



Test Monitoring Center

6555 Penn Avenue
Pittsburgh, PA 15206-4489

MEMORANDUM: 00-163

DATE: November 20, 2000

TO: Mr. Jerry Wang, Chairman CBT Surveillance Panel

FROM: Tom Schofield

SUBJECT: TMC Corrosion Bench Reference Testing
From April 1, 2000 through September 30, 2000

I respectfully submit the TMC's ASTM D02.B02 Corrosion Bench Reference Test Monitoring Semiannual Report, broken down by test area (Attachment 1).

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 previous periods. The TMC estimates test precision by standard deviation (s) for a single oil, and test severity by mean Δ/s , where:

s = Standard deviation for a single reference oils

(i.e., Test precision by oil)

$\Delta/s = [(\text{Result}) - (\text{Target mean})] / (\text{Target } s)$

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

Mean $\Delta/s = [\Sigma (\Delta/s)] / n$ (severity across reference oils)

(i.e., Overall test severity; "On average, how many standard deviations from the target mean are all the operationally valid calibration tests for each period?")

Notice that because severity calculations are normalized into standard deviations, the severity estimates are oil independent. This simplifies the interpretation by allowing us to estimate the severity performance of HTCBT across both reference oil performance levels. Also, note that Δ/s and Mean Δ/s are calculated using the targets that were effective at the time of test completion (new CBT targets became effective 11/1/98 and new HTCBT targets became effective 3/16/99). Because there is only one reference oil for CBT, and the two HTCBT reference oils perform very differently from each other, we look at the standard deviations for each oil separately to estimate overall precision rather than try to pool the standard deviation across oils.

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HTCBT mean and standard deviation calculations for ΔCu were done by transforming the reported calibration results to a natural log, forcing a more normal distribution of the data. Without this transformation, the data distribution in original units is skewed unacceptably to severe. (CBT data is not transformed.)

The tables in Attachment 1 comparing current and previous period precision and severity have become too large to conveniently show the entire prior report periods. To keep the information succinct some of the oldest annual comparison periods have been deleted.

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 my last report, and should remain the same lab in future reports. Operationally valid calibration test data and severity plots are available on the TMC website. Please contact me if you require further information.

TMS/tms
Attachments

c: CBT Surveillance Panel
J. Zalar
M. Lane
<ftp://tmc.astm.cmri.cmu.edu/docs/bench/cbt/semiannualreports/mem00-163>

ASTM Test Monitoring Center

Semiannual Report

**ASTM D02.B02 Corrosion Bench Reference Test Monitoring
From April 1, 2000 through September 30, 2000**

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443**STATUS**

Table 1 summarizes the reference tests reported to the TMC this period (3 labs reporting).

TABLE 1

	No. of Tests
Statistically Acceptable and Operationally Valid	92
Operationally Valid but Failed Acceptance Criteria	7
Operationally Invalid or Aborted	6
Total	105

Table 2 is breakdown of the statistically unacceptable tests.

TABLE 2

Reason for Fail	No. of Tests
Δ Cu Mild (TMC OIL 1005)	1
Δ Cu Severe (TMC OIL 1005)	2
Δ Cu Severe (TMC OIL 42)	1
Δ Pb Severe (TMC OIL 1005)	2
Δ Pb Severe (TMC OIL 42)	1

Fail Rate of Operationally Valid tests (both oils): 7.1%

48 operationally valid tests total run on TMC Oil 42 of which 2 failed statistically (4.2% fail rate).

51 operationally valid tests total run on TMC Oil 1005 of which 5 failed statistically (9.8% fail rate).

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continuedINDUSTRY SEVERITY, TMC OIL 42 & TMC OIL 1005 (combined statistics)

Table 3 shows the current severity for the Change in Metal Concentration test parameters, on all operationally valid tests, for the current and previous report periods. Initial Industry targets were based on the Matrix 2 data as reported January 13, 1998 and approved by the surveillance panel March 18, 1998. Industry targets were adjusted by the panel effective 3/16/99 based on the first 115 operationally valid reference tests completed through 3/10/99. Severity Δ/s for each individual test was calculated using the target mean and target precision that was effective at the time of test completion (the first HTCBT calibration test reported to the TMC was completed 7/4/98).

TABLE 3

TMC Oils 42 & 1005

	n	Parameter	Mean Δ/s
Industry 10/1/98 through 3/31/99	98	Δ Cu	-0.76
		Δ Pb	0.98
Industry 4/1/99 through 9/30/99	63	Δ Cu	-0.16
		Δ Pb	0.16
Industry 10/1/99 through 3/31/00	84	Δ Cu	-0.40
		Δ Pb	-0.27
Industry 4/1/00 through 9/30/00	99	Δ Cu	-0.05
		Δ Pb	-0.24

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continued

Table 4 shows the current overall period severity for the Change in Metal Concentration test parameters for each lab.

