

| MEMORANDUM: | 03-046 |
|-------------|---|
| DATE: | May 6, 2003 |
| TO: | Mr. Ted Selby, Chairman ASTM D02.B07 |
| FROM: | Thomas Schofield & Richard Grundza |
| SUBJECT: | TMC Bench Reference Test Monitoring from October 1, 2002 through March 31, 2003 |

We respectfully submit the TMC's ASTM D02.B07 Bench Reference Test Monitoring Semiannual Report, 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, where:

Pooled s = Standard deviation pooled across reference oils

(i.e., The pooled precision of the test this period.)

Δ/s = [(Result) - (Target mean)] / (Target s)

(i.e., "How many standard deviations from the target mean is this test?")

Mean Δ/s = [Σ (Δ/s)] / n (across reference oils 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?")

Notice that the period severity estimates (mean Δ /s) can be pooled across oils of different performance levels, because the individual test results used to calculate mean Δ /s have all been normalized into (target) standard deviations (Δ /s) for each corresponding reference oil. Using a pooled s for precision simplifies the interpretation of precision across all reference oil performance levels. These two calculations (pooled s and mean Δ /s) allow us to combine all calibration performance levels for each period into single 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.

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The 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.)

Beginning with the report period April 1, 2001 through September 30, 2001, we are reporting on consecutive six-month intervals for all test areas, rather than one-year intervals for some test areas and six-month for others. 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

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/mem03-046.pdf

Distribution: Email

Attachment 1

ASTM Test Monitoring Center

Semiannual Report

ASTM D02.B07 Bench Reference Test Monitoring From October 1, 2002 through March 31, 2003

D6417: Estimation of Engine Oil Volatility by Capillary Gas Chromatography

STATUS

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

| | No. of Tests |
|---|--------------|
| Statistically Acceptable and Operationally Valid | 14 |
| Operationally Valid but Failed Acceptance Criteria | 1 |
| Operationally Invalid (after informed of failing calibration) | 1 |
| Total | 16 |
| | |

TABLE 1

Fail Rate of Operationally Valid Tests: 6.7%

Table 2 is a breakdown of the statistically unacceptable tests.

| TABLE 2 | |
|------------------------|--------------|
| Reason for Fail | No. of Tests |
| Area % Volatized Mild | 1 |

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.)

| Area % Volatized @ 371°C | n n | df | Pooled s | Mean ∆/s |
|---------------------------|-----|-----|----------|----------|
| Initial Round Robin Study | 107 | 101 | 0.46 | |
| 10/5/00 through 3/31/01 | 18 | 15 | 0.50 | 1.42 |
| 4/1/01 through 9/30/01 | 16 | 13 | 0.54 | 0.65 |
| 10/1/01 through 3/31/02 | 13 | 10 | 0.44 | -0.45 |
| 4/1/02 through 9/30/02 | 16 | 13 | 0.34 | -0.29 |
| 10/1/02 through 3/31/03 | 15 | 12 | 0.39 | -0.47 |

TABLE 3

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.

| IADLE 4 | | | | |
|---------|---|----------|--|--|
| | n | Mean ∆/s | | |
| Lab A | 5 | -1.29 | | |
| Lab B | 2 | 0.40 | | |
| Lab D | 2 | -0.12 | | |
| Lab G | 2 | -0.36 | | |
| Lab H | 2 | 0.53 | | |
| Lab S | 2 | -0.72 | | |

TABLE 4

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

PRECISION AND SEVERITY

D6417 calibration testing precision is only directionally worse this period, and continues to be better than target. Overall severity continues mild of target performance, and is milder than last period. Severity is represented graphically in Figure 1. A strong three-test mild trend early in the period (starting at the 01OCT02 time-line) is followed by a series of tests that, overall, are closer to target. Lab A, with five tests (four more than 1 s mild and one slightly severe), is strongly influencing the overall mild trend, along with two additional fairly mild tests contributed from lab S.

Lab A's mild performance this period continues the significantly mild performance from last period (previous period average severity for Lab A was 1.19 s mild, n = 4). Labs S reported only one test last period; it was -0.94 s from target.

The fail rate of the operationally valid tests is good, with only one statistically unacceptable test reported this period.

TMC MEMORANDA

There were no TMC technical memoranda 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):

| TINDEL 5 | |
|---|--------------|
| | No. of Tests |
| Statistically Acceptable and Operationally Valid | 26 |
| Operationally Valid but Failed Acceptance Criteria | 8 |
| Operationally Invalid (initially reported as) | 0 |
| Operationally Invalid (after informed of failing calibration) | 3 |
| Total | 37 |
| | , |

TABLE 5

Fail Rate of Operationally Valid Tests: 23.5%

All 8 Statistically unacceptable test this period were by Procedure B

All 3 Operationally Invalid test reported this period were by Procedure C (all from Lab C)

Table 6 is a breakdown of the statistically unacceptable tests.

| TABLE 6 | | |
|---|--------------|--|
| Reason for Fail | No. of Tests | |
| Sample Evaporation Loss Severe | 8 | |
| (seven tests severe on Oil 58 and one severe on Oil 55) | | |

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 |
| Initial Round Robin Study | 180 | 175 | 0.51 | |
| New Targets Effective 9/26/00 | 178 | 175 | 0.56 | |
| 4/1/00 through 3/31/01 | 47 | 42 | 0.69 | 0.98 |
| 4/1/01 through 9/30/01 | 35 | 32 | 0.61 | 1.21 |
| 10/1/01 through 3/31/02 | 33 | 30 | 0.66 | 0.79 |
| 4/1/02 through 9/30/02 | 35 | 32 | 0.79 | 1.00 |
| 10/1/02 through 3/31/03 | 34 | 31 | 0.63 | 1.03 |

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 | 4 | 1 | 0.28 | -0.57 |
| Procedure B | 27 | 24 | 0.55 | 1.25 |
| Procedure C | 3 | 1 | 0.35 | 1.13 |

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.

| | n | Mean ∆/s |
|-------|---|----------|
| Lab A | 8 | 1.10 |
| Lab B | 5 | 0.95 |
| Lab D | 2 | 0.78 |
| Lab G | 8 | 1.96 |
| Lab H | 2 | 0.81 |
| Lab I | 3 | 0.56 |
| Lab J | 4 | -0.15 |
| Lab R | 2 | 0.73 |

| ΤA | BI | Æ | 9 |
|----|----|---|---|
| | | | |

PRECISION AND SEVERITY

Effective September 26, 2000, the TMC began monitoring the three Noack procedures under the latest D5800 test method. Also effective September 26, 2000, new reference oils, targets and acceptance bands were implemented for TMC calibration monitoring. Oils 51, 53 and 54 were dropped, oil 58 was introduced and targets for oils 52 & 55 were revised.

Overall precision, though slightly improved this period, continues to trend worse than target precision. Overall severity continues trending severe, at about the same level as before (1 s severe). The slopes of the plots in Figures 2A and 2B illustrate the continuous overall severe trend with D5800 calibration testing. Figure 2B shows that a strong severe trend that started a long time before new targets were established continues right on through the effective date of the new performance targets and up to the present time. A leveling to target would have been expected after the performance targets were updated in September 2000.

Testing failure rates on tests reported to the TMC as operationally valid for the last four report periods are 22.9%, 15.2%, 25.7% and, now, 23.5% (5% is "statistically expected"). The reason for the high fail rate is likely a result of the labs not meeting the acceptance bands for oil 58 (and, previously, on Oil 55 also). A previous round robin study does not indicate that oil 58 is any more variable in performance than oils 52 or 55, as some have suggested. Rather, it would appear that the target mean, at least for oil 58, and possibly for oil 55, is not accurate.

Industry performance on all three oils is severe of targets, with Oil 52 performance at 0.29 s severe of target, Oil 55 at 1.21 s severe and Oil 58 at 1.59 s severe (see Attachment 3A). Last period both oils 55 & 58 were severe, with oil 55 performing more severe than Oil 58. Attachment 3A shows a detailed comparison of the individual oil performances over time.

Given the overall consistent severe performance of D5800 reference tests, along with the consistently high statistical fail rate (particularly on Oil 58) the surveillance panel should question whether the test is actually performing severe (that is, is non-reference testing giving severer than expected results?) or whether the reference oil performance targets are incorrectly set.

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

TMC MEMORANDA

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

Memo 02-102, November 8, 2002 (correction issued December 3, 2002), D5800 A & B 2002 Post-Workshop Round-Robin Statistical Summary

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

STATUS

Table 10 summarizes the reference tests reported to the TMC this period (8 labs reporting):

| | No. of Tests |
|---|--------------|
| Statistically Acceptable and Operationally Valid | 27 |
| Operationally Valid but Failed Acceptance Criteria | 3 |
| Operationally Invalid (initially reported as) | 2 |
| Operationally Invalid (after informed of failing calibration) | 2 |
| Total | 34 |

TABLE 10 Reference Tests

Fail Rate of Operationally Valid Tests: 10.0%

Table 11 is a breakdown of the statistically unacceptable tests.

| TABLE 11 | |
|-----------------------|--------------|
| Reason for Fail | No. of Tests |
| Gelation Index Severe | 3 |

INDUSTRY PERFORMANCE

Table 12 shows the current Industry precision and severity for the Gelation Index and test parameter for all operationally valid tests for the report period. (First calibration test completed 4/20/96.) "Initial Tests" includes reference and donated tests; subsequent listings include only reference tests.

| TABLE 12 | | | | |
|--|-----|-----|----------|----------|
| Gelation Index | n | df | Pooled s | Mean ∆/s |
| Initial Tests 4/20/96 through 11/27/96 | 178 | 173 | 6.37 | |
| 4/20/96 through 3/31/97 | 60 | 55 | 5.40 | -0.06 |
| 4/1/97 through 3/31/98 | 64 | 59 | 5.20 | -0.12 |
| 4/1/98 through 3/31/99 | 68 | 63 | 6.67 | -0.07 |
| 4/1/99 through 3/31/00 | 62 | 57 | 6.30 | 0.09 |
| *4/1/00 through 3/31/01 | 65 | 60 | 5.93 | -0.15 |
| 4/1/01 through 9/30/01 | 33 | 28 | 2.84 | 0.13 |
| 10/1/01 through 3/31/02 | 30 | 26 | 4.76 | -0.02 |
| *4/1/02 through 9/30/02 | 32 | 28 | 2.15 | 0.43 |
| **10/1/02 through 3/31/03 | 30 | 26 | 2.02 | 0.87 |
| **10/1/02 through 3/31/03 | 29 | 25 | 2.02 | 0.59 |

*Excludes one data point as a rare event (for details, see the TMC's semiannual report for that period). **Summary statistics with and without LAB G result of 9 s severe of target, for comparison.

