



## Test Monitoring Center

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MEMORANDUM: 09-042

DATE: November 24, 2009

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, 2009, through September 30, 2009, for Test Areas  
D6417, D5800, D6335 (TEOST), D7097 (MTEOS), D5133 (GI), D6082,  
D874 and D7528 (ROBO)

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

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

Pooled  $s$  = Standard deviation pooled across labs and reference oils

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

$\Delta/s = [(\text{Single Test Result}) - (\text{Reference Oil's Target Mean Performance})] / (\text{R.O.'s Target Precision})$

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

Mean  $\Delta/s = [\sum (\Delta/s)] / n$  (across reference oils and labs, and over a period of time)

(i.e., "On average, how many standard deviations from the target mean are all 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.

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

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

#### Attachments

c: D02.B07 Bench Test Mailing List  
F. Farber, TMC  
J. Clark, TMC  
<ftp://ftp.astmtmc.cmu.edu/docs/bench/bo7semiannualreports/mem09-042.pdf>

Distribution: Email

**ASTM Test Monitoring Center**

**Semiannual Report**

**ASTM D02.B07 Bench Reference Test Monitoring  
From April 1, 2009 through September 30, 2009**

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

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

**MONITORED TESTING STATUS**

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

TABLE 1

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

Fail Rate of Operationally Valid Tests: 0.0%

Table 2 is a breakdown of the statistically unacceptable tests.

TABLE 2

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

**PRECISION AND SEVERITY**

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

TABLE 3

Area % Volatized @ 371°C	n	df	Pooled s	Mean Δ/s
Initial Round Robin Study	107	101	0.46	-----
10/1/05 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
10/1/07 through 3/31/08	14	11	0.29	0.84
4/1/08 through 9/30/08	14	11	0.34	0.54
10/1/08 through 3/31/09	14	11	0.23	-0.10
4/1/09 through 9/30/09	15	12	0.34	0.23

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	5	1.22
Lab B	2	-0.47
Lab D	2	-0.86
Lab G	2	0.06
Lab H	2	-0.29
Lab S	2	0.20

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

### **INDUSTRY PERFORMANCE**

D6417 reference testing is less precise, as measured by pooled s, compared to the previous period but remains more precise than the target precision (Table 3). Overall performance is slightly severe at 0.23 standard deviations. Severity is represented graphically in Figure 1 showing nearly on-target performance since the 01OCT08 timeline.

### **TMC MEMORANDA**

There was one TMC technical memo issued this report period for the D6417 test method:

TMC Memo 09-037, July 17, 2009, Subject: Updated Test Method D6417-09

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

**MONITORED TESTING STATUS**

Table 5 summarizes the reference tests reported to the TMC this period (8 labs reporting):

TABLE 5

	No. of Tests
Statistically Acceptable and Operationally Valid	31
Operationally Valid but Failed Acceptance Criteria	5
Operationally Invalid (initially reported as)	1
Operationally Invalid (after informed of failing calibration)	0
<b>Total</b>	<b>37</b>

Fail Rate of Operationally Valid Tests: 13.9%

Table 6 is a breakdown of the statistically unacceptable tests.

TABLE 6

Reason for Fail	No. of Tests
Sample Evaporation Loss Severe	5*
Sample Evaporation Loss Mild	0

\*4 severe results on oil 52 and one on oil 55

**PRECISION AND SEVERITY**

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

TABLE 7

Sample Evaporation Loss, mass %	n	df	Pooled s	Mean Δ/s
New Targets Effective 7/21/2003	102	99	0.70	-----
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
10/1/07 through 3/31/08	34	31	0.50	0.75
4/1/08 through 9/30/08	36	33	0.54	0.82
10/1/08 through 3/31/09	36	33	0.84	0.51
4/1/09 through 9/30/09	36	33	0.56	0.88

\*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.

TABLE 8

Sample Evaporation Loss, mass %	n	df	Pooled s	Mean Δ/s
Procedure A	0	0	---	---
Procedure B	33	30	0.56	0.87
Procedure C	3	0	---	1.00

**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

	<b>n</b>	<b>Mean <math>\Delta/s</math></b>
Lab A	5	0.97
Lab B	8	0.46
Lab D	2	0.22
Lab F	4	0.88
Lab G	5	0.61
Lab H	2	0.80
Lab I	6	1.43
Lab J	4	1.47

**INDUSTRY PERFORMANCE**

D5800 reference testing precision, as measured by pooled s, is more precise than the previous period, similar to the three report periods prior to last period, and is more precise than the target precision. Overall performance remains severe this period with all eight participating labs performing severe and fifteen of thirty-six operationally valid tests more than 1 s severe, nine of those fifteen were on oil 52. Only six tests were mild of targets, all by less than 1 s. Severity is graphically represented in Figures 2A and 2B. Figure 2A shows a clear long-term severe trend with an unexplained increase in severity since the 01JUL06 timeline. Oil 52, the mildest performing reference oil, continues to perform substantially severe at 1.32 s. Testing severity on Oil 52 has gradually shifted over the years from around the target mean of 13.8 to 14.6, or about 1.3 s severe of the expected target performance (Attachment 3). Labs H and J were the most severe performing labs last period, while labs I and J are the most severe performers this period.

A widely observed severe trend continues since at least January 2007, with another increase in overall severe performance for this period. Whereas precision had worsened last period, it appears to match historic levels again this period. Oil 52 is (and has been) performing 1.3 s mild of the target mean.

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, too few to estimate pooled s precision.

**TMC MEMORANDA**

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

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

**MONITORED TESTING STATUS**

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

TABLE 10  
Reference Tests

	No. of Tests
Statistically Acceptable and Operationally Valid	30
Operationally Valid but Failed Acceptance Criteria	3
Operationally Invalid (initially reported as)	3
Operationally Invalid (after informed of failing calibration)	0
<b>Total</b>	<b>36</b>

Fail Rate of Operationally Valid Tests: 9.1%

Table 11 is a breakdown of the statistically unacceptable tests.

TABLE 11

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

**PRECISION AND SEVERITY**

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

TABLE 12

Gelation Index	n	df	Pooled s	Mean Δ/s
Revised Targets Effective 20030715 (Oils 58 & 62 targets unchanged, added oil 1009, dropped oils 52 & 53)	68	65	2.86	-----
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
10/1/07 through 3/31/08	26	23	4.13	-0.31
4/1/08 through 9/30/08	27	24	3.54	0.18
10/1/08 through 3/31/09	24	21	2.32	0.10
4/1/09 through 9/30/09	33	30	2.79	-0.10



**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

	<b>n</b>	<b>GI Mean <math>\Delta/s</math></b>
Lab A	8	0.53
Lab B	5	-0.41
Lab D	2	0.35
Lab G	3	-1.49
Lab H	1	-0.87
Lab I	6	0.57
Lab S	8	-0.55

**INDUSTRY PERFORMANCE**

D5133 reference testing is less precise, as measured by pooled s, compared to last period but is nearly equal to the target precision. Overall performance has only a slight mild bias. Severity is graphically represented in Figures 3A and 3B showing nearly on-target performance.

Eight tests this period were more than 1 s mild, and six tests were 1 s or more severe, with three tests falling outside the acceptance bands. Last period all operationally valid tests passed calibration with only one test more than 1 s mild and four tests more than 1 s severe.

Compare this to the prior report period where twelve of twenty-seven operationally valid tests were 1 s or more from target (severe or mild), and individual tests as much as 2, 3 and 5 s mild or severe, and a fail rate of 14.8%. Three report periods prior had seventeen of twenty-six operationally valid tests at 1 s or more from target (severe or mild) and a fail rate of 7.1%.

**TMC MEMORANDA**

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

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

**MONITORED TESTING STATUS**

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

TABLE 14

	No. of Tests
Statistically Acceptable and Operationally Valid	13
Operationally Valid but Failed Acceptance Criteria	1
Operationally Invalid (initially reported as)	1
Operationally Invalid (after informed of failing calibration)	1
<b>Total</b>	<b>16</b>

Fail Rate of Operationally Valid Tests: 7.1%

Table 15 is a breakdown of the statistically unacceptable tests.

TABLE 15

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

**PRECISION AND SEVERITY**

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

TABLE 16

Total Deposits	n	df	Pooled s	Mean $\Delta/s$
Initial Round Robin Study	54	52	4.18	-----
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
10/1/07 through 3/31/08	22	20	9.65	0.92
4/1/08 through 9/30/08	15	13	6.99	0.20
10/1/08 through 3/31/09	18	16	4.90	0.98
4/1/09 through 9/30/09*	14	10	8.24	0.32
4/1/09 through 9/30/09*	13	9	3.71	0.68

\*Period statistics with and without a single unusually severe result included

## **D6335: TEOST, continued**

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

TABLE 17

	<b>n</b>	<b>Mean <math>\Delta/s</math></b>
Lab A	4	0.38
Lab B	4	1.15
Lab D	2	0.29
Lab G	2	0.32
Lab V	2	-1.45

## **INDUSTRY PERFORMANCE**

Reference testing precision, as measured by pooled  $s$ , is significantly worsened compared the previous report period and compared to target precision (Table 16). Performance is moderately severe at 0.32  $s$ . Severity is graphically represented in Figure 4 (attached) with an overall severe trend since the 01APR08 timeline.

