U.S. Department of Energy

Mixed-Analyte Performance Evaluation Program (MAPEP)

MAPEP TEST SESSION 16

CLOSING DATE: October 5, 2006, 12:00 noon MST

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Radioactive Decay Correction Date for All Radiological Samples:
July 1, 2006, 12:00 Mountain Standard Time (MST).

PLEASE NOTE THAT THE TURNAROUND TIME FOR REPORTING RESULTS IS <u>ABOUT 60 DAYS</u>

PLEASE READ ALL INSTRUCTIONS

CAREFULLY BEFORE ANALYZING SAMPLE

IMMEDIATELY UPON RECEIPT OF SAMPLES
CHECK FOR BREAKAGE AND SHIPPING ERRORS;
SAMPLE REPLACEMENT TAKES AT LEAST
2 TO 3 DAYS

INSTRUCTIONS FOR MAPEP TEST SESSION 16

1. MAPEP PERFORMANCE EVALUATION (PE) SAMPLES.

Water Sample:
Domestic laboratories performing radiological/inorganic and organic analyses may receive as many as three one-liter sample bottles (MaW – mixed analyte radiological and stable inorganic water, GrW – Gross alpha/beta water, and OrW – semi-volatile organics water sample). Samples are shipped in multiple boxes with various shipping dates. Please allow ample time for all samples to be received before assuming a sample is missing. Participants will be notified by email when a shipment is made. **It is critical that radiological and stable inorganic analyses utilize sample from ONLY the bottle marked for mixed analyte radiological and stable inorganic analyses (MaW). Gross alpha/beta analyses must utilize sample from ONLY the bottle marked for gross alpha/beta analyses (GrW). Organic analyses must utilize sample from ONLY the bottle marked for organic analyses (OrW)**. Failure to utilize the appropriate sample bottle will yield incorrect results.

Soil Sample:
Most laboratories performing radiological, stable inorganic, and/or organic analyses will receive one ~300 gram sample jar of soil. Laboratories that requested extra soil will receive more than one jar. The soil contains all analytes (radiological, stable inorganic, and semi-volatile organics) and is labeled as a mixed analyte soil (MaS).

Air Filters (47-mm glass fiber):
Air filters are spiked with radiological constituents only, i.e., they are not mixed analyte samples. Laboratories performing radiological and gross alpha/beta air filter analyses will receive one to three filter packets, dependent upon the analyses performed. Filters labeled RdF are radiological air filters with multiple radionuclide determinations required. A filter labeled GrF is for gross alpha/beta analyses only. Air filters that require multiple radionuclide determinations (RdF) will come in two filter packets. Each filter packet contains an identically spiked air filter sandwiched between upper and lower non-spiked filters. The spiked side of the middle filter is placed in the packet facing "up" toward the label. The second air filter may be used for screening, the non-sequential determination of Sr-90, gamma-ray spectrometry, or other analytical procedures as needed.

The gross alpha/beta air filter will come in one filter packet containing one filter (GrF). The spiked side of the filter is placed in the packet facing "up" toward the label. For gross calibration information, the gross alpha/beta filters are spiked with Th-230 and Sr-90. The RdF and GrF filters are not marked so carefully note the spiked filters and their orientation before removing them from the packets. Also note that 47-mm glass fiber air filters are being used for both RdF and GrF samples.

A blank 47-mm glass fiber filter is also provided. The blank filter can be used to prepare a gross alpha/beta filter calibration specific for the glass fiber filters used for the MAPEP gross alpha/beta filter unknown. Some participants have already prepared a MAPEP specific gross alpha/beta filter

calibration, but others have not. The bank filter is provided for those participants that desire to make this calibration.

Vegetation:
Laboratories that request a vegetation matrix will receive two samples: 1) a large sample of about 100 grams (about 400 mL) of finely milled grass hay spiked with only radiological constituents; 2) a smaller sample of less than 10 grams (about 50 mL) of the same vegetation matrix and identically spiked as the larger sample. The large sample is provided for gamma-ray spectrometry measurements and can be ashed to less than 10 grams for actinide and/or Sr-90 analyses. Other analytical methods that utilize the entire sample may also be useful, but ashing the vegetation is among the simplest. The small sample (less than 10 grams, about 50 mL volume) is provided primarily for those participants that cannot handle the larger sample size for actinide and/or Sr-90 analyses. Again, both the large and small samples are identically spiked for all targeted radionuclides. **The entire sample, whether large or small, must be used for analysis**. Use either the large or small vegetation sample, or use both, but results must be reported on a per sample basis. **Do not subdivide either sample**.