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

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

| TABLE 13 | | | | |
|----------|---|----------|--|--|
| | | GI | | |
| | n | Mean ∆/s | | |
| Lab A | 7 | 0.38 | | |
| Lab B | 5 | 1.13 | | |
| Lab D | 2 | 1.34 | | |
| *Lab G | 6 | 2.36 | | |
| *Lab G | 5 | 0.99 | | |
| Lab H | 2 | -0.55 | | |
| Lab I | 4 | 0.69 | | |
| Lab S | 3 | -0.52 | | |
| Lab U | 1 | 0.95 | | |

*Lab G with and without result of 9 s severe of target, for comparison

PRECISION AND SEVERITY

Effective October 24, 2001, new D5133 reference oils, targets and acceptance bands were implemented for TMC calibration monitoring. Oils 51 and 55 were dropped and oil 58 was introduced (targets for oils 52, 53 & 62 continue without revision). Current GI reference oils are 52, 53, 58 & 62.

Lab G reported a result this period on oil 52 (non-gelling), as operationally valid, which was 9 s severe of target. (The reported result is actually GI 6.7, with a target GI for oil 52 of 4.5 and acceptance bands 4.0 - 5.0. The test method indicates a GI less than 6 is non-gelling and should be reported as <6.) For comparison, I have included statistical summaries for the current period in Tables 12 & 13, and in Figures 3A & 3B with and without the extreme result included. Unless there is objection from the panel, the TMC will consider this result a rare event and exclude it (as a non-chartable test) from future period summaries and analyses due to the undue bias it creates in the precision and severity estimates for the period.

This is the third time the TMC has received a GI reference test with an unusually severe result. All three tests have been on TMC 52 (two from Lab A, 14 s & 20 s severe; one from Lab G, 9 s severe). It would appear from the reference data that oil 52 produces an occasional extremely severe result.

The 10.0% fail rate this period is comparable to last period (9% fail), though still higher than the statistically expected rate of 5%. Overall gelation index precision continues to be very good (with or without the extreme result) and remains considerably better than target. Overall severity continues to worsen (more severe), even with the severe result removed. Severity is graphically represented in Figures 3A & 3B (attached). Figure 3B (with the extreme result excluded) better shows the disturbing severe trend starting from the 01APR02 timeline.

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

An industry round-robin matrix was run on proposed GI reference oil 1009. The oil's performance in the matrix was somewhat milder than expected, but the results were reasonably precise across labs. The round-robin results were summarized in a separate TMC report issued October 2002 and we are waiting on a decision from the surveillance panel as to whether or not to amend the selection of reference oils to include oil 1009.

TMC MEMORANDA

There was one TMC technical memorandum issued this report period for the D5133 test method:

Memo 02-098, October 22, 2002, D5133 Round-Robin Results: Proposed Reference Oil 1009

D6335: 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):

| ΤA | BLE | 14 |
|----|-----|----|
| | | |

| | No. of Tests |
|---|--------------|
| Statistically Acceptable and Operationally Valid | 5 |
| Operationally Invalid (after informed of failing calibration) | 1 |
| Total | 6 |

Fail Rate of Operationally Valid Tests: 0.0%

INDUSTRY PERFORMANCE

Table 15 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 15 | | | | |
|---------------------------|----|----|----------|----------|
| Total Deposits | n | df | Pooled s | Mean ∆/s |
| Initial Round Robin Study | 54 | 52 | 4.18 | |
| 4/1/96 through 3/31/97 | 44 | 42 | 6.22 | 0.28 |
| 4/1/97 through 3/31/98 | 41 | 39 | 4.24 | -0.10 |
| 4/1/98 through 3/31/99 | 36 | 34 | 5.68 | -0.49 |
| 4/1/99 through 3/31/00 | 30 | 28 | 5.67 | 0.14 |
| 4/1/00 through 3/31/01 | 18 | 16 | 8.45 | 0.40 |
| 4/1/01 through 9/30/01 | 5 | 3 | 2.04 | 0.48 |
| 10/1/01 through 3/31/02 | 6 | 4 | 1.32 | 0.83 |
| 4/1/02 through 9/30/02 | 7 | 5 | 4.22 | 1.26 |
| 10/1/02 through 3/31/03 | 5 | 3 | 5.44 | 0.50 |

Table 16 shows the current severity for the Total Deposits parameter for each lab for all operationally valid tests in the report period.

| TABLE 16 | | | | |
|----------|---|----------|--|--|
| | n | Mean ∆/s | | |
| Lab A | 2 | 0.31 | | |
| Lab B | 2 | 0.15 | | |
| Lab G | 1 | 1.59 | | |

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

PRECISION AND SEVERITY

Calibration testing has dropped significantly with the introduction of the MHT-4 TEOST to replace TEOST-33C for GF-3/SL.

Overall precision has worsened again this period, and is worse than target. Severity is severe of targets, though much improved compared to the last two periods. Lab G hadn't calibrated in quite a long time came back into the system this period with a single rather severe (but passing) result. The severity trend is graphically represented in Figure 4 (attached). The plot shows some leveling this period compared to the severe trend from July 2001 through October 2002.

TMC MEMORANDA

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

TEOST MHT-4, Draft 17, 00.08.11: Determination of Moderately High Temperature Piston Deposits by Thermo-oxidation Engine Oil Simulation Test (MTEOS)

STATUS

Table 17 summarizes the reference tests reported to the TMC this period (7 labs reporting):

| | No. of Tests |
|---|--------------|
| Statistically Acceptable and Operationally Valid | 37 |
| Operationally Valid but Failed Acceptance Criteria | 5 |
| Operationally Invalid (initially reported as) | 1 |
| Operationally Invalid (after informed of failing calibration) | 4 |
| Total | 47 |

TABLE 17

Fail Rate of Operationally Valid Tests: 11.9%

Table 18 is a breakdown of the statistically unacceptable tests.

| TABLE 18 | | | |
|------------------------|--------------|--|--|
| Reason for Fail | No. of Tests | | |
| Total Deposits Mild | 4 | | |
| Total Deposits Severe | 1 | | |

INDUSTRY PERFORMANCE

Table 19 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.)

| | INDLL I. | / | | |
|----------------------------------|----------|----|----------|----------|
| Total Deposits | n | df | Pooled s | Mean ∆/s |
| Updated Targets Effective 6/1/01 | 80 | 76 | 5.40 | |
| 4/1/01 through 9/30/01 | 34 | 30 | 5.61 | -0.47 |
| 10/1/01 through 3/31/02 | 44 | 40 | 6.56 | -0.44 |
| 4/1/02 through 9/30/02 | 47 | 43 | 6.74 | -0.80 |
| 10/1/02 through 3/31/03 | 42 | 38 | 6.77 | -0.78 |

TABLE 19

Table 20 shows the current severity for the Total Deposits parameter for each lab for all operationally valid tests in the report period.

| TABLE 20 | | | | |
|----------|----|----------|--|--|
| | n | Mean ∆/s | | |
| Lab A | 11 | -0.13 | | |
| Lab AB | 2 | -0.15 | | |
| Lab B | 10 | -0.69 | | |
| Lab D | 2 | -1.12 | | |
| Lab G | 8 | -1.01 | | |
| Lab I | 1 | -0.80 | | |
| Lab V | 8 | -1.65 | | |

<u>TEOST MHT-4, Draft 17, 00.08.11: Determination of Moderately High Temperature Piston</u> Deposits by Thermo-oxidation Engine Oil Simulation Test (MTEOS), continued

PRECISION AND SEVERITY

Overall precision, though comparable to last period, is worse than target precision. Severity is substantially mild of target, comparable to last period, with all labs performing mild on the reference oils (though, as table 20 shows, some labs are substantially more mild than others). Severity is presented graphically in Figure 5 where an overall mild slope is observed (though it is interesting to note the short period on either side of the 01JAN03 timeline where more on-target, and even some severe results were reported, followed by an even steeper mild trend).

Last period a high number of operationally invalid tests were reported (often with the lab not realizing a problem until informed that they had failed on a TMC calibration oil). Last periods fail rate for tests reported as operationally valid was 21.3%, this periods fail rate is 11.9% (5% is statistically "expected").

As pointed out last report period, it appears, over time, that the precisions of the individual reference oils (Attachment 3A) have fluctuated substantially. Except for oil 74, there appears to be little consistency in the precision of the other three reference oils over time. Attachment 3A also shows that all four oils are again performing mild this period, with 432 performing significantly more mild (-1.7 s) than the other three oils. However, this performance is biased by three extreme results on 432 this period. Lab V has reported 3 failing results on oil 432 as operationally valid, with all three results reported more than 3 s mild of target. Removing these three results from the statistics causes the severity to drop to a more reasonable 0.86 s mild of target. Lab G also performs substantially mild on 432 this period (2 tests at more than 1.7 s mild) whereas Lab B seems to perform consistently near target on 432. Recent problems at one lab strongly suggests that oil 432 is extremely sensitive to operational conditions (more so than the other three reference oils) and, though difficult to maintain on-target performance, may prove to be a good indicator of operational problems or changes.

