Determination of Organophosphate and Brominated Flame Retardants in House Dust and Consumer Products by Gas Chromatography – Tandem Mass Spectrometry

1. SCOPE AND APPLICABILITY
This SOP describes the procedures for the analysis of house dust and consumer products (foam, batting and/or textiles) for organophosphate flame retardants (OPFRs) and polybrominated diphenyl ethers (PBDEs) using Gas Chromatography Tandem Mass Spectrometry.

2. DEFINITIONS
- BFR – Brominated Flame Retardant
- ECL – Environmental Chemistry Laboratory
- GC-MS/MS – Gas Chromatography Tandem Mass Spectrometry
- LCS – Laboratory Control Sample
- LCSD – Laboratory Control Sample Duplicate
- LIMS – Laboratory Information Management System
- MB – Method Blank
- MRM – Multiple Reaction Monitoring
- NIST SRM – National Institute of Standards and Technology Standard Reference Material
- OPFR – Organophosphate Flame Retardant
- PBDE – Polybrominated Diphenyl Ether
- PFTBA – Perfluorotributylamine
- RM – In House Reference Material
- RPD – Relative Percent Difference
- SB – Solvent Blank
- SDS – Safety Data Sheet

3. PRINCIPLE
House dust samples prepared by SOP “Sample Preparation for the Analysis of Organophosphate and Polybrominated Diphenyl Ethers in House Dust Using Sonication” and consumer product samples prepared by SOP “Sample Preparation for the Analysis of Organophosphate and Brominated Flame Retardants in Consumer Products Using Sonication” are analyzed on an Agilent
7890 series gas chromatograph coupled to an Agilent 7000 series triple quadrupole mass spectrometer.

4. **INTERFERENCES**

Trace-level analysis requires careful and clean preparation and analysis technique along with high-purity standards and solutions. Solvents, reagents, glassware, and other items used during sample analysis may introduce unexpected interferences or contamination to the sample. These materials are demonstrated to be free from interferences and contamination by analyzing a method blank with each sample batch.

4.1. **Glassware Cleaning**

4.1.1. Disposable Glassware

Glassware in this classification includes new and unused GC vials, glass tubes and Pasteur pipettes. Glassware in this classification is disposed of after use.

**Disposable glassware should be cleaned prior to use** by wrapping in aluminum foil and baking in muffle furnace at approximately 500°C for 3 hours.

When removing glassware from supplier box, check that no cardboard or other debris is transferred to aluminum foil. If supplies have been exposed to dust or other debris, clean glassware using method listed below for reusable glassware.

4.1.2. Reusable Glassware

Reusable glassware must be properly washed after use to prevent interference in subsequent analysis due to sample carryover.

Recommended procedures for cleaning glassware are described in SOP 02.0011.00, “Procedure for Washing Glassware for the Analysis of PCDDs/PCDFs, PCBs, OCPs, PBDEs, BFRs and Other Persistent Organic Pollutants in Environmental Samples”.

4.2. **Interferences**

Dust samples can contain variable amounts of matrix that can affect sample analysis. Matrix interference may cause signal suppression, enhancement and/or distorted peak shape. In addition, dirty samples will degrade the inlet, GC column and contaminate the mass spectrometer source.

Foam, batting and/or textile samples can contain high levels of target analytes that can contaminate autosampler components, inlet, GC column and the mass spectrometer source. If a high level sample is observed, subsequent samples must be checked for carryover and the analytical setup must be confirmed with a solvent blank. In addition, foam, batting and/or textile samples may contain variable amounts of matrix that will degrade the inlet, GC column and contaminate the mass spectrometer source. If contamination is suspected, solvent blank injections maybe required subsequent to each suspect sample injections.
4.3. **Sample Analysis**

The autosampler uses the same GC syringe to inject both standards and samples. To prevent carryover between samples, the syringe is rinsed before and after each sample injection with hexane and toluene from solvent reservoirs. The reservoirs should be rinsed three times with hexane or toluene and re-filled with fresh solvent before each analytical run. In addition, solvent blank injections are included in the run list to confirm system cleanliness. If there is evidence of carryover, it is recommended that the solvent rinse solutions, GC syringe, inlet liner and septa be replaced.