TABLE 4

TMC Oils 42 & 1005

	Parameter	n	Mean Δ/s
Lab A	Δ Cu	35	0.56
	Δ Pb	35	0.45
Lab B	Δ Cu	49	-0.87
	Δ Pb	49	-0.93
Lab G	Δ Cu	15	1.19
	Δ Pb	15	0.42

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continued**INDUSTRY PERFORMANCE, TMC OIL 42 ONLY**

Table 5 shows the precision and severity for the Change in Metal Concentration test parameters, on all operationally valid tests run on TMC Oil 42, for the current and previous report periods. Initial Industry targets were based on the Matrix 2 data as reported January 13, 1998 and approved by the surveillance panel March 18, 1998. Industry targets were adjusted by the panel effective 3/16/99 based on the first 55 operationally valid reference tests (on TMC Oil 42) completed through 3/10/99. Severity Δ/s for each individual test was calculated using the target mean and target precision that was effective at the time of test completion. (Units in parentheses are transformed to natural log to better normalize the data distribution; the first HTCBT calibration test reported to the TMC was completed 7/4/98). Initial targets and older report periods are not included to make room for current data. Refer to prior TMC reports for older period summaries.

TABLE 5
TMC Oil 42 Only

	n	Parameter	Mean (ppm)	s	Mean Δ/s
Reference Data Through 3/10/99	55	Δ Cu	(3.223) 25.1	(0.583)	(New Targets)
		Δ Pb	107.8	23.77	(New Targets)
Industry 10/1/98 through 3/31/99	47	Δ Cu	(3.240) 25.5	(0.600)	-2.23
		Δ Pb	106.4	25.08	0.77
Industry 4/1/99 through 9/30/99	32	Δ Cu	(3.189) 24.3	(0.264)	-0.06
		Δ Pb	115.6	11.68	0.33
Industry 10/1/99 through 3/31/00	42	Δ Cu	(2.999) 20.1	(0.273)	-0.39
		Δ Pb	104.1	16.03	-0.16
Industry 4/1/00 through 9/30/00	48	Δ Cu	(2.981) 19.7	(0.424)	-0.42
		Δ Pb	104.9	25.10	-0.12

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continued

Table 6 shows the current precision and severity for the Change in Metal Concentration test parameters for each lab.

TABLE 6

TMC Oil 42 Only

	Parameter	n	Mean (ppm)	s	Mean Δ/s
Lab A	Δ Cu	18	(3.050) 21.1	(0.38)	-0.30
	Δ Pb	18	121.3	12.13	0.57
Lab B	Δ Cu	24	(2.770) 16.0	(0.08)	-0.78
	Δ Pb	24	87.9	7.26	-0.84
Lab G	Δ Cu	6	(3.619) 37.3	(0.68)	0.68
	Δ Pb	6	124.0	48.8	0.68

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continued**INDUSTRY PERFORMANCE, TMC OIL 1005 ONLY**

Table 7 shows the precision and severity for the Change in Metal Concentration test parameters, on all operationally valid tests run on TMC Oil 1005, for the current and previous report periods. Initial Industry targets were based on the Matrix 2 data as reported January 13, 1998 and approved by the surveillance panel March 18, 1998. Industry targets were adjusted by the panel effective 3/16/99 based on the first 60 operationally valid reference tests (on TMC Oil 1005) completed through 3/10/99. Severity Δ/s for each individual test was calculated using the target mean and target precision that was effective at the time of test completion. (Units in parentheses are natural log transformed to better normalize the data distribution; the first HTCBT calibration test reported to the TMC was completed 7/4/98). Initial targets and older report periods are not included to make room for current data. Refer to prior TMC reports for older period summaries.

TABLE 7
TMC Oil 1005 Only

	n	Parameter	Mean (ppm)	s	Mean Δ/s
Reference Data Through 3/10/99	60	Δ Cu	(2.255) 9.5	(0.141)	(New Targets)
		Δ Pb	32.2	13.03	(New Targets)
Industry 10/1/98 through 3/31/99	51	Δ Cu	(2.251) 9.5	(0.151)	0.60
		Δ Pb	33.4	15.20	1.18
Industry 4/1/99 through 9/30/99	31	Δ Cu	(2.219) 9.2	(0.128)	-0.26
		Δ Pb	32.1	12.01	-0.01
Industry 10/1/99 through 3/31/00	42	Δ Cu	(2.197) 9.0	(0.154)	-0.42
		Δ Pb	27.2	6.76	-0.39
Industry 4/1/00 through 9/30/00	51	Δ Cu	(2.297) 9.9	(0.38)	0.29
		Δ Pb	27.7	12.27	-0.35

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continued

Table 8 shows the current precision and severity for the Change in Metal Concentration test parameters for each lab.