(It might be of interest that TMC Reference Oil 432 is the same oil and batch as TMC Reference Oil 58 that is used in D5800 calibration monitoring. The participating labs also have trouble maintaining target performance on this same oil in the D5800 calibration monitoring program. It has been pointed out to the TMC that the results for both D5800 and MTEOS are temperature dependent, and both tests run at roughly similar temperatures, 250°C vs. 285°C.)

Last period the TMC suggested that we start tracking Rod Batches (a critical hardware testing part in the MHT-4 TEOST). A serial number for each test rod is supplied to the TMC for each reported calibration test, but the TMC has no breakdown of how these serial numbers relate to manufacturer, or rod batches by a single manufacturer. Recently, the surveillance panel has asked the TMC to start collecting this data. The rods supplier has agreed to start supplying a rod batch ID with each shipment of rods, and the TMC is modifying the report package to include a field to collect this data. The TMC was also asked to correlate our current data base of rod serial numbers to rod batches, but, to date, the rod supplier has not divulged the rod batches to correspond with the rod serial numbers in our data base.

TMC MEMORANDA

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

D6082: High Temperature Foaming Characteristics of Lubricating Oils

On June 18, 2001, the section agreed to suspend the use of 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. A search for a suitable replacement oil has been ongoing.

Note that TMC 1007 has a Foam Stability (one minute after disconnect) target mean performance of zero ml and a target precision (standard deviation) of zero ml. Any negative (mild) result for this parameter is unlikely and any positive result would be "infinitely" severe in standard deviations (Δ /s). Therefore, for Foam Stability, it is preferable to simply note the number of non-zero occurrences in order to flag any severity trends.

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

STATUS

Table 21 summarizes the reference tests reported to the TMC this period (5 labs reporting):

| | No. of Tests |
|--|--------------|
| Statistically Acceptable and Operationally Valid | 11 |
| Operationally Valid but Failed Acceptance Criteria | 0 |
| Operationally Invalid | 0 |
| Total | 11 |

TABLE 21

Fail Rate of Operationally Valid Tests: 0.0%

D6082: High Temperature Foaming Characteristics of Lubricating Oils, continued

TMC 1007 INDUSTRY PERFORMANCE

Tables 22 and 23 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 22 | | | | |
|-------------------------------------|----|-------|-------|----------|
| 1007 Foam Tendency, ml | n | Mean | S | Mean Δ/s |
| Initial Round Robin Study (targets) | 28 | 65.71 | 19.28 | |
| 4/12/99 through 3/31/00 | 17 | 65.3 | 18.41 | -0.02 |
| 4/1/00 through 3/31/01 | 14 | 67.5 | 11.22 | 0.09 |
| 4/1/01 through 9/30/01 | 9 | 71.1 | 14.53 | 0.28 |
| 10/1/01 through 3/31/02 | 11 | 64.5 | 15.07 | -0.06 |
| 4/1/02 through 9/30/02 | 12 | 62.5 | 14.22 | -0.17 |
| 10/1/02 through 3/31/03 | 11 | 62.7 | 17.52 | -0.15 |

TABLE 23

| 1007 Foam Stability @ 1 min., ml | n | Mean | S | |
|----------------------------------|----|-------------------------|-------------|--|
| Initial Round Robin Study | 28 | 0.00 | 0.00 | |
| 4/12/99 through 3/31/00 | 17 | No non-zero | occurrences | |
| 4/1/00 through 3/31/01 | 17 | No non-zero occurrences | | |
| 4/1/01 through 9/30/01 | 9 | No non-zero | occurrences | |
| 10/1/01 through 3/31/02 | 11 | No non-zero | occurrences | |
| 4/1/02 through 9/30/02 | 12 | No non-zero | occurrences | |
| 10/1/02 through 3/31/03 | 11 | No non-zero | occurrences | |

Table 24 shows the current 1007 severity for the monitored result parameter for each lab for all operationally valid tests reported for the report period.

| TABL | LE 24 |
|------|-------|
| TMC | 1007 |

| 11/10 1007 | | | | |
|------------|---|------------------------------|--|--|
| | n | Foam Tendency Mean ∆/s | | |
| Lab A | 2 | 1.00 | | |
| Lab B | 4 | -0.69 | | |
| Lab D | 1 | -1.85 | | |
| Lab G | 2 | 0.22 | | |
| Lab I | 2 | 0.22 | | |

D6082: High Temperature Foaming Characteristics of Lubricating Oils, continued

PRECISION AND SEVERITY

Foam Tendency precision on 1007 is worse than last period, and better than the target precision. Severity is trending only slightly mild. There were no non-zero occurrences of Foam Stability on 1007; this would suggest Foam Stability precision is as expected. Foam Tendency severity is graphically represented in Figure 6 with some increased variability in the data this period.

A round-robin last period to select a severe performing reference oil was unsuccessful due to poor reproducibility. A teleconference was held in March to try to work out any operational discrepancies and another round-robin is pending.

TMC MEMORANDA

There was one TMC technical memorandum issued this report period for the D6082 test method:

Unapproved Minutes of the Test Method D6082 Teleconference "Workshop" Held o March 12, 2003 (minutes issued by Email on March 21, 2003).

D6557: Ball Rust Test (BRT)

Note that, for BRT, a positive Δ /s is mild, not severe (a higher AGV result is considered to be a more mild result while a lower AGV result is considered to be a more severe result.)

STATUS

Table 25 summarizes the reference tests reported to the TMC this period (4 labs reporting):

| | No. of Tests |
|--|--------------|
| Statistically Acceptable and Operationally Valid | 138 |
| Operationally Valid but Failed Acceptance Criteria | 5 |
| Aborted | 1 |
| Total | 144 |

TABLE 25

Fail Rate of Operationally Valid Tests: 3.6%

Table 26 is a breakdown of the statistically unacceptable tests.

| TABLE 26 | | | |
|--------------------|--------------|--|--|
| Reason for Fail | No. of Tests | | |
| Average AGV Mild | 4 | | |
| Average AGV Severe | 1 | | |

The aborted test was due to a pump malfunction.

INDUSTRY PERFORMANCE

Table 27 shows the current Industry precision and severity for the Average AGV test parameter for all operationally valid tests for the report period. (First calibration test completed 8/15/00.)

_ . _ _ _ _

| | TABLE 27 | 7 | | |
|-------------------------------------|----------|-----|----------|----------|
| Average AGV | n | df | Pooled s | Mean ∆/s |
| Initial Round Robin Study (targets) | 48 | 44 | 9.43 | |
| 8/15/00 through 9/30/00 | 28 | 25 | 10.50 | 0.38 |
| 10/1/00 through 3/31/01 | 112 | 109 | 8.48 | 0.42 |
| 4/1/01 through 9/30/01 | 156 | 153 | 8.90 | 0.36 |
| 10/1/01 through 3/31/02 | 116 | 113 | 12.46 | 0.67 |
| 4/1/02 through 9/30/02 | 138 | 135 | 11.38 | 0.76 |
| 10/1/02 through 3/31/03 | 143 | 140 | 7.76 | 0.69 |

Table 28 shows the current severity for the Average AGV parameter for each lab for all operationally valid tests for the report period.

D6557: Ball Rust Test (BRT), continued

| | n | Mean ∆/s |
|-------|----|----------|
| Lab A | 54 | 0.53 |
| Lab B | 51 | 1.11 |
| Lab G | 28 | 0.43 |
| Lab D | 10 | 0.10 |

TABLE 28

PRECISION AND SEVERITY

Precision this report period has improved when compared to the target matrix and the previous period. Overall severity is trending mild of target. Severity is graphically represented in Figure 7 (attached). All labs are trending mild of target, with the exception of lab D, which is on or near target. All five of the failing results were from oil 5A-3. Because of concerns of possible degradation of reference oil 5A-3, the panel is obtaining another severe reference oil to replace 5A-3.

TMC MEMORANDA

There were no technical memoranda issued this report period. As a result of a teleconference meeting of the Ball Rust Test Surveillance Panel, an information letter is being generated to address limits on the image analysis results obtained on the Calibration Reference Specimen at the beginning and end of the image analysis detailed in Test Method D6557.

Engine Oil Filterability Test (EOFT)

STATUS

Table 29 summarizes the reference tests reported to the TMC this period (3 labs reporting).

| | No. of Tests |
|--|--------------|
| Statistically Acceptable and Operationally Valid | 81 |
| Operationally Valid but Failed Acceptance Criteria | 0 |
| Aborted | 0 |
| Total | 81 |

TABLE 29

Fail Rate of Operationally Valid Tests: 0.0%

INDUSTRY PERFORMANCE

Table 30 shows the current Industry precision and severity for the Average % Change in Flow (CIF) test parameter for all operationally valid tests for the report period. (First calibration test completed 5/4/00.)

| TABLE 30 | | | | |
|-------------------------------------|-----|-----|----------|----------|
| Average % CIF | n | df | Pooled s | Mean ∆/s |
| Initial Round Robin Study (targets) | 24 | 22 | 5.76 | |
| 5/4/00 through 9/30/00 | 53 | 51 | 7.47 | 1.64 |
| 10/1/00 through 3/31/01 | 79 | 78 | 4.79 | 0.30 |
| 4/1/01 through 9/30/01 | 103 | 102 | 6.69 | -0.08 |
| 10/1/01 through 3/31/02 | 84 | 83 | 5.67 | -0.06 |
| 4/1/02 through 9/30/02 | 89 | 88 | 5.38 | 0.11 |
| 10/1/02 through 3/31/03 | 81 | 80 | 4.16 | -0.27 |

Table 31 shows the current severity for the Average % CIF parameter for each lab for all operationally valid tests for the report period.

| TABLE 31 | | | | |
|----------|----|----------|--|--|
| | n | Mean ∆/s | | |
| Lab A | 32 | -0.13 | | |
| Lab B | 25 | -0.10 | | |
| Lab G | 24 | -0.65 | | |

PRECISION AND SEVERITY

Precision this report period has improved when compared to the previous period and the target matrix. Overall severity trended mild for the period. Lab G trended mild, while Labs A and B were on or near target. Severity is graphically represented in Figure 8 (attached).

