

STATEMENT OF WORK

Analysis of Wastewater, Sludge/Biosolids, Leachate and other Complex Environmental Matrices for diethylhexyl phthalate (DEHP, CAS #117-81-7) and other priority phthalates

1.0 Background

The *Canadian Environmental Protection Act* (CEPA 1999) has the purpose of protecting the environment and the health and well-being of Canadians. A major part of the Act is to prevent pollution and address the exposure and potential effects of chemical substances (www.canada.ca). Scientific research and environmental monitoring provide the foundation for decision-making under CEPA.

Effluent discharges and land application of biosolids from Canadian wastewater treatment plants (WWTPs), and leachates from landfill sites, have been identified as important pathways of chemical substances to the aquatic and terrestrial environments. Environment and Climate Change Canada (ECCC) conducts monitoring programs to determine the occurrence and fate of these substances during treatment processes, and levels of these substances in environmental compartments. These monitoring programs supports the Chemicals Management Plan, the Whales Initiative, and other collaborations with federal, provincial, municipal, Indigenous, and academic partners to address issues related to chemical substances and the environment.

Diethylhexyl phthalate (DEHP, CAS #117-81-7) was concluded to be toxic under section 64(a) of CEPA (Canada 2020a). The proposed risk management action is a prohibition with some exceptions (Canada 2020b). In order to establish a baseline of concentrations, the monitoring program requires high-quality chemical analysis of DEHP that may be present at trace levels in environmental compartments such as, but not limited to, wastewater raw influent, treated effluent, environmental water, landfill leachate, raw sludge, treated biosolids, sediments, and biological tissues of aquatic and terrestrial non-human organisms. The results of this program contribute to the management of DEHP in Canada.

Other phthalates were also assessed in the Phthalates Substance Grouping (Canada 2020b). Some of these are associated with human health or ecological effects of concern; therefore, monitoring data on concentrations in different environmental compartments will also be of interest to ECCC.

2.0 Objective

Complete high-quality chemical analysis of DEHP (higher priority) and other phthalates (lower priority) in wastewater raw influent, treated effluent, raw sludge, treated biosolids, landfill leachate, environmental waters, sediments, and biological tissues of aquatic and terrestrial non-human organisms impacted by these discharges as part of monitoring programs on the occurrence and fate of chemical substances in the environment.

3.0 Definitions

CAS #	Chemical Abstract Service number (www.cas.org)
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Method Detection Limit (MDL)	The minimum measured concentration of a substances that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results (EPA 821-R-16-006 Dec 2016, e-CFR title 40 Part 136 Appendix B). https://www.ecfr.gov/current/title-40/chapter-I/subchapter-D/part-136#Appendix-B-to-Part-136
Sample-specific Detection Limit (SDL)	3 times the signal to noise ratio in the target channel converted to an equivalent sample concentration, or the concentration equivalent to the lowest calibration standard, whichever is greater.
Quarterly	Canada defines the quarterly periods as follows: 1 st Quarter 1 April to 30 June 2 nd Quarter 1 July to 30 September 3 rd Quarter 1 October to 31 December 4 th Quarter 1 January to 31 March

4.0 Scope of Work

- 4.1 Using validated analytical methods as per Canadian Association for Laboratory Accreditation (P07:2017 – CALA Application of Requirements in ISO/IEC 17025:2017, Revision 1.5, Sept. 30, 2020), the Contractor must conduct high-quality chemical analysis of DEHP and other phthalates in wastewater raw influent, treated effluent, raw sludge, treated biosolids, landfill leachate, environmental waters, sediments, and biological tissues of aquatic non-human organisms as part of ECCC monitoring programs on the occurrence and fate of chemical substances in the environment.
- 4.2 The Contractor must be able to begin receiving and analyzing samples from date of Contract Award.

5.0 Tasks

- 5.1 Sample Load
Up to 100 samples per year per environmental compartment.
- 5.2 Sampling Plan
The Technical Authority will provide a quarterly sampling plan to the Contractor within two (2) weeks of Contract Award; this plan will be update on a quarterly basis.
- 5.3 Sampling Protocol
The Contractor must provide sampling protocols for collection of aqueous, solids, and tissue samples. The sampling protocols must specify the type of container to use for collection of samples, the required sample volume or mass to achieve the Reporting Limits (RLs) stipulated in Table 1, and any preservation requirements to maintain sample integrity during collection and transit.
- 5.4 Submission Forms
The Contractor must provide submission form templates for collection of samples. Submission forms will include fields for Project Name; Client name, address and phone; Client Sample Identification; Matrix, Sampling Date; Container Type; Analyses Requested; Relinquished by with Date; Received by with Date.

5.5 Sample Transport

The Technical Authority will deliver samples to the Contractor by overnight courier. Temperature blanks will be included in each cooler so the Contractor can confirm sample temperature upon receipt.

