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Multi-Media, Multi-Concentration, Organic Analytical Service for Superfund (OLM04.3)

Office of Superfund Remediation and Technology Innovations
Analytical Operations/Data Quality Center (5204G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites, are a series of volatile, semivolatile, and pesticide/Aroclor (pesticide/PCB) compounds that are analyzed using Gas Chromatography coupled with Mass Spectrometry (GC/MS) and Gas Chromatography with Electron Capture (GC/EC). The Analytical Operations/Data Quality Center (AOC) of the Office of Superfund Remediation and Technology Innovations (OSRTI) offers an analytical service that provides data from the analysis of water and soil/sediment samples for organic compounds for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the organic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

The new organic analytical service is available as of March 2003. It provides a technical and contractual frame work for laboratories to apply EPA/CLP analytical methods for the isolation, detection, and quantitative measurement of 48 volatile, 65 semivolatile, and 28 pesticide/Aroclor target compounds in water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including Quality Assurance (QA), Quality Control (QC), and Standard Operating Procedures (SOPs), by which EPA evaluates the data. This service uses GC/MS and GC/EC methods to analyze the target compounds. Three data delivery turnarounds are available to CLP customers: 7, 14, and 21-day turnaround after laboratory receipt of the last sample in the set. In addition, there are 48 (for volatiles) and 72-hour (or semivolatiles and pesticides/Aroclors) preliminary data submission options available. The data associated with these Preliminary Results is due within the specified time after receipt of each sample at the laboratory. Options under this service include a closed system purge-and-trap method for low-level volatile soil analysis and methanol preservation for medium-level volatile soil analysis. In addition, data users may request modifications to the SOW that may include, but are not limited to, additional compounds, modified quantitation limits (e.g., lower volatile limits), additional cleanup options, and other requirements to enhance method performance.

DATA USES

This analytical service provides data which EPA uses for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of a hazardous waste site including site inspections, Hazard Ranking System scoring, remedial investigations/feasibility studies, remedial design, treatability studies, and removal actions. In addition, this service provides data that will be available for use in Superfund enforcement/litigation activities.

TARGET COMPOUNDS

The compounds for which this service is applicable and the corresponding quantitation limits are listed in **Table 1**. For water samples, the lowest reportable quantitation limits are 10 ppb for the volatile compounds, 10 ppb for the semivolatile compounds, and 0.05 ppb for the pesticide/Aroclor compounds. For soil samples, the lowest reportable quantitation limits are 10 ppb for the volatile compounds, 330 ppb for the semivolatile compounds, and 1.7 ppb for the pesticide/Aroclor compounds. Specific sample quantitation limits will be highly matrix-dependent. Compounds identified with concentrations below the quantitation limit will be reported as estimated concentration values.

Table 1. Target Compound List and Contract Required Quantitation Limits (CRQLs) For OLM04.3*

