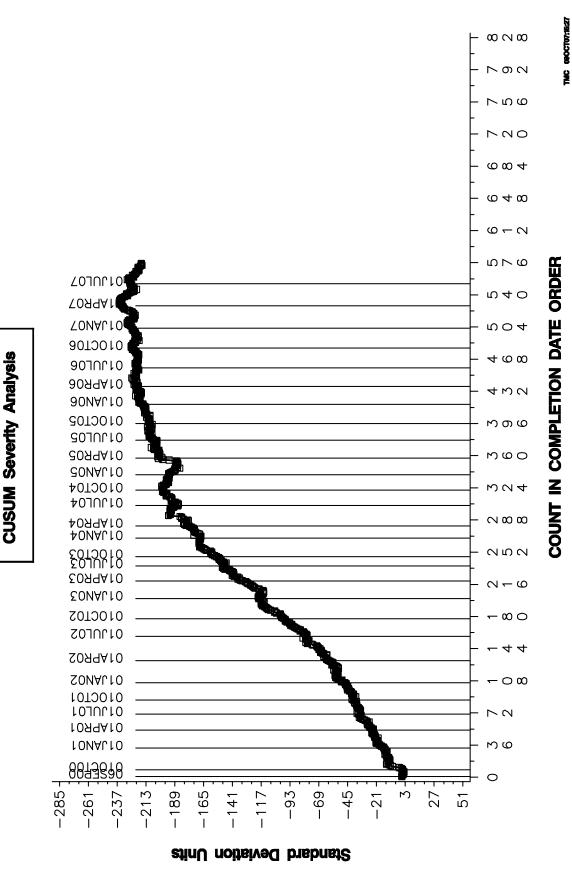


TOTAL DEPOSITS (mg)
CUSUM Severity Analysis

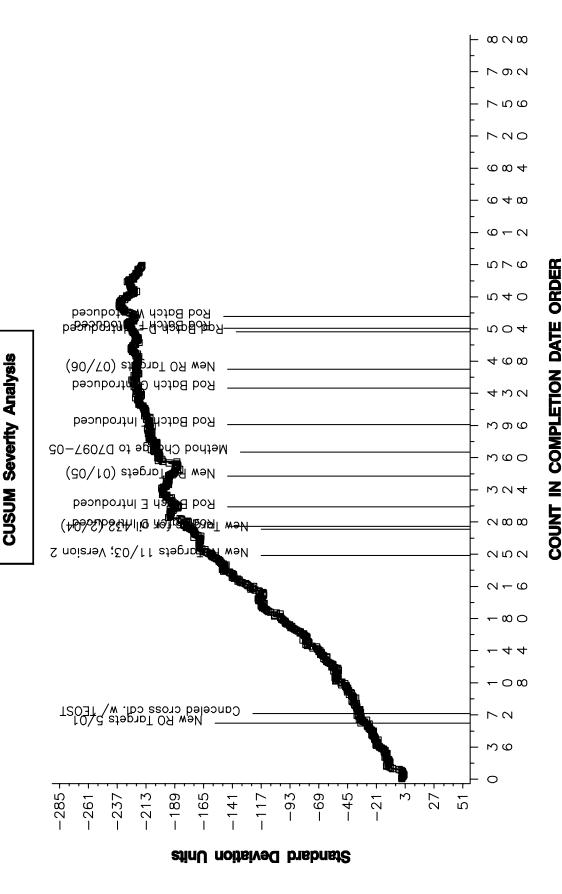


### MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA

TOTAL DEPOSITS (mg)



TOTAL DEPOSITS (mg)

