



# **HANDBOOK FOR THE DEPARTMENT OF ENERGY'S MIXED ANALYTE PERFORMANCE EVALUATION PROGRAM (MAPEP)**

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**U.S. Department of Energy  
Radiological and Environmental Sciences Laboratory  
1955 Fremont Drive, MS-4149  
Idaho Falls, ID 83415**

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**Guy Marlette, MAPEP Coordinator**

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# HANDBOOK FOR THE DEPARTMENT OF ENERGY'S MIXED ANALYTE PERFORMANCE EVALUATION PROGRAM (MAPEP)

## I. INTRODUCTION

Compliance and quality assurance issues associated with the Department of Energy (DOE) environmental programs typically require analytical services under contract with DOE to participate in a variety of performance evaluation programs (PEPs). The primary objective of the PEPs is to foster reliability and credibility for the analytical results used in the decision making process, particularly for those decisions affecting the environment, public health, and safety. Each PEP checks for specific analytical proficiencies in radiological, stable inorganic, or organic analyses. The performance evaluation (PE) samples used to test analytical proficiencies, however, frequently do not resemble the real-world samples analyzed for DOE. PE samples are frequently prepared with only a few target analytes in a concentrated or purified sample matrix, such as deionized or distilled water, with little chemical or other interference. The environmental samples submitted for analysis, however, typically have multiple target analytes in a whole-volume, non-concentrated and non-purified, natural matrix sample with numerous chemical or other interferences. Additionally, since the PE material is prepared for either radiological, stable inorganic, or organic analyses, the combined analytes are not in the same PE sample. Yet, the environmental samples that DOE must analyze typically contain constituents from each analytical category mixed together. Regulatory requirements frequently include analyses of radiological and non-radiological "mixed analytes" from the same environmental sample. DOE clearly needs PE material that contains mixed analytes in the same real-world sample matrix for testing the analytical proficiency of contracted services. A mixed analyte PEP, however, was previously not available. The Analytical Services Division of the DOE-HQ Office of Environmental Management (EM) established the MAPEP in 1994 to address this deficiency and to help assure the quality of analytical services across the DOE Complex.

The Radiological and Environmental Sciences Laboratory (RESL), under the program direction of the DOE-HQ Office of Health, Safety and Security (HSS), shall administer the MAPEP. MAPEP samples, distributed twice a year, include mixed analyte water and soil matrices with environmentally important radiological, stable inorganic, and organic constituents included in the same PE sample. Water and soil are typically among the most important matrices for DOE analytical services. Radiological air filter and vegetation matrices, and gross alpha/beta samples for water and air filter matrices, are also provided. Consolidating the major analytes of interest into a single PEP provides a more representative mixed analyte sample for the water and soil matrices and an efficient means for laboratories to demonstrate required proficiencies. The radiological vegetation and air filter samples address the quality assurance needs of DOE radiological programs, environmental monitoring, and long-term stewardship.

## II. PARTICIPATION

All laboratories that perform environmental analytical measurements for DOE (i.e., radiological, stable inorganic, and/or organic analyses, solely or in any combination) are required to participate in the MAPEP (Memorandum from the Assistant Secretary for Environmental Management, May 31, 1994, Newberry: 3-7615). MAPEP participation for radiological laboratories is also required by the *DOE Quality Systems for Analytical Services (QSAS), current version*. It is important to note that MAPEP water and soil samples are a mixed-**analyte** matrix, NOT a mixed **waste**: “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.” (Memorandum OCC-95-189, Office of the Chief Council, October 16, 1995). Participation is defined as requesting the performance sample materials, completing the appropriate analyses, reporting the results to RESL, and implementing any corrective actions. Participation may be requested by contacting the MAPEP Coordinator:

Guy M. Marlette  
Department of Energy  
Idaho Operations Office  
1955 Fremont Ave., MS-4149  
Idaho Falls, ID 83415

Phone: (208) 526-2532  
FAX: (208) 526-2548  
E-mail: marletgm@id.doe.gov

MAPEP applications are also available under the program information link on the MAPEP public website at <http://www.inl.gov/resl/mapep/>. A request for participation should include a shipping (do not use a post office box number) and correspondence address, a contact person for each, appropriate phone numbers, FAX number, e-mail address, any special shipping instructions, the current NRC or state license number for the laboratory or a statement of NRC license exemption, and the license or exemption expiration date. Since the MAPEP samples have a radioactive component, an NRC license or exemption is required for the receiving laboratories. Exemptions should specify the DOE contract number for the laboratory.

