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# Committee D19 on Water Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water

**Research Report: D19-1187** 

Interlaboratory Study to Establish Precision Statements for ASTM D7597, Determination of Diisopropyl Methylphosphonate, Ethyl Hydrogen Dimethylamidophosphate, Ethyl Methylphosphonic Acid, Isopropyl Methylphosphonic Acid, Methylphosphonic Acid and Pinacolyl Methylphosphonic Acid in Water by Liquid Chromatography/Tandem Mass Spectrometry

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#### 1. Introduction:

An Interlaboratory Study was conducted to establish a precision statement for D7597, Determination of Diisopropyl Methylphosphonate, Ethyl Hydrogen Dimethylamidophosphate, Ethyl Methylphosphonic Acid, Isopropyl Methylphosphonic Acid, Methylphosphonic Acid and Pinacolyl Methylphosphonic Acid in Water by Liquid Chromatography/Tandem Mass Spectrometry. Accompanying this report, find the list of participating laboratories, description of samples, interlaboratory study instructions, description of equipment, the data reporting forms, a data summary, and the complete analytical method.

### 2. Test Method:

The Test Method used for this ILS is D7597, 2009. To obtain a copy of D7597, go to ASTM's website, <a href="www.astm.org">www.astm.org</a>, 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 <a href="mailto:service@astm.org">service@astm.org</a>.

# 3. Participating Laboratories:

The following laboratories participated in this interlaboratory study

USEPA Region 1
 Technology Drive
 Mail Code: EIA
 North Chelmsford, MA 01863-2431
 Peter Philbrook
 617-918-8602
 philbrook.peter@epa.gov

2. USEPA Region 6 Houston Branch 10625 Fallstone Road Mail Code: 6MDHS Houston, TX 77099 Mathew Johnson 281-983-2132 mathew.johnson@epa.gov

3. USEPA Office of Pesticide Programs (OPP – MS) Biological and Economic Analysis Division Building 1105 Mail Code: 7503P Stennis Space Center, MS 39529 Elizabeth Flynt 228-688-2410 flynt.elizabeth@epa.gov

4. USEPA Office of Pesticide Programs (OPP – Fort Meade)
USEPA Environmental Science Center
701 Mapes Road
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Fort Meade, MD 20755-5350
Paul Golden
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- Virginia Division of Consolidated Laboratories (VA DCLS)
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   Richmond, VA 23219
   Christopher Retarides
   488-4480
   Christopher.retarides@dgs.virginia.gov
- 6. Lawrence Livermore National Laboratories (LLNL) 7000 East Ave. L-091 Livermore, CA 94550 Janel Owens 925-422-6888 owens33@llnl.gov
- 7. USEPA Region 5, Chicage Regional Laboratory (CRL)
  77 West Jackson Blvd
  Mail Code: ML-10C
  Chicago, IL 60604-3507
  Larry Zintek
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## 4. Description of Samples:

There were 24 samples of varying targeted results used for this study. Each sample was prepared by the individual laboratory, with neat standards provided by EPA Region 5 CRL. Below is a list of the samples.

- 1. 4 Precision and accuracy samples at 4 concentrations (16 total P&A)
- 2. 2 Surface water samples at 4 concentrations (8 total surface water)

### From the Study Plan:

Once laboratories have established instrument calibration ranges, precision and bias will be determined across the calibration. Laboratories will analyze four replicates of reagent water at each concentration level and two replicates of a local surface water source, at each of the concentration levels used for the reagent water. Laboratories will determine bias (as percent recovery) and precision (as either the standard deviation or relative percent difference of results) at each level. Specific concentration levels to be used will be provided in study-specific instructions, prior to initiation of laboratory activities. Concentration levels will likely target:

- Lowest calibration standard
- Second lowest calibration standard
- Mid-point calibration standard
- Between the second highest and highest calibration standards

### 5. Interlaboratory Study Instructions

Laboratory participants were emailed the test program instructions. For a copy of the instructions, please see Annex A.