TABLE 8

TMC Oil 1005 Only

	Parameter	n	Mean (ppm)	s	Mean Δ/s
Lab A	Δ Cu	17	(2.463) 11.7	(0.59)	1.47
	Δ Pb	17	36.5	14.02	0.33
Lab B	Δ Cu	25	(2.121) 8.3	(0.07)	-0.95
	Δ Pb	25	18.9	3.50	-1.02
Lab G	Δ Cu	9	(2.471) 11.8	(0.05)	1.52
	Δ Pb	9	35.5	6.16	0.25

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continuedPRECISION AND SEVERITY: Explanations & History

Severity trends for Change in Copper and Change in Lead are graphically represented in Figures 1 & 2 (attached).

Because the precision for each of the two HTCBT reference oils is very different it would be somewhat misleading to combine the precision analyses for the two oils into a pooled estimate of precision. To provide a more representative precision estimate of HTCBT calibration testing, precision estimates are not shown pooled across the two oils, but are instead broken down by oil (Tables 5 – 8). However, because severity estimates are normalized into standard deviations for each oil ("how many standard deviations is each test from target?"), combining the calibration performance of both oils for severity estimates gives a valid representation of overall calibration severity. Severity (mean Δ/s) estimates only are provided for both oils combined (Tables 3 & 4). Precision AND severity estimates are provided for each individual oil (Tables 5 & 7). Precision and severity estimates by lab & oil are also included (Tables 6 & 8). CUSUM plots (Figures 1 & 2), which graphically show severity trends, are shown only for both oils combined to give a better representation of overall industry severity. **Severity calculations and plots are made using the targets that were in place at the time of each test's completion.** That is, the matrix means and standard deviations were used in any severity calculations (and severity plotting) on tests completed before 3/16/99. Severity calculations (and plotting) for any test completed 3/16/99 or later use the new target means and standard deviation based on calibration testing.

Initially, mean performance and precision estimates for each of the reference oils were established from a matrix that was run in late 1997, before TMC monitoring. Since monitoring began on 7/4/98 these initial performance targets had not accurately reflected the observed overall Industry calibration performance of operationally valid testing for both reference oils (TMC Oils 42 & 1005) and both pass/fail parameters (Δ Cu & Δ Pb). Statistical failure rates on TMC Oil 42 were particularly high. The surveillance panel conducted exhaustive inquiries into why overall TMC calibration testing was not indicative of the original matrix performance. A rigorous explanation for underlying causes of the performance shifts (both in mean target performance, and overall precision) has proven to be elusive.

However, the surveillance panel agreed, on March 16, 1999, that HTCBT calibration performance had indeed shifted far from the targets established in the matrix, and the calibration data would better reflect current testing performance. The surveillance panel voted to adjust the target mean, precision and acceptance bands for the two reference oils to better reflect actual calibration performance. New performance targets and acceptance bands were implemented for tests completed after March 15, 1999 (see the official minutes of the panels March 16, 1999 teleconference for more details). The new targets and acceptance bands were based on all operationally valid TMC calibration test data completed from 7/4/98 (the start of TMC monitoring) through 3/10/99. The original matrix data was not used in setting the new targets.

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continued**PRECISION AND SEVERITY: Report Period Summary**

Three tests with abnormally severe results were reported as operationally valid this period. Lab A reported two tests more than 10 s severe of target for Δ Cu, both on oil 1005 (10.3 s & 14.3 s). Lab G reported one test 4.5 s severe for Δ Pb on oil 42.

Table 3 shows that overall calibration testing is running pretty much on target for Δ Cu this period (slight mild bias). However, the contribution of the two very extreme results from lab A indicates that less extreme milder tests are being reported more regularly, offsetting the extreme severe results. Figure 1 shows that the overall Δ Cu severity continued to run mild of targets even after the targets were changed on 3/16/99. More recently, from July 2000 through September 2000, the severity seemed to level much closer to target (excepting the two extreme results). However, there seems to be another strong mild trend developing after the end of this report period (9/30/00).

As mentioned, the targets were adjusted in March 1999 to correct for the mild trend observed up to that time. That these overall mild trends persist after the target adjustments should be of concern to the panel. The fact that there was some recent leveling from July to October 2000 (and a year earlier, July through November 1999) shows that the labs are capable of performing relatively on target for periods spanning several months (using the current adjusted targets).

A breakdown of the Δ Cu severity by oil (Tables 5 & 7) shows tests on oil 42 running moderately mild and tests on oil 1005 somewhat severe. Lab analysis of Δ Cu (Tables 6 & 8) shows two labs running mild and one severe on oil 42 but two labs severe (substantially so) and one lab mild on oil 1005. One thing that seems consistent with this test is that the lab severity is very inconsistent.

Overall Δ Pb severity for the period is 0.24 standard deviations mild of target (Table 3), comparable to last period. Two labs are comparably (and moderately) severe while one lab is significantly mild (Table 4). Figure 2 shows severity trends within the report period that the overall mean severity does not. In Figure 2, we see the Δ Pb going abruptly from extremely severe before the change in targets (on March 16, 1999) to substantially less severe after the change. Severity then slowly shifted to mild, with some recent leveling April through June 2000. July through October 2000 shows another mild trend, with significant worsening (slope increase) through the October timeline (marking the end of the current period). As with Δ Cu, severity for Δ Pb has some shifting trends within the current period, but favors overall mild.