Consumer Products: Sample dilutions maybe required if high concentrations of analytes are detected. Analytes measured at or above the highest calibration point will require dilution. Diluted samples maybe added to the sequence and are injected after a Solvent Blank (SB) and are followed by a check standard. See section 8.2.3 Sequence Monitoring Criteria.

5. **PRESERVATION AND HOLDING TIMES**

5.1. **Sample Extracts**

**Dust Samples:** Samples are processed for OPFRs and PBDEs according to ECL SOP “Sample Preparation for the Analysis of Organophosphate Flame Retardants and Polybrominated Diphenyl Ethers in House Dust Using Sonication”, DCN 05.0030.00.

**Consumer Products:** Samples are processed for OPFRs and BFRs according to ECL SOP “Sample Preparation for the Analysis of Organophosphate and Brominated Flame Retardants in Consumer Products Using Sonication”, DCN 05.0031.00.

Sample extracts shall be stored in sealed glass vials in the dark at $<$6°C when not in use. Septa on autosampler vials containing sample extracts should be replaced as soon as possible after puncture, and the vials returned promptly to dark storage at $<$6°C.

5.2. **Holding Times**

Due to the stability of these analytes, there is no holding time limit for dust or consumer product samples that are stored in the dark at $<$6°C. Foam, batting and textiles samples shall be stored in sealed glass containers or sealed plastic bags with the sample fully enclosed in aluminum foil at $<$6°C.

6. **EQUIPMENT AND SETUP**

For details on the recommended consumables and equipment, see Table 1.

6.1. **Description**

The instrument used for sample analysis is an Agilent 7890 series gas chromatograph coupled to an Agilent 7000 series triple quadrupole mass spectrometer (GC-MS/MS).
6.2. Operating Conditions

Sample data are acquired using the Agilent Technologies MassHunter GC/MS Acquisition program (Version B.07.00 or higher). Suggested settings for the GC and mass spectrometer are listed in Table 2. Samples are analyzed using electron ionization and multiple ion monitoring. A list of analytes, including example retention times, transitions and collision cell energies, can be found in Table 3.

6.3. Instrument Consumables

For details on the recommended instrument consumables, see Table 4.

7. STANDARDS AND REAGENTS

7.1. Solvents

For details on the recommended solvents, see Table 5.

7.2. Standards

7.2.1. Standards

For details on the recommended standards used (concentration, part number and supplier), see Table 6.

All standards shall be certified. When available, it is recommended to use certified analyte concentrations corrected for purity.

7.2.2. Calibration Standard

Example calibration standard concentrations are listed in Table 7. The calibration standard shall be prepared using certified standards and calibrated pipettors. Standard preparation details shall be recorded in the laboratory notebook.

7.2.3. Surrogate Standard, Laboratory Control Standard and Injection Standard

Information regarding standards, including suppliers, part numbers, and concentrations; are included in ECL SOP 05.0030.00: “Sample Preparation for the Analysis of Organophosphate Flame Retardants and Polybrominated Diphenyl Ethers in House Dust Using Sonication” for dust preparation, or ECL SOP 05.0031.00: “Sample Preparation for the Analysis of Organophosphate and Brominated Flame Retardants in Consumer Products Using Sonication” for consumer products.

8. METHOD PROCEDURE

8.1. Batch QC Requirements

**Dust:** The following Quality Control (QC) analyses must be performed for each sample batch: Method Blank (MB), Laboratory Control Sample (LCS), Standard Reference
Material (SRM), Standard Reference Material (SRM) Duplicate and Sample Duplicate: A sample batch is defined as up to 15 samples, including QC samples prepared together.

**Consumer Products**: The following Quality Control (QC) analyses must be performed for each sample batch: Method Blank (MB), Laboratory Control Sample (LCS), Laboratory Control Sample Duplicate (LCSD), Reference Material and Sample Duplicate. A sample batch is defined as up to 24 samples, including QC samples prepared together.