Quantitation Limits				Quantitation Limits			Quantitation Limits		
	Water (µg/L)	Low Soil (µg/Kg)	Modified Cal. [1] Levels (µg/L)		Water (µg/L)	Low Soil (µg/Kg)		Water (µg/L)	Low Soil (µg/Kg)
VOLATILES				**SEMIVOLATILES**			98. Carbazole	10	330
1. Dichlorodifluoromethane	10	10	0.50	49. Benzaldehyde	10	330	99. Di-n-butylphthalate	10	330
2. Chloromethane	10	10	0.50	50. Phenol	10	330	100. Fluoranthene	10	330
3. Vinyl Chloride	10	10	0.50	51. bis-(2-Chloroethyl)ether	10	330	101. Pyrene	10	330
4. Bromomethane	10	10	0.50	52. 2-Chlorophenol	10	330	102. Butylbenzylphthalate	10	330
5. Chloroethane	10	10	0.50	53. 2-Methylphenol	10	330	103. 3,3'-Dichlorobenzidine	10	330
6. Trichlorofluoromethane	10	10	0.50	54. 2,2'-oxybis (1-Chloropropane)	10	330	104. Benzo(a)anthracene	10	330
7. 1,1-Dichloroethene	10	10	0.50	55. Acetophenone	10	330	105. Chrysene	10	330
8. 1,1,2-Trichloro-1,2,2-trifluoroethane	10	10	0.50	56. 4-Methylphenol	10	330	106. bis-(2-Ethylhexyl)phthalate	10	330
9. Acetone	10	10	10	57. N-Nitroso-di-n-propylamine	10	330	107. Di-n-octylphthalate	10	330
10. Carbon Disulfide	10	10	0.50	58. Hexachloroethane	10	330	108. Benzo(b)fluoranthene	10	330
11. Methyl Acetate	10	10	0.50	59. Nitrobenzene	10	330	109. Benzo(k)fluoranthene	10	330
12. Methylene Chloride	10	10	0.50	60. Isophorone	10	330	110. Benzo(a)pyrene	10	330
13. trans-1,2-Dichloroethene	10	10	0.50	61. 2-Nitrophenol	10	330	111. Indeno(1,2,3-cd)pyrene	10	330
14. Methyl tert-Butyl Ether	10	10	0.50	62. 2,4-Dimethylphenol	10	330	112. Dibenz(a,h)anthracene	10	330
15. 1,1-Dichloroethane	10	10	0.50	63. bis-(2-Chloroethoxy) methane	10	330	113. Benzo(g,h,i)perylene	10	330
16. cis-1,2-Dichloroethene	10	10	0.50	64. 2,4-Dichlorophenol	10	330			
17. 2-Butanone	10	10	10	65. Naphthalene	10	330	**PESTICIDES/AROCLORS (PESTICIDES/PCBs)**	**Water (µg/L)**	**Soil (µg/Kg)**
18. Chloroform	10	10	0.50	66. 4-Chloroaniline	10	330			
19. 1,1,1-Trichloroethane	10	10	0.50	67. Hexachlorobutadiene	10	330			
20. Cyclohexane	10	10	0.50	68. Caprolactam	10	330	114. alpha-BHC	0.05	1.7
21. Carbon Tetrachloride	10	10	0.50	69. 4-Chloro-3-methylphenol	10	330	115. beta-BHC	0.05	1.7
22. Benzene	10	10	0.50	70. 2-Methylnaphthalene	10	330	116. delta-BHC	0.05	1.7
23. 1,2-Dichloroethane	10	10	0.50	71. Hexachlorocyclo-pentadiene	10	330	117. gamma-BHC (Lindane)	0.05	1.7
24. Trichloroethene	10	10	0.50	72. 2,4,6-Trichlorophenol	10	330	118. Heptachlor	0.05	1.7
25. Methylcyclohexane	10	10	0.50	73. 2,4,5-Trichlorophenol	25	830	119. Aldrin	0.05	1.7
26. 1,2-Dichloropropane	10	10	0.50	74. 1,1'-Biphenyl	10	330	120. Heptachlor epoxide	0.05	1.7
27. Bromodichloromethane	10	10	0.50	75. 2-Chloronaphthalene	10	330	121. Endosulfan I	0.05	1.7
28. cis-1,3-Dichloropropene	10	10	0.50	76. 2-Nitroaniline	25	830	122. Dieldrin	0.10	3.3
29. 4-Methyl-2-pentanone	10	10	10	77. Dimethylphthalate	10	330	123. 4,4'-DDE	0.10	3.3
30. Toluene	10	10	0.50	78. 2,6-Dinitrotoluene	10	330	124. Endrin	0.10	3.3
31. trans-1,3-Dichloropropene	10	10	0.50	79. Acenaphthylene	10	330	125. Endosulfan II	0.10	3.3
32. 1,1,2-Trichloroethane	10	10	0.50	80. 3-Nitroaniline	25	830	126. 4,4'-DDD	0.10	3.3
33. Tetrachloroethene	10	10	0.50	81. Acenaphthene	10	330	127. Endosulfan sulfate	0.10	3.3
34. 2-Hexanone	10	10	0.50	82. 2,4-Dinitrophenol	25	830	128. 4,4'-DDT	0.10	3.3
35. Dibromochloromethane	10	10	10	83. 4-Nitrophenol	25	830	129. Methoxychlor	0.50	17
36. 1,2-Dibromoethane	10	10	0.50	84. Dibenzofuran	10	330	130. Endrin ketone	0.10	3.3
37. Chlorobenzene	10	10	0.50	85. 2,4-Dinitrotoluene	10	330	131. Endrin aldehyde	0.10	3.3
38. Ethylbenzene	10	10	0.50	86. Diethylphthalate	10	330	132. alpha-Chlordane	0.05	1.7
39. Xylenes (Total)	10	10	0.50	87. Fluorene	10	330	133. gamma-Chlordane	0.05	1.7
40. Styrene	10	10	0.50	88. 4-Chlorophenyl-phenyl ether	10	830	134. Toxaphene	5.0	170
41. Bromoform	10	10	0.50	89. 4-Nitroaniline	25	830	135. Aroclor-1016	1.0	33
42. Isopropylbenzene	10	10	0.50	90. 4,6-Dinitro-2-methylphenol	25	830	136. Aroclor-1221	2.0	67
43. 1,1,2,2-Tetrachloroethane	10	10	0.50	91. N-Nitrosodiphenylamine	10	330	137. Aroclor-1232	1.0	33
44. 1,3-Dichlorobenzene	10	10	0.50	92. 4-Bromophenyl-phenylether	10	330	138. Aroclor-1242	1.0	33
45. 1,4-Dichlorobenzene	10	10	0.50	93. Hexachlorobenzene	10	330	139. Aroclor-1248	1.0	33
46. 1,2-Dichlorobenzene	10	10	0.50	94. Atrazine	10	330	140. Aroclor-1254	1.0	33
47. 1,2-Dibromo-3-chloropropane	10	10	0.50	95. Pentachlorophenol	25	830	141. Aroclor-1260	1.0	33
48. 1,2,4-Triclorobenzene	10	10	0.50	96. Phenanthrene	10	330			
				97. Anthracene	10	330			