At this time, only TMC 78 is being assigned as TMC calibration oil. The panel is pursuing a replacement oil for TMC 77, which had been providing results significantly mild of target.

Engine Oil Filterability Test (EOFT), continued

TMC MEMORANDA

There were no technical memoranda issued this report period nor were any information letters issued this report period.

Engine Oil Water Tolerance Test (EOWT): 0.6% Water Treat Level

STATUS

Table 32 summarizes the reference tests reported to the TMC this period (3 labs reporting): TABLE 32

| TABLE 52 | |
|--|--------------|
| | No. of Tests |
| Statistically Acceptable and Operationally Valid | 88 |
| Operationally Valid but Failed Acceptance Criteria | 1 |
| Total | 89 |
| Eail Data of Operationally, Valid Testa: 1 | 10/ |

Fail Rate of Operationally Valid Tests: 1.1%

Table 33 is a breakdown of the statistically unacceptable tests.

| TABLE 33 | | | |
|--|--------------|--|--|
| Reason for Fail | No. of Tests | | |
| Average % Change in Flow Mild (Oil 77) | 1 | | |

INDUSTRY PERFORMANCE

Table 34 shows the current Industry precision and severity for the Average % Change in Flow (CIF) test parameter for all operationally valid tests for the report period. (First calibration test completed 5/4/00.)

| Average % CIF | n | df | Pooled s | Mean ∆/s |
|-------------------------------------|-----|-----|----------|----------|
| Initial Round Robin Study (targets) | 24 | 22 | 5.93 | |
| 5/4/00 through 9/30/00 | 34 | 32 | 6.25 | -0.039 |
| 10/1/00 through 3/31/01 | 101 | 99 | 5.61 | -0.173 |
| 4/1/01 through 9/30/01 | 123 | 121 | 6.28 | 0.047 |
| 10/1/01 through 3/31/02 | 88 | 86 | 6.12 | -0.048 |
| 4/1/02 through 9/30/02 | 102 | 100 | 4.50 | 0.181 |
| 10/1/02 through 3/31/03 | 89 | 87 | 4.86 | -0.075 |

Table 35 shows the current severity for the Average % CIF parameter for each lab for all operationally valid tests for the report period.

| TABLE 35 | | | |
|----------|----|----------|--|
| | n | Mean ∆/s | |
| Lab A | 37 | -0.62 | |
| Lab B | 24 | 0.22 | |
| Lab G | 28 | 0.40 | |

PRECISION AND SEVERITY

Precision is essentially unchanged when compared with the previous period and has improved when compared to the target matrix. Severity was on or near target for the period. Severity is graphically represented in Figure 9 (attached). Lab A trended mild, while labs B and G trended severe for the period.

Engine Oil Water Tolerance Test (EOWT): 1.0% Water Treat Level

STATUS

Table 36 summarizes the reference tests reported to the TMC this period (3 labs reporting):

| ΤA | RI | E | 36 | |
|----|----|------|----|--|
| ID | DL | , Li | 50 | |

| | No. of Tests |
|--|--------------|
| Statistically Acceptable and Operationally Valid | 88 |
| Operationally Valid but Failed Acceptance Criteria | 1 |
| Total | 89 |
| | 10/ |

Fail Rate of Operationally Valid Tests: 1.1%

Table 37 is a breakdown of the statistically unacceptable tests.

| TABLE 37 | | | |
|--|--------------|--|--|
| Reason for Fail | No. of Tests | | |
| Average % Change in Flow severe (Oil 78) | 1 | | |

INDUSTRY PERFORMANCE

Table 38 shows the current Industry precision and severity for the Average % Change in Flow (CIF) test parameter for all operationally valid tests for the report period. (First calibration test completed 5/4/00.) TABLE 38

| Average % CIF | n | df | Pooled s | Mean ∆/s |
|-------------------------------------|-----|-----|----------|----------|
| Initial Round Robin Study (targets) | 24 | 22 | 5.81 | |
| 5/4/00 through 9/30/00 | 33 | 31 | 6.98 | 0.12 |
| 10/1/00 through 3/31/01 | 99 | 97 | 5.85 | -0.19 |
| 4/1/01 through 9/30/01 | 115 | 113 | 5.79 | 0.26 |
| 10/1/01 through 3/31/02 | 89 | 87 | 7.20 | 0.02 |
| 4/1/02 through 9/30/02 | 105 | 103 | 4.30 | 0.25 |
| 10/1/02 through 3/31/03 | 89 | 87 | 3.42 | 0.25 |

Table 39 shows the current severity for the Average % CIF parameter for each lab for all operationally valid tests for the report period.

| TABLE 39 | | | |
|----------|----|----------|--|
| | Ν | Mean ∆/s | |
| Lab A | 37 | 0.06 | |
| Lab B | 24 | 0.00 | |
| Lab G | 28 | 0.89 | |

PRECISION AND SEVERITY

Precision has improved when compared to the previous period and historical rates. Industry data is trending severe. Lab G trended severe, while labs A and B were on or near target this report period. Severity is graphically represented in Figure 10 (attached).

Engine Oil Water Tolerance Test (EOWT): 2.0% Water Treat Level

STATUS

Table 40 summarizes the reference tests reported to the TMC this period (3 labs reporting):

| TABLE 40 | |
|--|--------------|
| | No. of Tests |
| Statistically Acceptable and Operationally Valid | 87 |
| Operationally Valid but Failed Acceptance Criteria | 2 |
| Total | 89 |

Fail Rate of Operationally Valid Tests: 2.2%

Table 41 is a breakdown of the statistically unacceptable tests.

| TABLE 41 | | | |
|--|--------------|--|--|
| Reason for Fail | No. of Tests | | |
| Average % Change in Flow severe (Oil 77) | 1 | | |
| Average % Change in Flow mild (Oil 78) | 1 | | |

T () D () ()

INDUSTRY PERFORMANCE

Table 42 shows the current Industry precision and severity for the Average % Change in Flow (CIF) test parameter for all operationally valid tests for the report period. (First calibration test completed 5/4/00.)

| Average % CIF | n | df | Pooled s | Mean ∆/s |
|-------------------------------------|-----|-----|----------|----------|
| Initial Round Robin Study (targets) | 24 | 22 | 7.08 | |
| 5/4/00 through 9/30/00 | 31 | 29 | 5.63 | -0.07 |
| 10/1/00 through 3/31/01 | 100 | 98 | 6.25 | -0.16 |
| 4/1/01 through 9/30/01 | 114 | 112 | 6.57 | 0.22 |
| 10/1/01 through 3/31/02 | 89 | 87 | 5.75 | -0.02 |
| 4/1/02 through 9/30/02 | 103 | 101 | 3.76 | 0.09 |
| 10/1/02 through 3/31/03 | 89 | 87 | 5.77 | 0.11 |

TABLE 42

Table 43 shows the current severity for the Average % CIF parameter for each lab for all operationally valid tests for the report period.

| TABLE | 43 |
|-------|----|
|-------|----|

| | n | Mean ∆/s |
|-------|----|----------|
| Lab A | 37 | -0.13 |
| Lab B | 24 | -0.14 |
| Lab G | 28 | 0.63 |

Engine Oil Water Tolerance Test (EOWT): 2.0% Water Treat Level continued

PRECISION AND SEVERITY

Precision for this period has degraded when compared to the previous period, but has improved when compared to the target estimates. Severity was on or near target for the period. Lab G trended severe for the period, while labs A and B were slightly mild for the period. Severity is graphically represented in Figure 11 (attached).

Engine Oil Water Tolerance Test (EOWT): 3.0% Water Treat Level

STATUS

Table 44 summarizes the reference tests reported to the TMC this period (3 labs reporting):

| TABLE 44 | |
|-----------|--|
| I ADLL TT | |

| | No. of Tests |
|--|--------------|
| Statistically Acceptable and Operationally Valid | 86 |
| Operationally Valid but Failed Acceptance Criteria | 3 |
| Total | 89 |
| | |

Fail Rate of Operationally Valid Tests: 3.3%

Table 45 is a breakdown of the statistically unacceptable tests.

| TABLE 45 | |
|--|--------------|
| Reason for Fail | No. of Tests |
| Average % Change in Flow Severe (Oil 78) | 1 |
| Average % Change in Flow Severe (Oil 77) | 1 |
| Average % Change in Flow Mild (Oil 78) | 1 |

INDUSTRY PERFORMANCE

Table 46 shows the current Industry precision and severity for the Average % Change in Flow (CIF) test parameter for all operationally valid tests for the report period. (First calibration test completed 5/4/00.)

TADIE 16

| Average % CIF | n | df | Pooled s | Mean ∆/s |
|-------------------------------------|-----|-----|----------|----------|
| Initial Round Robin Study (targets) | 24 | 22 | 5.79 | |
| 5/4/00 through 9/30/00 | 32 | 30 | 5.71 | 0.23 |
| 10/1/00 through 3/31/01 | 98 | 96 | 5.71 | -0.01 |
| 4/1/01 through 9/30/01 | 122 | 120 | 6.46 | 0.34 |
| 10/1/01 through 3/31/02 | 89 | 87 | 5.82 | 0.31 |
| 4/1/02 through 9/30/02 | 108 | 106 | 4.69 | 0.56 |
| 10/1/02 through 3/31/03 | 89 | 87 | 5.09 | 0.50 |

Table 47 shows the current severity for the Average % CIF parameter for each lab for all operationally valid tests for the report period.

| TABLE 47 | | | | |
|----------|----|----------|--|--|
| | n | Mean ∆/s | | |
| Lab A | 36 | 0.33 | | |
| Lab B | 25 | 0.10 | | |
| Lab G | 28 | 1.05 | | |

.

Engine Oil Water Tolerance Test (EOWT): 3.0% Water Treat Level continued

PRECISION AND SEVERITY

Precision has degraded slightly when compared to the previous period and compares well with the target matrix. Severity trended severe of target for the period. Severity is graphically represented in Figure 12 (attached). Laboratories A and G trended severe of target during the period. Lab B was on or near target for the period.