### 8.1.1. Method Blank

*The* MB is analyzed at a minimum frequency of one per sample preparation batch and is subjected to the entire analytical process. *The* MB is prepared as follows; 50 mg (±10 mg) of sodium sulfate (Dust) or 50 mg (±5 mg) analyte-free foam (Consumer Products) are weighed and spiked with the appropriate surrogate standard (see Dust or Consumer Products Sample Preparation SOP for composition and concentration).

The MB demonstrates that the analytical process itself does not introduce contamination and is within defined limits. Small amounts of analytes may be detected in blanks. If the amount measured is greater than the LOQ, the source of contamination should be located and corrected.

### 8.1.2. Laboratory Control Sample

Each LCS is processed with 50 mg (±10 mg) of sodium sulfate (Dust) or 50 mg (±5 mg) of analyte-free foam (Consumer Products) spiked with the appropriate LCS standard and surrogate standard and subjected to the entire analytical process (See Dust or Consumer Products Sample Preparation SOP for composition and concentration). The LCS is used to test inter-batch precision and to determine expected analyte recoveries. The percent recovery of the target analytes in the LCS standard must be within 50-150%.

**Consumer Products**: The LCS and LCSD must have a relative percent difference (RPD) of ≤30% for each target analyte.

### 8.1.3. Standard Reference Material (SRM)/Reference Material (RM)

**Dust**: The SRM and SRM duplicate are analyzed at a minimum frequency of one set per sample preparation batch and are subjected to the entire analytical process. The SRM is prepared as follows: 50 mg (±10 mg) of NIST SRM 2585 is weighed and spiked with Surrogate Standard. It is analyzed to test the method accuracy. NIST SRM 2585 provides certified values for select PBDEs and reference values for select OPFRs. Certified and reference values are shown in the NIST Certificate of Analysis (see reference 7). PBDE results must be within 70-130% of certified values. The SRM and SRM duplicate must have a relative percent difference (RPD) of ≤30% for each target analyte.

**Consumer Products**: The RM is analyzed at a minimum frequency of one per sample preparation batch and is subjected to the entire analytical process. The RM is prepared as follows: 50 mg (±5 mg) of RM foam is weighed
and spiked with Surrogate Standard. RM results must be within 70-130% of defined value for cryomilled RM and 50-150% for unmilled RM.

8.1.4. Sample Duplicate

**Dust:** A **duplicate sample** is analyzed at a minimum frequency of one per batch. The Sample Duplicate is prepared as follows; 50 mg (± 10 mg) of dust is weighed and spiked with the appropriate surrogate standard and subjected to the entire analytical process (see Dust Sample Preparation SOP for composition and concentration). A duplicate sample is used to determine the intra batch precision of the analysis. The Sample Duplicate must have a relative percent difference (RPD) of ≤30% for each target analyte. Failure to meet the acceptance criteria due to an unavoidable lack of sample homogeneity must be noted in the case narrative of the final report.

**Consumer Products:** A **duplicate sample** is analyzed at a minimum frequency of one per batch. The Sample Duplicate is prepared as follows; 50 mg (± 5 mg) of foam and or textile is weighed and spiked with the appropriate surrogate standard and subjected to the entire analytical process (see Consumer Products Sample Preparation SOP for composition and concentration). A duplicate sample is used to determine the intra batch precision of the analysis.

Cryomilled duplicate samples must have a relative percent difference (RPD) of ≤30% for each target analyte. Failure to meet the acceptance criteria, due to the inability of cryomilling to completely homogenize the sample must be noted in the case narrative of the final report.

Un-milled duplicate samples must have a relative percent difference (RPD) of ≤50%. Failure to meet the acceptance criteria due to an unavoidable lack of sample homogeneity must be noted in the case narrative of the final report.

8.2. Instrument QC Requirements

8.2.1. Tuning of Mass Spectrometer

The Agilent GC-MS/MS is auto-calibrated using PFTBA anytime instrument maintenance is performed (such as changing the column, source cleaning or any maintenance procedure requiring instrument venting and or power shutdown).