Δ Pb severity by oil (Tables 5 & 7) shows both oils to be somewhat mild of targets. A breakdown by lab and oil (Tables 6 & 8) shows Labs A & G performing severe to different degrees on both oils and Lab B performing substantially mild on both oils.

Overall Δ Pb precision (Tables 5 & 7) is significantly worse again this period compared to last, but approximates the target precision for both oils (somewhat worse for oil 42 and directionally better for oil 1005). Precision comparisons between the labs (Tables 6 & 8) show very different performance between the labs. The precision of Lab G on oil 42 is particularly alarming. Lab B precision, looking at both oils, is particularly good this period.

High Temperature Corrosion Bench Test (HTCBT) by ASTM RR D02-1443, continued

PROCEDURE UPGRADE STATUS

Mr. Lyle Bowman has facilitated the upgrade of the test method to an ASTM Standard. As of this writing, the proposed standard has passed all committee ballots and is very nearly ready for publication. The method will be given the ASTM Standard designation D6954. The TMC will issue a technical memorandum of the procedure upgrade when the method is available for purchase from ASTM Headquarters.

REFERENCE OIL SUPPLY

The TMC has approximately 16.4 gallons of Oil 42, having used approximately 4.4 gallons in the previous 12 months. We also have a single drum of TMC Oil 1005 set aside solely for HTCBT reference testing. This drum has approximately 45.3 gallons remaining.

At the request of the surveillance panel (3/16/99), the TMC now mixes the drums of Oils 42 & 1005 before pouring aliquots for shipping as HTCBT reference samples.

TECHNICAL MEMORANDUMS

There were no HTCBT information letters or technical memorandums issued this report period.

Corrosion Bench Test (CBT) by ASTM D5968**STATUS**

All reference tests reported this period were run on TMC Oil 43.

Table 9 summarizes the reference tests reported to the TMC this period (4 labs reporting).

TABLE 9

	No. of Tests
Statistically Acceptable and Operationally Valid	31
Operationally Valid but Failed Acceptance Criteria	2
Operationally Invalid	4
Total	37

Fail rate of operationally valid tests: 6.1%

Table 10 is breakdown of the statistically unacceptable tests.

TABLE 10

Reason for Fail	No. of Tests
Change in Pb Mild	2

Corrosion Bench Test (CBT) by ASTM D5968, continued**INDUSTRY PERFORMANCE, TMC OIL 43**

Table 11 shows the precision and severity for the Change in Metal Concentration test parameters, on all operationally valid tests run on TMC Oil 43, for the current and previous report periods. Initial Industry targets and acceptance bands were based on a matrix introducing Oil 43 and a new coupon Batch C (with a new lead alloy), with lab severity bias on the previous CBT reference Oil 40 factored in. Industry targets were adjusted by the panel effective 11/1/98 based on the matrix data with no bias factors. **Severity Δ /s for each individual test was calculated using the target mean and target precision that was effective at the time of test completion.** CUSUM plots (Figures 4 & 5) include both Oils 40 and 43 to better show the severity history of the test. Initial targets and older report periods are not included in the tables to make room for current data. Refer to prior TMC reports for older period summaries.

TABLE 11
TMC Oil 43 Only

	n	Parameter	Mean (ppm)	s	Mean Δ /s
Revised Matrix	9	Δ Cu	18.28	2.94	-----
Targets 11/1/98		Δ Pb	119.60	14.68	-----
Industry 10/1/98 through 3/31/99	35	Δ Cu	18.2	1.54	0.02
		Δ Pb	112.0	12.90	-0.58
Industry 4/1/99 through 9/30/99	26	Δ Cu	17.1	1.94	-0.39
		Δ Pb	110.6	14.25	-0.61
Industry 10/1/99 Through 3/31/00	33 *32	Δ Cu	17.3	3.04	-0.33
		Δ Pb	117.5 *112.3	36.59 *21.18	-0.14 *-0.50
Industry 4/1/00 Through 9/30/00	33	Δ Cu	18.1	2.26	-0.06
		Δ Pb	116.6	17.80	-0.20

*With single extreme severe Δ Pb result excluded.

Corrosion Bench Test (CBT) by ASTM D5968, continued

Table 12 shows the current precision and severity for the Change in Metal Concentration test parameters for each lab.

TABLE 12
TMC Oil 43 Only

	Parameter	n	Mean (ppm)	s	Mean Δ/s
Lab A	Δ Cu	19	16.9	2.21	-0.46
	Δ Pb	19	125.7	13.24	0.41
Lab B	Δ Cu	3	19.7	0.58	0.47
	Δ Pb	3	88.5	9.26	-2.12
Lab G	Δ Cu	11	19.7	1.10	0.49
	Δ Pb	11	108.7	14.80	-0.74

Note: A fourth lab, Lab X, has reported three calibration tests this period as operationally valid. However, all three tests have been statistically unacceptable. Because the lab has been unable to achieve initial calibrated status, the TMC has elected to not include the results from lab X in the statistics. Once Lab X begins to calibrate successfully and routinely, we will begin to include their data in our statistical summaries.

