

U.S. Department of Energy

Mixed-Analyte Performance Evaluation Program (MAPEP)

MAPEP TEST SESSION 39

**CLOSING DATE: November 14, 2018, 19:00 Coordinated Universal Time (UTC)
or 12:00 Noon Mountain Standard Time (MST)**

**Radioactive Decay Correction Date for All Radiological Samples:
August 1, 2018, 19:00 Coordinated Universal Time (UTC)
or 12:00 Noon Mountain Standard Time (MST)**

**PLEASE NOTE THAT THE TURNAROUND TIME FOR REPORTING
RESULTS IS ABOUT 60 DAYS FROM THE SHIP DATE**

**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 39

1. SAMPLE AVAILABILITY.

The amount of proficiency testing (PT) material prepared for each MAPEP distribution is limited. MAPEP will not honor requests for additional PT samples without compelling analytical justification. Participants should use appropriate sampling methods and the maximum specific activities or concentrations listed on the sample descriptions to help select the optimum amount of sample for each analysis. This will help ensure that sufficient sample is available for all of the MAPEP determinations.

DO NOT SUBDIVIDE VEGETATION OR AIR FILTER SAMPLES.

2. MAPEP PERFORMANCE EVALUATION (PE) SAMPLES.

Water Sample:

Domestic laboratories performing radiological and inorganic analyses may receive a one-liter sample bottle for each water matrix (MaW – mixed analyte radiological and inorganic water, and XaW– alkaline radiological water, and ScR = Screen alpha/beta/gamma water. 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 inorganic analyses utilize sample from only the bottle marked for mixed analyte radiological and inorganic analyses (MaW). Radiological analyses with alkaline water (e.g., I-129) must utilize sample from only the bottle marked alkaline radiological water (XaW). Screening alpha/beta/gamma analyses must utilize sample from only the bottle marked for alpha/beta/gamma analyses (ScR). **Failure to utilize the appropriate sample bottle will yield incorrect results.** Thoroughly mix the entire water sample before performing analyses.

Soil Sample:

Most laboratories performing radiological and inorganic analyses will receive one ~300 gram sample of soil. The soil contains all targeted radiological and inorganic analytes 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 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. 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 RdF 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 each of the samples. **Do not subdivide the air filter samples.**

Vegetation:

Laboratories that request a vegetation matrix will receive two samples: 1) a large sample of about 95 grams (about 400 mL) of finely milled hay spiked with only radiological constituents; 2) a smaller sample of less than 10 grams (about 40 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 40-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 vegetation sample.**

The 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). 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 40-mL sample). Since both samples are identically spiked, either sample may be used if the results are reported in Bq/sample. **Do not subdivide either vegetation sample.**

Special Radiological Matrix (XrM):

The XrM sample is an undisclosed sample matrix and may contain some radionuclides that are not listed in the MAPEP Handbook. Alpha-emitting radionuclides have a total activity of less than 2 Bq/sample, beta-emitting radionuclides have a total activity of less than 4 Bq/sample, and gamma-emitting radionuclides have a total activity of less than 10 Bq/sample. The sample is not “radioactive” according to the U.S. Department of Transportation criteria, and other known hazardous constituents will not be added. If, for some unforeseen reason, a hazardous constituent is required for sample preservation, stability, etc., the identity and quantity of the hazardous constituent will be disclosed prior to shipment. Performance testing for the XrM sample will not include inorganic analytes. A limited number of radionuclides can be determined by mass methods and reported in mass units. The sample matrix and radionuclides will vary for each test session. **The entire sample must be used for analysis. Do not subdivide the XrM sample. The results must be reported on a per sample basis. The specific activity for all results must be reported in Bq/sample.**

3. SAMPLE DESCRIPTIONS.

Sample descriptions for the samples associated with this study are found at the end of these instructions. Analyze the samples for those analytes identified on the sample description that are within the scope of your routine function, capability, compliance requirements, and/or contractual obligations. Thoroughly mix the entire water sample before performing analyses. Report all uncertainties at one standard deviation.