The grass hay is a real-world vegetation matrix for environmental analytical services. A relatively large sample size is typically collected to ensure that the sample is representative and to provide adequate counting statistics and/or meet appropriate detection limits. For gamma-ray spectrometry, the vegetation may be compressed to create a slightly smaller geometry (e.g., 350 mL), or the density may be decreased to create a slightly larger geometry (e.g., 450 mL). Extra sample may be requested if an even larger geometry is required. However, specific activity for all results must be **reported in Bq/sample** (i.e., Bq per single large 400 mL sample or Bq per single small 50 mL sample). Since both samples are identically spiked, either sample may be used if the results are reported in Bq/sample.

2. SAMPLE DESCRIPTIONS.

Sample descriptions for the water, soil, air filters, and vegetation associated with this study are found toward the end of these instructions. Analyze the sample for those analytes that comprise your routine function or constitute your compliance requirements.

3. REPORTING ANALYTICAL RESULTS.

REPORTING RADIOLOGICAL ANALYTES:
Radiological analyses should report results for only the analytes listed on the sample description. Other analytes may be detectable but will not be evaluated. Conversely, some of the radiological analytes listed on the sample description may not be detected. Report the actual results obtained for all analyses performed, including negative numbers, even if the radionuclide was not detected (i.e., do not report results as “Less Than” or “Not Detected”). Do not report a “0.0” (zero) result or uncertainty. The result and total propagated uncertainty are required for sensitivity determinations and false positive testing. Failure to report results for requested analyses may result in a “Not Acceptable” performance evaluation if the analysis is within the scope of your routine function or contractual obligations. Report all results in Becquerels per unit, i.e., Bq/L (water), Bq/kg (soil), Bq/sample (filter and vegetation).

REPORTING STABLE INORGANIC ANALYTES:
Stable inorganic analyses should report results for only the analytes listed on the sample description. Other analytes may be detectable but will not be evaluated. Conversely, some of the stable inorganic analytes listed on the sample description may not be detected. Report the actual results obtained, or if applicable, the detection limit value. "Less Than" ('<') with an established Detection Limit is acceptable for reporting results for stable inorganic analytes. **DO NOT USE 0.00 (zero) as a reportable value.** Results reported as zeros will be flagged as "Not Acceptable". Total uranium, uranium-238, and uranium-235 can be reported when utilizing mass spectrometric techniques under the reporting section for stable inorganic analytes. Report as mg/L (water), mg/kg (soil), and µg/sample (filter and vegetation; for mass spec uranium results only).

REPORTING SEMIVOLATILE ORGANIC ANALYTES:
Effective December 1, 2005, the performance of participating laboratories that perform semi-volatile organic analyses in water and soil will be evaluated in accordance with the NELAC Performance Criteria as specified in the appropriate Field of Testing (FoT) tables associated with non-potable water (organics in water) and RECRA solids (soils). The semi-volatile organics water standards will be evaluated using the spiked value as the true value "T" (reference value). The acceptance limits for the water standard will be generated utilizing the linear regression line found in the June 2005 Non-potable water Field of Testing tables.

NOTE: "Less Than" ('<') with an established Detection Limit should be used for reporting results for organic analytes. Less than values signify that the laboratory has calibrated for the component, but was unable to detect it above the stated limit. Values reported as less than a detection limit will be evaluated against the true value. In the absence of a stated detection limit (i.e. the field is left blank), MAPEP will assume a calibration was NOT performed and the facility was unable to determine a value. Report all results in micrograms per unit, i.e., µg/L (water) and µg/kg (soil).

DO NOT USE CLP reporting flags (U, J, etc.). DO NOT USE 0.00 (zero) as a reportable value. Results reported as zeros will be flagged as "Not Acceptable".

FOR ALL ANALYTES:
You are required to report only one result for each appropriate analyte. If the reported result is actually a mean of several replicate analyses, the reported uncertainty should also be the mean of the individual uncertainties. Do not propagate the individual uncertainties for replicate measurements. For example, assume three replicate analyses provided the following results and individual uncertainties: 101 +/- 12, 108 +/- 15, 110 +/- 16. The mean result is (101+108+110)/3=106 and the mean individual uncertainty is (12+15+16)/3=14. The reported result and uncertainty is 106 +/- 14. The reported total uncertainty should be at one standard deviation. Propagated uncertainties are strongly encouraged. Please also ensure that the Method Code is entered correctly for each reported result. Method Codes are used in proficiency testing and an inappropriate Method Code may result in a "Not Acceptable" performance evaluation.