PRECISION AND SEVERITY

All CBT calibration data this period was run on the TMC Oil 43/Batch "C" coupon combination (currently the only allowable combination for TMC calibrations). Both Oil 40 and Batch A coupons are obsolete and calibration data on that combination was last summarized in the TMC's semiannual report of May 17, 1999 (TMC memo 99-90 on CBT Testing October 1, 1998 Through March 31, 1999).

Due to an unacceptably high number of failing reference tests when Oil 43 was introduced, the surveillance panel voted to change the targets and acceptance bands of Oil 43 for all tests started after November 1, 1998. The first calibration test evaluated using the new targets and acceptance bands was completed November 10, 1998 (CMIR 32840). The CUSUM plots (Figures 3 & 4) include all operationally valid data (both Oils 40 & 43) to illustrate the entire severity history of the test. **In all cases, severity calculations and plots are made using the targets that were in place at the time of each test's completion.**

Table 11 shows overall precision is improved compared to last period on both parameters. Δ Cu precision is better than target while Δ Pb is somewhat worse. Δ Cu severity is near target (slight mild bias) while Δ Pb is running somewhat mild. Both parameters are closer to target than last period.

Corrosion Bench Test (CBT) by ASTM D5968, continued

Table 12 shows the current period precision and severity by lab. For Δ Cu severity, Lab A is running moderately mild while Labs B & G are running equally moderately severe. For Δ Pb we see the opposite trends, with Lab A running severe and Labs B & G running substantially mild (Lab B is extremely mild). Unlike last period, there are no lab precision issues this period; all labs appear to be running near or better than the target precision for both parameters.

Severity trends for Δ Cu and Δ Pb (for both oils 40 & 43 combined) are graphically represented in Figures 3 & 4. Figure 3 shows Δ Cu severity to be reasonably level throughout the report period, with only minor trends developing within the period. Figure 4 shows a leveling of Δ Pb severity throughout most of the report period, but with a disturbing mild trend developing again at the end of the period. Keep in mind that targets were adjusted on November 1, 1998 to account for the persistently strong mild trend for Δ Pb.

REFERENCE OIL SUPPLY

Per surveillance panel approval, the referencing labs had been contacted some time ago to dispose of all remaining reference samples of TMC Oil 40. The TMC currently has sixteen 130 ml samples of reference Oil 40 left in our inventory. The TMC will store these samples for approximately six more months then dispose of them if the surveillance panel has no further use for them.

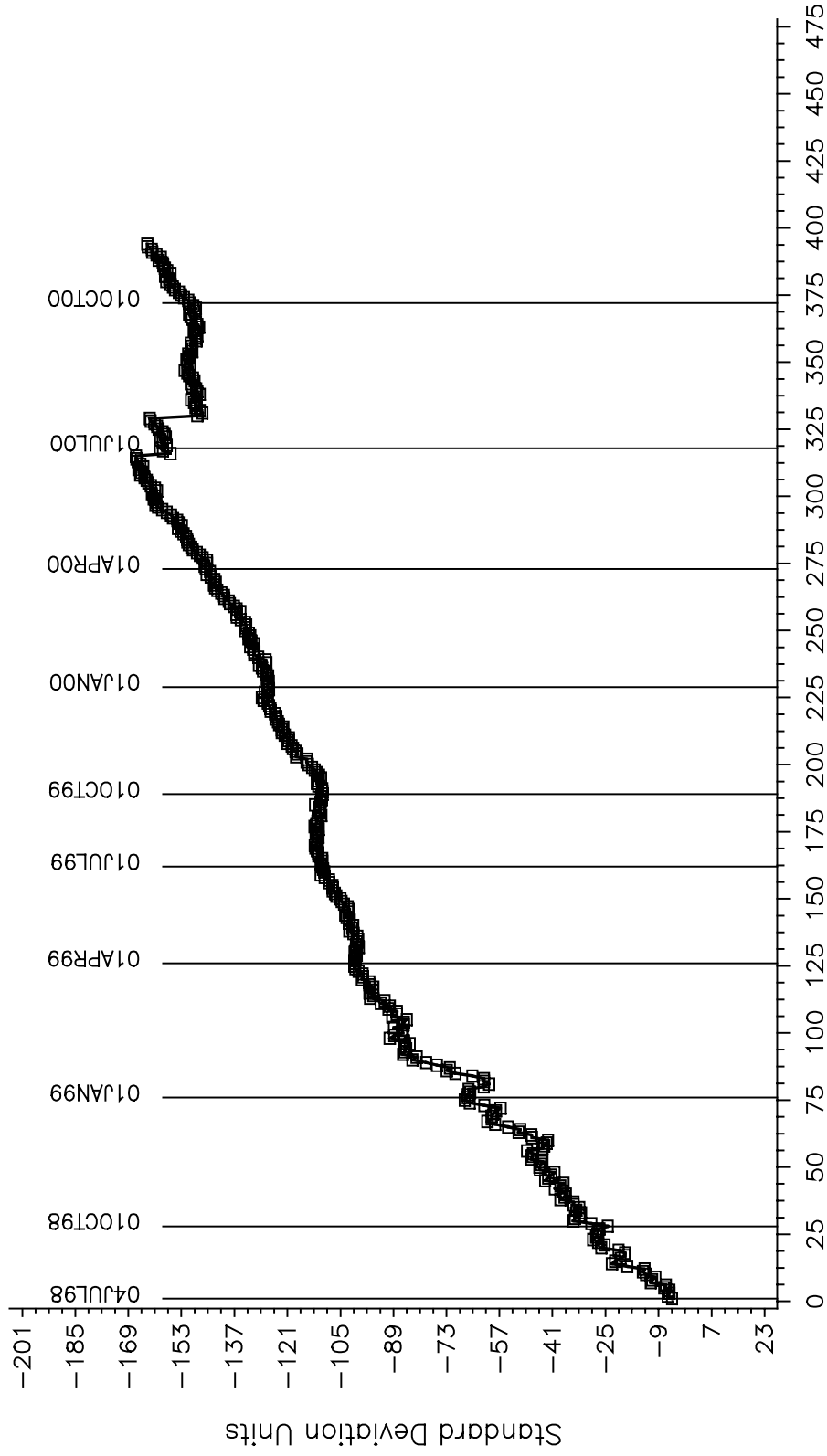
The TMC has approximately 68.3 gallons of TMC Oil 43 left. We shipped approximately 2.4 gallons in the past 12 months. There is ample supply of TMC Oil 43 at this time.

