

This Research Report is issued under the fixed designation RR: D02-1655. You agree not to reproduce or circulate or quote, in whole or part, this document outside of ASTM International Committee/Society activities, or submit it to any other organization or standards body (whether national, international or other) except with the approval of the Chairman of the Committee having jurisdiction and the written authorization of the President of the Society. If you do not agree to these conditions, please immediately destroy all copies of this document. *Copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. All rights reserved.*

**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](http://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](mailto:service@astm.org).

**3. Participating Laboratories:**

The following laboratory participated in this pilot study:

<p>1. Merck KGaA Frankfurterstrasse 250 D-64293 Darmstadt Germany</p> <p>Ms C Lindhardt +49 (0)6151 72 4611 <a href="mailto:Charlotte.lindhardt@merck.de">Charlotte.lindhardt@merck.de</a></p>
--

**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 ( $\pm$  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<sup>1</sup>:**

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.

---

<sup>1</sup> 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:

<b>Sample ID</b>	<b>S1</b>	<b>S2</b>	<b>S3</b>	<b>S4</b>
<b>Average</b>	7244.4	687.8	2355.6	64.1
<b>r</b>	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.

*ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this research report. Users of this research report are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This research report is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this research report may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or serviceastm.org (e-mail); or through the ASTM website (www.astm.org).*