However, one result reported this period was -4.36  $s$  mild of target (Lab V, Oil 71), contributing significantly to the very poor precision, but also offsetting the otherwise more severe overall performance of the other tests this period. The bottom row in table 16 shows the period statistics with this result excluded. With the exclusion, precision improves to the best in years, but the overall performance estimate becomes more severe at 0.68  $s$ . This is the only failing result this period of all the tests reported as operationally valid, so the fail rate of 7.1% translates into only one in 14 tests, which is quite good.

It would appear that, excluding the occasional errant result, the last three report periods since the April 2008 workshop shows increasingly improved precision with TMC calibration tests. Also, a revised test method was published during the current report period that includes clarifications and suggestions from the workshop participants, perhaps those improvements also contribute to the improving precision seen in the calibration test data.

All tests this period are reported as using Rod Batch H.

Oils 71 and 72 have virtually run out at the TMC, so replacement oils 71-1 and 72-1 have been distributed. There were three results on 72-1 and one result on 71-1 this period, the first results to be reported on either oil. There are too few results reported at this time for a rigorous comparison of the performance of the "reblends" to the original blends, but there is nothing worrisome about the results so far.

## **TMC MEMORANDA**

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

TMC Memo 09-038, August 12, 2008, Subject: Updated Test method D6335-09

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

**MONITORED TESTING STATUS**

Table 18 summarizes the reference tests reported to the TMC this period (6 labs reporting):

TABLE 18

	No. of Tests
Statistically Acceptable and Operationally Valid	47
Operationally Valid but Failed Acceptance Criteria	1
Operationally Invalid (initially reported as)	2
Operationally Invalid (after informed of failing calibration)	1
<b>Total</b>	<b>51</b>

Fail Rate of Operationally Valid Tests: 2.1%

Table 19 is a breakdown of the statistically unacceptable tests.

TABLE 19

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

**PRECISION AND SEVERITY**

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

TABLE 20

Total Deposits	n	df	Pooled s	Mean Δ/s
Updated Targets Effective 7/31/06	90	87	5.62	-----
10/1/06 through 3/31/07	47	44	7.53	-0.17
4/1/07 through 9/30/07	48	45	7.68	0.32
10/1/07 through 3/31/08	46	43	7.41	-0.21
4/1/08 through 9/30/08	46	43	6.09	0.01
10/1/08 through 3/31/09	53	50	5.25	0.73
4/1/09 through 9/30/09	48	45	4.35	-0.08

**D7097: Determination of Moderately High Temperature Piston Deposits by Thermo-oxidation 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

	<b>n</b>	<b>Mean <math>\Delta/s</math></b>
Lab A	15	-0.10
Lab AK	2	1.11
Lab B	12	-0.17
Lab D	6	-0.22
Lab G	11	0.05
Lab V	2	-0.76

**INDUSTRY PERFORMANCE**

D7097 reference testing overall precision, as measured by pooled s, has improved compared to the prior report period and remains more precise than the updated target precision. This is the fourth consecutive report period with improvement in overall precision, with large improvements the past two periods, since the workshop held in April 2008, and again since the recent publication of an updated test method with improvements and clarifications from the workshop participants. Overall performance this period was on target. The fail rate of operationally valid tests is a remarkable 2.1%.

The MTEOS severity is graphically represented in Figures 5A & 5B, with Figure 5B showing when the new performance targets were implemented, when the monitored test method was changed and when new rod batches are introduced. Figure 5A shows the period severity with overall severe performance for the report period.

All operationally valid tests reported this period were run using rod batch H, and all but two operationally valid tests are reported as using catalyst batch 0902C. The severity issue with catalyst batch 0802 reported last period appears to have been corrected with the replacement 0902C catalyst batch.

**TMC MEMORANDA**

There was one TMC technical update issued this report period for the D7097 test method:

TMC Memo 09-039, August 12, 2009, Subject: Updated Test Method D7097-09

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

**MONITORED TESTING 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)	1
<b>Total</b>	<b>11</b>

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.

**TMC 1007 PRECISION AND SEVERITY**

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

TABLE 23

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

\*Period statistics with and without extreme results included.

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

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

TABLE 24

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

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

	<b>n</b>	<b>Foam Tendency Mean <math>\Delta/s</math></b>
Lab A	2	0.21
Lab B	4	-0.32
Lab G	2	-0.58
Lab I	2	-0.32

## **INDUSTRY PERFORMANCE**

The D6082 Foam Tendency precision, as measured by standard deviation (s) on TMC oil 1007, is significantly improved over last period, though unchanged if we exclude an extreme result from last period (Table 23). Overall precision is significantly more precise than the target precision (nearly half). Overall performance is somewhat 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 Sulfated Ash from Lubricating Oils and Additives**

**MONITORED TESTING STATUS**

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

TABLE 26  
Reference Tests

	No. of Tests
Statistically Acceptable and Operationally Valid	6
Operationally Valid but Failed Acceptance Criteria	1
Operationally Invalid (initially reported as)	0
Operationally Invalid (after informed of failing calibration)	0
<b>Total</b>	<b>7</b>

Fail Rate of Operationally Valid Tests: 14.3%

Table 27 is a breakdown of the statistically unacceptable tests.

TABLE 27

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

**PRECISION AND SEVERITY**

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

TABLE 28

Gelation Index	n	df	Pooled s	Mean Δ/s
Initial Round Robin Targets	81	79	0.07	-----
4/1/07 through 9/30/07	2	1	0.01	-0.50
10/1/07 through 3/31/08	5	2	0.11	-0.41
4/1/08 through 9/30/08	6	3	0.04	-0.62
10/1/08 through 3/31/09	6	3	0.07	-1.23
4/1/09 through 9/30/09	7	4	0.03	-0.41

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

TABLE 29

	n	Mean Δ/s
Lab A	2	0.09
Lab B	2	0.42
Lab G	3	-1.29



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

### **INDUSTRY PERFORMANCE**

D874 precision, as measured by pooled s, is more precise than the target precision but performance is mild of targets, mostly influenced by Lab G's very mild performance. Severity is graphically represented in Figure 7.

Lab G continues to run substantially mild, as it has in prior periods. Lab A is calibrating nearly on target and Lab B is somewhat severe.

### **TMC MEMORANDA**

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

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

**MONITORED TESTING STATUS**

Table 30 summarizes the reference tests reported to the TMC this period (8 labs reporting):

TABLE 30  
Reference Tests

	No. of Tests
Statistically Acceptable and Operationally Valid	26
Operationally Valid but Failed Acceptance Criteria	0
Operationally Invalid (initially reported as)	11
Operationally Invalid (after informed of failing calibration)	3*
<b>Total</b>	<b>40</b>

Fail Rate of Operationally Valid Tests: 0.0%

\*Nineteen additional tests were reported as operationally valid but were on rigs that either had not previously achieved calibrated status to begin with, or had a changed VCV set position and had trouble re-calibrating on the second runs resulting in another change to the VCV set position, so the results were excluded from the overall statistical estimates for this period.

Table 31 is a breakdown of the statistically unacceptable tests.

TABLE 31

Reason for Fail	No. of Tests
MRV Viscosity Mild	0
MRV Viscosity Severe	0

**PRECISION AND SEVERITY**

Note: A Box-Cox analysis of the initial round robin study suggested that mathematical transform of the MRV Viscosity test parameter was suggested to better normalize the data distribution. A natural log (ln) transformation is applied to each MRV Viscosity test result before any statistical analyses are performed.

Table 32 shows the current Industry precision and severity for the transformed MRV Viscosity test parameter for all operationally valid tests for the report period. (The first calibration test, completed August 31, 2008, was given retroactive calibrated status as part of a round robin study.)

TABLE 32

Natural Log (MRV Viscosity)	n	df	Pooled s	Mean Δ/s
Initial Round Robin Targets	42	39	0.2309	-----
8/31/08 through 3/31/09	22	19	0.2302	-0.47
4/1/09 through 9/30/09	26	23	0.1872	-0.58

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

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

TABLE 33

	<b>n</b>	<b>Mean <math>\Delta/s</math></b>
Lab A	7	-1.12
Lab AM	3	-0.35
Lab AN	3	-0.19
Lab AO	1	0.80
Lab B	4	-0.68
Lab D	3	-0.41
Lab G	4	-0.74
Lab Q	1	0.41

### **BACKGROUND NOTES ON INDUSTRY PERFORMANCE ESTIMATES**

On October 9, 2009, the ROBO surveillance panel agreed to shorten the calibration period from 100 days or 25 runs (test starts) to 50 days or 15 runs (test starts). This was made retroactive for rigs that were calibrated at that time. Therefore, the shortened reference period affected rigs with date completed 20090806 and later, and the frequency of calibration testing has increased as of October 9, 2009.