INFORMATION LETTERS

There were no CBT information letters or technical memorandums issued this report period.

HIGH TEMP CBT INDUSTRY OPERATIONALLY VALID DATA
TMC Oils 42 & 1005 (combined statistics)
REF. COPPER CHANGE IN CONCENTRATION [μ](ppm)

CUSUM Severity Analysis

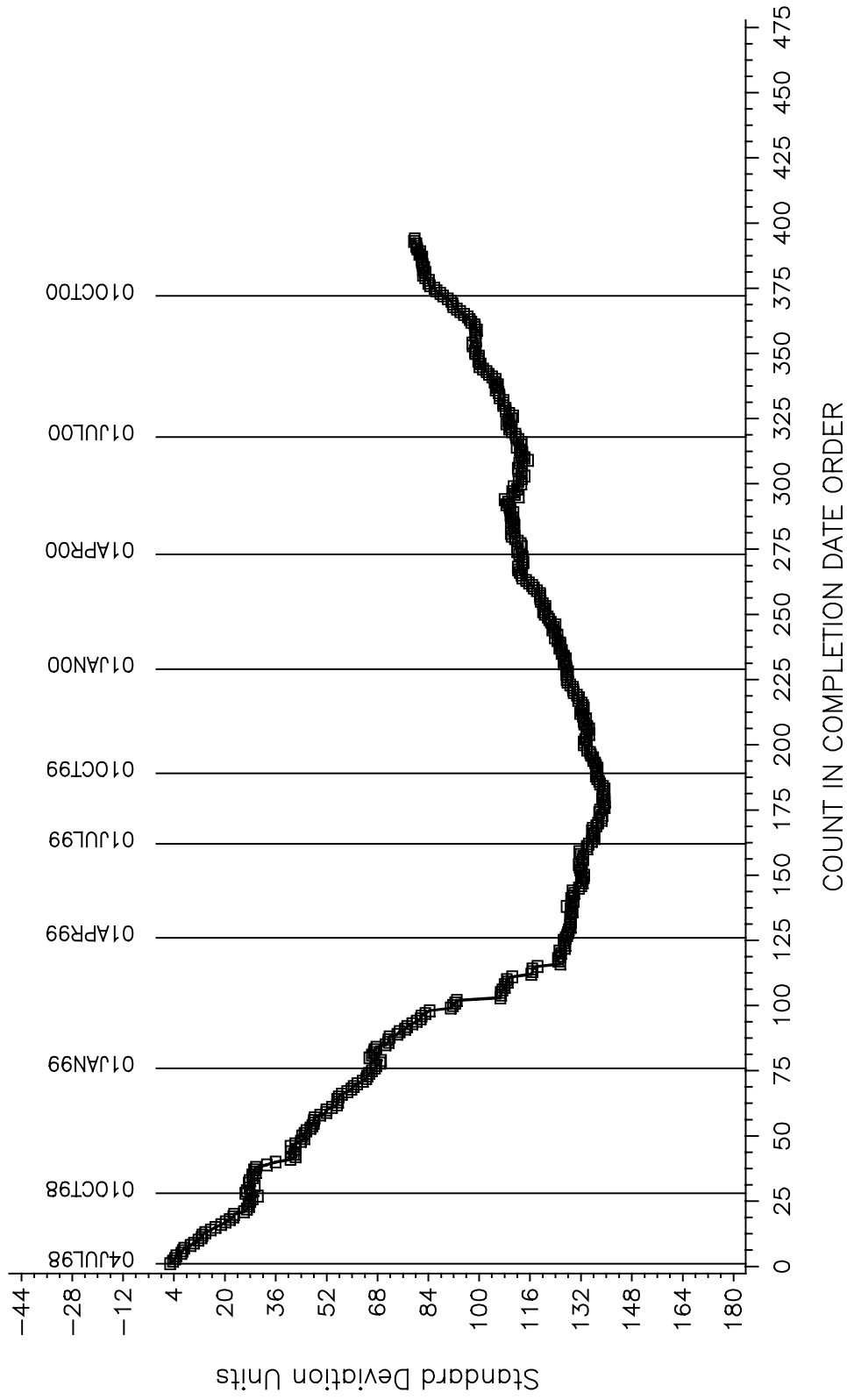


TMC 15NOV00:17:26
COUNT IN COMPLETION DATE ORDER