REFERENCE OIL SUPPLIES

There is adequate supply of PCEOCP Bench Test reference oils on hand at the TMC. Table 48 lists the PCEOCP bench test reference oils currently on hand at the TMC.

| Table 48 | | | | |
|----------|----------------------|----------------------------|--|--|
| Oil | For Tests | Quantity Left (gallons) | Quantity Used Last 12 Months (gallons) | |
| 5A-3 | BRT | 1787.2 | 0.6 | |
| 51 | GI | 94.6 | 0.0 | |
| 52 | D6417, D5800, GI | 71.1 | 12.0 | |
| 53 | GI | 96.8 | 0.2 | |
| ^54 | Obsolete Volatility | 97.8 | 0.0 | |
| 55 | D6417, D5800 | 76.1 | 11.8 | |
| ^57 | Volatility Candidate | 51.2 | 0.0 | |
| 58 | D6417, D5800, GI | 128.9 | 12.1 | |
| 62 | GI | 2.0 | 0.2 | |
| ^66 | D6082 Candidate | 105.6 | 2.4 | |
| 71 | TEOST | 4.6 | 0.4 | |
| 72 | TEOST | 4.6 | 0.2 | |
| 74 | MTEOS | 2.3 | 0.4 | |
| 77 | EOWT | 172.1 | 26.8 | |
| 78 | EOFT, EOWT | 132.8 | 40.2 | |
| ^80 | BRT | 26.5 | 0.0 | |
| 81 | BRT | 19.1 | 1.4 | |
| ^82 | BRT | 10 | 0.0 | |
| **432 | MTEOS | Adequate | | |
| **433 | MTEOS | Adequate | | |
| 1006 | BRT, MTEOS | 44.8 | | |
| *1007 | FOAM | Est. 30 | | |

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

*One drum of oil is set aside for bench calibration testing; the TMC has a larger supply of this oil. **Five gallon aliquot set aside for bench testing; hard to get an inventory reading on amount set aside.

REFERENCE OIL SUPPLIES, continued

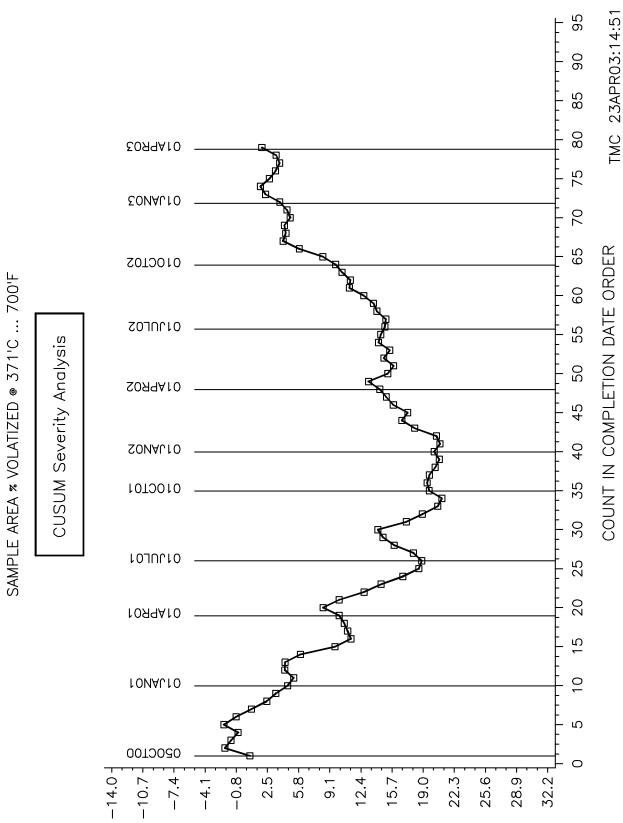
Shipping aliquots are:

| D6417 | 1 ml |
|-------|--------|
| D5480 | 4 ml |
| D5800 | 100 ml |
| GI | 25 ml |
| MTEOS | 17 ml |
| TEOST | 125 ml |
| D6082 | 525 ml |
| EOFT | 290 ml |
| EOWT | 290 ml |
| BRT | 30 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 (like test development studies, test performance studies and reference oil selection 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. If your lab should require additional information on this type of data reporting, please contact Tom Schofield at (412) 365-1011 or Rich Grundza at (412) 365-1031.



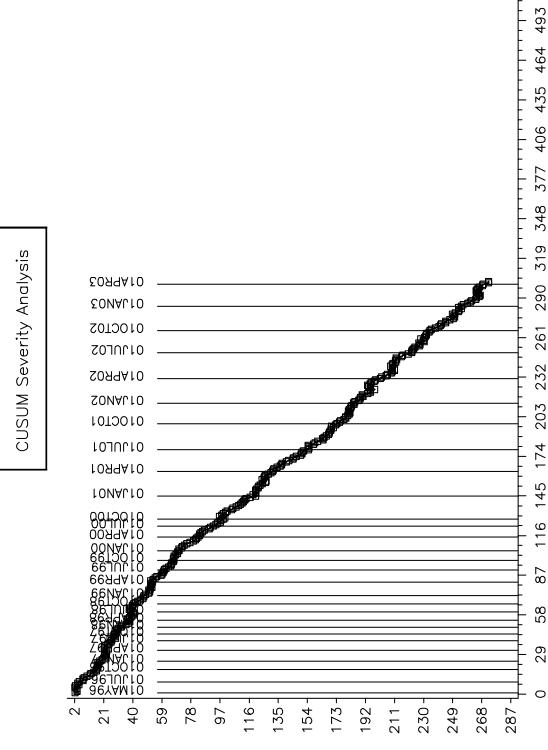
Standard Deviation Units





D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA

TEST OIL SAMPLE EVAPORATION LOSS, MASS³



TMC 24APR03:15:37

COUNT IN COMPLETION DATE ORDER

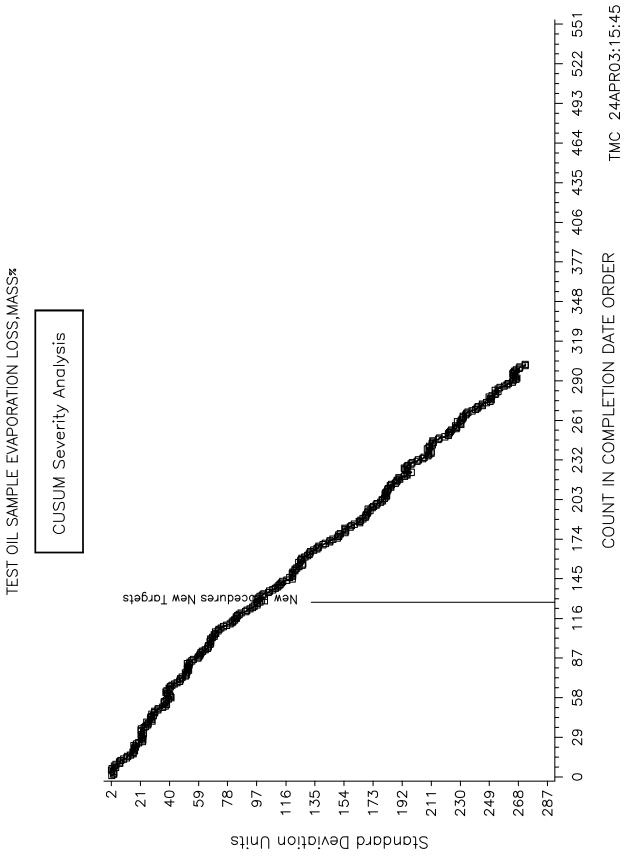
551

522

Standard Deviation Units



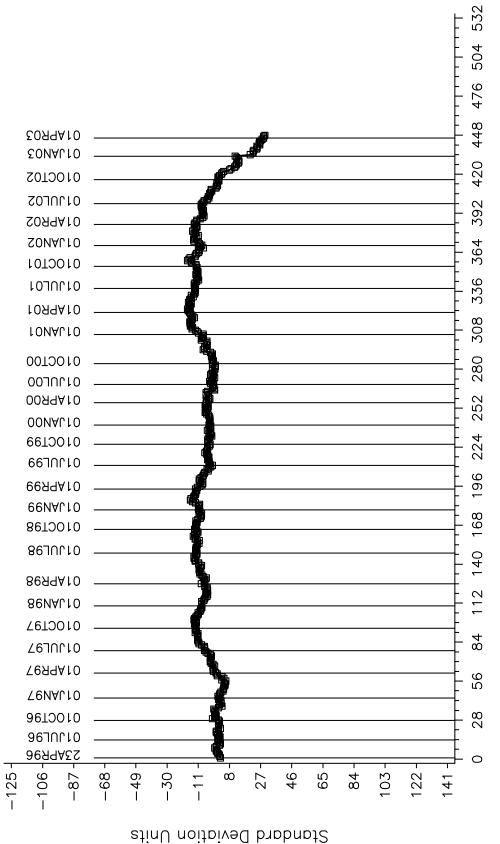
D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA





With One Exceptionally Severe Data Point Included (LAb G, Oil 52, CMIR 44338) D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA **GELATION INDEX**



