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Test Monitoring Center

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MEMORANDUM: 24-025

DATE: August 27, 2024

TO: D02.B0.07 Section and Surveillance Panel Chairs

FROM: John G. Loop

SUBJECT: Examination of ASTM D 6557 (BRT) Reference Oils

On May 2, 2024, the Surveillance Panel governing the Ball Rust Test (BRT) voted to suspend the use of Reference Oils 86 and 87 until an investigation as to why labs have seen an increase in failing calibration results with these two reference oils could be completed. TMC was able to recover a few BRT reference oil retains from the labs and complete a quality assessment of these retains as well as survey specific lab calibration test results over the last 12 months. Unfortunately, TMC could not identify any one root cause or reason for the increase in failing calibration results found to occur in the April/May 2024 time period.

A summary of TMC's investigation and conclusions are presented within this document.

Five test labs were running calibrated BRT testing on all four Reference Oils (1006, 82-1, 86 and 87) at the time when one lab came forward with concerns about recent numbers of failures with Reference Oil 86. A summary of the fails occurring within the previous 12 months for all five labs is shown below in TABLE 1.

TABLE 1: RO 86 & 87 Fail Rate Summary (between June 2023 and June 2024)

LAB ID	RO 86 Fails	RO 86 Fail Rate	RO 87 Fails	RO 87 Fail Rate
A	4	9%	0	0%
B	1	25%	0	0%
D	3	43%	3	43%
G	3	10%	3	10%
L	0	0%	0	0%

Labs were using the same batch (same drum source) of Reference Oils 86 & 87 for all runs well before and during the time when occurrences of failing tests spiked. Labs went through many different Acid Batches over this same time period. Most labs were on Acid Batches Z9 or Z10 with most fails occurring with Acid Batch Z10.

TABLE 2 (shown below) lists the specific combination of Reference Oil Batch ID with the Acid Batch ID for all failed calibrations with RO 86 and RO 87 over 12 months preceding the decision to suspend testing on 86 and 87.

Note: Lab L had three test runs with RO's 86 & 87, but no calibration run failures.

TABLE 2: Reference Oil and Acid Batch ID's for Failing Test Results

LAB ID	RO 86 Fails Batch ID	Acid Batch Fails Batch ID	RO 87 Fails Batch ID	Acid Batch Fails Batch ID
A	4 20170901-1-1	1 GMA-01-Z10 3 GMA-13-Z10	None (20170906-1-2)	N/A (many Z9, Z10)
B	1 20170901-1-1	1 GMA-02-Z10	None (20170906-1-2)	N/A (GMA-02-Z10)
D	3 20170901-1-1	1 Z9 2 Z10	3 20170906-1-2	1 Z9 2 Z10
G	3 20170901-1-1	1 GMA-07-Z9 1 GMA-02-Z7 1 GMA-12-Z10	3 20170906-1-2	2 GMA-11-Z10 1 GMA-12-Z10
L	None (20170901-1-1)	N/A (GMA-13-Z9) (GMA-07-Z10)	N/A (20170906-1-2)	N/A (GMA-07-Z10)

TABLE 3 (shown below) summarizes the range of Reference Oil Storage Days versus Storage Days when failing tests occurred. No correlation is observed between "Storage Days (at Labs) and Calibration Test Fails".

Note: Some labs have successfully stored samples for over a year (>365 days) and still achieved passing calibration test results.

TABLE 3: Storage Days vs Failing Tests

LAB ID	RO 86 Storage Days Range	RO 86 Storage Day Fails	RO 87 Storage Days Range	RO 86 Storage Day Fails
A	23 to 115	76, 98, 106, 112	24 to 136	None
B	204 to 406	208	393 to 420	None
D	127 to 463	127, 196, 343	51 to 640	448, 576, 640
G	14 to 168	14, 55, 67	19 to 157	90, 98, 140
L	1022 to 1167	None	1065	None

TMC was able to obtain six retains of reference oil samples after the surveillance panel had decided to suspend RO 86 and 87. TABLE 4 (shown below) summarizes the reference oil retain samples recovered.

TABLE 4: Summary of Reference Oil Retains recovered and Analyzed by TMC

	Reference Oil 82-1	Reference Oil 86	Reference Oil 87
TESTKEY	187576	187586	187596
Ship Date	20240327	20240327	20240327
LTMS Date	20240430	20240521	20240521
Result	Yi = -0.256 (Pass)	Yi = -4.913 (Fail)	Yi = +0.259 (Pass)
TESTKEY	187577	185041	185022
Ship Date	20240327	20231201	20231201
LTMS Date	20240516	20240502	20240503
Result (sd)	Yi = +4.195 (Fail)	Yi = -0.544 (Pass)	Yi = -0.212 (Pass)

Quality testing conducted on the retains included Metals by ICP and FTIR.

ICP testing showed no significant difference between metals found for the recovered retain samples, a sampling of the inventory drum currently stored at TMC and the historical metals profile for all three reference oils. This would indicate that the concentrations of the detergents and anti-wear agents remain consistent in the samples sent out for testing as well as the reference oil in storage at TMC.

Differential FTIR scans were also compared between the recovered retains and the current TMC inventory. The results show matching spectra with only a small increase in moisture for the samples returned from the lab. However, this amount of moisture found was quite small and is typically observed by TMC when reference oil samples (which were open to atmosphere) are returned and analyzed by FTIR.

Note: There were both passing and failing results for the 82-1 and 86 recovered reference oil samples, but nothing remarkable was found regarding their ICP or FTIR profiles. And these samples came from the same drum and were tested around the time of the spike in RO 86 and 87 fails at the labs.

Other factors that may have contributed to the recent increase in failures but were not studied by TMC include the specific batches of Acid Solutions, the condition of the Batch D ball bearings, the condition of the lab air supply, and the condition of the shaker tables and syringe pumps that were used to run the calibration tests.

Conclusions: TMC investigated the Ball Rust Test (ASTM D 6557) but was unable to identify a specific reason why there was an increase in failing calibration test results in the Spring of 2024. Quality Control (QC) checks of TESTKEY retains recovered from one lab did not show signs of contamination or degradation which could have caused low AGV test results. A survey of the timing and quantity of fails at the five test labs was wholly inconsistent. Some labs had failing results with Reference Oil 86 alone, some labs had failing results with both Reference Oils 86 and 87, one lab had passes and fails with Reference Oil 86, and one lab had no fails with either Reference Oil 86 or 87. All labs have been using TESTKEYs from single drum sources of Reference Oil 86 and Reference Oil 87 well before the fails started happening. But for those labs with failures, the failure rate ramped up at about the same time suggesting that there is/was a common cause for failures. Unfortunately, however, the root cause of these failures is not apparent (at this time). TMC recommends resuming Reference Oils 86 and 87 assignments for BRT calibration testing because there isn't evidence to indicate that this is solely an issue with the reference oils. And TMC recommends that labs hold on to BRT TESTKEY retains (and possibly Acid Solution, and Used Test Oils) for a selected time period so that if failures continue, there will be a better selection of potential investigation samples available for a future root cause analysis study.

https://www.astmtmc.org/ftp/docs/bench/brt/memos/mem24-025.jgl.pdf

JGL/jgl/mem24-025.jgl.docx

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