



Test Monitoring Center

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MEMORANDUM: 18-029

DATE: September 6, 2018

TO: D02.B07 Volatility Mailing List

FROM: Tom Schofield

SUBJECT: D6417 (Volatility by GC) Technical Update: New TMC Calibration Requirements Effective October 1, 2018

On August 30, 2018, the ASTM D02.B0.07 Volatility Surveillance Panel voted by teleconference to approve the attached TMC Calibration Requirements which explain the new requirements needed to calibrate a D6417 GC instrument with the TMC.

These requirements are effective October 1, 2018.

TMS/tms

Attachment

c: <http://www.astmtmc.cmu.edu/ftp/docs/bench/d6417/memos/mem18-029.pdf>

Distribution: Email

D6417 (Volatility by GC) TMC Calibration Requirements
Surveillance Panel Approved Version 20180830

The following are the specific D6417 (Volatility by GC) TMC calibration test requirements as approved by the ASTM D02.B0.07 Volatility Surveillance Panel by Teleconference vote on 20180830, and effective 20181001.

A. Reference Oils and Critical Parameters

1. The critical pass/fail parameter is Area % Volatility Loss at 371°C. The reference oils, performance targets and acceptance criteria required for the test stand calibration with the TMC are listed in Table 1 and have been approved by the ASTM D02.B0.07 Volatility Surveillance Panel.

Table 1
D6417 (Volatility by GC) Reference Oil Targets and Acceptance Bands Effective 20001002

Test	Oil Code	Parameter	n	Mean	sR	Acceptance Bands*	
						Lower	Upper
GC Volatility By 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

*95% Acceptance Bands = Mean +/- (1.960 x sR)

B. Test Stand Defined

1. A test stand is defined as a single channel on a Gas Chromatograph (GC) instrument. If labs wish to calibrate multiple channels in an instrument, each channel must be registered with the TMC as a separate test stand.
2. Tracking and Reporting Stand ID's
 - a. Tracking a stands calibration status will be effected by tracking and reporting to the TMC the instrument serial number and, for instruments with multiple channels, an "F" (Front) or "B" (Back) as a suffix to uniquely identify each channel. Instrument ID shall be the instrument serial number and shall not change for the entire history of a TMC monitored test stand (instrument ID variations will be allowed for existing instruments that were calibrated prior to the serial number ID requirement).

C. Acceptance Criteria

1. New Laboratory/Test Stand(s)
 - a. The TMC calibration system calibrates individual test stands (single GC channel) at individual laboratories. There are no special requirements to bring a LAB into TMC calibrated status, there are only requirements to bring individual test stands into TMC calibrated status, as follows:
 - b. A minimum of two (2) operationally valid calibration tests which fall within the acceptance bands for the oils assigned are required to calibrate a stand for the first time. These must be back-to-back consecutive runs on the same test stand, though exceptions can be made at the sole discretion of the TMC for operational fails for reasons that would be considered to have had no bearing on the

operational performance of the test stand for subsequent tests (for example, a power failure).

- c. Passing two back-to-back consecutive TMC calibrations places the new test stand in TMC calibrated status. Both tests must pass on operational and statistical criteria.
- d. TMC calibrated status of a test stand is valid for no more than 90 days from date completed of a valid TMC calibration (that is, the end of the full elution of the test sample). To renew the calibration at the end of the calibration period, see Item 2 for Existing Laboratory/Test Stand(s).

2. Existing Laboratory/Test Stand(s)

- a. An existing TMC calibrated test stand, or one where the TMC calibrated status had expired within the past 90 days, can renew its TMC calibrated status by demonstrating a successful calibration on another single TMC blind calibration audit. The test must pass on both operational and statistical criteria.
- b. TMC calibrated status of an existing test stand is valid for no more than 90 days from date completed of a valid TMC calibration (that is, the end of the full test sample elution). Test stands that exceed these time/run specifications are considered to be out of calibration for TMC monitoring purposes.
- c. A stand that has been out of TMC calibration for more than 90 days from the prior TMC calibration expiration date will require New Test Stand calibration as listed in C.1.b through C.1.d. of this document.
- d. A stand must pass the TMC calibration within two operationally valid test runs. If a stand cannot produce a calibration test that falls into the acceptance bands for the assigned oil within two operationally valid runs, renewing calibration on that stand will require the two-test calibration as listed in C.1.b through C.1.d.
- e. Changing the injector flow controller, changing the electronic pneumatic control (EPC) module or changing the flame ionization detector (FID) on a stand for any reason voids the current TMC calibrated status. Renewing calibration on that stand will require a two-test calibration as listed in C.1.b through C.1.d.

3. Reference Oil Assignment

- a. Of the two tests required to bring a new stand into TMC calibrated status, the tests shall be conducted on reference oil 55 and either oils 52 or 58, or reblends as approved by the surveillance panel, assigned in random order. Once a stand has attained TMC calibrated status (existing test stand), 100% of the scheduled calibration tests shall be conducted on an assigned blind reference oil sample from the currently accepted set of reference oils. There shall be no preference for any one reference oil in blind calibration runs, and each shall be assigned for approximately 1/3 of the passing runs on each stand.

4. Mandatory Daily QC Check Sample

- a. All TMC-monitored instruments must utilize TMC reference oil 58 to comply with the Reference Materials section of the ASTM D6417 test method. TMC reference oil 58 is to be run daily, or immediately prior to each session of D6417 runs on each test stand to confirm performance of the test stand before commencing with TMC blind reference, candidate or non-reference runs. The daily QC reference

material run must meet the acceptance bands shown in Table 1 for TMC oil 58. If the result falls outside those approved bands, the problem must be resolved, and additional TMC oil 58 runs performed as needed to confirm that the daily QC check sample reliably meets the acceptance range in Table 1. The successful Reference Material run result for the session that includes the TMC blind calibration run is to be reported to the TMC with the blind calibration data using the approved TMC reporting format.

- b. Four-ounce aliquots of TMC oil 58, for use as a D6417 daily QC check fluid, are available for purchase from the TMC.
5. Calibration Test Evaluation:
 - a. The calibration status of the stands will be based on a review of operational parameters for compliance with the test method, followed by a statistical evaluation of the critical parameter test result against the acceptance ranges in Section A (commonly referred to as a Shewhart severity evaluation). Unless otherwise noted, the acceptance bands in Section A are based on a 95% confidence treatment of round robin test results with data exclusions as approved by the surveillance panel.
 6. Removal of Test Stands from the System
 - a. The laboratory must notify the TMC when removing a stand from the system. No reference oil data shall be removed from the TMC's data base of prior TMC calibrations or calibration attempts. Return of the stand to the system will be evaluated based on section C.1.b through C.1.d above.
 7. Introduction of New or Re-Blended Reference Oils
 - a. Introduction of new or replacement reference oils will be conducted at the discretion of the surveillance panel. Participating laboratories may be asked to donate tests on the new oil(s) to establish baseline performance in the D6417 GC test. The number of tests requested will be sufficient to rigorously evaluate the oil's performance. Preliminary statistical performance targets and acceptance criteria will be established by the surveillance panel, and those values will be re-assessed as the TMC collects additional calibration data.