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MEMORANDUM: 06-055

DATE: August 9, 2006

TO: D02.B07 TEOST Mailing List

FROM: Tom Schofield

SUBJECT: MTEOS Technical Update: Section 10.7 Test Procedure Clarification

Since the introduction of the D7097 (MHT TEOST) test method, the TMC has been caught in the middle of conflicting advisement about the exact procedural intent of Section 10.7 of the test method. Sections 10.6 and 10.7 of D7097-06 read:

> 10.6 Using a microlitre syringe, add the pre-calculated mass of catalyst required to make 8.5 g of sample-catalyst mixture based on the certified value of the catalyst and record the mass to the nearest 0.0001 g. The range for the mass of catalyst to be added shall be +/-0.0003 g of the mass required.
>
> Note 7-- The mass of oil required for the appropriate mixture of catalyst-to-oil ratio is stated on the label of the vial of certified catalyst
>
> 10.7 Again, tare the balance and add the required mass of oil to the sample flask to make the sample-catalyst mixture total 8.5 g. The range for the mass of the oil to be added shall be +/-0.01 g of the mass required. If more oil than required is added, make a new sample.

TMC monitoring has exposed operational discrepancies resulting from the statement in Section 10.7 “The range for the mass of the oil to be added shall be +/-0.01 g of the mass required.” That statement, as written, is ambiguous, particularly if one understands the chemistry behind the need to add a catalyst very exactly. The TMC has had a few labs reporting tests where the catalyst is added to +/-0.0003 g of the target amount, and then the “required” mass of neat sample is simply added to bring the total catalyzed sample mixture up to 8.5 g. Other labs (the majority) are reporting tests where the catalyst is added to +/- 0.0003 g of the target amount, and then the target amount of “required” neat sample to meet the correct catalyst-to-sample ratio is recalculated, based on the actual amount of catalyst added to the flask, and that calculated amount of neat sample is added to +/-0.01 g (though, this interpretation might mean the final catalyzed sample mass could be somewhat more or less than 8.5 g).

After extensive and robust discussion with knowledgeable parties, the TMC’s position is that the test method, as presently written, could correctly be interpreted either way. In the attached memo, Ted Selby has weighed in rather conclusively on the topic. He is correct in pointing out that the sentence “The range for the mass of the oil to be added shall be +/-0.01 g of the mass required” is ambiguous as to the subject of the sentence (the mass required for what, to reach the correct ratio, or to reach the 8.5 g final sample mass?).

Ted also points out in his memo that the recommended practice would be to try to maintain the catalyst-to-sample ratio with the potential compromise of not exactly obtaining an 8.5 g final catalyzed sample mass.

In trying to monitor conformance and compliance to the test method, the TMC recognizes there is justifiable confusion among operators, and we will presently accept EITHER interpretation of Section 10.7 of the test method as operationally valid. However, please be aware that the RECOMMENDED interpretation is to first weigh out the catalyst to within +/- 0.0003 g of the target amount required (accurately weighed to +/- 0.0001 g), then to RECALCULATE the target mass of neat test sample to be added to meet the target ratio, and add that amount of sample to the flask to +/- 0.01 g.

Greg Miiller, chair of D02.09, is presently balloting a clarification to the test method as follows:

CURRENT TEXT
10.7 Again, tare the balance and add the required mass of oil to the sample flask to make the sample-catalyst mixture total 8.5 g. The range for the mass of the oil to be added shall be ±0.01 g of the mass required. If more oil than required is added, make a new sample.

PROPOSED TEXT
10.7 Again, tare the balance and add the required mass of oil to the sample flask to make the sample-catalyst mixture total of 8.5 g ±0.05g. The range for the mass of the oil to be added shall be ±0.01 g of the mass required to obtain the catalyst/oil ratio shown on the catalyst bottle. If more oil than required is added, make a new sample.

If the ballot passes in Subcommittee 9 and the change is published in an updated test method, the TMC will then accept ONLY the clarified procedure as operationally valid. As mentioned, until that time, the TMC will accept either interpretation, although it is recommended to follow Ted Selby's advice on this matter.

I am well aware that the distinctions between the interpretations of Section 10.7 are subtle, and therefore potentially confusing. Please contact me if you have any questions.

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Attachment

c: Mike Lane
ftp://ftp.astmtmc.cmu.edu/docs/bench/mteos/memos/mem06-055.pdf

Distribution: Email

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To: Susan Milczewski, Chair, Section 7, TEOST Surveillance Panel, ASTM D02, SC B
Tom Schofield, TMC Liaison to Section 7, ASTM D02, SC B
Mike Habitz, Savant Lab Manager
Greg Miiller, Co-Chairman, Section G, ASTM D02, SC 9

Members of Team TEOST MHT:

The interchange of E-mails by all of you who are involved and knowledgeable about the TEOST MHT shows the need to clarify the technique required for the TEOST MHT ASTM D 7097 Test Method.

Considering this, I thought it would be helpful for all if I would review the background of the use and application of the catalyst as it was first applied and as it has been modified to a simpler technique for the last round robin producing ASTM Test Method D 7097. It probably would have forestalled some of the 'controversy' that Tom Schofield mentions in his E-mail of July 6.

We are all aware of the simplifying change in the TEOST MHT technique brought by the use of the sample flask which becomes part of the sample circulation system and makes weighing and introducing the sample so much easier (in contrast to the initial 'syringe' technique requiring the complex technique shown in the attached section and figure from the earlier test method written in 1999). Besides the awkwardness of the ‘syringe’ protocol, one of the significant difficulties of that method was limitation in the sensitivity with which the injected sample could be weighed since many balances could not handle either (or both) the size of the syringe or its weight combined with the sample. As a consequence, weights had to be based on the use of scales and the precision of the operating fluid weight of the sample injected was ±0.04g or ±0.5%. In contrast, the flask and its contents can be weighed at least to ±0.0010g. (Unfortunately, advantage of this greater precision was not addressed in the present method but can be in a future round robin if considered necessary.)

The catalyst and its concentration in the test fluid has always been very important to the precision of the testing an oil sample. Thus, the ratio between the catalyst and the oil is similarly important. In comparison, although it is important, the final weight of the test oil plus catalyst is less important than the catalyst/oil ratio.

What obviously needs to be clarified is the subject of the word 'required' in Section 10.7 of ASTM D 7097. I suggest that this part of the section be changed to read: "The range of the mass of the oil to be added to the catalyst shall be ±0.01g of the mass required *to obtain the proper catalyst/oil ratio shown on the catalyst bottle"* (italicized portion added).

I realize that this is a somewhat lengthy set of comments but, with hope, it will bring some further clarification of the formal ASTM Method D 7097. Perhaps this situation shows that despite all efforts to be clear in our test method development and description, there is always opportunity for improvement. It certainly shows that we must be very careful in the transition from one technique to another in a given method to prevent older imprecision to carry over to the newer version. This can best be done by clear definitions.

With all this said, if there are any comments, questions and – particularly – corrections, please let me and the other team members know.

Best regards,

Ted Selby,
Team TEOST MHT Member

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