Participating laboratories are required to have appropriate radiological control measures and a QA/QC plan. Guidance for the QA/QC plan can be found in *DOE Quality Systems for Analytical Services (QSAS), (current version)*. Furthermore, in performing sample analyses the participating laboratory accepts title and ownership of the MAPEP sample and becomes the generator of any resulting waste or sample residues.

### III. SAMPLE PREPARATION, CHARACTERIZATION, AND VERIFICATION

Liquid MAPEP samples are prepared from radiological and stable inorganic standards traceable to the National Institute of Standards and Technology (NIST). Final concentrations for these analytes are calculated from the NIST certified standard value and the standard dilution(s) used. A known quantity of standard is combined and diluted to a known final volume with 2-5% (v/v) nitric acid and characterized natural ground or surface water. The nitric acid is high quality double distilled with certified concentrations for known contaminants. Organic analytes in water and soil are added to a separate whole-volume sample. The organic water and soil samples contain no radiological or stable inorganic components. All sample containers are acid-washed polyethylene or pre-cleaned glass bottles.

Solid samples are prepared from natural soil matrices spiked with NIST traceable standards for the various analytes of interest. The sample is characterized, homogeneity is assessed, and target analyte concentrations are verified prior to sample distribution. Known values for the radiological and stable inorganic analytes are calculated from the NIST certified standard values and the standard dilution(s) used. Rarely, a known value is derived from the sample characterizations in accordance with ISO Guide 43. Known values for organic analytes are derived from vendor certified standards and procedures that are in accordance with ISO Guide 43 (see Appendix H). Sample handling and storage procedures are similar to those for the liquid sample. Appendix H delineates the requirements for MAPEP PE sample material preparation and verification in accordance with a Proficiency Testing Provider operating under a Quality System that complies with, and is accredited to, ISO 9001:2000, ISO 17025:2005 and ILAC G13:2000 (ISO 43).

The U.S. Department of Transportation (DOT) does not typically classify MAPEP samples as radioactive. Other hazardous components are typically below the Resource Conservation and Recovery Act (RCRA) regulatory concentrations. Participants are provided sample descriptions that delineate the major analytes of interest, concentration ranges, and other important sample information. Each participant is responsible for reviewing their own compliance requirements and must determine if the analytical procedures utilized result in a mixed waste. Analysis shall not proceed without full compliance to all applicable regulatory authorities.

### IV. SAMPLE DISTRIBUTION

Samples are distributed semi-annually. Sample instructions containing a sample description will be available on the Internet prior to sample distribution (see Appendix B for a typical sample description) and provided with each sample. The MAPEP Coordinator must be notified of any special shipping requirements or other problems pertaining to the sample prior to shipment. The participants must ensure that they are authorized to receive a mixed analyte sample and that their standard operating procedures incorporate appropriate sample management and waste disposal practices. Acceptance of the sample means that the participating laboratory takes title and ownership of the sample. Excess sample or associated residues cannot be returned to RESL. Sample analysis shall not be initiated if approved treatment, storage, or disposal options are not available.

## V. SAMPLE ANALYSES

Analyses are required for only those analytes that are a component of the participant's routine analytical workload or compliance requirements (i.e., a complete sample analysis may not be appropriate). Laboratories must, however, report results for a targeted analyte if the determination is typically given by the analytical methodology utilized. For example, if Pu-238 and Pu-239 are targeted analytes, and results for Pu-239 are reported utilizing alpha spectrometry, the results for Pu-238 must also be reported. The receiving laboratory can split the sample and subcontract analyses if this is routinely performed. If a subcontracted laboratory is already participating in the MAPEP, a sample split is not required. The subcontracting laboratory can simply request, with the Additional Reports option in the MAPEP data entry program, that a copy of their MAPEP performance report be sent to the contracting laboratory. This will document MAPEP participation for all interested parties and prevents redundant analyses. The same analytical procedures employed for routine analyses should also be utilized for MAPEP samples. MAPEP, however, may also be used to develop new analytical methods or demonstrate proof of process. Participants are typically allowed about 60 calendar days to complete those analyses not controlled by regulatory holding times. The deadline for reporting results will be specified for each sample distribution.