Since about half the results last period were used to set performance targets on the reference oils, the performance estimates for the last report period were not wholly independent of the data used to set performance targets. This is the first period where the calibration test results are independent of the data used to set the performance targets.

Labs must calibrate new rigs by passing two consecutive blind reference oil tests. Also, if the vacuum pump is changed or the vacuum control valve (VCV) set position is changed after calibrated status is attained, the lab must recalibrate with two passing tests, as if it were a new rig. Once the operation of a rig is established as calibrated, and the VCV set position is not changed, subsequent audits require only periodic one-test calibrations.

### **INDUSTRY PERFORMANCE**

ROBO precision, as measured by pooled  $s$ , is more precise than the target precision but performance is mild of targets at -0.58 s. Severity is graphically represented in Figure 8. Only Lab A is running more than 1 s from targets on average (Table 33).

Nineteen tests were reported this period as operationally valid on rigs that were never previously TMC calibrated, or where the vacuum system was changed after a successful TMC calibration, but the tests failed to meet the TMC statistical acceptance criteria. These tests were not included in the period statistics as operationally valid because the labs had not adequately demonstrated that the rigs could pass an initial two-test blind calibration series.

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

**TMC MEMORANDA**

There were two TMC technical updates issued this report period for the D7528 test method:

TMC Memo 09-036, July 8, 2009, Subject: Test Method D7528-09

Email from Tom Schofield, October 12, 2009, Subject: ROBO TMC Calibration New Requirements  
(Shortened Rig Calibration Period)

### **D6922 Determination of Homogeneity and Miscibility in Automotive Engine Oils**

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

**REFERENCE OIL SUPPLIES**

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

Table 34A  
Current Reference Oils

<b>Oil</b>	<b>For Tests</b>	<b>Quantity Left (gallons)</b>	<b>Quantity Used Last 12 Months (gallons)</b>
52	D6417, D5800	64.7	0.9
55	D6417, D5800	69.7	0.94
58	D6417, D5800, GI	120.9	1.1
62	GI	1.99	0.2
66	D6082 (Discrimination)	95.7	1.7
71	TEOST	4 samples	---
71-1	TEOST	1.5	0.45
72	TEOST	2 samples	---
72-1	TEOST	1.2	0.8
74	MTEOS	1.0	0.2
90	D874 & D874 Daily Check	40.9	3.0
91	D874	4.6	0.0
**432	MTEOS	Adequate Supply	----
**434	MTEOS	Adequate	----
**820-2	D874	Adequate	----
*1007	D6082	Est. 20	----
**1009	GI	Adequate	----
*434-1	ROBO	Adequate	----
<b>**435</b>	<b>ROBO</b>	<b>Nearly Gone</b>	<b>----</b>
*435-1	ROBO	Adequate	----
*438	ROBO	Adequate	----

\*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**

Table 34B  
Obsolete or Test Development Reference Oils

<b>Oil</b>	<b>For Tests</b>	<b>Quantity Left (gallons)</b>	<b>Quantity Used Last 12 Months (gallons)</b>
^51	Obsolete Vol. & GI	94.6	0.0
^53	Obsolete Vol. & GI	96.8	0.0
^54	Obsolete Volatility	97.8	0.0
^83	ROBO (RR)	47.3	0.0
^84	ROBO (RR)	3.3	0.0
^85	ROBO (RR)	3.3	0.0
^**433	Obsolete MTEOS	Adequate Supply	-----

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

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

Table 34C  
Homogeneity and Miscibility Reference Oils

<b>Oil</b>	<b>For Tests</b>	<b>Quantity Left (gallons)</b>	<b>Quantity Used Last 12 Months (gallons)</b>
HMA	H&M (D6922)	182.0	3.3
HMB	H&M (D6922)	186.0	3.3
HMC	H&M (D6922)	172.0	3.3
HMD	H&M (D6922)	180.0	3.3
HME	H&M (D6922)	165.0	3.3
HMF	H&M (D6922)	187.8	3.3