Before each analytical sequence, an instrument check tune is performed. If the system passes check tune, the sequence can be run. If check tune fails, auto-calibration should be performed again. Example check tune values are shown in Figure 1.

8.2.2. Calibration Curves

Calibration curves, consisting of seven standards with concentrations ranging from 25 to 1050 pg/µL, are generated by MassHunter Quantitative Analysis software (Version B.08.00 or higher) using the ratio of the peak area of the analyte to the assigned labeled standard plotted against concentration. MassHunter Workstation calculated calibration curve concentrations must be within 80-120% percent of the method defined values. Calibration curves must be continuous and have an R-
squared value equal to or greater than 0.990. A minimum of five points must be used to build the calibration curves and a minimum of five calibration standards are used to calculate the average qualifier response ratios for both native and labelled compounds. Qualifier/Quantifier relative response ratios must be within 80-120\% of the expected value for both native and labelled compounds that have an expected relative response ratio greater than 50. Qualifier/Quantifier relative response ratios must be within 70-130\% of the expected value for both native and labelled compounds that have an expected relative response ratio less than 50. Surrogate concentration are calculated by MassHunter Quantitative Analysis software using the average response factors of a minimum of five points. If the aforementioned conditions are not met, check instrument performance.

For consumer products, the range of reportable values is determined by the lowest and highest calibration curve concentrations. For dust, the range of reportable values is determined by the peak area of the lowest and highest calibration curve standards. Reportable values must be bracketed.

A new calibration curve is generated for each sample batch.

### 8.2.3. Sequence Monitoring Criteria

A calibration check standard (concentrations equivalent to calibration standard 4) is analyzed at the end of each batch of samples and is injected after one or more Solvent Blank (SB) injection. The calibration check standard is quantified with the calibration curve and must be within 70-130\% of the expected value. If the calibration check standard does not meet acceptance criteria it can be rerun one time.

### 8.3. Sample Analysis

#### 8.3.1. Data Acquisition

The sample data are acquired using the Agilent Technologies MassHunter GC/MS Acquisition software (Version B.07.00 or higher). A sequence table shall contain the following columns; Vial, Name, Data File, Method File, Method Path, Type, Dilution, Amount, Total Amount and Data Path. A sequence table is prepared with the following recommended sample order:

1) Solvent Blank  
2) Calibration Standards  
3) Solvent Blank  
4) Method Blank  
5) Laboratory Control Sample  
6) Laboratory Control Sample Duplicate (Consumer Products)  
7) SRM/RM Sample  
8) SRM Duplicate (Dust)  
9) Batch Samples  
10) Solvent Blank  
11) Calibration Check Standard
One or more solvent blank injections may be required subsequent to sample injections with high level of target analytes and/or matrix to confirm no carry over is observed. See section 4.2 Interferences.

8.3.2. Integration of Chromatograms

Chromatograms are processed using Agilent Technologies MassHunter Workstation software Quantitative Analysis for QqQ (Version B.08.00 or higher). The data are processed in batches which include the calibration curve standards, QC, samples and calibration check standard. Each analyte peak should be checked for correct integration, Qualifier/Quantifier peak ratio and retention time.

8.3.3. Data Processing

Once the integration check is complete, a summary report is generated for each batch. Analyte concentrations are calculated in ug/kg dust or mg/kg consumer product.

8.4. Data Reporting

8.4.1. Data Files

A log file is created for each run and is located in the data folder. After run completion, the log file shall be printed and placed into the GC sample log book. Additionally, the run information should be transferred to the electronic sample log file (Sample Counts.xlsx) stored on the GC-MS/MS computer.

Data files are stored on the GC-MS/MS computer by creating a subdirectory named according to the ECL Authorization Number and or other unique identifiers. The subdirectory shall contain all relevant data, including the sequence log, MRM data results, check tune report, and other relevant data. Data backups will be created according to ECL procedures.