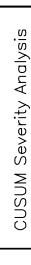


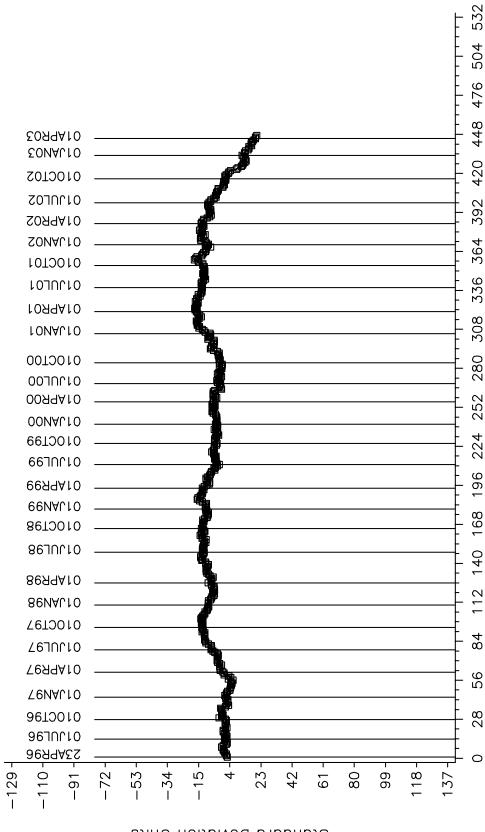
TMC 29APR03:14:12

COUNT IN COMPLETION DATE ORDER

Figure 3B

With One Exceptionally Severe Data Point Removed (Lab G, Oil 52, CMIR 44338) D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA **GELATION INDEX**





TMC 29APR03:13:32

COUNT IN COMPLETION DATE ORDER

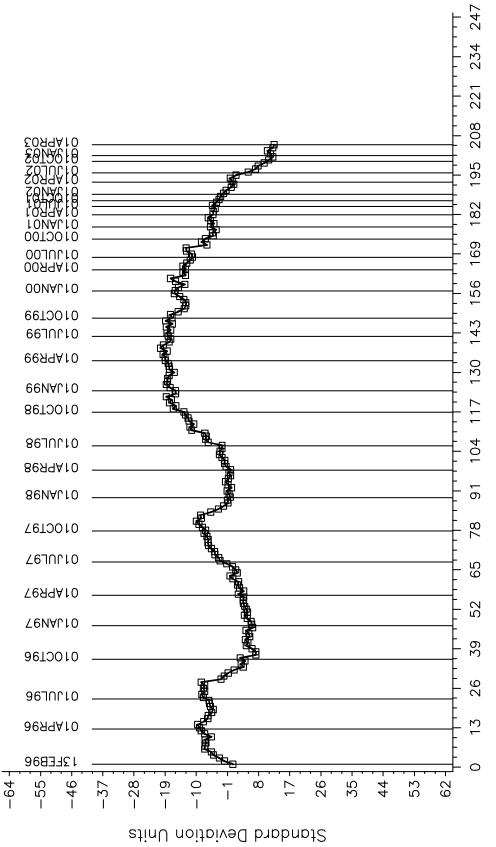
Standard Deviation Units



TEOST-33C INDUSTRY OPERATIONALLY VALID DATA

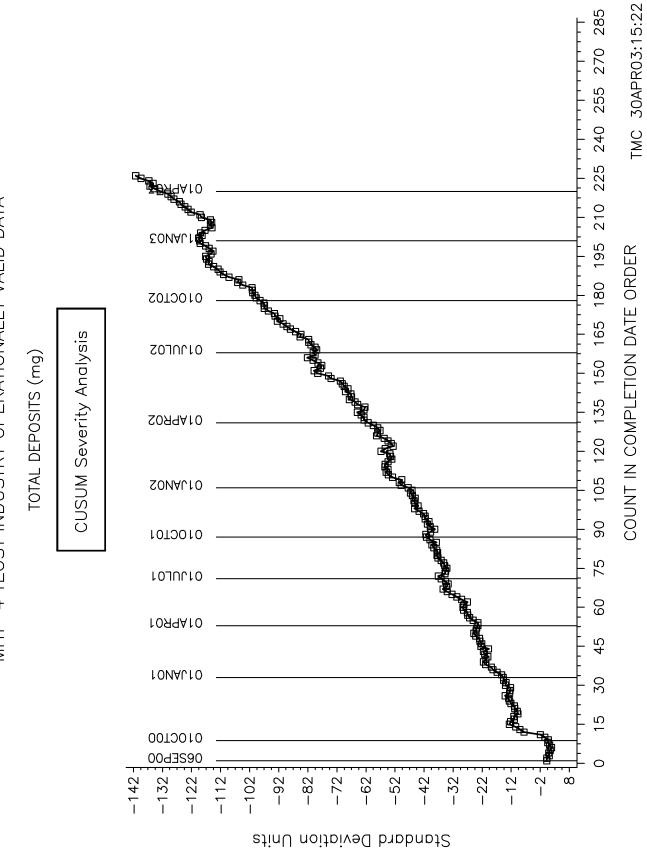
TOTAL DEPOSITS (mg)





TMC 29APR03:15:17

COUNT IN COMPLETION DATE ORDER



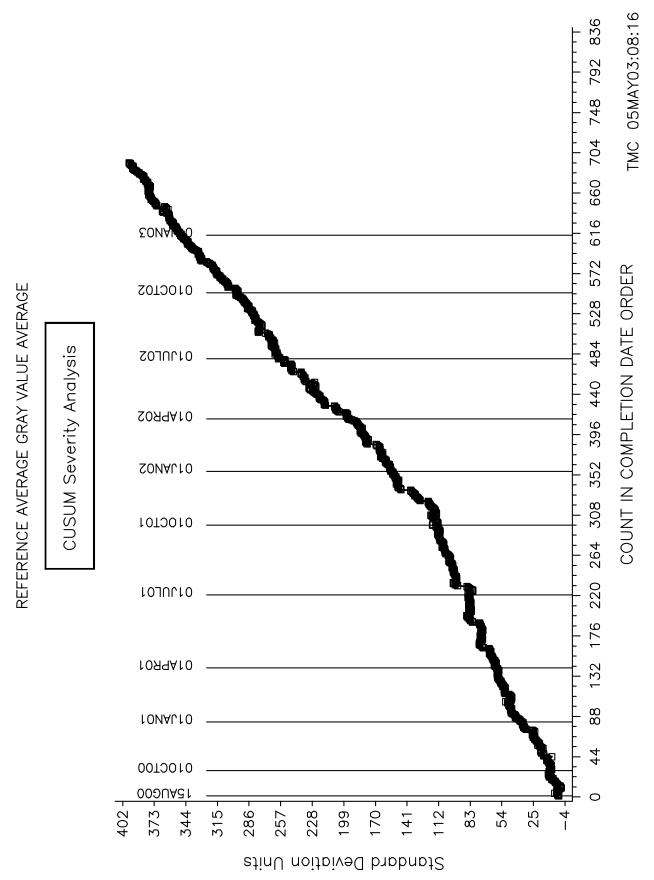
MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA

TMC 01MAY03:11:12 85 80 75 20Я9A10 70 EONALIO FOAM TENDENCY, IMMEDIATELY BEFORE DISCONNECT STATI 65 COUNT IN COMPLETION DATE ORDER 010CT02 60 0110105 55 **CUSUM Severity Analysis** 20A9A10 50 d d d SONALIO 45 10TO010 40 35 0170101 10A9A10 30 10NAL10 25 0100100 011000 20 00A9A10 15 OONALIO 0 010C199 011NF36 ഹ 12APR99 0 -22.0 --18.8--15.6 --0.9-22.8 --2.8-0.4 10.0-19.6 --12.4 -9.2 3.6 0 8 9 13.2 16.4