**Shipping aliquots are:**

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

**MISCELLANEOUS**

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

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

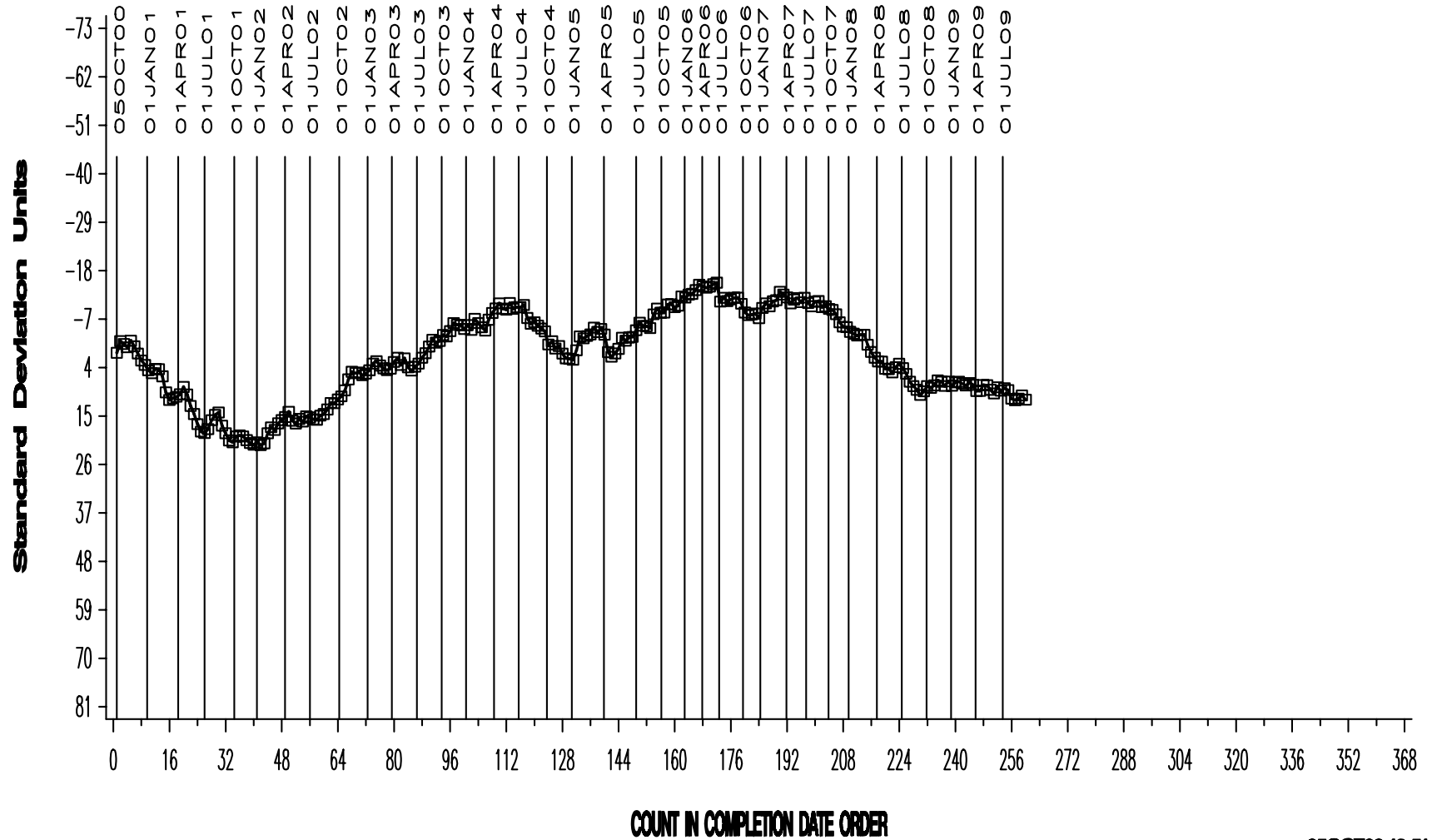


**D6417 VOLATILITY BY GC INDUSTRY OPERATIONALLY VALID DATA**

Figure 1

**SAMPLE AREA % VOLATIZED @ 371'C ... 700'F**

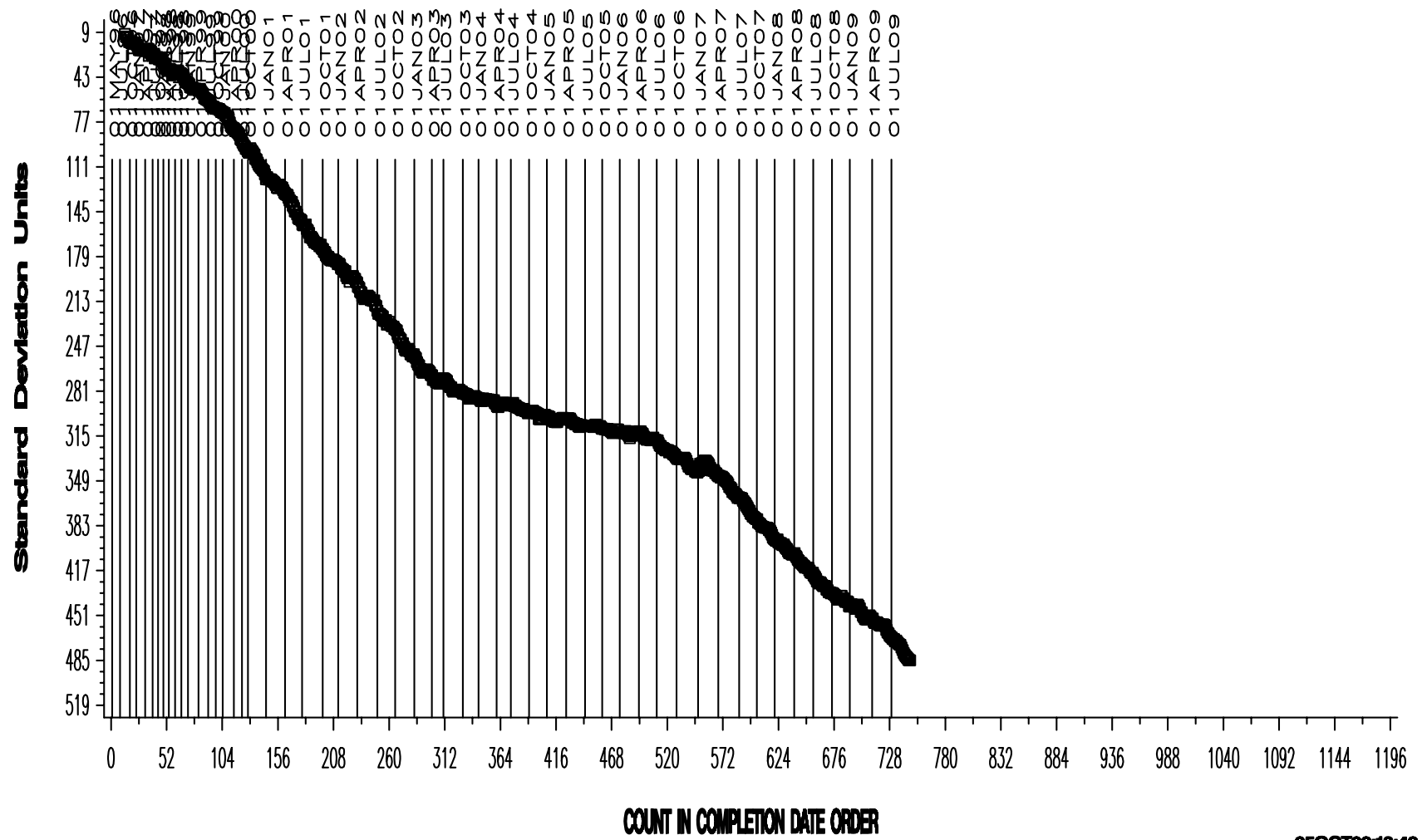