Although analytical methods are not prescribed by MAPEP, standard analytical procedures will be utilized to independently characterize and verify the MAPEP samples. These analytical techniques include alpha spectrometry, beta counting, gamma spectrometry, inductively coupled plasma (ICP) atomic emission spectroscopy, ICP mass spectrometry, gas chromatography, gas chromatography/mass spectrometry, and other common analytical methods.

Activities for radiological analytes are typically sufficient to provide less than 10% (and usually <5%) counting uncertainty with a reasonable sample size and count time. Similar uncertainties should be achievable for most stable inorganic/organic analytes. The amount of sample is, however, limited. Therefore, the activity and concentration ranges indicated in the sample description must be used to select the optimum quantity of sample for each analysis.

## VI. REPORTING RESULTS

Analytical results are reported to RESL via the Internet. Data entry and edit screens are available for reporting the analytical results, and a hard copy record can be printed for laboratory records and/or review. Data entry and editing is allowed any time prior to the closing date for the particular study. The data entry program guides the user through selection of Method Codes for radiological (see Appendix C), stable inorganic (see Appendix D), and organic (see Appendix E) analyses. Data are entered directly into the MAPEP database via the Internet. Specific instructions for using the data entry program are provided in Appendix G.

The MAPEP will NOT accept any hard copy, floppy disks, or other electronic media without prior authorization. A laboratory's performance report can be sent to additional contacts if desired. Participants must keep their respective address and contact information current.

Participants are required to report only ONE result for each appropriate analyte. Each reported radiological result must be accompanied by an estimate of its uncertainty in the units of measurement (NOT as a percent), and both numbers should follow the rules for significant figures. Do not report a zero (0.0) result or uncertainty. The MAPEP strongly encourages that all results, including stable inorganic and organic analyses, be reported with uncertainty estimates. If the reported result is actually a mean of several replicate analyses, the reported uncertainty should also be the MEAN of the INDIVIDUAL uncertainties at one standard deviation. Do NOT combine the variances associated with the individual uncertainties for replicate measurements, even though this should typically be performed. The larger individual uncertainties associated with a single analysis are of interest to MAPEP since they are more indicative of routine performance. 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 result and total uncertainty as reported for MAPEP is 106 +/- 14. The total uncertainty is reported at one standard deviation.

The uncertainty characterizes the range about the result within which the true value is expected to lie (result +/- uncertainty). The uncertainty provides a probabilistic statement about the extent to which the result may be inaccurate. Because of Poisson counting statistics, a unique uncertainty can be propagated for each radiological result. This is not necessarily the case for stable element analyses where average uncertainties may be assigned for different analytes and concentration ranges. The exact method for estimating the uncertainty is not prescribed here since the reported uncertainty for MAPEP analyses should reflect the actual methods used for data generated on routine real-world samples. For guidance, however, it is preferred to estimate all uncertainty components, including those derived from a complete statistical analysis (Type A,  $s_A$ ) and those evaluated by other means (Type B,  $s_B$ ), as approximations to standard deviations. This convention follows that proposed by the Bureau International des Poids et Mesures (BIPM) and as suggested in several standard references (NIST Technical Note 1297, 1994; ISO/IEC/OIML/BIPM Guide to the Expression of Uncertainty in Measurement: 1995; NCSL Information Manual - Determining and Reporting Measurement Uncertainties, RP-12, 1994; ANSI N42.14-1991; NCRP Report No. 58, second edition, 1985). It allows all of the uncertainty components to be propagated into a total combined uncertainty by statistical rules and the combination of variances:

$$\mu = \sqrt{s_A^2 + s_B^2}$$

where  $\mu$  = the combined uncertainty and the other variables are as described above.