*For volatiles, quantitation limits for medium soils are approximately 130 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 30 times the quantitation limits for low soils.

[1]Modified quantitation limits are available under the Flexibility Clause.

The list of target compounds for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since inception of the CLP, compounds have been added to and removed from the Target Compound List (TCL), based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program.

For drinking water and groundwater type samples, use of the low concentration organic analytical service is recommended.

METHODS AND INSTRUMENTATION

For volatile water samples, 5 mL of water sample is added to a purge and trap device and purged with an inert gas at room temperature. For volatile low-level soil samples, a 5 g aliquot of soil is added to a purge and trap device with 5 mL of reagent water then purged with an inert gas at 40°C; or a 5 g aliquot (pre-weighed in the field) is purged from a closed-system purge and trap device at 40°C. For volatile medium-level soil samples, a measured amount is collected/extracted with methanol and an aliquot of the methanol extract is added to reagent water and purged at room temperature. For both water and soil samples, the volatiles purged from the sample are trapped on a solid sorbent. They are subsequently desorbed by rapidly heating the sorbent and then introduced into a GC/MS system.

For semivolatile and pesticide/Aroclor water samples, a 1 L aliquot is extracted with methylene chloride using a continuous liquid-liquid extractor or separatory funnel [for pesticides/Aroclors only]. For low-level semivolatile soil and pesticide/Aroclor soil samples, a 30 g soil/sediment sample is extracted with methylene chloride/acetone using sonication, automated soxhlet, or pressurized fluid extraction techniques. For medium-level semivolatile soil samples, a 1 g aliquot is extracted with methylene chloride using the techniques mentioned above for low-level soil samples. For both water and soil samples, the extract is concentrated, subjected to fraction-specific cleanup procedures, and analyzed by GC/MS for semivolatiles or GC/EC for pesticides/Aroclors. **Table 2** summarizes the methods and instruments used in this analytical service.