**CUSUM Severity Analysis**



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

**TEST OIL SAMPLE EVAPORATION LOSS, MASS%**

**CUSUM Severity Analysis**



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

**TEST OIL SAMPLE EVAPORATION LOSS, MASS%**

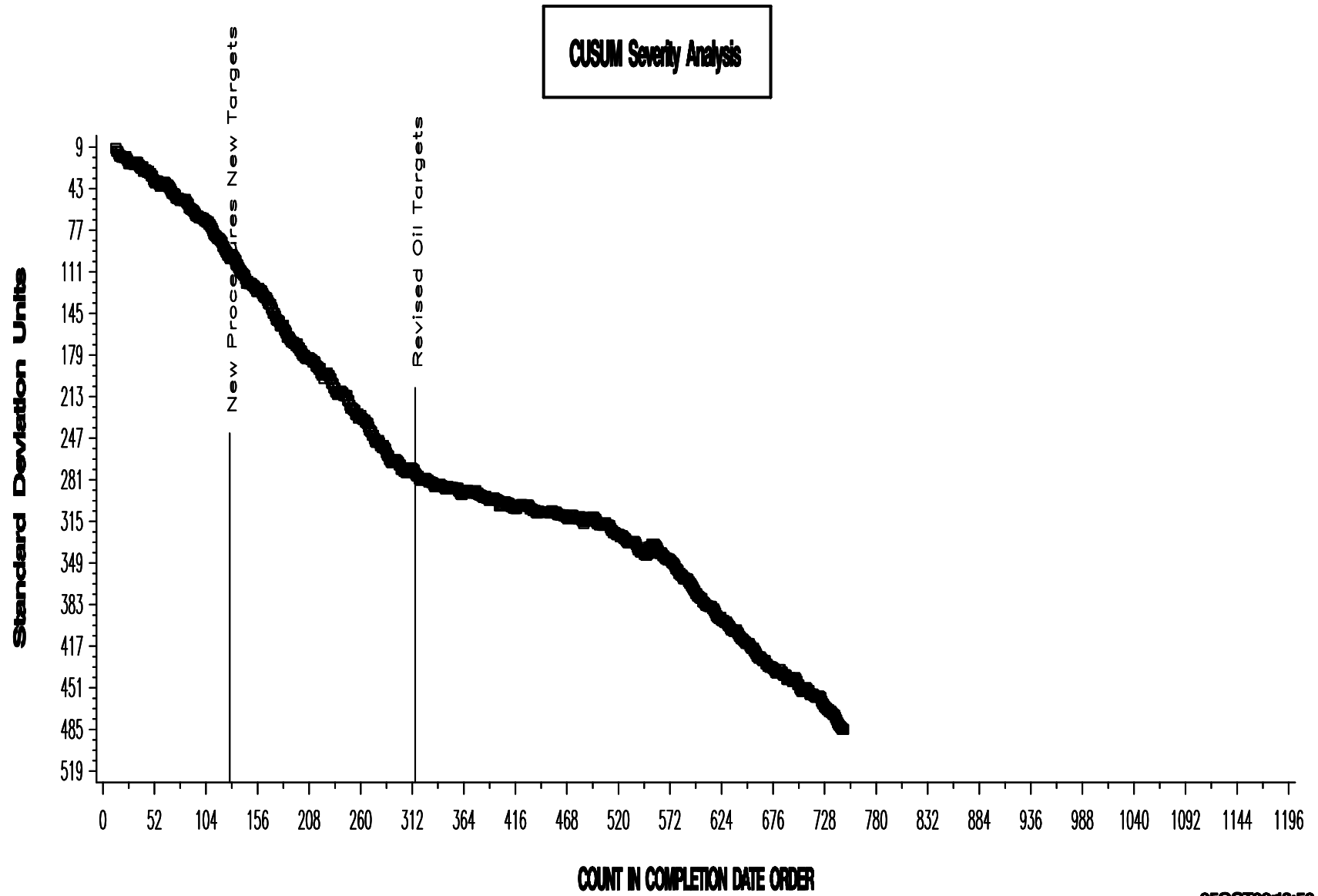


Figure 3A

D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA

GELATION INDEX

CUSUM Severity Analysis

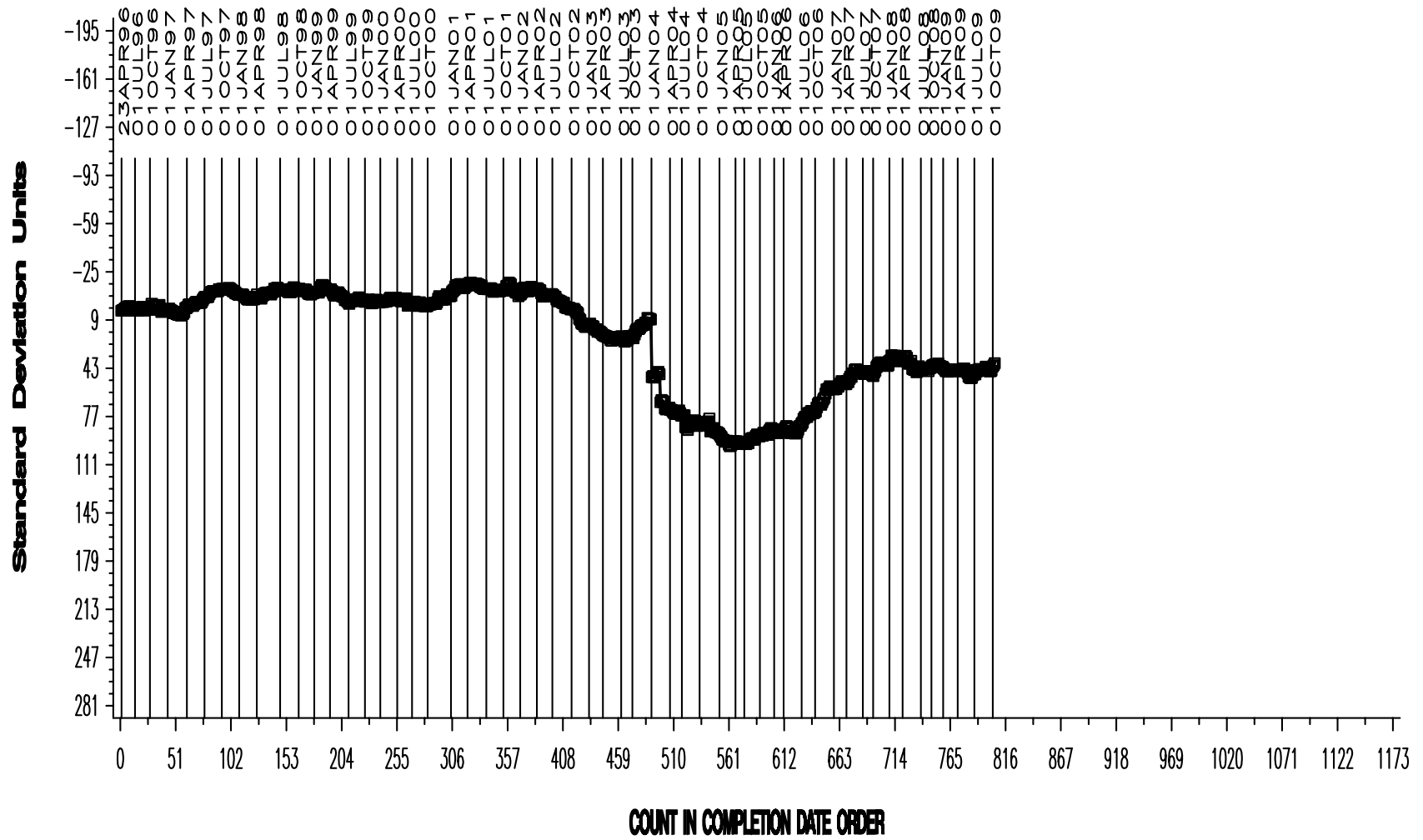
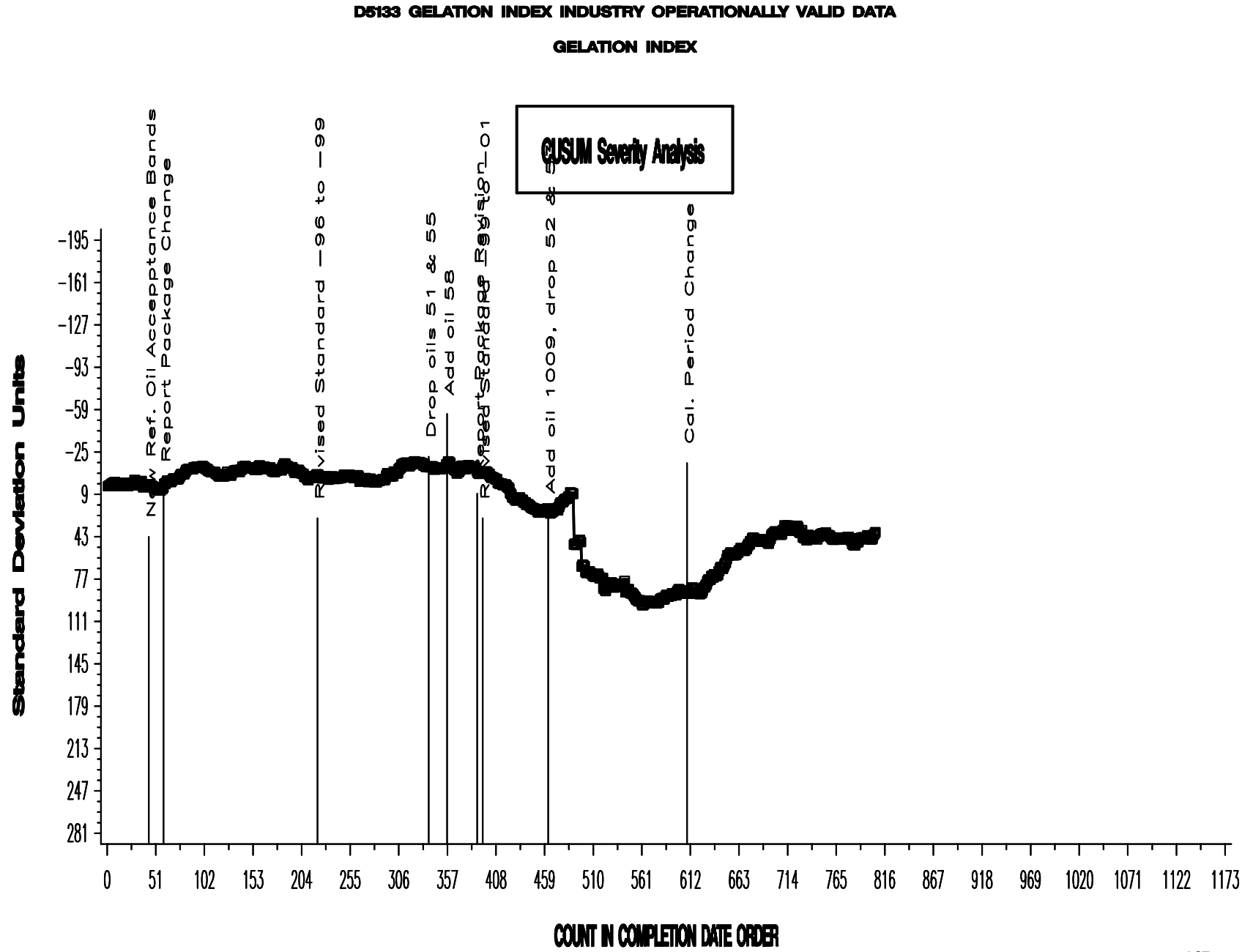