For example, let R = the analytical result,  $\Delta R$  = the total combined uncertainty in the result, U1 = an uncertainty component involved in the calculation of the result (such as a pipette calibration),  $\Delta U1$  the uncertainty in the pipette calibration derived statistically as the standard deviation of 10 measurements, i.e., an example of Type A uncertainty, U2 = a second uncertainty component, such as the value of a calibration standard used in calculating the result,  $\Delta U2$  = the uncertainty of the calibration standard obtained from a standard certificate at one standard deviation, i.e., an example of Type B uncertainty, U3 = a third uncertainty component, such as a weight measurement,  $\Delta U3$  the uncertainty in the weight measurement, U4 = a fourth uncertainty component, such as a volume measurement,  $\Delta U4$  = the uncertainty in the volume measurement, etc. Note that all uncertainty

components, including Type B uncertainty, should be estimated at one standard deviation. The equation used to calculate the total combined uncertainty in the result is given by:

$$\Delta R = R * \sqrt{\left[\frac{\Delta U1}{U1}\right]^2 + \left[\frac{\Delta U2}{U2}\right]^2 + \left[\frac{\Delta U3}{U3}\right]^2 + \left[\frac{\Delta U4}{U4}\right]^2 + \dots}$$

This example is for illustrative purposes only; frequently the uncertainty components cannot be derived directly but must rely on the mathematical manipulation of other measurable quantities. In this event, the specific error propagation formulas for the various mathematical functions, i.e., addition, subtraction, multiplication, division, exponential, etc., must be utilized. These formulas and a detailed discussion on error propagation can be found in the references cited above and other statistical and analytical references. A statistician may also be consulted.

When entering inorganic and organic analytical results, the uncertainty field associated with the result is optional for input. If the laboratory propagates uncertainties for the analytes being reported, then the uncertainty field must be used to record the uncertainty result for the inorganic or organic analyte. It is important to report all uncertainties at one standard deviation in the units of measurement, NOT in percent. Many MAPEP participants utilize EPA methodology and therefore may not routinely report uncertainties. The MAPEP, however, stresses the importance of determining the uncertainty of a measurement as outlined in the ISO, NIST, and other references cited above. Understanding the uncertainty of measurements is crucial for quality control and the improvement of radiological, stable inorganic, and organic analytical methods.

The MAPEP does not require a laboratory to calibrate for more organic components than they typically perform for other DOE work. Laboratories may utilize “less than” values for inorganic and organic target analytes to signify a calibrated component when the results are below the detection limit. Laboratories must not report a result for those components that are not routinely analyzed (i.e., leave blank). Failure to follow this rule may result in inappropriately derived performance flags for a target analyte.

## VII. PERFORMANCE EVALUATION

Acceptance criteria for MAPEP were developed from a review of precision and accuracy data compiled by other PEPs, the analytical methods literature, from several MAPEP pilot studies, and from what is considered reasonable, acceptable, and achievable for routine analyses among the more experienced laboratories. The acceptance criteria are designed to be pragmatic in approach and may be changed as warranted. The typical performance evaluation and acceptance criteria for targeted analytes are:

**For each reported radiological and stable inorganic analyte**, the laboratory result and the RESL reference value is used to calculate a relative bias:

$$\% \text{ BIAS} = \frac{(100)(\text{LABORATORY RESULT} - \text{RESL REFERENCE VALUE})}{\text{RESL REFERENCE VALUE}}$$

The relative bias places the laboratory result in one of three categories for the radiological and stable inorganic analytes:

- 1) ACCEPTABLE..... BIAS <= 20%
- 2) ACCEPTABLE WITH WARNING.... 20% < BIAS <= 30%
- 3) NOT ACCEPTABLE..... BIAS > 30%

**For each reported organic analyte**, effective December 1, 2005, the laboratory result is graded in accordance with NELAC Institute Performance Criteria as specified in the appropriate Field of Proficiency Testing (FoPT) tables associated with each matrix. The semi-volatile organics water standard is evaluated using the spiked value as the true value “T” (assigned value). The acceptance limits for this standard are generated utilizing the linear regression line found in the July 2007 Non-potable water FoPT tables. The MAPEP does not require a laboratory to calibrate for more organic components than they typically perform for other DOE work. See the above discussion regarding the use of “less than” values and when not to report results. A laboratory’s failure to detect a spiked component (i.e., a “less than” value is reported, but the component’s concentration is above that limit) is a false negative and is flagged as “Not Acceptable” (N).