4. REPORTING ANALYTICAL RESULTS.

REPORTING RADIOLOGICAL ANALYTES (for all samples excluding XrM):

Report results for only the analytes listed on the sample description. Other analytes may be detectable but will not be evaluated. This includes any chemical or spectral interference deliberately added to the sample. 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”). Small negative values are expected, but a statistically significant negative value is “Not Acceptable” because it implies a real negative activity instead of a random fluctuation about zero. **Do not report a “0.0” (zero) result or uncertainty.** The result and total propagated uncertainty are required for sensitivity evaluations 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, capability, compliance requirements, and/or contractual obligations. Report all results in Becquerels per unit, i.e., Bq/L (water), Bq/kg (soil), Bq/sample (filter and vegetation). Report the total uncertainty at one standard deviation.

All targeted radionuclides determined by a given analytical method must be reported together. Specifically, if Pu-239 is determined by alpha spectrometry, then Pu-238 must also be reported. The same is true for U-238 and U-234 if they are determined by alpha spectrometry. The same alpha spectrum for the first isotope provides information about the second. Similarly, failure to report results for all targeted gamma-emitting radionuclides is "Not Acceptable" if the laboratory reported results for at least one targeted gamma emitter. The same efficiency curve used to determine one gamma emitter can be used to determine the others, and MAPEP is specifically testing the efficiency curve used for these spectrometry measurements. The targeted gamma emitters are Co-57, Cs-134, Cs-137, Mn-54, Zn-65, and Co-60 for radiological air filter and vegetation sample matrices, with the addition of K-40 in the mixed-analyte soil and water matrices. Am-241 is not considered a targeted gamma emitter for reporting purposes, but gamma ray spectrometry can be used for its determination. Also, failure to report the results of a false positive test or sensitivity evaluation for an otherwise reportable radionuclide is “Not Acceptable”. For example, failure to report the results for a Sr-90 false positive test is “Not Acceptable” if Sr-90 was reported within the last two test sessions, and the laboratory historically reports Sr-90 results. For testing purposes, MAPEP defines a result as statistically positive when the Abs [Result ÷ Total Uncertainty] > 3. The probability of this occurring by chance alone is less than 1%.

Alpha/Beta/Gamma Screening Water Analytes:

The MAPEP ScR PT water sample is a whole volume, real world sample, with dissolved solids and multiple radionuclides. Report the screening result for the alpha activity present in the MAPEP ScR PT sample which will be compared to the total known alpha activity and the bias will be determined for the alpha measurement. Similarly, report the screening result for the beta/gamma activity present in the ScR PT sample which will be compared to the known beta/gamma activity and the bias will be determined for the measurement. Along with bias, participants will be provided the activities of the individual alpha/beta/gamma-emitting radionuclides which will aid in the performance evaluation. The specific activity for all results must be reported in Bq/L. Report the total uncertainty at one standard deviation.

REPORTING SPECIAL RADIOLOGICAL MATRIX (XrM) ANALYTES:

Individual radionuclides are not identified on the XrM sample description, so participants do not have a list of targeted analytes. Participants should report results for those radionuclides that they can detect and that are within the scope of their routine function or compliance requirements. Participants can also perform research in areas such as method development or proof of process work. Detected radionuclides must also be available on the pick list for reporting MAPEP radiological results. Other radionuclides might be detected, but will not be evaluated. The performance evaluation associated with the XrM sample is designed to encourage participants to fully test their analytical capabilities without the fear of being punished for participating. Reported results for the XrM sample are not graded or evaluated by MAPEP. Each reported result will have the bias from the RESL known value calculated, but the reported results will not be flagged or evaluated by any acceptance criteria, i.e., reported results will not be flagged or evaluated as “Acceptable”, “Acceptable with Warning”, or “Not Acceptable”. Participants will see the bias of their reported result from the known value and can evaluate their own performance. MAPEP will not issue Letters of Concern for performance associated with the XrM sample. Participation will not be punitive. Report all activity measurements for the XrM sample as Bq/sample and all mass results as ug/sample. Report all uncertainties at one standard deviation.

REPORTING INORGANIC ANALYTES:

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 inorganic analytes listed on the sample description may not be detected. Total Uranium, Uranium-238, Uranium-235, and Technicium-99 can be reported when utilizing mass spectrometric techniques under the reporting section for inorganic analytes. Report inorganic results as mg/L (water), mg/kg (soil), and µg/sample (filter and vegetation).