8.4.2. Final Data Submission

Once all sample processing and analysis for the sample batch or project are complete, the analyst shall review and confirm the data are correct. Failed samples shall be re-extracted and analyzed, if sample material is available. Once data are confirmed by analyst, the batch or project data shall be peer reviewed. If suspect data are found, the peer review analyst will ask sample analyst for confirmation. Once the data are confirmed by peer review analyst, the data and or report shall be submitted to supervisor for final approval.
9. MAINTENANCE AND TROUBLESHOOTING

9.1. Agilent GC Triple Quadrupole Mass Spectrometer

9.1.1. Maintenance

Prior to sample analysis, rinse autosampler vials with hexane or toluene and fill to capacity or replace when deemed necessary. Run instrument check tune and verify criteria shown in Figure 1 are met.

**Weekly:** Recommend replacement of septa, glass inlet liner, and o-ring weekly or when deemed necessary. After replacement, check the system for leaks by running an Air and Water Check or by running an instrument check tune. If a leak is found, the inlet and or column connections should be checked.

**Six Months:** Foreline pump oil is recommended to be changed every 6 months. The tune solution vial volume should be checked and more added, if necessary.

**As needed:** Recommend replacement of the analytical column and source cleaning be performed when sample injections reach 1000 or when deemed necessary by analyst. Instrument consumables and parts (e.g. filaments, electron multipliers, etc.) and carrier gas traps should be replaced when needed. Sample syringe is recommended to be replaced when sample injections reach 500 or when deemed necessary by analyst.

The system is maintained by analyst, unless a system failure occurs (low sensitivity, electronic faults, etc.). In the case of system failures, it is recommended to contact an Agilent service technician. After maintenance requiring a system vent or power off, an auto-tune shall be performed to verify the analytical system is calibrated and operating within normal parameters.

9.1.2. Standards Check

Before using a new standard, it is recommended to verify that the new standard meets the method performance QC limits. A Reference Standard is prepared to check the Internal Standard and LCS Standard by spiking appropriate amounts of each into toluene and quantifying the new standard using the aforementioned data acquisition and quantitation methods.

9.1.3. Trouble Shooting


10. REFERENCES


2. ECL SOP 02.0011.00: “Procedure for Washing Glassware for the Analysis of PCDDs/PCDFs, PCBs, OCPs, PBDEs, BFRs and Other Persistent Organic Pollutants in Environmental Samples.”
3. ECL SOP 05.0030.00: “Sample Preparation for the Analysis of Organophosphate Flame Retardants and Polybrominated Diphenyl Ethers in House Dust Using Sonication.”

4. ECL SOP 05.0031.00: “Sample Preparation for the Analysis of Organophosphate and Brominated Flame Retardants in Consumer Products Using Sonication.”