**For the organics in water PE sample**, a Z-Score is generated using the calculated mean and calculated standard deviation derived from the FoPT:

$$Z\text{-Score} = \frac{(\text{LABORATORY RESULT} - \text{Calculated MEAN})}{\text{Calculated STANDARD DEVIATION}}$$

**The soil reference values for the organic target analytes** are determined as the biweight mean<sup>1,2</sup> of all laboratory data for the analyte. The acceptance limits for this standard are generated utilizing the linear regression line found in the July 2007 solids/soil FoPT tables. The uncertainties for the semi-volatile reference values are derived in accordance with these tables. Where the population mean is required for determining a reference value, the calculation used is analogous to the classical confidence interval for the mean of a population:

$$T_{bi} \pm (t_{(0.95, n-1)}) * \frac{S_{bi}}{\sqrt{n}}$$

Where  $T_{bi}$  = Biweight mean  
 $S_{bi}$  = Biweight standard deviation  
 n = Number of observations

1 Kafadar, K. (1982), “A Biweight Approach to the One-Sample Problem”, *Journal of the American Statistical Association*, 77, 416-424.

2 ISO Guide 43, *Proficiency Testing by Interlaboratory Comparisons*, Second Edition, 1997

$t_{(0.95, n-1)}$  = Student's t value at the 95% confidence interval and n-1 degrees of freedom.

**For the organics in soil PE sample**, the mean of all reported results and the standard deviation of all results (less outliers) are used to calculate a Z-Score:

$$Z\text{-Score} = \frac{(LABORATORY\ RESULT - MEAN\ OF\ ALL\ DATA)}{\text{Standard Deviation of all data OR Calculated Standard Deviation from FOT tables.}}$$

The Z-Score for the semi-volatile organics places the laboratory result in one of two categories:

- 1) ACCEPTABLE (A) .....  $0.0 < Z\text{-Score} \leq 3.0$
- 2) NOT ACCEPTABLE (N) ....  $Z\text{-Score} > 3.0$

For all results, the reported uncertainty is not currently used as part of the typical MAPEP acceptance criteria, but it is used to flag a potential area of concern. Reported uncertainties that appear unreasonably low or suspiciously high are flagged. Participants with flagged uncertainties, particularly if they are numerous, should review their methods and ensure that the reported uncertainties are appropriate.

Reported total uncertainties are used to evaluate performance in special radiological false positive/negative tests and sensitivity evaluations (see Appendix H). False positive results are a very important quality concern for DOE since they typically initiate needless investigations, require additional sampling and analysis, and are used to formulate erroneous decisions, thereby increasing DOE's liability risk and taxpayer costs. False positive/negative tests are also performed for stable inorganic and organic results. Since total uncertainties are not typically reported for these analytes, the special performance evaluations for inorganic/organic results must use a different methodology than the radiological tests.

## VIII. PERFORMANCE REPORTS

Participants will receive e-mail notification when their respective performance reports are available for review. The participant can also indicate other contacts, such as sample management offices, field offices, contracting laboratories, etc., where copies of the report should be sent. The participant's report will include the RESL reference value for the analyte of interest, the laboratory reported value, acceptance status, and the grand mean for all laboratories. Other pertinent or helpful information may also be included. MAPEP participants will not be scored or ranked. Performance, however, will be monitored and corrective actions may be called for as required. MAPEP routinely issues Letters of Concern to point out potential quality issues. It is MAPEP's intent to inform each laboratory of potential quality concerns revealed by MAPEP participation. It is the responsibility of each laboratory to investigate their consistent "NOT ACCEPTABLE" or "ACCEPTABLE with WARNING" performance evaluations. Each notified laboratory should determine the cause(s) for the identified quality concern and make the appropriate procedural changes necessary to improve future data quality.

MAPEP data will also be forwarded to the DOE-HSS Analytical Services Program Manager and other DOE-HQ contacts, DOE Field Offices, Sample Management Offices, the DOE Consolidated Audit Program (DOECAP), and other MAPEP stakeholders. DOECAP may review the overall performance of the laboratory in concert with other performance evaluation programs and identify any additional concerns. Corrective actions will strive to be more focused on technical assistance rather than punitive. They may range from simple correspondence to a DOECAP on-site assessment.