PRESERVATION OF VOLATILE SOIL SAMPLES

All soil sample containers that are not chemically preserved should be frozen upon receipt at the laboratory. This includes closed-system vials received from the sampler; closed-system vials generated by the laboratory; low headspace vials containing sample for moisture determination (or for analysis by the old Method 5030 protocol); and all QC samples.

DATA DELIVERABLES

Data deliverables for this service include hardcopy data reporting forms and supporting raw data. In addition to the hardcopy deliverable, contract laboratories must also submit the same data electronically. The laboratory must submit data to EPA within 7, 14, or 21 days after laboratory receipt of the last sample in the set. Preliminary data must be submitted within 48 hours (for volatiles) or 72 hours (for semivolatiles and pesticides/Aroclors) after receipt of each sample at the laboratory. EPA then processes the data through an automated Data Assessment Tool (DAT). DAT is a complete CLP data assessment package. DAT incorporates Contract Compliance Screening (CCS) and Computer-Aided Data Review and Evaluation (CADRE) to provide EPA Regions with PC-compatible reports, spreadsheets, and electronic files within 24 to 48 hours from the receipt of the data. This automated tool facilitates the transfer of analytical data into Regional databases. In addition to the Regional electronic reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has 6 days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

QUALITY ASSURANCE

The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of the quality required.

During the implementation of the data collection effort, QA activities ensure that the QC system is functioning effectively and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions.

Each contract laboratory prepares a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, and functional guidelines, as well as the QA and QC activities designed to achieve the data quality requirements for this analytical service.

QUALITY CONTROL

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analytical service are shown in **Table 3.**

Table 2. Methods and Instruments

Fraction	Water	Soil
Volatiles	Purge-and-trap followed by GC/MS analysis.	Purge-and-trap or closed-system purge-and-trap followed by GC/MS analysis.
Semivolatiles	Continuous liquid-liquid extraction followed by GC/MS analysis.	Sonication, automated soxhlet, or pressurized fluid extraction followed by GC/MS analysis.
Pesticides/Aroclors	Continuous liquid-liquid or separatory funnel extraction followed by dual column GC/EC analysis.	Sonication, automated soxhlet, or pressurized fluid extraction followed by dual column GC/EC analysis.

Table 3. Quality Control

QC Operation	Frequency
System Monitoring Compounds (volatiles)	Added to each sample, standard, and blank.
Surrogates (for semivolatiles and pesticides/Aroclors)	Added to each sample, standard, and blank.
Method Blanks (volatiles)	Analyzed at least every 12 hours for each matrix and level.
Method Blanks (semivolatiles and pesticides/Aroclors)	Prepared with each group of 20 samples or less of same matrix and level, or each time samples are extracted by the same procedure.
Instrument Blank (volatiles)	Analyzed after a sample that contains compounds at concentrations greater than the calibration range.
Instrument Blank (pesticides/Aroclors)	Every 12 hours on each GC column used for analysis.
Storage Blanks (volatiles)	Prepared and stored with each set of samples.
GC/MS Mass Calibration and Ion Abundance Patterns (volatiles and semivolatiles)	Every 12 hours for each instrument used for analysis.
GC Resolution Check (pesticides/Aroclors)	Prior to initial calibration, on each instrument used for analysis.
Initial Calibration	Upon initial set up of each instrument, whenever instrument maintenance/modification has been performed, and each time continuing calibration fails to meet the acceptance criteria.
Continuing Calibration	Every 12 hours for each instrument used for analysis.
Internal Standards (volatiles and semivolatiles)	Added to each sample, standard, and blank.
Matrix Spike and Matrix Spike Duplicate	Once every 20 or fewer samples of same fraction, matrix, and level in an SDG.

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by AOC and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, GC/MS and/or GC/EC tape audits, and evaluates laboratory performance through the use of blind performance evaluation samples.

For more information, or for suggestions to improve this analytical service, please contact:

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