5.6 Analyses

- 5.6.1 Wastewater influents and effluents and landfill leachates are challenging matrices because of the elevated levels of suspended solids and organic content compared to typical environmental waters. Treated wastewater effluents can contain suspended solids up to 60 mg/L depending on the treatment type. Raw wastewater influents can contain suspended solids up to 200 mg/L. These solids are an integral part of the sample because they may contain significant levels of the compounds of interest, particularly if the compounds are hydrophobic. Therefore, sample preparation and extraction methods that are able to accommodate the solids (e.g. liquid/liquid extraction) are preferred where possible. However if the solids must be removed by filtration prior to extraction (e.g. solid phase extraction) it is unlikely that separate analysis of the solids is feasible due to the amount of material and the cost of the additional analysis. All results from wastewater influent and effluent samples, landfill leachates, and other aqueous matrices must be reported on a mass/volume basis (e.g. ng/L or $\mu\text{g/L}$).
- 5.6.2 Raw sludge and treated biosolids are challenging matrices because of the high moisture and organic content compared to typical sediment samples. These samples can contain anywhere from 2% to 30% solids, and 50% to 75% organic material. The solids are the important phase of these samples; therefore if phase separation is required for sample preparation and extraction the Contractor must analyze the solid phase. However sample preparation and extraction methods that avoid the need for phase separation are preferred where possible. All results from raw sludge, treated biosolids, and sediments must be reported on a mass/mass and dry weight basis (e.g. ng/g dw or $\mu\text{g/g dw}$).
- 5.6.3 Tissue samples can contain high levels of lipid. All results from tissues must be reported on a mass/mass and wet weight basis (e.g. ng/g ww or $\mu\text{g/g ww}$). The Contractor must also report the percentage of lipid in the sample by volume.
- 5.6.4 The Contractor must use analytical methods that reflect the current state of analytical technology, i.e. mass discrimination techniques to maximize analyte identification and quantification. The Contractor must provide a copy of their complete analytical method, including all quality assurance and quality control elements such as acceptable ranges for blank levels, laboratory spike recoveries, surrogate recoveries, and duplicate sample results.
- 5.6.5 As described in the Risk Management Approach (Canada 2020b) the predicted no-effect concentration (PNEC) of DEHP for aquatic organisms in water is 0.07 $\mu\text{g/L}$. Analytical methods that can achieve RLs equal to or lower than this value in surface water and wastewater effluents will be preferred. Methods must employ, at a minimum, the labeled surrogate standards listed in Table 1, or equivalent, for analyte quantification.



- 5.6.6 The Contractor must communicate any anomalous situations with respect to sample integrity or analytical challenges to the Technical Authority by email within three (3) business days of discovering such situation.

5.7 Storage and Disposal

The Contractor must adhere to the maximum sample holding time and storage conditions as specified in the analytical method.

5.8 Quality Assurance / Quality Control (QA/QC)

- 5.8.1 The Contractor must analyze samples in a batch system, with each batch consisting of a method blank, spiked blank, and replicate sample. These QA/QC elements must comprise 5% or more of each analytical batch, i.e. every batch of 20 samples or fewer must contain a blank, spike, and replicate. Blank corrections or blank subtractions must not be used. Results of method blanks shall be quantified and reported.
- 5.8.2 The Contractor must consider field duplicates, field blanks, and equipment blanks submitted by ECCC as samples. Method blanks, spiked blanks, and laboratory replicate analyses must be conducted as part of the Contractor's Quality Assurance/Quality Control (QA/QC) program and are not billed as samples submitted.
- 5.8.3 Laboratory raw data, chromatograms, and all relevant laboratory notes must be retained by the Contractor for a minimum period of 36 months following submission of samples. Raw data must include chromatograms and area tables for all instrument calibrations including linearity, resolution, and sensitivity checks showing date and time of analysis, and evidence that all QA/QC specifications have been met; and aliquot masses, volumes, suspended solids content and moisture content for all samples, including original and re-analyses, dilutions, and other details of the analytical procedure.
- 5.8.4 The Contractor must provide consultation on sampling procedures, delivery schedules, unexpected analytical results, and other contingencies as requested by the Technical Authority.

5.9 Reports

- 5.9.1 The Contractor must electronically provide sample submittal confirmation to the Technical Authority within five (5) business days of sample receipt.
- 5.9.2 Sample Data Reports
 - 5.9.2.1 The Contractor must deliver Sample Data Reports to the Technical Authority within eight (8) weeks of receiving the samples. Sample Data Reports must include the following:
 - 5.9.2.1.1 Concentrations of each analyte in the samples and replicates.
 - 5.9.2.1.2 Concentrations of each analyte in the method blank.
 - 5.9.2.1.3 Per cent recoveries in spiked blanks.
 - 5.9.2.1.4 The reporting limit for each analyte.
 - 5.9.2.1.5 Percent recovery of surrogates.
 - 5.9.2.1.6 Any problems with samples or data, including corrective actions taken, resolutions, and explanation of flagged data.
 - 5.9.2.2 Sample Data Reports are subject to the acceptance and approval of the Technical Authority.
- 5.9.3 Final Data Report
 - 5.9.3.1 The Contractor must deliver a final report to the Technical Authority. Final Data Report must include the following.