Figure 3B

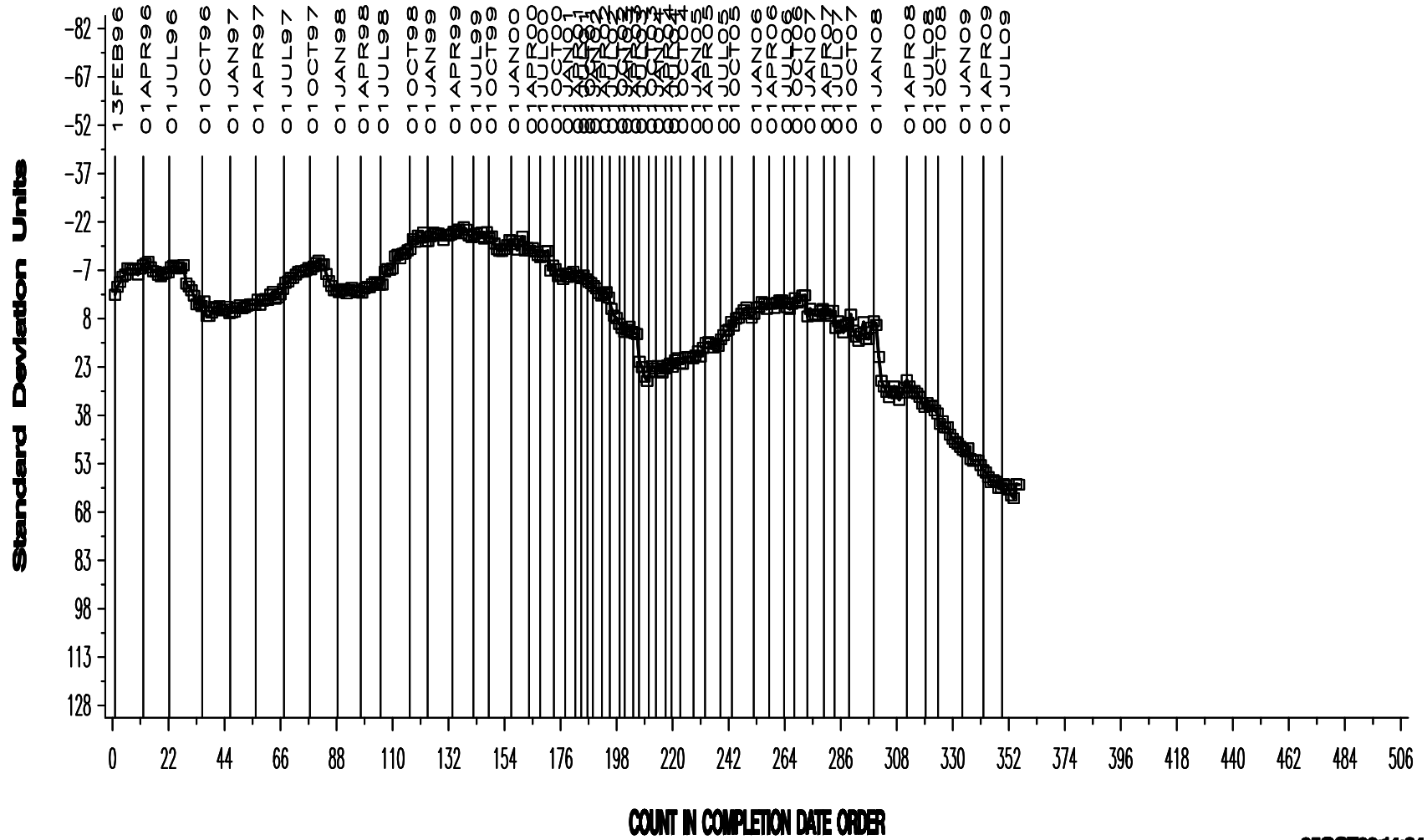


TEOST-33C INDUSTRY OPERATIONALLY VALID DATA

Figure 4

TOTAL DEPOSITS MG

CUSUM Severity Analysis

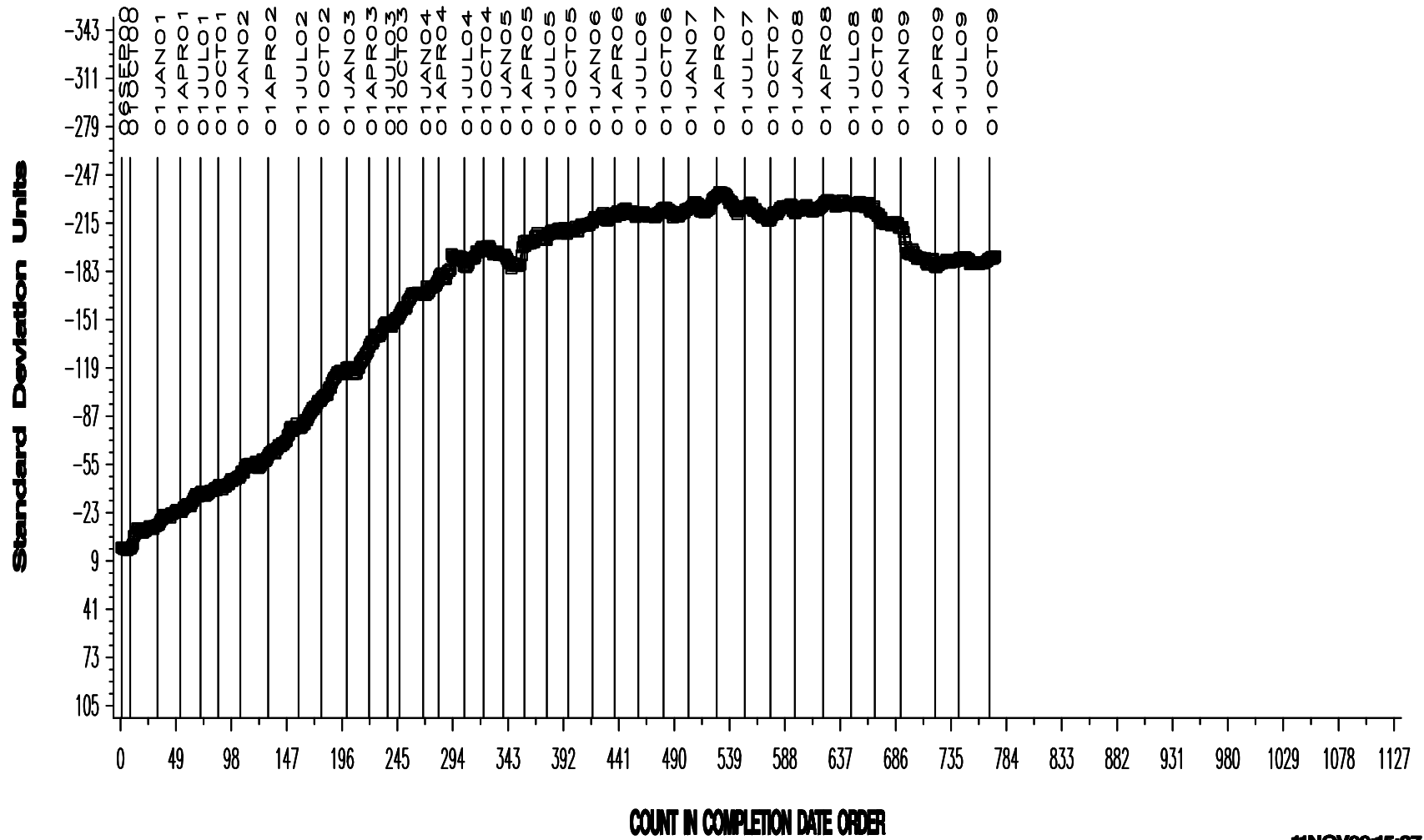


MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA

Figure 5A

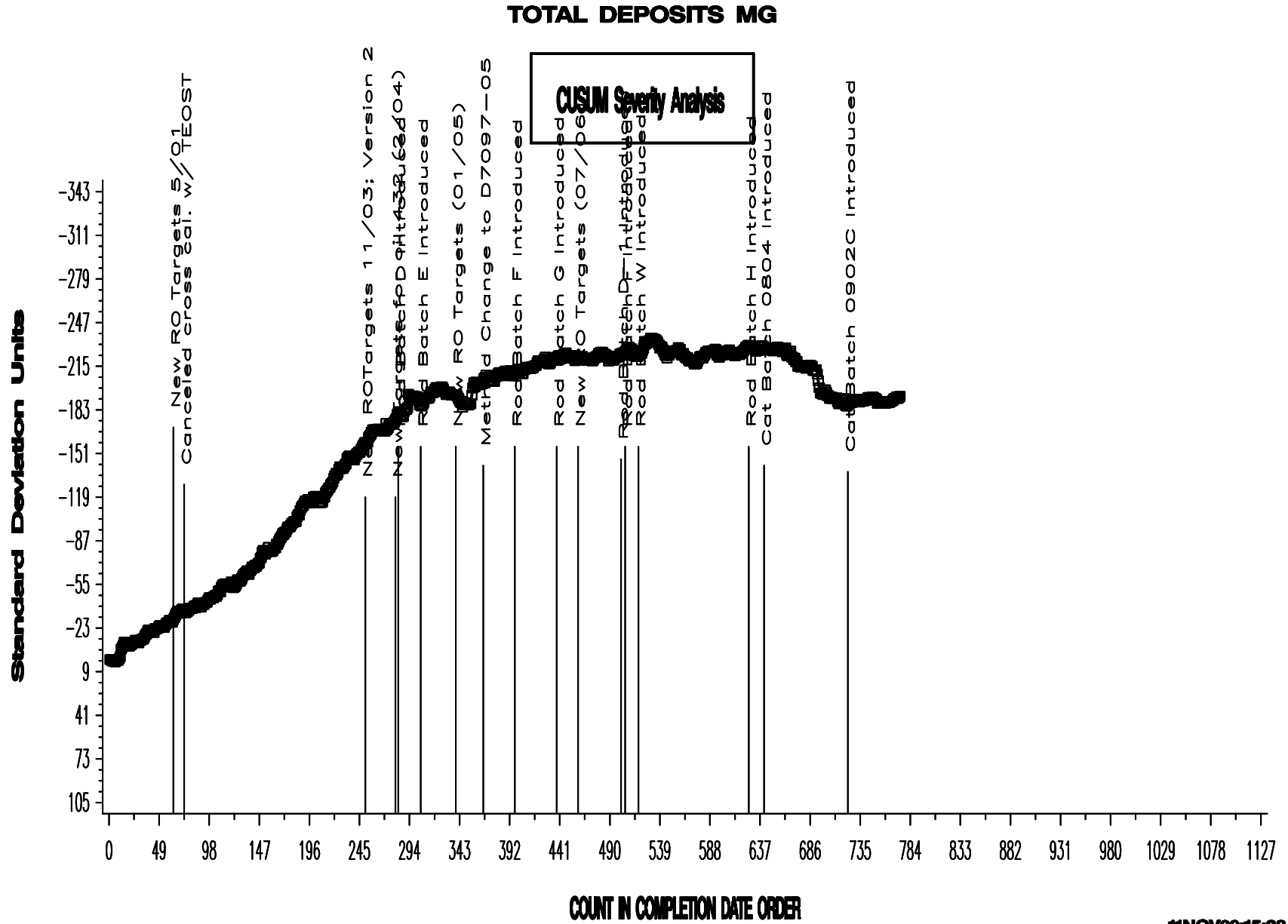
TOTAL DEPOSITS MG

CUSUM Severity Analysis



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

Figure 5B





**D6082 HIGH TEMPERATURE FOAM INDUSTRY OPERATIONALLY VALID DATA  
 IND=1007  
 FOAM TENDENCY, IMMEDIATELY BEFORE DISCONNECT STATI**