### 11. TABLES

#### Table 1: Recommended Consumables and Supplies

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
<th>Supplier</th>
<th>Part/Model #</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Glassware</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GC vial 300 µL</td>
<td>300 µL amber vial with insert and writing patch</td>
<td>Wheaton¹</td>
<td>225328</td>
</tr>
<tr>
<td>GC vial 1.5 mL</td>
<td>Micro-V Vial Amber, 1.5mL, 12x32MM</td>
<td>VWR²</td>
<td>66064-914</td>
</tr>
<tr>
<td>Pasteur pipettes</td>
<td>Borosilicate glass, overall length 22.9 cm (9&quot;)</td>
<td>VWR</td>
<td>14673-043</td>
</tr>
<tr>
<td>Volumetric flask – 3 mL</td>
<td>Volumetric Cylindrical Flask, ±0.015 mL</td>
<td>VWR</td>
<td>14209-628</td>
</tr>
<tr>
<td>Volumetric flask – 5 mL</td>
<td>Volumetric Cylindrical Flask, ±0.02 mL</td>
<td>VWR</td>
<td>14204-538</td>
</tr>
<tr>
<td>Volumetric flask – 10 mL</td>
<td>Volumetric Cylindrical Flask, ±0.02 mL</td>
<td>VWR</td>
<td>14203-258</td>
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<tr>
<td><strong>Plastics</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pipette bulbs</td>
<td>Small-volume, natural rubber latex, 2 mL</td>
<td>VWR</td>
<td>82024-554</td>
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<tr>
<td>20 µL pipette tips</td>
<td>ART Barrier, Low Retention 20 µL Barrier (2749-05-HR)</td>
<td>VWR</td>
<td>89031-352</td>
</tr>
<tr>
<td>200 µL pipet tips</td>
<td>ART Barrier, Low Retention 200 µL Barrier (2769-05-HR)</td>
<td>VWR</td>
<td>89031-374</td>
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<tr>
<td>300 µL pipet tips</td>
<td>ART Barrier, Low Retention 300 µL Barrier (2739-05-HR)</td>
<td>VWR</td>
<td>89031-394</td>
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<td>1000 µL pipet tips</td>
<td>LTS 1 ml Tips, RT-LTS-A-L1000µL-1F</td>
<td>Mettler-Toledo³</td>
<td>30389212</td>
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<tr>
<td>5000 µL pipet tips</td>
<td>LTS 5 mL Tips, RT-LTS-A-5000µL-192/8</td>
<td>Mettler-Toledo³</td>
<td>30389256</td>
</tr>
<tr>
<td>GC vial caps</td>
<td>ABC cap with PTFE/ Silicone Septum</td>
<td>Wheaton</td>
<td>W225332-0204</td>
</tr>
<tr>
<td><strong>Other</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>20 µL pipette</td>
<td>Electronic Pipette, LTS E4-20XLS+ 2-20 µL</td>
<td>Mettler-Toledo</td>
<td>17014487</td>
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<tr>
<td>100 µL pipette</td>
<td>Electronic Pipette, LTS E4-100XLS+ 10-100 µL</td>
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<td>300 µL pipette</td>
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<td>5 mL pipette</td>
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<td>17012312</td>
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¹wheaton.com, ²us.vwr.com, ³us.mt.com
### Table 2: Recommended Instrumentation and Analytical Conditions for GC-MS/MS

<table>
<thead>
<tr>
<th>GC</th>
<th>Agilent 7890A or 7890B Series</th>
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</thead>
<tbody>
<tr>
<td>Autosampler</td>
<td>Agilent 7000 Series</td>
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<tr>
<td>Analytical column</td>
<td>DB-5ms 30 m x 0.25 mm I.D. x 0.25µm film thickness (P/N 122-5532UI)</td>
</tr>
<tr>
<td>Initial column flow rate</td>
<td>1.5 mL/min (constant flow)</td>
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<tr>
<td>Carrier gas</td>
<td>Helium</td>
</tr>
<tr>
<td>Oven temperature program</td>
<td>90°C (1 min), 15°C/min to 200°C (3 min), 5°C/min to 250°C (0 min), 15°C/min to 300°C (8 min)</td>
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<tr>
<td>Run time</td>
<td>32.667 min</td>
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**Front Inlet Split/Splitless Parameters**

<table>
<thead>
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<th>Mode</th>
<th>Pulsed Splitless</th>
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<tr>
<td>Injection volume</td>
<td>1.5 µL</td>
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<tr>
<td>Inlet liner</td>
<td>Agilent Ultra Inert Liner, splitless, single taper, glass wool (P/N 5190-3163)</td>
</tr>
<tr>
<td>Inlet temperature</td>
<td>250°C</td>
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<tr>
<td>Inlet pressure</td>
<td>15.494 psi</td>
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<tr>
<td>Septum purge flow</td>
<td>3 mL/min</td>
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<tr>
<td>Gas saver</td>
<td>20 mL/min after 3 min</td>
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<tr>
<td>Injection pulse pressure</td>
<td>20 psi until 1 min</td>
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<tr>
<td>Purge flow to split vent</td>
<td>30 mL/min at 1 min</td>
</tr>
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**Triple Quadrupole Mass Parameters**