Report your results electronically via the MAPEP World Wide Web application at **http://mapep.inel.gov/.** Login information, including user ID and password, were previously emailed to the MAPEP point of contact (POC) for your laboratory. Please ensure that your lab code, points of contact, addresses, and NRC license information are entered correctly in the data

entry program. The shipping distribution list and correspondence mailing list will utilize the address and POC information exactly as you enter it here. You are a U.S. Federal Laboratory only if your employees are federal government workers (i.e., EPA, USGS, NRC, etc.). If you are a primary contractor for a DOE National Laboratory you may have a DOE exemption and, if so, enter your DOE contract number.

4. ADDITIONAL INFORMATION.

4.1 The laboratory may choose the analytical method.

4.2 The amount of sample is limited. The laboratory should use the maximum specific activities and concentration ranges listed on the sample description to select the optimum amount of sample for each analysis to ensure that sufficient sample is available for all of the analyses.

4.3 Excess sample or residues shall not be returned to RESL. Do not initiate analysis of the sample if approved waste treatment, storage, or disposal options are not available.

"MAPEP samples are analytical standards or a "product" generated for the purpose of securing and evaluating analytical services; they are not hazardous waste and they are not samples of hazardous waste... Thus, a laboratory participating in the MAPEP is in the process of establishing its eligibility and credentials to do DOE analytical work. It follows, therefore, that the laboratory is the "generator" of the waste resulting when the samples and the resulting residues are to be discarded." (MEMORANDUM OCC-95-189, Office of Chief Counsel, October 16, 1995)

4.4 The reference date for radioactive decay correction for all radiological analyses is July 1, 2006, 12:00 Mountain Standard Time (MST). Sample-holding time is based upon the RECEIPT date of the sample by the participating laboratory. EPA recommends mercury holding times of 28 days for acidified aqueous samples and as soon as possible for refrigerated solids.

4.5 Results are due by October 5, 2006, 12:00 noon MST. Late results will not be included in the final report.

Please address any questions to the appropriate point of contact:

Jim Dahlgran (208-526-6243, dahlgrjr@id.doe.gov): data entry and organic analyses;
Leon Jensen (208-526-4591, jensenll@ id.doe.gov): stable inorganic analyses;
David Sill (208-526-8031, sillds@ id.doe.gov): radiological analyses.

MAPEP-06-MaW16 WATER SAMPLE DESCRIPTION

The analytes for the MAPEP water, and their maximum specific activities and concentration ranges, are listed in the following tables. Each radiological/stable inorganic sample contains approximately one liter of 5% (v/v) nitric acid in water.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
^{241}Am, ^{238}Pu, ^{239}Pu, ^{234}U, ^{238}U	< 15 Bq/L	^{57}Co, ^{134}Cs, ^{137}Cs, ^{55}Fe, ^{63}Ni, ^{54}Mn, ^{65}Zn, ^{60}Co	< 2000 Bq/L
^{90}Sr, ^{99}Tc	< 100 Bq/L	^{3}H	<1000 Bq/L

NOTE: The ^{234}U and ^{238}U isotopes may not be in equilibrium. Some of the radionuclides listed on the sample description may not be detected, but if included in your sample analyses, the result and total propagated uncertainty must be reported for sensitivity evaluation and/or false positive testing.

STABLE INORGANIC CONSTITUENT DESCRIPTION

Analyte	Concentration Range	Analyte	Concentration Range
As, Cr (Total), Pb, Ag	0.01 – 4.9 mg/L	Sb, Ni, Tl, V, Zn	0.01 – 6.9 mg/L
Cd, Se, Be	0.005 – 0.74 mg/L	Co, Cu	0.025 – 24 mg/L
Hg	0.0002 – 0.15 mg/L	Ba	0.2 – 95 mg/L
^{238}U, ^{Total}U	0.01 – 1.23 mg/L	^{235}U	0.0001 – 0.018 mg/L

NOTE: Some of the stable inorganic constituents listed in the above table may not be present in the sample. Laboratories should report results and associated uncertainties for those constituents quantitated above the minimum concentration range listed for that analyte. For sensitivity evaluation and/or false positive testing, the actual analytical or detection limit values should be reported for those constituents with results found to be less than the lower concentration range. Failure to report analytical results as instructed may result in a false positive or false negative performance evaluation.

