

| MEMORANDUM: | 03-058 |
|-------------|--|
| DATE: | May 27, 2003 |
| TO: | Mr. Frank Gotto, Chair D02.B07 High Temperature Foam Surveillance Panel |
| FROM: | Tom Schofield |
| SUBJECT: | D6082 Round-Robin Results: Proposed Reference Oil TMC 66 Second Round-Robin |

A second D6082 High Temperature Foam round-robin matrix was completed by participating TMC monitored labs, under the auspices of the ASTM D02.B07 High Temperature Foam Surveillance Panel, on proposed TMC reference oil 66. Initial screener tests on oil 66 gave expectations of severe performance with respect to the current API SL passing limit of 100 ml Static Foam Tendency (Immediately Before Air Disconnect). The failing (severe) oil would be expected to compliment the current passing D6082 reference oil, TMC 1007, and replace TMC 1002 which was discontinued as a reference oil because of its inappropriately severe and imprecise performance. The round robin was expected to provide data to propose initial performance targets and acceptance bands on oil 66.

A first round-robin was conducted in 2002 and reported in my memo 02-069 of September 16, 2002. The results of that study showed unacceptably poor precision in the data. A teleconference "workshop" was completed on March 12, 2003, to try to improve operational conformance between the participants. A follow-up second round robin was then conducted on Oil 66.

The second round-robin test results for the proposed new D6082 reference oil, TMC 66, have been reported to the TMC by the participating laboratories. The matrix consisted of four TMC monitored laboratories each running a sample of TMC 66 in duplicate using the D6082 test method for a total of eight results. This is the same design as the first matrix except that the participants agreed that instead of running the duplicate runs out of the same sample at each lab concurrently, separate samples should be run two days or more apart.

The individual test results of the second round-robin are included in the attached table. Table 1 is a summary of the reported results:

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| Table 1 TMC Oil 66 D6082 Round-Robin 2 Summary | | | | | | | | | |
|---|----------------|---|-------|------|---|--|--|--|--|
| | | n | FTIB | FS1M | _ | | | | |
| | Max | | 380 | 0 | | | | | |
| | Avg | 8 | 226 | 0 | | | | | |
| | S _R | 8 | 94.41 | 0.00 | | | | | |
| | Sr | 8 | 62.75 | 0.00 | | | | | |
| | Min | | 140 | 0 | | | | | |

FTIB = Foam Tendency Immediately Before Air Disconnect, ml FS1M = Foam Stability 1 Minute After Air Disconnect, ml

For comparison, Table 2 is a summary of the first round-round robin:

| Table 2 TMC Oil 66 D60 <u>82 Round-Robin 1 Sum</u> mary | | | | | | | | | | |
|--|----------------|---|--------|------|--|--|--|--|--|--|
| | | n | FTIB | FS1M | | | | | | |
| | Max | | 400 | 0 | | | | | | |
| | Avg | 8 | 256 | 0 | | | | | | |
| | S _R | 8 | 107.03 | 0.00 | | | | | | |
| | Sr | 8 | 13.69 | 0.00 | | | | | | |
| | Min | | 120 | 0 | | | | | | |

FTIB = Foam Tendency Immediately Before Air Disconnect, ml FS1M = Foam Stability 1 Minute After Air Disconnect, ml

As with the first round-robin, the range of data in Table 1 is unreasonably broad and standard deviation of reproducibility (s_R ; between labs) is unreasonably poor. The standard deviation of repeatability (s_r ; within labs) also degraded compared to the first round-robin.

The TMC's opinion is that the second round-robin data is also too variable to be used for setting any realistic acceptance bands on TMC oil 66. Using a 95% confidence treatment of the results (mean +/- 1.960 s_R), the acceptance range for Foam Tendency Immediately Before Disconnect would be 41 to 411 ml. This would not provide a useful range to allow discrimination from oil 1007 (range 28 to 103), or to verify a lab's ability to discriminate between an API SL category passing and failing oil.

It might appear, from these two round-robins, and from the TMC reference data on severe oil 1002, that this test method may not be sufficiently precise on severe performing oils for the application of any meaningful statistical monitoring. In both Oil 66 round-robins, however, all labs could discriminate that oil 66 performed well above the current D4485 Oil Specification limit of 100 ml (static foam tendency FTIB). Perhaps oil 66 could be introduced purely as a discrimination oil for calibration monitoring purposes.

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TMS/tms

Attachment

 c: D02.B07 (D6082) High Temperature Foam Surveillance Panel D02.B07 (D6082) High Temperature Foam Mailing List D02.B07 (D6082) High Temperature Foam Participants Dr. John Zalar, TMC <u>ftp://www.astmtmc.cmu.edu/docs/bench/d6082/memo3-058.pdf</u>

Distribution: Email

| Comments | | | | | | | | IT IS UNUSUAL TO SEE 0 KINETIC FOAM. | | | | | | trument that was not TMC calibrated; lab declined to rerun on calibrated instrument) |
|------------|-------------------|-------------------|--------------------|--------------------|-------------------|-------------------|-----------------|---|--------|------|-----|-------|-------|--|
| | -65.3 (house air) | -65.3 (house air) | | | | | (Bottled) | IT IS U (Bottled)FOAM. | | | | | | lined to re |
| | -65.3 | -65.3 | 4600 Not Reported | 4950 Not Reported | 4870 Not Reported | 4375 Not Reported | Zero Air | Zero Air | | | | | | rated; lab dec |
| DIFFPER | 4263 | 4280 | 4600 | 4950 | 4870 | 4375 | 3886 | 3886 | | | | | | ot TMC calib |
| | 23 | 22 | 22 | 19 | 24 | 23 | 21 | 21 | | | | | | t that was no |
| | 22381 | 21811 | 23158 | 23711 | 22000 | 22000 | 23800 | 23800 | | | | | | n instrumen |
| | 740.4 | 737.4 | 747.1 | 738.6 | 0 Not Reported | 0 Not Reported | 742.0 | 747 | | | | | | samples on a |
| FS1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0.00 | 0.00 | an the |
| FTIBy i | 150-0.808 | 200-0.278 | 170-0.596 | 340 1.205 | 150-0.808 | 140-0.914 | 280 0.569 | 380 1.629 | Ot | 36 | 30 | .5 | 11 | the lab r |
| | 0 15 | | | | | | | | 140 | 226 | 380 | 62.75 | 94.41 | scause |
| | 47747 20030402 66 | 477482003040466 | G 4774520030421 66 | G 4774620030423 66 | 477492003050266 | 4775020030508 66 | 477522003042366 | A 4775120030408 66 | Min | Mean | Max | sr | sR | Lab D data excluded because the lab ran the samples on an inst |
| | B 477 | B 477 | G 477 | G 477 | I 477 | I 477 | A 477 | A 477 | | | | | | -ab D (|
| | | | | - | | | | | | | | | | |

D6082 Round-Robin 2 Test Results for Proposed Reference Oil TMC 66 (April-May 2003 Study)