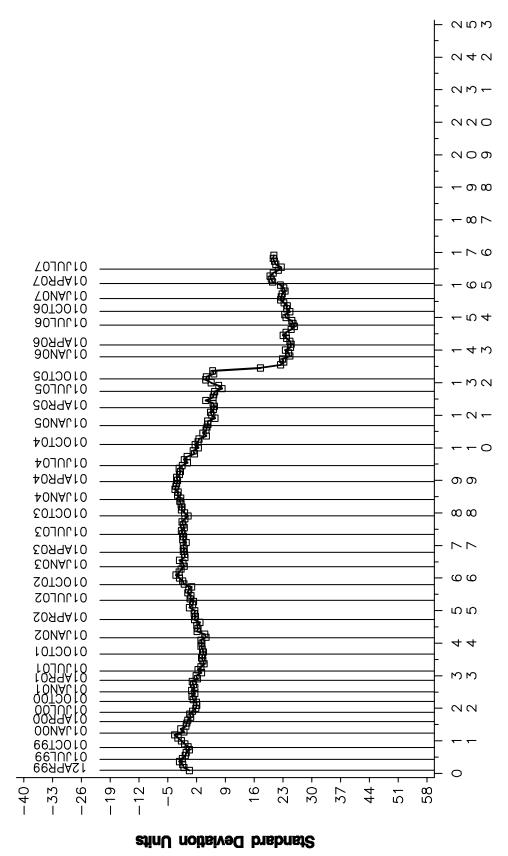


94OCT07:18:90

¥







TIMC 040CT07;16:26

ORDER

DATE

COMPLETION

Z

COUNT

TMC Monitored Bench Tests Reference Oil Test Targets and Acceptance Bands

						Acceptance Bands	e Bands *
						36	95%
Test	Oil Code	Parameter	п	Mean	sR	Lower	Upper
D6417	52	area % volatility loss	18	6.97	0.31	6.4	9.7
	55	area % volatility loss	18	11.68	0.51	10.7	12.7
	58	area % volatility loss	18	5.61	0.30	2.0	6.2
D5800	52	mass % volatility loss	33	13.75	0.61	12.6	14.9
New Targets	55	mass % volatility loss	32	17.09	92.0	15.6	18.6
7/21/2003	58	mass % volatility loss	37	15.20	0.72	13.8	16.6
TEOST by	7.1	Total Deposit wt. (mg)	27	51.79	4.79	42.4	61.2
D6335	72	Total Deposit wt. (mg)	27	26.72	3.46	19.9	33.5
MTEOS by	74	Total Deposit wt. (mg)	30	12.85	69'9	1.9	23.8
D7097	432	Total Deposit wt. (mg)	30	47.04	4.50	38.2	55.9
New Targets	434	Total Deposit wt. (mg)	30	27.37	6.57	14.5	40.2
20060731							
GI by	58	Gelation Index	17	5.8	69.0	4.4	7.2
D5133	62	Gelation Index	35	17.0	3.90	9.4	24.6
New Targets	1009	Gelation Index	16	7.3	0.68	0.9	8.6
7/15/2003							
D6082	1007	Tendency (ml)	28	99	19	29	103
(HT FOAM)	1007	Stability (ml)	28	0	0	0	0
D6082	66 (DISCRIM)	Tendency (ml)	ł	-		>100	-
(HT FOAM)	66 (DISCRIM)	Stability (ml)	ŀ	-		0	0
D874	06	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

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

				Targets	10		4/1/06	4/1/06 - 9/30/06	9		10/1/06	10/1/06 - 3/31/07	70		4/1/07	4/1/07 - 9/30/07	7
	Oil								Mean				Mean				Mean
Test	Code	Parameter	L	Mean	SR	_	Mean	sR	∆/s	_	Mean	sR	∆/s	L	Mean	sR	∆/s
D6417	52	Area % Volatized	18	26.9	0.31	2	7.1	0.27	0.29	3	7.0	0.23	0.20	2	7.1	0.31	0.48
	22	Area % Volatized	18	11.68	0.51	3	11.6	0.21	-0.22	9	11.3	0.65	-0.71	3	12.0	0.35	0.56
	58	Area % Volatized	18	5.61	0.30	4	5.9	0.68	0.88	3	5.8	0.45	0.52	4	5.5	0.28	-0.37
D5800	52	% volatility loss	33	13.75	0.61	13	14.1	0.46	0.61	12	14.2	09.0	0.81	12	14.3	0.45	0.94
*	22	% volatility loss	32	17.09	0.76	10	17.6	0.85	0.61	15	17.4	1.42	0.46	13	18.0	0.51	1.24
	58	% volatility loss	37	15.20	0.72	12	15.5	0.55	0.42	12	15.0	0.55	-0.22	11	15.6	0.52	0.53
TEOST	71	Deposit wt. (mg)	27	51.79	4.79	4	51.5	6.46	-0.06	9	48.8	6.13	-0.62	4	9.09	10.89	-0.24
(D6335)	72	Deposit wt. (mg)	27	26.72	3.46	6	25.9	4.10	-0.23	9	29.8	10.60	0.89	9	29.8	8.72	0.88
MTEOS	432	Deposit wt. (mg)	30	47.04	4.50	18	47.2	4.58	-0.05	13	45.9	5.01	-0.25	20	48.7	5.87	0.38
(D7097)	434	Deposit wt. (mg)	30	27.37	6.57	14	26.4	7.29	-0.17	16	27.0	9.07	-0.05	14	30.4	10.59	0.46
* * *	74	Deposit wt. (mg)	30	12.85	5.59	1	12.4	6.23	-0.06	18	11.6	7.50	-0.23	14	13.4	6.45	0.09
Ō	58	Gelation Index	17	5.8	0.69	6	6.1	1.07	0.42	00	0.9	0.57	0.22	6	6.2	0.75	0.64
(D5133)	62	Gelation Index	35	17.0	3.90	13	13.8	5.45	-0.82	∞	14.1	90.9	-0.75	8	13.2	5.71	-0.96
***	1009	Gelation Index	16	7.30	0.68	7	6.7	0.59	-0.95	13	6.5	0.97	-1.20	7	6.8	0.67	-0.69
D6082	1007	Tendency (ml)	28	65	19	12	99	16	-0.01	6	61	12	-0.26	10	63	18	-0.16

<sup>\*\*</sup>D5800 Targets Adjusted 10/2/00; new oils selected; new procedures approved; targets adjusted again 7/21/03 \*\*\*MTEOS Targets Adjusted: 6/1/01 (matrix); 11/1/03 (SC9 RR2); 2/18/04 (add 432); 1/12/05 (add 434, drop 433 & 1006); 6/30/05 (Batch E ref. data); 6/31/06 (updated ref. data n=30) \*\*\*\*\*GI: Added oil 1009 and dropped oils 52 & 53 10/15/03; added oil 58 10/24/01; dropped oils 51 & 55 7/2/01