HIGH TEMP CBT INDUSTRY OPERATIONALLY VALID DATA
TMC Oils 42 & 1005 (combined statistics)
REF. LEAD CHANGE IN CONCENTRATION [\leq](ppm)

CUSUM Severity Analysis

Figure 2

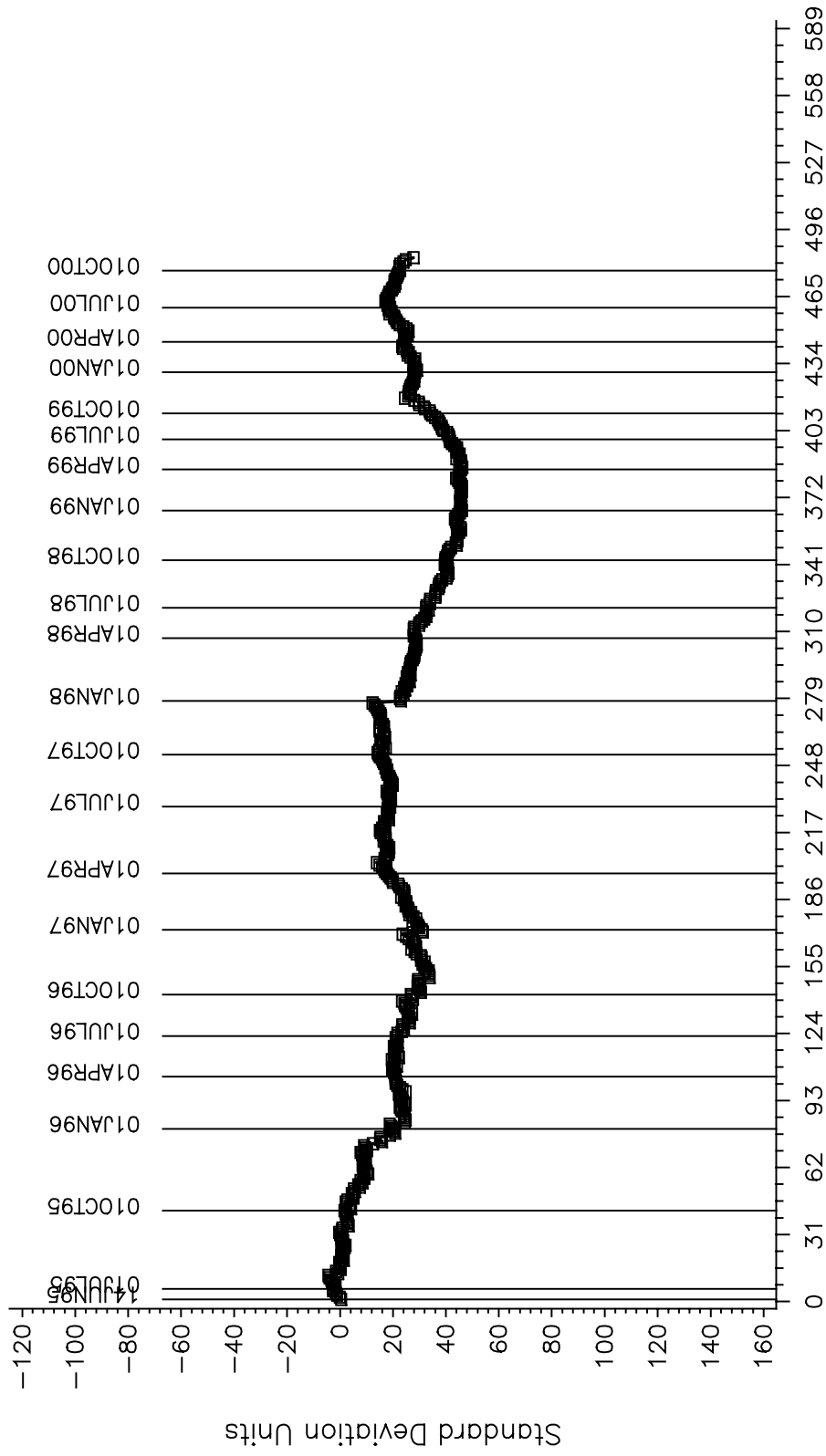


TMC 15NOV00:17:26

CBT INDUSTRY OPERATIONALLY VALID DATA
Oils 40 & 43 (combined statistics)
REF. COPPER CHANGE IN CONCENTRATION [$<1=0$] (PPM)

