1 December 2008

Committee D02 on Fuels and Lubricants
Subcommittee D02.14 on Particulate contamination

Research Report D02-1655

Intralaboratory Study to Establish repeatability statement for ASTM D7463, Standard Test Method for determining Adenosine Triphosphate (ATP) concentration in liquid fuels and fuel-associated water

Technical contact:
Ms. Charlotte Lindhardt,
Merck KGaA
Darmstadt,
Germany
+49 (0)6151 72 46 11
Charlotte.lindhardt@merck.de

ASTM International
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959

1. Introduction:

A data evaluation was conducted to establish an interim Repeatability Statement for D7463, Standard Test Method for determining Adenosine Triphosphate (ATP) concentration in liquid fuels and fuel-associated water.

2. Test Method:

The test method used for this study is ASTM D7463. To obtain a copy of D7463, go to ASTM's website, www.astm.org, or contact ASTM Customer Service by phone at 610-832-9585 (8:30 a.m. - 4:30 p.m. Eastern U.S. Standard Time, Monday through Friday) or by email at service@astm.org.

3. Participating Laboratories:

The following laboratory participated in this pilot study:

1. Merck KGaA
Frankfurterstrasse 250
D-64293 Darmstadt
Germany

Ms C Lindhardt
+49 (0)6151 72 4611
Charlotte.lindhardt@merck.de

4. Description of Samples:

The samples consisted of four (4) bulk samples with varying bioburden (microbial) loads. Each bulk sample was approximately two (2) gallons in volume and consisted of pooled samples of naturally (microbially) contaminated fuels (Marine diesel).

For the interim repeatability study, each bulk sample was mixed well by shaking prior to dispensing ten (10) aliquots of approx 500 ml (± 50 ml) into factory new plastic containers (32 oz volume) for analysis.

5. Study Instructions

The program instructions used for this Interim Repeatability Study were that of the Work Item 12289 for fuel samples with < 1% visible free water. For a copy of the Work Item 12289 refer to Section 2 of this report.

6. Description of Equipment/Apparatus[1]:

The equipment/apparatus used to collect the data was a HY-LiTE 2 luminometer which is manufactured by Merck KGaA, and other materials as described in the ASTM Work Item 12289.

[1] The equipment listed was used to develop a precision statement for D7463-08. This listing is not an endorsement or certification by ASTM International.

7. Data Report Forms:

A copy of the raw data is provided in Annex A.

8. Statistical Data Summary:

This was a single laboratory data collection in order to develop an interim Repeatability Statement for ASTM Work Item 12289. The data was submitted to CS94 statistician for review. The D2PP precision program version 5.1.13 was used for this analysis. A summary of the statistics calculated from the raw data returned by the participating laboratory are provided in Annex B.

9. Precision and Bias Statement:

Interim Repeatability—The difference between repetitive results, from non-diluted samples, obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

Repeatability (r) = 0.9243* (X - 16) RLU, where X = average of the two results

Based on the above the precision estimates examples of precision for the 4 samples are shown in Table 1:

Table 1:

Sample ID	S1	S2	S3	S4
Average	7244.4	687.8	2355.6	64.1
r	6681.4	620.9	2162.5	44.5

Bias—Since there is no accepted reference material suitable for determining the bias of the procedure in ASTM Work Item 12289, bias cannot be determined.