<table>
<thead>
<tr>
<th>Spectrometer</th>
<th>Agilent 7000 Series</th>
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<tbody>
<tr>
<td>Source</td>
<td>Electron Impact Extractor (EIEX)</td>
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<td>Electron Energy (eV)</td>
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<tr>
<td>Tune file</td>
<td>atune_250.eiex.tune.xml</td>
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<tr>
<td>Transfer line temperature</td>
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<tr>
<td>Solvent delay</td>
<td>5 min</td>
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<tr>
<td>Source temperature</td>
<td>250°C</td>
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<tr>
<td>Quadrupole temperature</td>
<td>Q1 and Q2 = 150°C</td>
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<tr>
<td>Gain factor</td>
<td>50</td>
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**MRM Mode Conditions**

<table>
<thead>
<tr>
<th>MS1 resolution</th>
<th>1.2 amu</th>
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</thead>
<tbody>
<tr>
<td>MS2 resolution</td>
<td>1.2 amu</td>
</tr>
<tr>
<td>Dwell times</td>
<td>Variable from 40 to 150 ms</td>
</tr>
<tr>
<td>Collision gas flow</td>
<td>Nitrogen at 1.5 mL/min, Helium at 2.25 mL/min</td>
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**Software**

<table>
<thead>
<tr>
<th>Data acquisition</th>
<th>Agilent MassHunter Data Acquisition Software (Ver. B.07.00. or higher)</th>
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<tbody>
<tr>
<td>Qualitative analysis</td>
<td>MassHunter Workstation Software for Qualitative Analysis (Ver. B.06.00 or higher)</td>
</tr>
<tr>
<td>Quantitative analysis</td>
<td>MassHunter Workstation Software for Quantitative Analysis (Ver. B.08.00 or higher)</td>
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<tr>
<td>Compound Abbreviation</td>
<td>Compound Name</td>
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<tr>
<td>-----------------------</td>
<td>----------------------------------------</td>
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<tr>
<td>dTPP</td>
<td>Tri-n-propyl phosphate-d21</td>
</tr>
<tr>
<td>TPP</td>
<td>Tri-n-propyl phosphate</td>
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<tr>
<td>dTNBP</td>
<td>Tri-n-butyl phosphate-d21</td>
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<tr>
<td>TNBP</td>
<td>Tri-n-butyl phosphate</td>
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<tr>
<td>dTCEP</td>
<td>Tris(2-chloroethyl) phosphate-d21</td>
</tr>
<tr>
<td>TCEP</td>
<td>Tris(2-chloroethyl) phosphate</td>
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<tr>
<td>dTCIPP</td>
<td>Tris(2-chloroisopropyl) phosphate-d24</td>
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<tr>
<td>TCIPP</td>
<td>Tris(1-chloro-2-propyl) phosphate</td>
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<tr>
<td>BDE-17</td>
<td>2,2',4-Tribromodiphenyl ether</td>
</tr>
<tr>
<td>BDE-28</td>
<td>2,4,4'-Tribromodiphenyl ether</td>
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Table 4: Recommended GC-MS/MS Consumables and Supplies

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⁵airgas.com, ⁶agilent.com

Table 5: Recommended Solvents

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⁷avantormaterials.com
### Table 6: Recommended Native and Labelled Standards

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*well-labs.com, isotope.com, accustandard.com*
Table 7: **Recommended** Calibration Standard Concentrations

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*EH-TBB, BEH-TEBP and corresponding labelled standards are not included in dust analysis.
12. FIGURES

Figure 1. Example GC-MS/MS System Check Tune

Triple Quadrupole GC/MS System Verification - Tune

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**Instrument Actuals**

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<th>Ionization mode</th>
<th>Rough Vacuum</th>
<th>Value (mTorr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>El+</td>
<td>High Vacuum</td>
<td>1.47E+2</td>
</tr>
<tr>
<td>Source Temperature</td>
<td>251 °C</td>
<td>7.43E-5</td>
</tr>
<tr>
<td>Quad. 1 Temperature</td>
<td>150 °C</td>
<td>100.0 %</td>
</tr>
<tr>
<td>Quad. 2 Temperature</td>
<td>150 °C</td>
<td>21.538 W</td>
</tr>
<tr>
<td>Emission Current</td>
<td>35 uA</td>
<td></td>
</tr>
</tbody>
</table>