**CUSUM Severity Analysis**

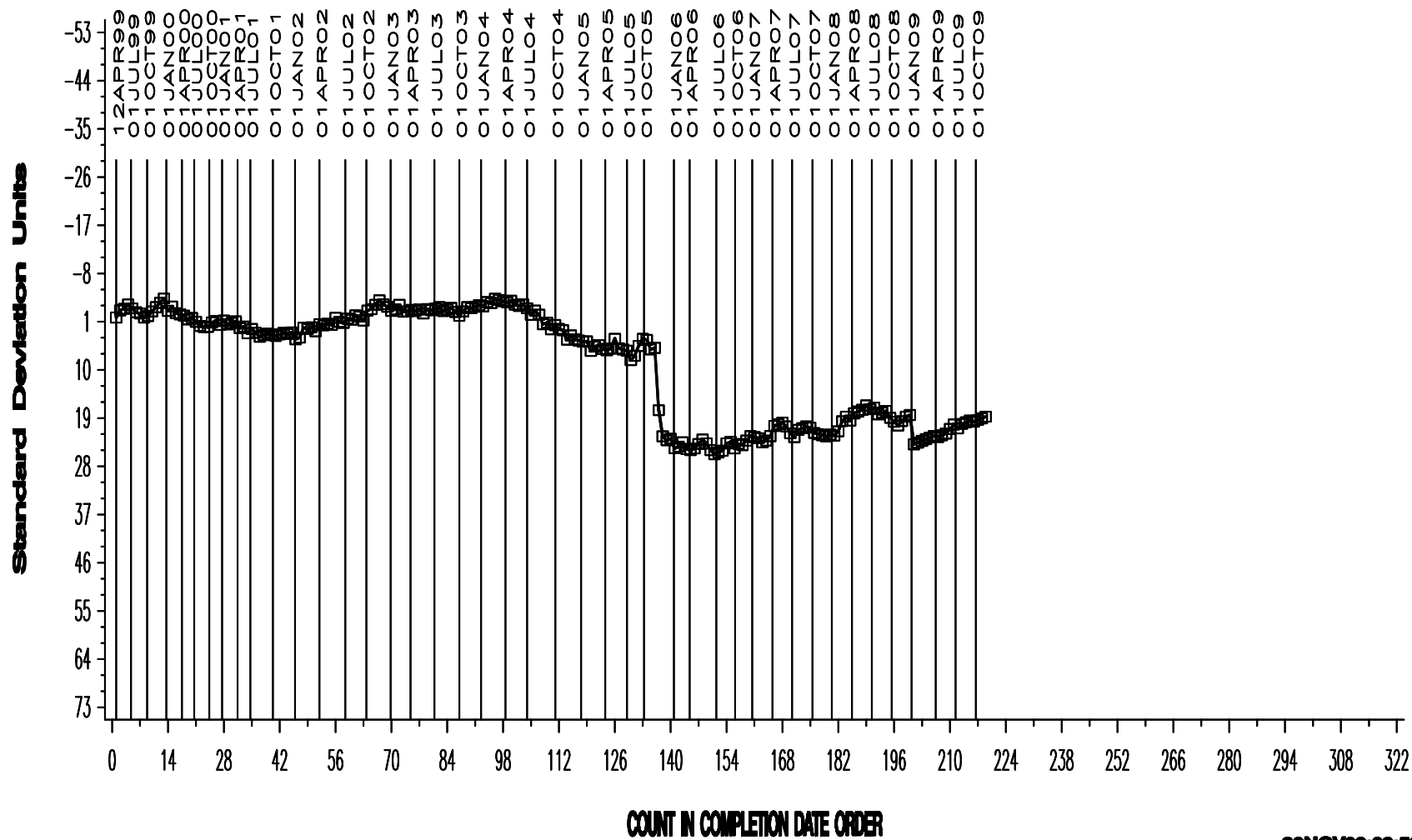


Figure 7

**D874 INDUSTRY OPERATIONALLY VALID DATA**  
**TEST SAMPLE PERCENT SULFATED ASH [<]**

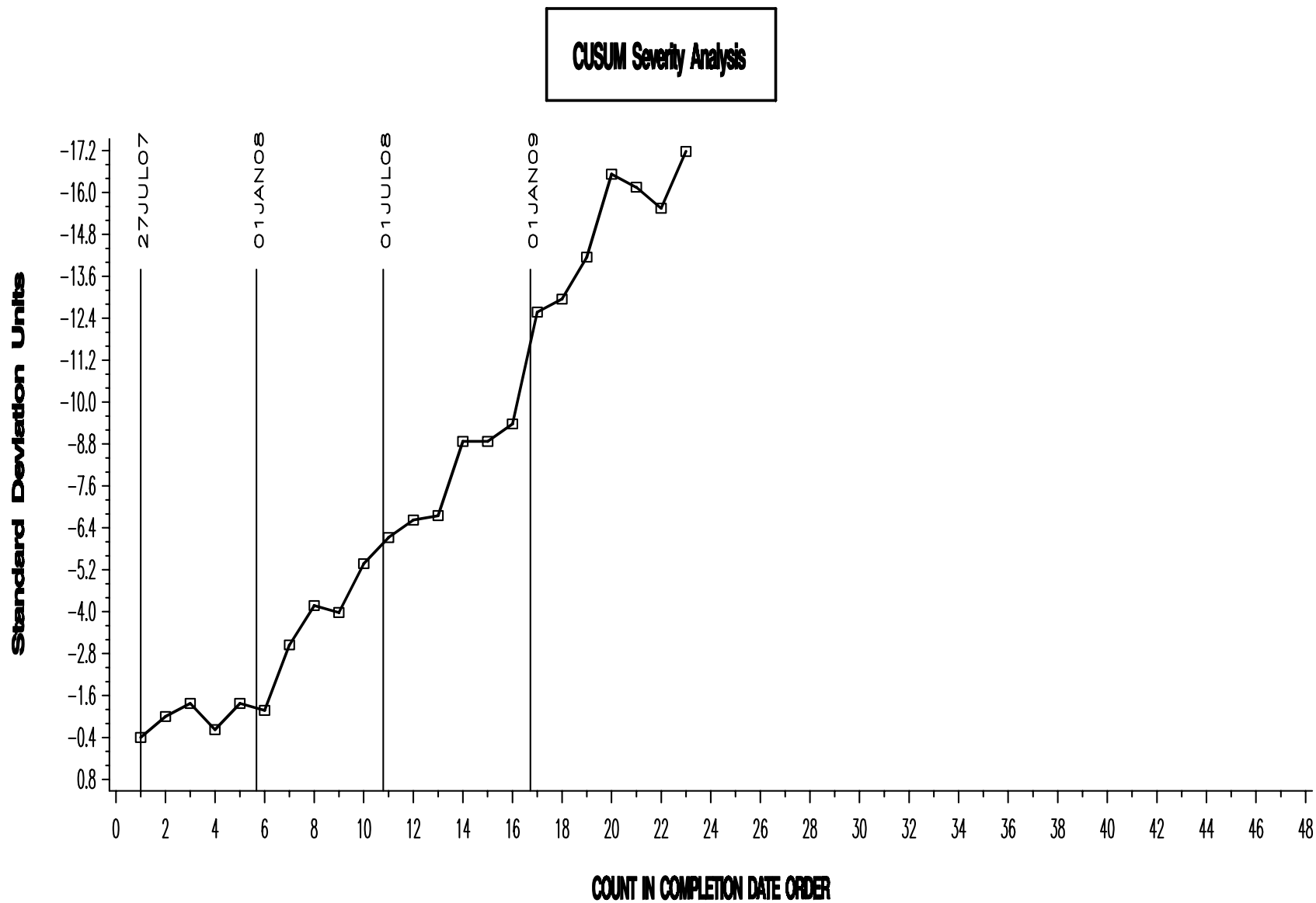
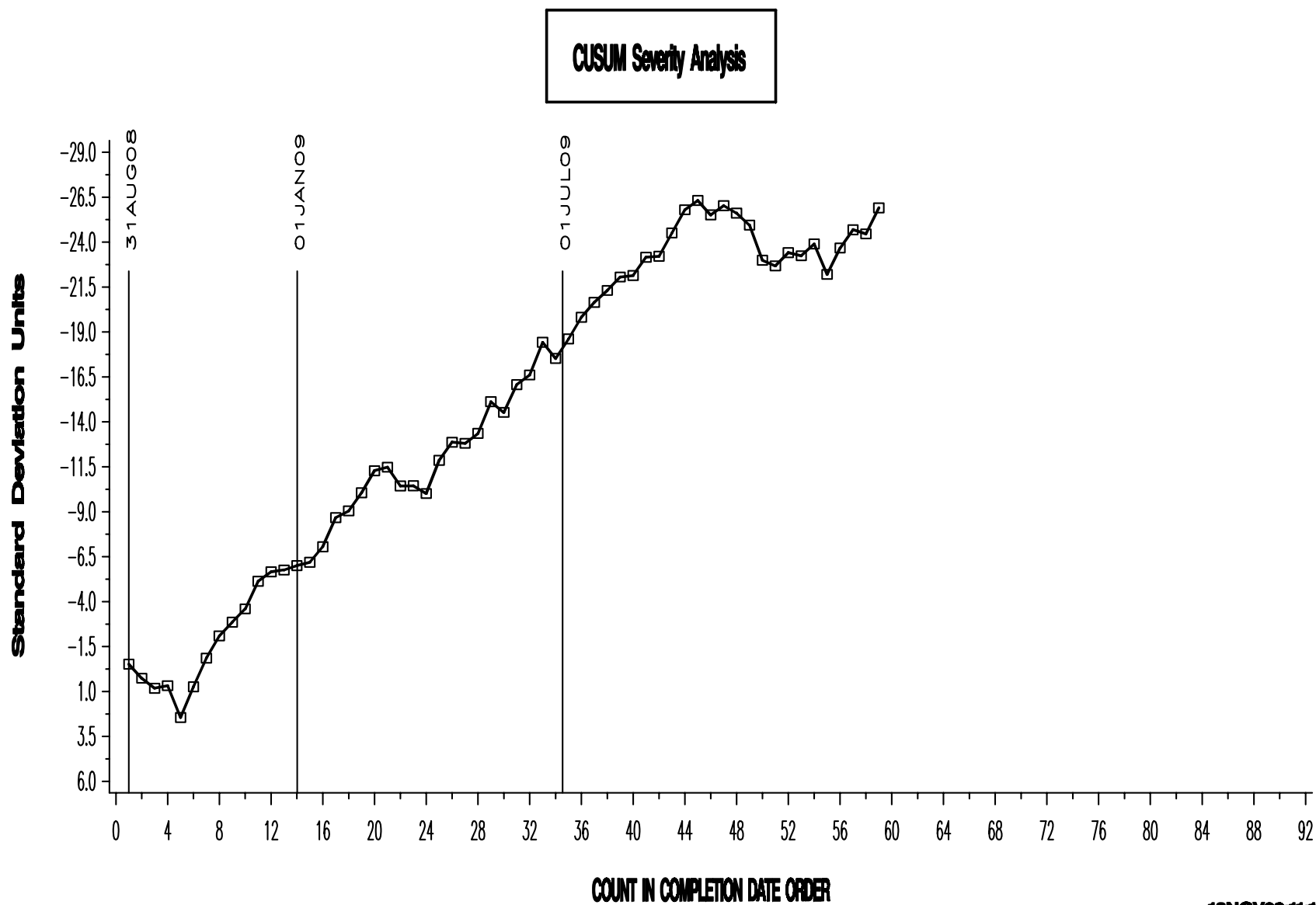