Figure 3

CUSUM Severity Analysis



TMC 15NOV00:17:08

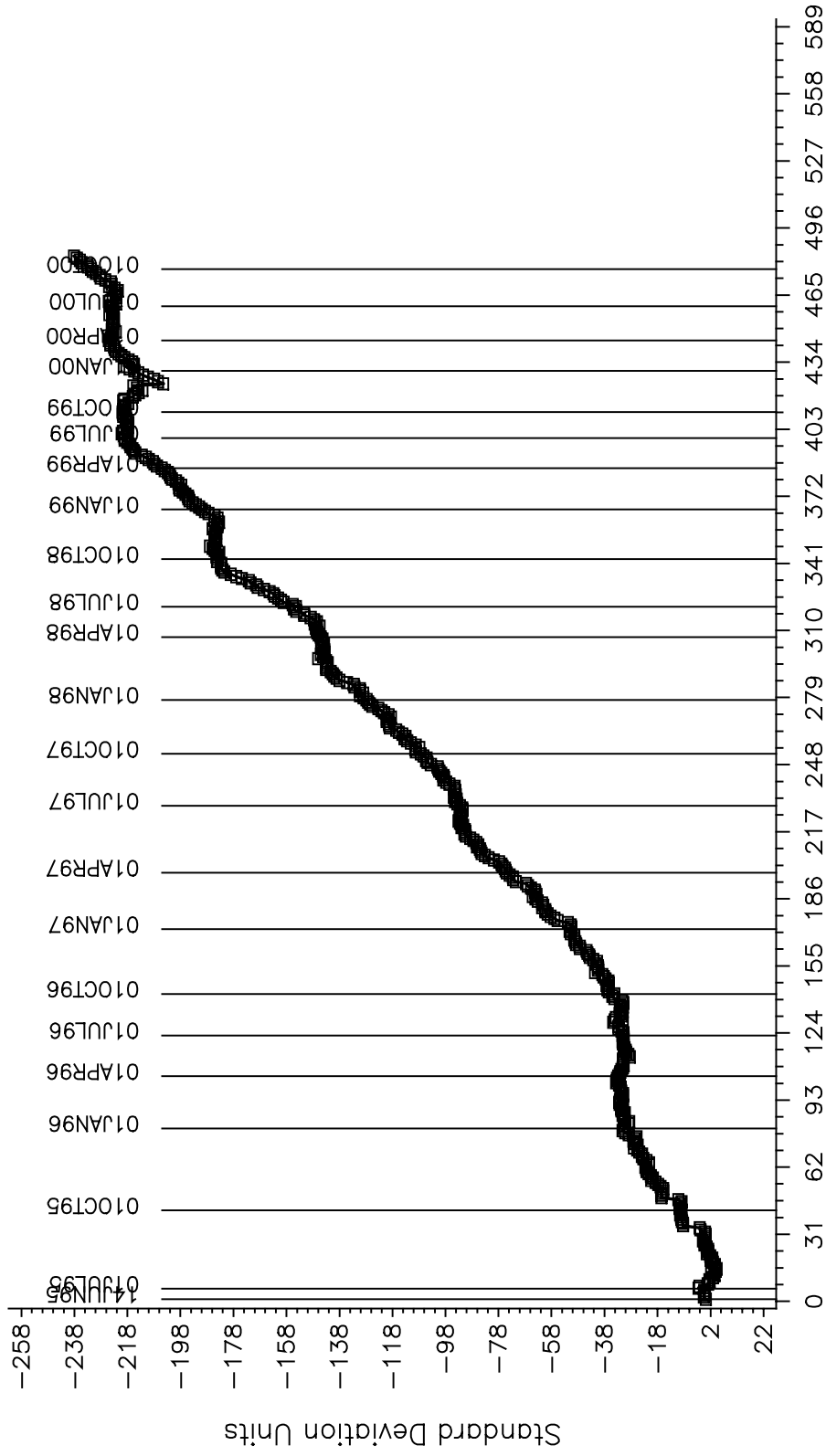
CBT INDUSTRY OPERATIONALLY VALID DATA

Oils 40 & 43 (combined statistics)

REF. LEAD CHANGE IN CONCENTRATION [$<1=0$] (PPM)

CUSUM Severity Analysis

Figure 4



COUNT IN COMPLETION DATE ORDER

TMC 15NOV00:17:08