**MS1 Checktune Results**

| Low mass assignment (target 69.00, actual 69.00) | 0.00 | <= 0.20 | OK     |
| Mid mass assignment (target 264.00, actual 264.00) | 0.00 | <= 0.20 | OK     |
| High mass assignment (target 502.00, actual 502.00) | 0.00 | <= 0.20 | OK     |
| Low mass isotope position (target 70.00, actual 70.00) | 0.00 | <= 0.20 | OK     |
| Mid mass isotope position (target 365.00, actual 365.00) | 0.00 | <= 0.20 | OK     |
| High mass isotope position (target 503.00, actual 503.00) | 0.00 | <= 0.20 | OK     |
| Low mass isotope ratio | 1.10% | >= 0.5% and <= 1.6% | OK |
| Mid mass isotope ratio | 5.72% | >= 4.2% and <= 5.9% | OK |
| High mass isotope ratio | 9.99% | >= 7.5% and <= 12.3% | OK |
| Ratio of mid mass to low mass | 18.65% | >= 5.0% | OK |
| Ratio of high mass to low mass | 5.52% | >= 0.8% | OK |
| Low mass precursor ratio | 0.08% | <= 3.00% | OK |
| Mid mass precursor ratio | 0.31% | <= 6.00% | OK |
| High mass precursor ratio | 0.17% | <= 12.00% | OK |

**MS2 Checktune Results**

| Low mass assignment (target 69.00, actual 69.00) | 0.00 | <= 0.20 | OK     |
| Mid mass assignment (target 264.00, actual 264.00) | 0.00 | <= 0.20 | OK     |
| High mass assignment (target 502.00, actual 502.00) | 0.00 | <= 0.20 | OK     |
| Low mass isotope position (target 70.00, actual 70.00) | 0.00 | <= 0.20 | OK     |
| Mid mass isotope position (target 365.00, actual 365.00) | 0.00 | <= 0.20 | OK     |
| High mass isotope position (target 503.00, actual 503.00) | 0.00 | <= 0.20 | OK     |
| Low mass isotope ratio | 1.08% | >= 0.5% and <= 1.6% | OK |
| Mid mass isotope ratio | 5.67% | >= 4.2% and <= 6.9% | OK |
| High mass isotope ratio | 10.03% | >= 7.5% and <= 12.3% | OK |
| Low mass precursor ratio | 0.45% | <= 3.00% | OK |
| Mid mass precursor ratio | 0.03% | <= 6.00% | OK |
| High mass precursor ratio | 0.10% | <= 12.00% | OK |

**Detector**

<table>
<thead>
<tr>
<th>ENV</th>
<th>Maximum gain factor</th>
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</thead>
<tbody>
<tr>
<td>1652</td>
<td>&lt; = 2900 OK</td>
</tr>
<tr>
<td>1059</td>
<td>&gt; = 100 OK</td>
</tr>
</tbody>
</table>

**Air and Water Check**

<table>
<thead>
<tr>
<th></th>
<th>Abundance</th>
<th>Relative Abundance</th>
<th>Limit</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>PFIDA (69.00)</td>
<td>1533003</td>
<td>1.57%</td>
<td>&lt;= 20.00%</td>
<td>OK</td>
</tr>
<tr>
<td>Water</td>
<td>24007</td>
<td>0.29%</td>
<td>&lt;= 2.50%</td>
<td>OK</td>
</tr>
<tr>
<td>Oxygen</td>
<td>4399</td>
<td>1.07%</td>
<td>&lt;= 10.00%</td>
<td>OK</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>16393</td>
<td>0.29%</td>
<td>&lt;= 2.50%</td>
<td>OK</td>
</tr>
</tbody>
</table>

* Nitrogen values are calculated from oxygen abundance
13. REVIEW

<table>
<thead>
<tr>
<th>Signatures</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>___________________</td>
<td></td>
</tr>
<tr>
<td>___________________</td>
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<tr>
<td>___________________</td>
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</tbody>
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