## **IX. COMMUNICATION WITH MAPEP PARTICIPANTS AND STAKEHOLDERS**

MAPEP shall communicate with participants and stakeholders primarily with notifications from email and information posted on the MAPEP websites. The communications shall include routinely scheduled items for each test session, such as enrollment periods, PE sample selection(s), shipping dates, closing dates, sample descriptions, test session instructions, individual performance reports, and final PE reports. Performance evaluation reports and program information are provided on the MAPEP public website at <http://www.inl.gov/resl/mapep/>. MAPEP participants and stakeholders may also use the MAPEP password protected website at <http://mapep.inl.gov/> where several database tools are available to track and trend historical performance, auditors can prepare for DOECAP Audits, and participants receive the MAPEP Letters of Concern regarding potential quality issues.

## **X. CRITERIA FOR LETTERS OF CONCERN**

The following provides a brief overview of the policies and processes associated with issuing and responding to a Mixed Analyte Performance Evaluation Program (MAPEP) Letter of Concern, and its significance to the Department of Energy’s Consolidated Audit Program (DOECAP).

The MAPEP issues a Letter of Concern to a participating laboratory upon identification of a potential analytical data quality problem in the MAPEP results, in order to help participants identify, investigate, and resolve potential quality issues. Letters of Concern have been issued since 1996, shortly after the beginning of the MAPEP program. A copy of the Letter of Concern is also sent to DOE/contractor oversight Points of Contact (POCs), including DOE Field Office and Headquarters POCs, and contractor Sample Management POCs. Issued to be informative and not punitive, each Letter of Concern states, *"This letter is solely intended to alert your laboratory to a potential quality concern that you may wish to investigate for corrective action."* A Letter of Concern is issued to any participating laboratory that demonstrates:

“Not Acceptable” performance for a targeted analyte in a given sample matrix for the two most recent test sessions (e.g., Pu-238 in soil test 13 “+N” (+36% bias), Pu-238 in soil test 14 “-N” (-43% bias));

“Not Acceptable” performance for a targeted analyte in two or more sample matrices for the current test session (e.g., Cs-137 in water test 14 “+N” (+38%), Cs-137 in soil test 14 “+N” (+45%));

Consistent bias, either positive or negative, at the “Warning” level (greater than +/-20% bias) for a targeted analyte in a given sample matrix for the two most recent test sessions (e.g., Sr-90 in air filter test 13 “+W” (+26%), Sr-90 in air filter test 14 “+W” (+28%));

Quality issues (flags other than “Acceptable”) that weren’t identified by the above criteria for a targeted analyte in a given sample matrix over the last three test sessions (e.g., Am-241 in soil test 12 “-N” (-47%), Am-241 in soil test 13 “+W” (+24%), Am-241 in soil test 14 “-N” (-38%));

Any other performance indicator and/or historical trending that demonstrate an obvious quality concern (e.g., consistent “False Positive” results for Pu-238 in all tested matrices over the last three test sessions).

A review period of about two weeks is provided at the close of each MAPEP test session, prior to the release of final results to DOE stakeholders and the general public, when any laboratory may question or appeal performance evaluation results. All laboratories have the opportunity to respond to a Letter of Concern by contacting the MAPEP Coordinator, and many frequently do so. In addition, laboratories can request additional MAPEP standards at any time for verification of measurement processes, and many have utilized this option.

Letters of Concern specifically address an area of significance to the DOECAP, as laboratory participation in performance evaluation (PE) programs is typically assessed during a DOECAP audit. The DOECAP QSAS, Revision 2.1, (i.e., pages 83 and 84) identifies the corrective action and documentation required for a laboratory to address PE program failure. For two consecutive failures, the laboratory is required to develop and document corrective action(s) to address the cause(s) within 21 days. Corrective action documentation must be available for review during DOECAP audits, and the same documentation should be available for any clients or other stakeholders. If the DOECAP issues a finding in the area of PE performance, including any finding derived from or associated with a MAPEP Letter of Concern, the laboratory has the opportunity to respond and perform corrective actions through the DOECAP process.

In addition to issuing Letters of Concern, the MAPEP Team provides technical assistance whenever requested, to both MAPEP participants and DOE/contractor oversight personnel. That assistance has helped resolve many quality issues, thereby improving the quality of analytical services and ultimately reducing potential DOE liability. MAPEP Letters of Concern are instrumental in this process by providing a method of communication that focuses attention on analytical performance, and when used as intended, assists laboratories and DOE/contractor oversight personnel avoid potential quality problems and/or correct quality issues in a timely manner.