Each reported inorganic result must be accompanied by an estimate of its total uncertainty in the units of measurement. Report the actual results obtained for all analyses performed, including negative numbers, even if the analyte was not detected (i.e., do not report results as “Less Than” or “Not Detected”). False positive testing and sensitivity evaluations are performed as described in the radiological analytes section (see above). Report the total uncertainty at one standard deviation. **Do not report a zero result or uncertainty, and do not report the uncertainty as a percentage.**

FOR ALL ANALYTES:

You are required to report only one result for each target analyte. If the reported result is a mean of several replicate analyses, the reported uncertainty should also be the mean of the individual uncertainties. In this scenario, do not propagate the individual uncertainties for the replicate measurements. For example, assume three replicate analyses provided the following results and individual uncertainties: 101 +/- 12, 108 +/- 15, and 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. Report the total uncertainty at one standard deviation. MAPEP is interested in the uncertainty reported for a single measurement, and therefore requests the mean individual uncertainty of replicate measurements. 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 <https://mapep.inl.gov/>. 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 points of contact (POC) information exactly as you enter it here. For NRC licensing information, 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 NRC license exemption and, if so, enter your DOE contract number instead of an NRC license number.

5. ADDITIONAL INFORMATION.

5.1 The laboratory may choose the analytical method.

5.2 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)

5.3 Sample-holding time is based upon the receipt date of the sample by the participating laboratory.

5.4 Late results will not be included in the final report.

Please address any questions to the appropriate point of contact:

Primary email address: MAPEP@id.doe.gov

Shane Steidley (208-526-8249, steidlsd@id.doe.gov): MAPEP Coordinator;

Vanessa Jim (208-526-6243, jimvq@id.doe.gov): inorganic analyses;

David Sill (208-526-8031, sillds@id.doe.gov): radiological analyses;

MAPEP-18-MaW39 WATER SAMPLE DESCRIPTION

The analytes for the MAPEP water, and their maximum specific activities or concentrations, are listed in the following tables. Each radiological/inorganic sample contains approximately one liter of 5% (v/v) nitric acid in water. Thoroughly mix the entire sample before performing analyses.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
²⁴¹ Am, ²³⁸ Pu, ²³⁹ Pu, ²³⁴ U, ²³⁸ U, ²²⁶ Ra	< 25 Bq/L	⁵⁷ Co, ⁶⁰ Co, ¹³⁴ Cs, ¹³⁷ Cs, ⁵⁵ Fe, ⁴⁰ K, ⁶³ Ni, ⁵⁴ Mn, ⁶⁵ Zn,	< 2000 Bq/L
⁹⁰ Sr, ⁹⁹ Tc	< 200 Bq/L	³ H	<1500 Bq/L

NOTE: The U-234 and U-238 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. Failure to report analytical results as instructed may result in a “Not Acceptable” performance evaluation.

INORGANIC CONSTITUENT DESCRIPTION

Analyte	Concentration	Analyte	Concentration
As, Cr (Total) Pb, Tl	< 4.9 mg/L	Ba, Co, Cu, Ni, Sb, V, Zn	< 20 mg/L
Hg	< 0.19 mg/L	Cd, Se	< 0.9 mg/L
⁹⁹ Tc	< 0.00032 mg/L	²³⁸ U, TotalU	< 2.1 mg/L
Be	<5.5 mg/L	²³⁵ U	< 0.02 mg/L

NOTE: Some of the inorganic constituents 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. Failure to report analytical results as instructed may result in a “Not Acceptable” performance evaluation.

MAPEP-18-XaW39 ALKALINE RADIOLOGICAL WATER SAMPLE DESCRIPTION

The alkaline radiological water sample contains only I-129 as the target analyte with the maximum specific activity listed in the following table. Other non-target radionuclides and interferences may be present. Each alkaline water sample contains approximately one liter of water. Thoroughly mix the entire sample before performing analyses.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity
¹²⁹ I	< 100 Bq/L

MAPEP-18-ScR39 ALPHA/BETA/GAMMA SCREENING WATER SAMPLE DESCRIPTION

The maximum specific activity for the MAPEP alpha/beta/gamma screening water sample is listed in the following table. Each sample contains approximately one liter of 5% (v/v) nitric acid in water. Thoroughly mix the entire sample before performing analyses.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity
Alpha-emitting	< 25 Bq/L
Beta-emitting	< 200 Bq/L
Gamma-emitting	< 2000 Bq/L

MAPEP-18-MaS39 SOIL SAMPLE DESCRIPTION

The analytes for the MAPEP soil, and their maximum specific activities or concentrations, are listed in the following tables. Most participants will receive a single sample containing approximately 300 grams of soil. Chemical or spectral interferences may or may not be deliberately added.