- 5.9.3.1.1 The project name.
- 5.9.3.1.2 Sample site name.
- 5.9.3.1.3 Date of sample receipt.
- 5.9.3.1.4 Sample temperatures upon receipt.
- 5.9.3.1.5 Reporting conventions and laboratory qualifiers.
- 5.9.3.1.6 QA/QC notes.
- 5.9.3.1.7 Analytical discussion.
- 5.9.3.1.8 Correlation table showing client and Contractor sample identifiers, and analysis reports for each sample and substance.

5.9.4 Annual Project Meeting

The Contractor must attend a yearly project meeting with ECCC representatives. The meeting must take place within one month of commencement of sample collection.

Notes to Table:

- A. The Contractor is invited to offer additional analytes as long as the cost of the analysis does not increase because of the additions.

Table 1: Reporting limits for DEHP and other phthalates in environmental matrices

Name*	Required reporting limit wastewater influent, landfill leachate (ng/L)	Required reporting limit wastewater effluent, surface water (ng/L)	Required reporting limit sludge, biosolids (ng/g dry weight)	Required reporting limit sediment (ng/g dry weight)	Required reporting limit tissues (ng/g wet weight)	Labeled surrogate for quantification
79P	100	10	100	10	50	
B79P	100	10	100	10	50	
B84P	100	10	100	10	50	
BBP	100	10	100	10	50	
BCHP	100	10	100	10	50	
BIOP	100	10	100	10	50	
C9-rich	100	10	100	10	50	
CHIBP	100	10	100	10	50	
DBP	100	10	100	10	50	D ₄ -DBP
DBzP	100	10	100	10	50	D ₄ -DBzP
DCHP	100	10	100	10	50	D ₄ -DCHP
DEHP (highest priority)	100	10	100	10	50	
DEP	100	10	100	10	50	D ₄ -DEP
DIBP	100	10	100	10	50	
DIHepP	100	10	100	10	50	
DINP	100	10	100	10	50	
DIOP	100	10	100	10	50	
DMCHP	100	10	100	10	50	
DMP	100	10	100	10	50	
DnHP	100	10	100	10	50	



DnOP	100	10	100	10	50	
DPrP	100	10	100	10	50	D ₄ -DPrP

*see Canada 2020b, Annex A for full chemical names

6.0 Deliverables

Deliverable	Due Date
6.1 Sampling Protocol	Within two weeks of Contract Award
6.2 Submission Forms	Within two weeks of Contract Award
6.3 Yearly project meeting	Within one month of commencement of sample collection (normally May)
6.4 Sample Data Reports	Within six (6) weeks of sample receipt
6.5 Final Data Reports	Within four (4) weeks of acceptance of the Sample Data Report by the Technical Authority

7.0 Format of Deliverables:

7.1 Sample Data Reports

- 7.1.1 The Contractor must deliver the Sample Data Reports in Microsoft Excel .xlsx spreadsheet format, or equivalent compatible format electronically to the Technical Authority.
- 7.1.2 The Sample Data Reports must be separated by sampling site, e.g. WWTP.

7.2 Final Data Report

- 7.2.1 The Contractor must deliver the Final Data Report in PDF format including a cover letter signed by the analyst electronically to the Technical Authority.

8.0 Government Supplied Material

All sampling activities, equipment, supplies, and shipping will be provided by ECCC.

ECCC will generate trip blanks, field blanks, and equipment blanks as part of this Contract, which will be submitted and invoiced as samples.

9.0 Language of Work

All written and verbal communication must be in English or French.

10.0 Work Location

The work will take place at the Contractor's facilities.

11.0 Travel

Travel is not required to perform the Work.

12.0 Sustainable Procurement Considerations

The Contractor should make an effort to ensure that their operations and performance of the Work align with the Treasury Board [Policy on Green Procurement](#) and [Greening Government](#)

[Strategy](#). Procurement documents will specify the green procurement criteria and standards to be met and provide guidelines for the evaluation of proposals with respect to those criteria and standards.

The following green procurement criteria and standards must form part of the Work:

- Provide all correspondence and deliverables including (but not limited to) documents, reports and invoices in electronic format.

13.0 Accessibility Considerations

The Government of Canada strives to ensure that the goods and services it procures are inclusive by design and accessible by default, in accordance with the [Accessible Canada Act](#), its associated regulations and standards, and Treasury Board Contracting Policy. Procurement documents will specify the accessibility criteria and standards to be met and provide guidelines for the evaluation of proposals with respect to those criteria and standards.

The following accessibility criteria and standards must form part of the Work:

- All written reports must be created in a format that is screen reader and adaptive technology friendly.

14.0 References

Canada. 2020a. Dept. of the Environment and Climate Change, Dept. of Health. Screening assessment for the phthalate substance grouping.

Canada. 2020b. Dept. of the Environment and Climate Change, Dept. of Health. Risk management approach for 1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester (DEHP). CAS RN 117-81-7.