

**Alaska Department of Environmental Conservation
Division of Spill Prevention and Response
Contaminated Sites Program**

Technical Memorandum – 16-001

Date: June 15, 2016

Laboratory Approval Program Request for Method Modification

Purpose:

The Alaska Department of Environmental Conservation (DEC) has added a method modification request process to the Contaminated Sites (CS) Laboratory Approval Program (LAP). Currently, method modifications have been for reduced volume methods only, but this procedure can be tailored to any method modification presented to Laboratory Approval Program. For example, a new extraction method for soils can be presented to the Laboratory Approval Program for consideration. Method modifications are accepted on the basis of method performance, rather than relying on consistent technique.

Method Modification Request Requirements for All Methods:

1. Examples of modifications requiring LAP approval:
 - Sample extraction conditions
 - Glassware (including sampling containers)
 - Sample size
 - Surrogate choice

2. A validation study must be submitted to the Laboratory Approval Program for review. The validation study must contain, at a minimum:
 - a. Standard Operating Procedure (SOP);
 - b. Method Detection Limit (MDL) study to establish limits of detection (LODs, must be 99% confidence) and limits of quantitation (LOQs);
 - c. Initial Demonstration of Capability (IDC) - Four LCS samples yielding results within the LCS control limits the laboratory uses to determine extraction batch acceptability. Also, analysis of a standard at the proposed reporting limit (RL) yielding results within the CCV control limits the laboratory uses to determine analytical batch acceptability. The RL must meet the minimum cleanup level as given in Table 1 in the CS regulations (18AAC 75.341);
 - d. Passing Proficiency Test (PT) - All modified methods require Proficiency Tests (PTs) for Laboratory Approval. This is in exception to the Alaska regulations where only AK 101/102/103 and BTEX Proficiency Tests are required. All analytes affected by the method modification must be included in the PT sample. *PTs must be verified annually;* and
 - e. Any other data that illustrates the ability of the laboratory to produce reliable data by the method modification.

3. Only the methods that appear on the Alaska Laboratory Approval Application will be considered for modification. Link to the application: <http://dec.alaska.gov/eh/docs/lab/CS/USTapp.pdf>
4. Modifications to chromatographic conditions or settings, columns, or traps do not require LAP approval.
5. The Laboratory Approval Program will respond to method modification requests in order of receipt. Please note that a request submission does not guarantee Approval by the Program. A final decision on allowance of the method modification in the Contaminated Sites Program will be made 30-45 days from receipt of the request.
6. The following modifications will NOT be approved:
 - Integration range
 - Sampling container materials – e.g. Teflon-lined lids, glass, or high-density polyethylene
 - Holding times

Reduced Volume Modification Request Requirements for Semivolatile Organics in waters:

1. The minimum volume for all reduced volume semivolatile organic methods is 100mL. Any method modification request for a smaller volume will not be considered.
2. For reduced volume semivolatile organic methods, such as 8270 and 8082A, the extraction must be performed by the use of the separatory funnel method EPA 3510C or by the Continuous LLE method by EPA 3520C. The use of the EPA 3511 method is not allowed. There are no exceptions to this requirement.
3. The PTs for 8260 and 8270 must pass 80% of their respective full analyte lists. The NELAC method of selectively spiking the PT is acceptable for the full list only.
4. The PTs for PAH methods must include the following compounds. *All compounds must be present (spiked) in the PT sample, and the results must pass for all analytes:*

1-Methylnaphthalene	Benzo(k)fluoranthene
2-Methylnaphthalene	Chrysene
Acenaphthene	Dibenz(a,h)anthracene
Acenaphthylene	Fluoranthene
Anthracene	Fluorene
Benzo(a)anthracene	Indeno(1,2,3-cd)pyrene
Benzo(a)pyrene	Naphthalene
Benzo(b)fluoranthene	Phenanthrene
Benzo(g,h,i)perylene	Pyrene

Low Level Modification Request Requirements for Volatile Organics in waters and soils:

1. The minimum list of analytes for a low level volatile organics analysis modification is the following. *All compounds must be present (spiked) in the PT sample, and the results must pass for all analytes.*

1,1,1,2-Tetrachloroethane	Dichlorodifluoromethane
1,2,4-Trichlorobenzene	Ethylbenzene
2-Butanone (MEK)	Methyl-t-butyl ether (MTBE)
4-Isopropyltoluene	Tetrachloroethene
Benzene	Toluene
Bromobenzene	Trichloroethene
Bromoform	Trichlorofluoromethane
Carbon Tetrachloride	Vinyl chloride
Carbon disulfide	Xylenes (total)

2. PT sample concentrations for validating any low level volatile method are the following:

Matrix	Range (most analytes)	Range (MTBE and MEK)
Waters	0.1 - 10 ppb	0.5 - 50 ppb
Soils	1 - 50 ppb	5 - 250 ppb

Any questions regarding this memorandum should be directed to Shera Hickman, Laboratory Approval Officer at declabcert@alaska.gov or 907-375-8210.