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 U-234 and U-238 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. Failure to report analytical results as instructed may result in a “Not Acceptable” performance evaluation.

INORGANIC CONSTITUENT DESCRIPTION

Analyte	Concentration	Analyte	Concentration
Ba, Co, Cu, Ni, V, Zn	< 1950 mg/kg	Hg	< 3.8 mg/kg
Ag, As, Cr (Total), Pb	< 95 mg/kg	Sb	< 450 mg/kg
Be	< 70 mg/kg	Tl	< 650 mg/kg
Cd, Se	< 19 mg/kg	²³⁸ U, Total U	< 24 mg/kg
⁹⁹ Tc	< 0.0016 mg/kg	²³⁵ U	< 0.17 mg/kg

NOTE: Some of the inorganic constituents 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. Failure to report analytical results as instructed may result in a “Not Acceptable” performance evaluation.

MAPEP-18-RdF39 RADIOLOGICAL AIR FILTER SAMPLE DESCRIPTION

The analytes for the MAPEP radiological air filters and their maximum specific activities or concentrations are listed in the following tables. 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		

INORGANIC CONSTITUENT DESCRIPTION

Analyte	Concentration	Analyte	Concentration
^{238}U , Total U	< 160 $\mu\text{g}/\text{sample}$	^{235}U	< 1.2 $\mu\text{g}/\text{sample}$

MAPEP-18-XrM39 SPECIAL RADIOLOGICAL MATRIX SAMPLE DESCRIPTION

The maximum specific activity for the MAPEP special radiological matrix is listed in the following table. Most participants will receive two samples of the special radiological matrix.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity
Alpha-emitting	< 2 Bq/sample
Beta-emitting	< 4 Bq/sample
Gamma-emitting	< 10 Bq/sample

MAPEP-18-RdV39 RADIOLOGICAL VEGETATION SAMPLE DESCRIPTION

Analytes for the radiological vegetation and their maximum specific activities or concentrations are listed in the following tables. Laboratories that request vegetation will receive two samples: 1) a large sample of about 95 grams (about 400 mL) of milled grass hay spiked with only radiological constituents, and 2) a smaller sample of less than 10 grams (about 40 mL) of the same vegetation 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 40-mL volume) is provided primarily for those participants that cannot handle the larger sample size for actinide and/or Sr-90 analyses. 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.** The 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 40-mL 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		

INORGANIC CONSTITUENT DESCRIPTION

Analyte	Concentration	Analyte	Concentration
²³⁸ U, Total U	< 160 µg/sample	²³⁵ U	< 1.2 µg/sample

SAFETY DATA SHEETS ARE FOUND AT:

<http://www.id.energy.gov/resl/mapep/describe.html>



Department of Energy

Idaho Operations Office
1955 Fremont Ave
Idaho Falls, Idaho 83415-2112

Radiological and Environmental Sciences Laboratory

MAPEP Series 39

TO: MAPEP Participants

SUBJECT: Conformity Certificate MAPEP Standards

The MAPEP standards provided for the current test session 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,

Shane Steidley
MAPEP Coordinator
United States Department of Energy
Radiological and Environmental Sciences Laboratory



Department of Energy
Idaho Operations Office
1955 Fremont Ave
Idaho Falls, Idaho 83415-2112

Radiological and Environmental Sciences Laboratory

MAPEP Series 39

Quality Certificate - MAPEP Standards

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

Sincerely,

Shane Steidley
MAPEP Coordinator
United States Department of Energy
Radiological and Environmental Sciences Laboratory

COLLUSION WARNING

Collusion, both between participants or between individual participants and MAPEP, is contrary to professional scientific conduct and serves only to nullify the benefits of proficiency testing. By reporting MAPEP results, you attest to the fact that the reported analytical results were generated by your facility and are not a result of collusion with any other analytical body. Any participant found guilty of collusion will be in breach of conduct and will fail the MAPEP test session for all reported results and may face other adverse actions.

END OF INSTRUCTIONS