

U.S. Department of Energy

Mixed-Analyte Performance Evaluation Program (MAPEP)

MAPEP TEST SESSION 46

**CLOSING DATE: May 11, 2022, 18:00 Coordinated Universal Time (UTC)
or 12:00 Noon Mountain Daylight Time (MDT)**

**Radioactive Decay Correction Date for All Radiological Samples:
February 1, 2022, 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 46

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 GrW – Gross alpha/beta 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). Gross alpha/beta analyses must utilize sample from only the bottle marked for gross alpha beta water (GrW). **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 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, 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 each of the RdF and GrF samples.

If a gross alpha/beta filter sample is requested, a blank 47-mm glass fiber filter is also provided in a sealed packet. The blank filter can be used for blank subtraction and to prepare a gross alpha/beta filter calibration specific 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. **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.**

Urine Sample

Laboratories that request a urine matrix will receive a 34 gram sample of synthetic urine salts. The synthetic urine salts must be diluted to 1000 mL with 2% nitric acid prior to analyses. A single jar containing blank urine salts has also been provided. Analytical results should not be reported for the blank samples; however, **the blank sample results should be subtracted from results obtained for the PE samples.**

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:

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/L (urine), Bq/kg (soil), Bq/sample (filter, fecal 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 $\text{Abs} [\text{Result} \div \text{Total Uncertainty}] > 3$. The probability of this occurring by chance alone is less than 1%.

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 $\mu\text{g}/\text{sample}$ (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 send any questions to the MAPEP email address: MAPEP@id.doe.gov

MAPEP-22-MaW46 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, Be, 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
		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-22-RdF46 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 , TotalU	< 160 $\mu\text{g}/\text{sample}$	^{235}U	< 1.2 $\mu\text{g}/\text{sample}$

MAPEP-22-MaS46 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 ^{228}Th , ^{230}Th , ^{232}Th	< 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 , TotalU	< 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-22-GrW46 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. Thoroughly mix the entire sample before performing analyses.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity
Gross Alpha (Th-230)	< 5 Bq/L
Gross Beta (Sr-90)	< 15 Bq/L

MAPEP-22-GrF46 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	Specific Activity
Gross Alpha (Th-230)	< 5 Bq/sample
Gross Beta (Sr-90)	< 5 Bq/sample

MAPEP-22-MaSU46 SYNTHETIC URINE SAMPLE DESCRIPTION

The analytes for the MAPEP urine, and their maximum specific activities or concentrations, are listed in the following table.

RADIOLOGICAL CONSTITUENT DESCRIPTION

Analyte	Specific Activity	Analyte	Specific Activity
^{241}Am , ^{238}Pu , ^{239}Pu , ^{234}U , ^{238}U , ^{244}Cm	< 25 Bq/L	^{57}Co , ^{60}Co , ^{63}Ni , ^{134}Cs , ^{137}Cs , ^{40}K , ^{54}Mn , ^{65}Zn ,	< 2000 Bq/L
^{90}Sr , ^{99}Tc	< 200 Bq/L		

NOTE: See ANSI/HPS N13.30 for synthetic urine matrix recipe.

MASS CONSTITUENT DESCRIPTION

Analyte	Concentration	Analyte	Concentration
^{239}Pu , ^{240}Pu	< 80 pg/L	^{238}U , Total U	< 10 ug/L
		^{235}U	< 5 ng/L

NOTE: Some of the mass 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-22-RdV46 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 $\mu\text{g}/\text{sample}$	^{235}U	< 1.2 $\mu\text{g}/\text{sample}$

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 46

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
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 46

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
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