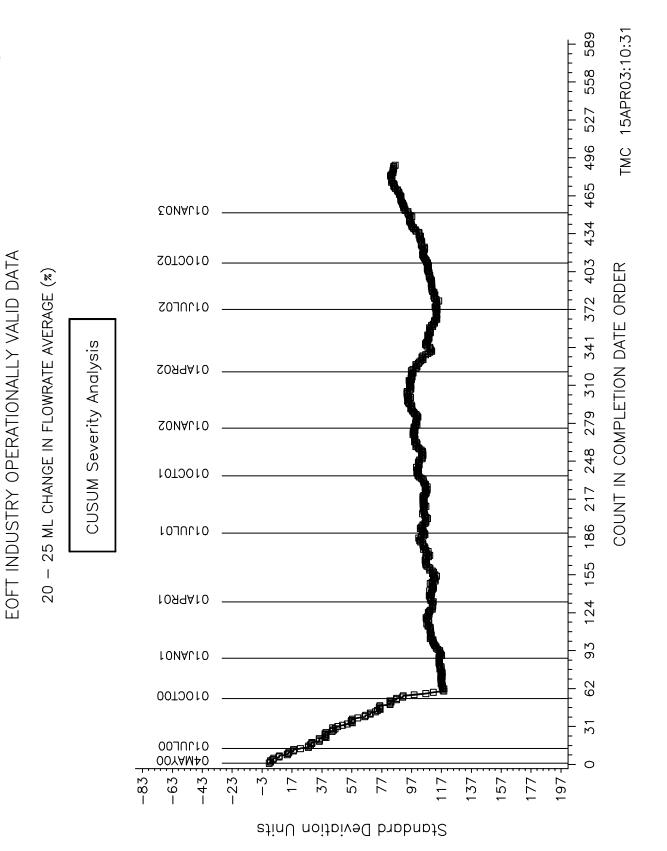
95

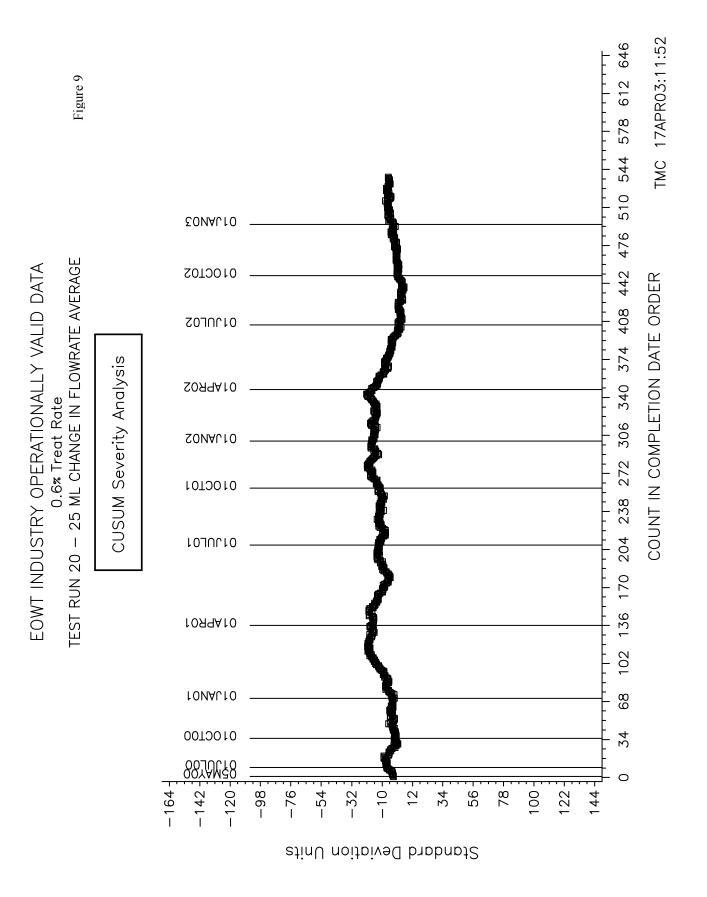
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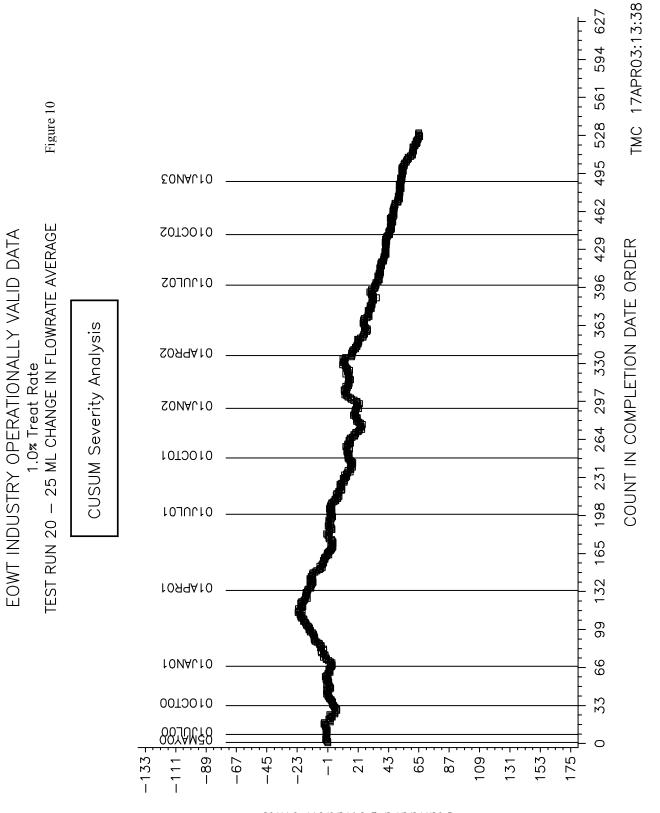
D6082 HIGH TEMPERATURE FOAM INDUSTRY OPERATIONALLY VALID DATA Oil 1007 Data Only

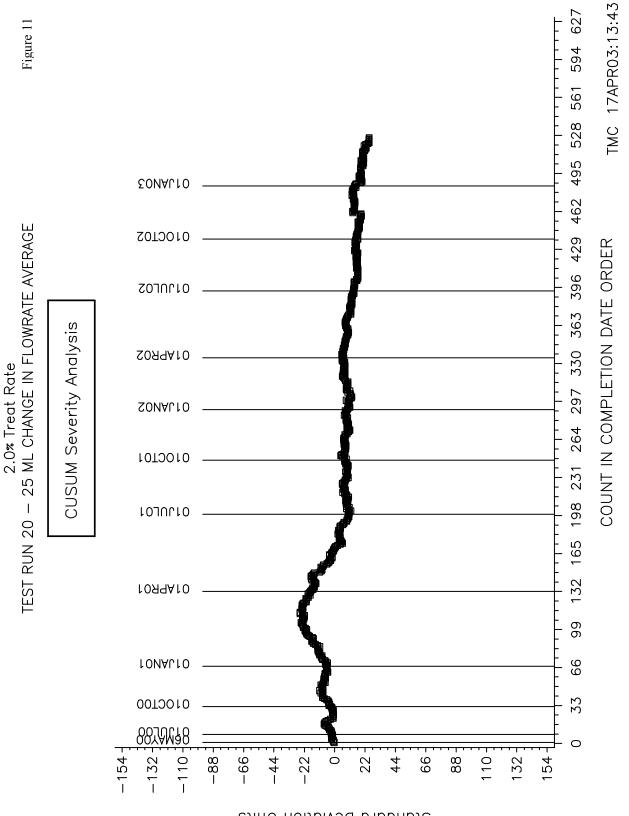


BALL RUST TEST INDUSTRY OPERATIONALLY VALID DATA

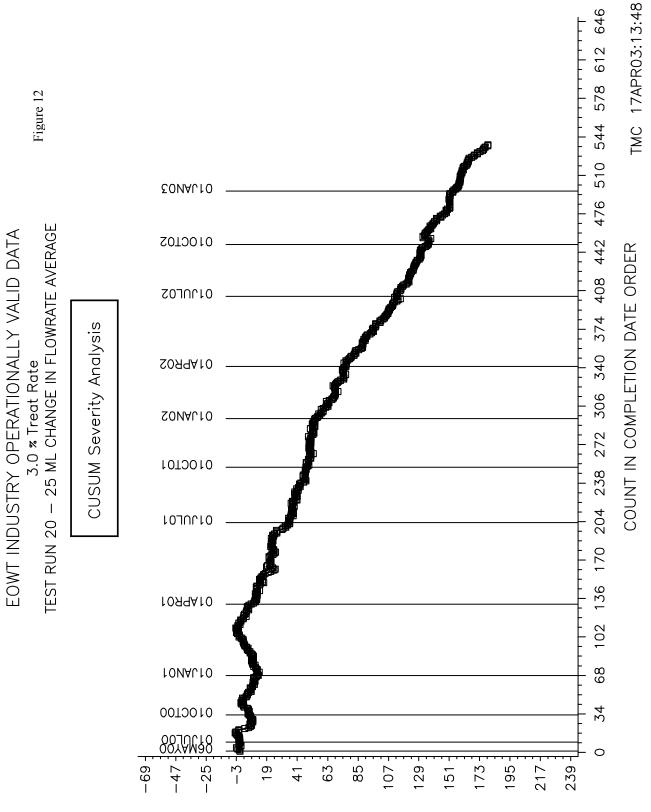








EOWT INDUSTRY OPERATIONALLY VALID DATA



TMC Monitored Bench Tests Reference Oil Test Targets and Acceptance Bands

| | | | | | | Acceptane | ce Bands * |
|-------------------|----------|--------------------------------|----|--------|-------|-----------|------------|
| | | | | | | 95 | 5% |
| 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 | 59 | 13.61 | 0.49 | 12.6 | 14.6 |
| New Targets | 55 | mass % volatility loss | 60 | 16.39 | 0.66 | 15.1 | 17.7 |
| 9/26/00 | 58 | mass % volatility loss | 59 | 14.46 | 0.52 | 13.4 | 15.5 |
| TEOST by | 71 | 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) | 20 | 16.84 | 5.28 | 6.5 | 27.2 |
| Draft 17 00.08.11 | 432 | Total Deposit wt. (mg) | 18 | 50.13 | 4.88 | 40.6 | 59.7 |
| New Targets | 433 | Total Deposit wt. (mg) | 18 | 50.28 | 5.26 | 40.0 | 60.6 |
| 6/1/01 | 1006 | Total Deposit wt. (mg) | 24 | 34.53 | 5.93 | 22.9 | 46.2 |
| GI by | 52 | Gelation Index | 35 | 4.5 | 0.24 | 4.0 | 5.0 |
| D5133 | 53 | Gelation Index | 37 | 44.7 | 4.64 | 35.6 | 53.8 |
| | 58 | Gelation Index | 17 | 5.8 | 0.69 | 4.4 | 7.2 |
| | 62 | Gelation Index | 35 | 17.0 | 3.90 | 9.4 | 24.6 |
| D6082 | 1007 | Tendency (ml) | 28 | 65.71 | 19.28 | 28 | 103 |
| (HT FOAM) | 1007 | Stability (ml) | 28 | 0.00 | 0.00 | 0 | 0 |
| BRT by | 81 | Average AGV | 12 | 112 | 14.00 | 85 | 140 |
| D02-1483 | 1006 | Average AGV | 12 | 128 | 7.21 | 114 | 142 |
| (D6557) | 5A-3 | Average AGV | 12 | 76 | 6.47 | 63 | 89 |
| EOFT by | 77 | Δ Flowrate (%) | 12 | -45.55 | 4.36 | -54.10 | -37.00 |
| (Draft 6) | 78 | ∆ Flowrate (%) | 12 | 15.74 | 6.87 | 2.27 | 29.21 |
| EOWT by | 77 | 0.6% H20 ∆ Flowrate (%) | 12 | -24.90 | 5.68 | -36.03 | -13.77 |
| (Draft 5) | 77 | 1.0% H20 Δ Flowrate (%) | 12 | -17.94 | 5.45 | -28.62 | -7.26 |
| | 77 | 2.0% H20 Δ Flowrate (%) | 12 | -17.96 | 8.47 | -34.56 | -1.36 |
| | 77 | 3.0% H20 Δ Flowrate (%) | 12 | -18.23 | 6.83 | -31.62 | -4.84 |
| EOWT by | 78 | 0.6% H20 Δ Flowrate (%) | 12 | 10.87 | 6.16 | -1.20 | 22.94 |
| (Draft 5) | 78 | 1.0% H20 Δ Flowrate (%) | 12 | 7.54 | 6.15 | -4.51 | 19.59 |
| | 78 | 2.0% H20 Δ Flowrate (%) | 12 | 5.17 | 5.33 | -5.27 | 15.62 |
| | 78 | 3.0% H20 ∆ Flowrate (%) | 12 | -0.54 | 4.52 | -9.40 | 8.32 |

