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HTCBT INFORMATION LETTER 05-1
Sequence No. 6

January 7, 2005

***ASTM consensus has not been obtained on this information letter. An appropriate ASTM ballot will be issued in order to achieve such consensus.***

TO: CBT Mailing List

SUBJECT: Coupon Cleaning Solvent
Evaporation Loss Measurement and Calculation
Donated Reference Oil Test Programs
Precision Estimate

Coupon Cleaning Solvent

As approved by the Corrosion Bench Test Surveillance Panel, the pretest coupon cleaning and storing solvent for Test Method D 6594 has changed from tetrahydrofuran to acetone, and the post test copper coupon cleaning solvent has changed from tetrahydrofuran to heptane. Sections 7.7, 8.1.1, 8.3.3, 8.3.4, 8.3.5 and 10.2 have been revised and are attached. Section 7.8 has been deleted and Section 7.9 has been renumbered. This change is effective December 7, 2004.

Evaporation Loss Measurement and Calculation

As approved by the Corrosion Bench Test Surveillance Panel, the measurement and calculation methodology for evaporation loss has changed. Sections 6.3.5, 9.4 through 9.9, and 10.5.1 are affected and are attached. It should be noted that this is a measurement and calculation change only; no operational changes to the test have been made. This change is effective December 7, 2004.

Donated Reference Oil Test Programs

On November 8, 2004, ASTM Subcommittee D02.B approved a recommendation from the Test Monitoring Board to revise test methods monitored by the Test Monitoring Center regarding the surveillance panels' use of donated reference oil test programs. This revision provides consistent language for the procedures and clarification to the end users. Accordingly, Section 11.2 has been added and is attached.

Precision Estimate

At the request of ASTM Section D02.B0.09, the definitions of Intermediate Precision and Reproducibility in Test Method D 6594 have been revised. Note 4 has been added to Section 13.1.1, Sections 13.1.1.1 and 13.1.2.1 have been updated, Section 13.1.3 has been added, and a footnote has been added to Table 1. All new and revised sections are attached. This change is effective the date of this information letter.

Joe Franklin
Chairman
CBT Surveillance Panel

John L. Zalar
Administrator
ASTM Test Monitoring Center

Attachment

c: ftp://ftp.astmtmc.cmu.edu/docs/bench/htcbt/procedure_and_ils/htcbtil05-1.pdf

Distribution: Email

**(Revises Test Method D 6594-04a)**

6.3.5 Ruler, 30 cm minimum length, 1-mm graduations.

**7.7 *Heptane.* (Warning – Flammable. Health hazard.)**

**Delete Section 7.8**

**Renumber old Section 7.9 as new Section 7.8**

**Renumber old Section 7.10 as new Section 7.9**

8.1.1 Rinse all items and the air tube adapter with cleaning solvent to remove residual oil, and air-dry.

8.3.3 Store the polished metal specimens in acetone.

8.3.4 Just prior to a test start, remove each specimen from the acetone, and clean all metal dust from the specimen using 100% cotton. Rub with a light-to-medium touch to remove the particles but do not polish the specimen further.

8.3.5 Wash specimens in acetone and allow them to dry in a desiccator.

**Delete Section 9.4 and renumber Sections 9.5 through 9.9 (and subsections) as Sections 9.4 through 9.8 (and subsections). Upon completing renumbering, change text of sections 9.4, 9.8.2, and 9.8.3 as follows:**

9.4 Add 100 +/- 1 mL of oil volumetrically to the test tube by syringe, measure the oil level in the test tube to the nearest 1 mm and record as the initial level of oil.

9.8.2 Remove sample tube from the bath, allow it to cool to room temperature, and wipe off the outside of the tube with a cloth dampened with cleaning solvent.

9.8.3 Re-measure the oil level to the nearest 1 mm and compute the percentage of level loss resulting from evaporation of oil and potential leakage (see 10.5.1). If the loss is greater than 8%, leakage is present. Correct the leak, and repeat the determination, using fresh oil sample and new coupons.

10.2 Using forceps or gloves, wash the copper specimen in heptane, and discard the other specimens.

10.5.1 Evaporation Loss:

$$L = [(V_2 - V_1) / V_2] \times 100 \quad (1)$$

where:

L = percentage evaporation loss,

$V_1$ = final level of the oil in the test tube, and

$V_2$ = initial level of oil in the test tube.

11.2 *Donated Reference Oil Test Programs* - The surveillance panel is charged with maintaining effective reference oil test severity and precision monitoring. During times of new parts introductions, new or re-blended reference oil additions, and procedural revisions, it may be necessary to evaluate the possible effects on severity and precision levels. The surveillance panel may choose to conduct a program of donated reference oil tests in those laboratories participating in the monitoring system, in order to quantify the effect of a particular change on severity and precision. Typically, the surveillance panel requests its panel members to volunteer enough reference oil test results to create a robust data set. Broad laboratory participation is needed to provide a representative sampling of the industry. To ensure the quality of the

data obtained, donated tests are conducted on calibrated test baths. The surveillance panel shall arrange an appropriate number of donated tests and ensure completion of the test program in a timely manner.

13.1.1 *Intermediate Precision Conditions* – Conditions where test results are obtained with the same test method using the same test oil, with changing conditions such as operators, measuring equipment, test stands, test engines, and time.

Note 4 – Intermediate precision is the appropriate term for this method rather than repeatability which defines more rigorous within-laboratory conditions.

13.1.1.1 *Intermediate Precision Limit (i.p.)* – The difference between two results obtained under intermediate precision conditions that would, in the long run, in the normal and correct conduct of the test method, exceed the values shown in Table 1 in only one case in twenty. When only a single test result is available, the Intermediate Precision Limit can be used to calculate a range (test result ± Intermediate Precision Limit) outside of which a second test result would be expected to fall about one time in twenty.

13.1.2.1 *Reproducibility Limit (R)* – The difference between two results obtained under reproducibility conditions that would, in the long run, in the normal and correct conduct of the test method, exceed the values shown in Table 1 in only one case in twenty. When only a single test result is available, the Reproducibility Limit can be used to calculate a range (test result ± Reproducibility Limit) outside of which a second test result would be expected to fall about one time in twenty.

13.1.3 The test precision, as of January 1, 2005 is shown in Table 1.

**TABLE 1 Reference Oil Statistics [A]**

| Test Result | Intermediate Precision | | Reproducibility | |
|---|---|---|---|---|
| | $S_{i.p.}$ | i.p. | $S_R$ | R |
| Δ Copper, ln(ppm)[B] | 0.318 | 0.890 | 0.349 | 0.977 |
| Δ Lead, ppm | 15.54 | 43.51 | 17.16 | 48.05 |

| Legend: | |
|---|---|
| $S_{i.p.}$ | = intermediate precision standard deviation |
| *i.p.* | = intermediate precision limit [C] |
| $S_R$ | = reproducibility standard deviation |
| *R* | = reproducibility limit [C] |

[A] These statistics are based on results obtained on Test Monitoring Center Reference Oils 42 and 1005.
[B] This parameter is transformed using a natural log. When comparing two test results on this parameter, first apply this transformation to each test result. Compare the absolute difference between the transformed results with the appropriate (intermediate or reproducibility) precision limit.
[C] This value is obtained by multiplying the standard deviation by 2.8.