MAPEP-06-OrW16 SEMI-VOLATILE ORGANIC WATER SAMPLE DESCRIPTION

Analyte Class	Concentration Range
Acids & Base Neutrals	10 to 200 µg/L
Chlorinated Pesticides	0.5 to 20 µg/L
µg = micrograms	L=liter

NOTE: Sample-holding time is based upon the RECEIPT date of the sample by the participating laboratory.

MAPEP-06-MaS16 SOIL SAMPLE DESCRIPTION

The analytes for the MAPEP soil, and their maximum specific activities and concentration ranges, are listed in the following tables. Most participants will receive a single sample containing approximately 300 grams of soil.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
^{57}Co, ^{134}Cs, ^{137}Cs, ^{54}Mn, ^{65}Zn, ^{60}Co, ^{40}K	< 4000 Bq/kg	^{55}Fe, ^{63}Ni	< 2000 Bq/kg
^{90}Sr, ^{99}Tc	< 1000 Bq/kg	^{241}Am, ^{238}Pu, ^{239}Pu ^{234}U, ^{238}U	< 300 Bq/kg

NOTE: The ^{234}U and ^{238}U isotopes may NOT be in equilibrium. Some of the radionuclides listed on the sample description may not be detected, but if included in your sample analyses, the result and total propagated uncertainty must be reported for sensitivity evaluation and/or false positive testing.

STABLE INORGANIC CONSTITUENT DESCRIPTION

Analyte	Concentration Range	Analyte	Concentration Range
Co, Cu, Tl, Ni, V, Sb, Zn	1 – 400 mg/kg	Ba	5 – 1900 mg/kg
Ag, As, Cr (Total), Pb	0.2 – 95 mg/kg	Be	0.1 – 50 mg/kg
Cd, Se	0.1 – 19 mg/kg	Hg	0.004 – 3.8 mg/kg
^{238}U, ^{Total}U	0.1 – 24.5 mg/kg	^{235}U	0.001 – 0.35 mg/kg

NOTE: Some of the stable inorganic constituents listed in the above table may not be present in the sample. Laboratories should report results and associated uncertainties for those constituents quantitated above the minimum concentration range listed for that analyte. For sensitivity evaluation and/or false positive testing, the actual analytical or detection limit values should be reported for those constituents with results found to be less than the lower concentration range. Failure to report analytical results as instructed may result in a false positive or false negative performance evaluation.

SEMI-VOLATILE ORGANIC SAMPLE DESCRIPTION

Analyte Class	Concentration Range
Chlorinated pesticides	5 to 500 µg/kg
Acids & Base Neutrals	500 to 15000 µg/kg
µg = micrograms	kg = kilograms

NOTE: Sample-holding time is based upon the RECEIPT date of the sample by the participating laboratory.

MAPEP-06-RdF16 RADIOLOGICAL AIR FILTER SAMPLE DESCRIPTION

The analytes for the MAPEP radiological air filters and their maximum specific activities are listed in the following table. Each filter packet contains an identically spiked 47-mm glass fiber air filter sandwiched between upper and lower non-spiked filters. The spiked side of the middle filter is placed in the packet facing "up" toward the label.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
^{241}Am, ^{238}Pu, ^{239}Pu, ^{234}U, ^{238}U	< 2 Bq/sample	^{57}Co, ^{134}Cs, ^{137}Cs, ^{54}Mn, ^{65}Zn, ^{60}Co	< 10 Bq/sample
^{90}Sr	< 4 Bq/sample		

MAPEP-06-GrW16 GROSS ALPHA/BETA WATER SAMPLE DESCRIPTION

The maximum specific activity for the MAPEP gross alpha/beta water is listed in the following table. Each sample contains approximately one liter of 5% (v/v) nitric acid in water.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Concentration Range
Gross Alpha (Th-230)	< 2 Bq/L
Gross Beta (Sr-90)	< 3 Bq/L

MAPEP-06-GrF16 GROSS ALPHA/BETA AIR FILTER SAMPLE DESCRIPTION

The maximum specific activity for the MAPEP gross alpha/beta air filter is listed in the following table. The filter packet contains one 47-mm glass fiber filter. The spiked side of the filter is placed in the packet facing "up" toward the label.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Concentration Range
Gross Alpha (Th-230)	< 2 Bq/sample
Gross Beta (Sr-90)	< 3 Bq/sample