Attachment 3A

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

| | | | | Targets | v | L | 10/1/01 | 10/1/01 - 3/31/01 | 01 | | 4/1/02 | 4/1/02 - 9/30/02 | 5 | | 10/1/02 | 10/1/02 - 3/31/03 | 33 |
|---------|-----------|--|-----|-----------|--------|-------|-----------------------------------|-------------------|-------------|----|--------|------------------|-------|----|---------|-------------------|-------|
| | Oil | | | |) | | | 5 | Mean | | | 5000 | Mean | | | | Mean |
| Test | Code | Parameter | L | Mean | sR | c | Mean | sR | Δ /s | c | Mean | sR | Δ/s | c | Mean | sR | Δ/s |
| D6417 | 52 | Area % Volatized | 18 | 6.97 | 0.31 | З | 6.9 | 0.15 | -0.12 | 7 | 6.9 | 0.38 | -0.36 | 2 | 6.6 | 0.21 | -1.03 |
| | 55 | Area % Volatized | 18 | 11.68 | 0.51 | 9 | 11.2 | 0.57 | -0.84 | 5 | 11.7 | 0.33 | 0.00 | 7 | 11.2 | 0.51 | -0.91 |
| | 58 | Area % Volatized | 18 | 5.61 | 0.30 | 4 | 5.6 | 0.29 | -0.12 | 4 | 5.4 | 0.24 | -0.53 | 9 | 5.7 | 0.19 | 0.24 |
| D5800 | 52 | % volatility loss | 59 | 13.61 | 0.49 | 11 | 13.8 | 0.60 | 0.48 | 10 | 13.7 | 0.73 | 0.10 | 12 | 13.8 | 0.55 | 0.29 |
| ** | 55 | % volatility loss | 60 | 16.39 | 0.66 | 6 | 16.6 | 0.58 | 0.34 | 14 | 17.3 | 0.85 | 1.42 | 6 | 17.2 | 0.61 | 1.21 |
| | 58 | % volatility loss | 59 | 14.46 | 0.52 | 13 | 15.2 | 0.75 | 1.36 | 11 | 15.1 | 0.76 | 1.28 | 13 | 15.3 | 0.71 | 1.59 |
| TEOST | 71 | Deposit wt. (mg) | 27 | 51.79 | 4.79 | 3 | 55.6 | 0.29 | 0.79 | 5 | 61.2 | 4.71 | 1.96 | 3 | 51.7 | 6.66 | -0.01 |
| (D6335) | 72 | Deposit wt. (mg) | 27 | 26.72 | 3.46 | 3 | 29.8 | 1.84 | 0.88 | 2 | 25.0 | 0.28 | -0.50 | 2 | 31.1 | 0.28 | 1.27 |
| MTEOS | 1006 | Deposit wt. (mg) | 24 | 34.53 | 5.93 | 14 | 36.1 | 5.11 | 0.26 | 7 | 31.8 | 6.32 | -0.46 | 14 | 31.4 | 8.72 | -0.52 |
| *** | 432 | Deposit wt. (mg) | 18 | 50.13 | 4.88 | 1 | 46.9 | 5.98 | -0.67 | 16 | 42.5 | 7.05 | -1.56 | ø | 41.8 | 6.62 | -1.70 |
| | 433 | Deposit wt. (mg) | 18 | 50.28 | 5.26 | 12 | 46.5 | 9.18 | -0.72 | 1 | 48.3 | 8.31 | -0.37 | 13 | 47.2 | 5.00 | -0.59 |
| | 74 | Deposit wt. (mg) | 20 | 16.84 | 5.28 | 7 | 11.5 | 4.01 | -1.01 | 13 | 14.7 | 4.80 | -0.41 | 7 | 13.5 | 4.95 | -0.63 |
| ß | 52 | Gelation Index | 35 | 4.5 | 0.24 | 4 | 4.3 | 0.13 | -1.04 | 8 | 4.4 | 0.12 | -0.36 | 7 | 4.6 | 0.20 | 0.36 |
| (D5133) | 53 | Gelation Index | 37 | 44.7 | 4.64 | 7 | 47.6 | 5.90 | 0.63 | 6 | 49.9 | 3.12 | 1.13 | 5 | 47.0 | 3.73 | 0.50 |
| **** | 58 | Gelation Index | 17 | 5.8 | 0.69 | ი | 5.9 | 1.18 | 0.11 | ∞ | 6.4 | 0.66 | 0.80 | 8 | 6.6 | 1.00 | 1.09 |
| | 62 | Gelation Index | 35 | 17.0 | 3.90 | 10 | 16.3 | 6.41 | -0.19 | 7 | 17.2 | 2.85 | 0.04 | 6 | 18.5 | 2.21 | 0.37 |
| D6082 | 1007 | Tendency (ml) | 28 | 65.71 | 19.28 | 11 | 64.5 | 15.08 | -0.06 | 12 | 62.5 | 14.22 | -0.17 | 11 | 62.7 | 17.52 | -0.15 |
| **D5800 | Targets / | **D5800 Targets Adjusted 10/2/00; new oils | N O | Is select | ed: ne | v pro | selected: new procedures approved | approv | 'ed | | | | | | | | |

MTEOS Targets Adjusted 10/2/00, new oils selected, new procedures approved ***MTEOS Targets Adjusted 6/1/01 per direction of TEOST Surveillance Panel (based on completed Matrix 6 data) *GI Added new oil 58 10/24/01; dropped oils 51 & 55 7/2/01

Attachment 3B

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

| | | | | Tangets | (0 | 10/1 | 10/1/00 - 3/31/01 | 31/01 | 4/1/ | 4/ 1/ 01 - 9/ 30/ 01 | 30/ 01 | 10/1 | 10/1/01 - 3/31/02 | 31/02 | 4/1/ | 4/1/02 - 9/30/02 | 30/02 | 1 0/ 1 | 10/1/02 - 3/31/03 | 31/03 |
|-------------------|------|------------------|----|---------|-------|------|-------------------|-------|------|----------------------|--------|------|-------------------|-------|------|------------------|-------|--------|-------------------|-------|
| | 0 | | | | | | | | | | | | | | | | | | | |
| Test | Code | Parameter | ۲ | Mean | sR | c | Mean | Яŝ | c | Mean | sR | c | Mean | sR | c | Mean | ЯS | ۲ | Mean | R |
| BRT | 1006 | Average AGV | 12 | 128 | 7.21 | 26 | 123.7 | 6.79 | 39 | 124.4 | 5.77 | 29 | 125.0 | 5.19 | 30 | 123.0 | 9.11 | 38 | 126.0 | 5.09 |
| | 5A-3 | Average AGV | 12 | 76 | 6.47 | 31 | 81.6 | 13.72 | 38 | 83.4 | 12.60 | 28 | 87.6 | 15.69 | 38 | 89.7 | 15.52 | 23 | 85.9 | 14.43 |
| | 81 | Average AGV | 12 | 112 | 14.00 | 55 | 121.0 | 6.06 | 79 | 117.8 | 7.99 | 59 | 121.2 | 13.22 | 70 | 121.3 | 9.38 | 82 | 124.6 | 5.98 |
| EO FT | 77 | Avg. % CF | 12 | -45.55 | 4.36 | 5 | -18.5 | 7.03 | 0 | | | 0 | | | 0 | | | 0 | | |
| | 78 | Avg. % CF | 12 | 15.74 | 6.87 | 74 | 15.1 | 4.64 | 103 | 15.2 | 6.67 | 84 | 15.3 | 5.67 | 89 | 16.5 | 5.39 | 81 | 13.9 | 4.16 |
| EO W ⁻ | 77 | 0.6 H2O Avg. %CF | 12 | -24.90 | 5.68 | 53 | -23.8 | 4.71 | 63 | -24.8 | 5.64 | 47 | -24.6 | 5.45 | 61 | -24.5 | 4.15 | 48 | -24.5 | 4.15 |
| | 77 | 1.0 H2O Avg. %CF | 12 | -17.94 | 5.45 | 45 | -17.8 | 5.25 | 59 | -16.3 | 5.71 | 41 | -17.3 | 6.70 | 52 | -16.4 | 4.17 | 47 | -16.2 | 2.49 |
| | 77 | 2.0 H2O Avg. %CF | 12 | -17.96 | 8.47 | 50 | -17.0 | 6.72 | 56 | -16.1 | 6.25 | 47 | -17.9 | 5.34 | 47 | -16.7 | 3.87 | 36 | -15.1 | 5.21 |
| | 77 | 3.0 H2O Avg. %CF | 12 | -18.23 | 6.83 | 48 | -18.4 | 6.19 | 60 | -17.7 | 6.44 | 46 | -17.0 | 5.46 | 50 | -16.9 | 5.70 | 46 | -16.2 | 4.53 |
| EO W ⁻ | 78 | 0.6 H2O Avg. %CF | 12 | 10.87 | 6.16 | 48 | 8.6 | 6.46 | 60 | 11.4 | 6.90 | 41 | 9.9 | 6.80 | 41 | 13.0 | 5.00 | 41 | 10.5 | 5.41 |
| | 78 | 1.0 H2O Avg. %CF | 12 | 7.54 | 6.15 | 54 | 5.3 | 6.30 | 56 | 8.9 | 5.87 | 48 | 7.1 | 7.61 | 52 | 8.9 | 4.42 | 42 | 8.6 | 4.23 |
| | 78 | 2.0 H2O Avg. %CF | 12 | 5.17 | 5.33 | 50 | 2.8 | 5.75 | 58 | 6.4 | 6.85 | 42 | 4.9 | 6.18 | 56 | 5.4 | 3.67 | 53 | 4.9 | 6.12 |
| | 78 | 3.0 H2O Avg. %CF | 12 | -0.54 | 4.52 | 50 | -0.6 | 5.22 | 62 | 2.2 | 6.48 | 43 | 1.5 | 6.18 | 58 | 3.4 | 3.59 | 43 | 2.6 | 5.64 |