Figure 8

**ROBO TEST INDUSTRY OPERATIONALLY VALID DATA**  
**AGED OIL D4684 MRV APPARENT VISCOSITY [<, >]**



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

Test	Oil Code	Parameter	n	Mean	sR	Acceptance Bands *	
						95%	
						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 New Targets 20030721	52	mass % volatility loss	33	13.75	0.61	12.6	14.9
	55	mass % volatility loss	32	17.09	0.76	15.6	18.6
	58	mass % volatility loss	37	15.20	0.72	13.8	16.6
TEOST by D6335	71	Total Deposit (mg)	27	51.79	4.79	42.4	61.2
	71-1	Total Deposit (mg)	27	51.79	4.79	42.4	61.2
	72	Total Deposit (mg)	27	26.72	3.46	19.9	33.5
	72-1	Total Deposit (mg)	27	26.72	3.46	19.9	33.5
MTEOS by D7097 New Targets 20060731	74	Total Deposit (mg)	30	12.85	5.59	1.9	23.8
	432	Total Deposit (mg)	30	47.04	4.50	38.2	55.9
	434	Total Deposit (mg)	30	27.37	6.57	14.5	40.2
GI by D5133 New Targets 7/15/2003	58	Gelation Index	17	5.8	0.69	4.4	7.2
	62	Gelation Index	35	17.0	3.90	9.4	24.6
	1009	Gelation Index	16	7.3	0.68	6.0	8.6
D6082 (HT FOAM)	1007	Tendency (ml)	28	66	19	29	103
	1007	Stability (ml)	28	0	0	0	0
D6082 (HT FOAM)	66 (DISCRIM)	Tendency (ml)	--	-----	-----	>100	-----
	66 (DISCRIM)	Stability (ml)	--	-----	-----	0	0
D874	90	mass % Sulfated Ash	27	1.07	0.08	0.91	1.23
	91	mass % Sulfated Ash	27	0.82	0.05	0.72	0.92
	820-2	mass % Sulfated Ash	27	1.57	0.08	1.40	1.73
ROBO	434-1	In MRV, ln(mPa-s)	13	10.6599 (42612)	0.1672	10.3322 (30706)	10.9875 (59130)
	435	In MRV, ln(mPa-s)	15	11.4895 (97685)	0.2932	11.0021 (60000)	12.0642 (173546)
	438	In MRV, ln(mPa-s)	14	10.2676 (28785)	0.2037	9.8683 (19308)	10.6669 (42912)

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

Test	Oil Code	Parameter	Targets			4/1/08 - 9/30/08				10/1/08 - 3/31/09				4/1/09 - 9/30/09			
			n	Mean	sR	n	Mea n	sR	Mean Δ/s	n	Mean	sR	Mean Δ/s	n	Mean	sR	Mean Δ/s
D6417	52	Area % Volatized	18	6.97	0.31	4	6.9	0.28	-0.31	6	7.0	0.22	-0.01	4	7.0	0.17	0.18
	55	Area % Volatized	18	11.68	0.51	6	12.0	0.46	0.66	4	11.4	0.21	-0.65	4	12.0	0.39	0.73
	58	Area % Volatized	18	5.61	0.30	4	6.0	0.10	1.22	4	5.7	0.28	0.30	7	5.6	0.37	-0.03
D5800	52	% volatility loss	33	13.75	0.61	12	14.6	0.43	1.35	13	14.5	0.68	1.28	16	14.6	0.50	1.32
	55	% volatility loss	32	17.09	0.76	12	17.6	0.73	0.68	10	17.1	1.01	0.00	15	17.5	0.63	0.59
	58	% volatility loss	37	15.20	0.72	12	15.5	0.39	0.44	13	15.3	0.84	0.13	5	15.4	0.53	0.31
TEOST D6335	71	Deposit wt. (mg)	27	51.79	4.79	8	49.6	9.06	-0.45	10	56.6	4.38	1.01	6	52.4	11.51	0.13
	71-1	Deposit wt. (mg)		51.79	4.79	--	---	---	---	---	---	---	---	1	46.4	--	-1.13
	72	Deposit wt. (mg)	27	26.72	3.46	7	30.0	3.19	0.94	8	30.0	5.50	0.94	4	29.8	2.22	0.89
	72-1	Deposit wt. (mg)		26.72	3.46	--	---	---	---	--	---	---	---	3	28.2	0.85	0.42
MTEOS D7097	432	Deposit wt. (mg)	30	47.04	4.50	19	49.1	7.34	0.47	17	60.0	6.05	2.87	19	48.4	5.16	0.29
	434	Deposit wt. (mg)	30	27.37	6.57	10	24.5	6.52	-0.44	21	26.2	5.76	-0.18	13	26.1	4.05	-0.20
	74	Deposit wt. (mg)	30	12.85	5.59	17	11.5	3.90	-0.24	15	10.4	3.01	-0.43	16	10.5	3.42	-0.42
GI D5133	58	Gelation Index	17	5.8	0.69	10	6.5	1.39	1.00	6	6.2	0.51	0.51	14	5.9	0.98	0.19
	62	Gelation Index	35	17.0	3.90	9	16.2	5.85	-0.22	10	18.3	3.52	0.33	11	16.6	4.68	-0.09
	1009	Gelation Index	16	7.30	0.68	8	7.0	1.17	-0.39	8	7.0	0.26	-0.48	8	6.9	0.40	-0.62
D6082	1007	Tendency (ml)	28	65	19	10	65	16	-0.05	11	72	34	0.31	10	61	10	-0.26
D874	820-2	Sulfated Ash m%	27	1.57	0.08	2	1.54	0.02	-0.31	1	1.40	--	-2.12	3	1.59	0.01	0.25
	90	Sulfated Ash m%	27	1.07	0.08	2	1.00	0.02	-0.94	2	1.04	0.01	-0.44	2	0.91	0.04	-2.00
	91	Sulfated Ash m%	27	0.82	0.05	2	0.79	0.06	-0.60	3	0.75	0.08	-1.47	2	0.83	0.03	0.20
ROBO	434-1	ln (MRV Vis)	13	10.6599	0.1672	--	--	--	--	8	10.5971	0.1692	-0.38	7	10.5242	0.1288	-0.81
	435	ln (MRV Vis)	15	11.4895	0.2932	--	--	--	--	9	11.3553	0.3078	-0.46	11	11.3302	0.1960	-0.54
	438	ln (MRV Vis)	14	10.2676	0.2037	--	--	--	--	5	10.1330	0.1101	-0.66	8	10.1778	0.2144	-0.44