MAPEP-06-RdV16 RADIOLOGICAL VEGETATION SAMPLE DESCRIPTION

The analytes for the MAPEP radiological vegetation and their maximum specific activities are listed in the following table. Laboratories that request a vegetation matrix will receive two samples: 1) a large sample of about 100 grams (about 400 mL) of finely milled grass hay spiked with only radiological constituents; 2) a smaller sample of less than 10 grams (about 50 mL) of the same vegetation matrix and identically spiked as the larger sample. The large sample is provided for gamma-ray spectrometry measurements and can be ashed to less than 10 grams for actinide and/or Sr-90 analyses. The small sample (less than 10 grams, about 50 mL volume) is provided primarily for those participants that cannot handle the larger sample size for actinide and/or Sr-90 analyses. Again, both the large and small samples are identically spiked for all targeted radionuclides. **The entire sample, whether large or small, must be used for analysis**. Use either the large or small vegetation sample, or use both, but results must be reported on a per sample basis. **Do not subdivide either sample**. The specific activity for all results must be **reported in Bq/sample** (i.e., Bq per single large 400 mL sample or Bq per single small 50 mL sample). Since both samples are identically spiked, either sample may be used if the results are reported in Bq/sample.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
^{241}Am, ^{238}Pu, ^{239}Pu, ^{234}U, ^{238}U	< 2 Bq/sample	^{57}Co, ^{134}Cs, ^{137}Cs, ^{54}Mn, ^{65}Zn, ^{60}Co	< 15 Bq/sample
^{90}Sr	< 4 Bq/sample		

MAPEP Sample Semi-Volatile Organic Target Compounds

Soil (MaS) and water (OrW) samples may contain any or all of the following compounds.

Acids	***Base/Neutrals***		
4-Chloro-3-methylphenol	2-Chloronaphthalene	Dimethylphthalate	Pyrene
2-Chlorophenol	1,3-Dichlorobenzene	Diethylphthalate	Benzo(a)anthracene
2,4-Dichlorophenol	1,4-Dichlorobenzene	Di-n-butylphthalate	Chrysene
2,4-Dimethylphenol	1,2-Dichlorobenzene	Butylbenzylphthalate	Benzo(b)fluoranthene
2,4-Dinitrophenol	Hexachlorobenzene	Bis(2-ethylhexyl)phthalate	Benzo(k)fluoranthene
4,6-Dinitro-2-methylphenol	Hexachlorobutadiene	Di-n-octylphthalate	Benzo(a)pyrene
2-Methylphenol	Hexachlorocyclopentadiene	2-methylnaphthalene	Indeno(1,2,3-c,d)pyrene
4-Methylphenol	Hexachloroethane	Naphthalene	Dibenzo(a,h)anthracene
2-Nitrophenol	1,2,4-Trichlorobenzene	Acenaphthylene	Benzo(g,h,i)perylene
4-Nitrophenol	Dibenzofuran	Acenaphthene	
Pentachlorophenol	Nitrobenzene	Fluorene	
Phenol	2,4-Dinitrotoluene	Phenanthrene	
2,4,5-Trichlorophenol	2,6-Dinitrotoluene	Anthracene	
2,4,6-Trichlorophenol	Isophorone	Fluoranthene	

Pesticides			
alpha-BHC	beta-BHC	Lindane	delta-BHC
Heptachlor	Aldrin	Heptachlor epoxide	Endosulfan I
4,4'-DDE	Dieldrin	Endrin	4,4'-DDD
Endosulfan II	4,4'-DDT	Endrin Aldehyde	Endosulfan Sulfate

MATERIAL SAFETY DATA SHEETS ARE ENCLOSED

Department of Energy
Idaho Operations Office
1955 Fremont Ave
Idaho Falls, Idaho 83415-4149

Radiological and Environmental Sciences Laboratory

July 2006

TO: MAPEP Participants

SUBJECT: Conformity Certificate MAPEP Standards

The MAPEP standards, supplied in the shipment dated July 2006 are accurately described in the associated sample description and Quality Certificate.

The MAPEP standards are not radioactive in accordance with U.S. Department of Transportation regulations.

Sincerely,

Guy M. Marlette
MAPEP Coordinator

Department of Energy
Idaho Operations Office
1955 Fremont Ave
Idaho Falls, Idaho 83415-4149

Radiological and Environmental Sciences Laboratory

July 2006

Quality Certificate - MAPEP Standards

The Radiological and Environmental Sciences Laboratory hereby states that the standard(s) described by MAPEP and delivered to participants in this study have been tested and no fault or discrepancy from that description was found.

Sincerely,

Guy M. Marlette
MAPEP Coordinator

END OF INSTRUCTIONS