It is also important to note that the DOE field site management/personnel, and/or its DOE contractor, that enter into a contractual agreement with an analytical laboratory for field data services, have an important responsibility. They are responsible for assuring that the corrective actions needed to remedy the data discrepancy, as identified by the performance evaluation and testing of MAPEP, satisfy the Department’s obligations and provide confidence in the quality, validity, and reliability of the analytical data.

## Appendix A.

### List of MAPEP Target Analytes

#### Radiochemical Analytes

Actinium-228	Americium-241	Antimony-124
Antimony-125	Barium-133	Bismuth-212
Bismuth-214	Cadmium-109	Carbon-14
Cerium-139	Cerium-144	Cesium-134
Cesium-137	Cobalt-57	Cobalt-58
Cobalt-60	Curium-244	Europium-152
Europium-154	Europium-155	Hydrogen-3
Iodine-129	Iron-55	Lead-212
Lead-214	Manganese-54	Neptunium-237
Nickel-63	Plutonium-238	Plutonium-239/240
Polonium-210	Potassium-40	Protactinium-234m
Radium-226	Radium-228	Ruthenium-106
Selenium-75	Silver-110m	Strontium-90
Sulfur-35	Technetium-99	Thallium-208
Thorium-227	Thorium-228	Thorium-230
Thorium-232	Tin-113	Uranium-234/233
Uranium-235	Uranium-238	Yttrium-88
Zinc-65	Zirconium-95	

**Appendix A (continued).**

**List of MAPEP Target Analytes**

**Stable Inorganic Analytes**

Aluminum	Antimony	Arsenic
Barium	Beryllium	Cadmium
Calcium	Chromium	Cobalt
Copper	Iron	Lead
Magnesium	Manganese	Mercury
Molybdenum	Nickel	Potassium
Selenium	Silver	Sodium
Thallium	Uranium-Total	Uranium-235
Uranium-238	Vanadium	Zinc

**Appendix A (continued).**

**List of MAPEP Target Analytes**

The MAPEP semi-volatile organics and volatile organics can be found in the NELAC PT Fields of Proficiency Testing FoPT for “Non-potable water” and “Solid and Chemical Materials”

## Appendix B.

### Typical Sample Descriptions

#### MAPEP-10-MaW23 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
<sup>241</sup> Am, <sup>238</sup> Pu, <sup>239</sup> Pu, <sup>234</sup> U, <sup>238</sup> U	< 15 Bq/L	<sup>57</sup> Co, <sup>134</sup> Cs, <sup>137</sup> Cs, <sup>55</sup> Fe, <sup>63</sup> Ni, <sup>54</sup> Mn, <sup>65</sup> Zn, <sup>60</sup> Co	< 2000 Bq/L
<sup>90</sup> Sr, <sup>99</sup> Tc	< 100 Bq/L	<sup>3</sup> H	<1000 Bq/L

NOTE: The <sup>234</sup>U and <sup>238</sup>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
<sup>238</sup> U, Total U	0.01 – 1.23 mg/L	<sup>235</sup> 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-10-OrW23 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.

## Appendix B (continued).

### Typical Sample Descriptions

#### MAPEP-10-MaS23 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
<sup>57</sup> Co, <sup>134</sup> Cs, <sup>137</sup> Cs, <sup>54</sup> Mn, <sup>65</sup> Zn, <sup>60</sup> Co, <sup>40</sup> K	< 4000 Bq/kg	<sup>55</sup> Fe, <sup>63</sup> Ni	< 2000 Bq/kg
<sup>90</sup> Sr, <sup>99</sup> Tc	< 1000 Bq/kg	<sup>241</sup> Am, <sup>238</sup> Pu, <sup>239</sup> Pu, <sup>234</sup> U, <sup>238</sup> U	< 300 Bq/kg

NOTE: The <sup>234</sup>U and <sup>238</sup>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
<sup>238</sup> U, Total U	0.1 – 24.5 mg/kg	<sup>235</sup> 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.

#### MAPEP-10-OrS23 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.

**Appendix B (continued).**

**Typical Sample Descriptions**

**MAPEP-10-RdF23 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
<sup>241</sup> Am, <sup>238</sup> Pu, <sup>239</sup> Pu, <sup>234</sup> U, <sup>238</sup> U	< 2 Bq/sample	<sup>57</sup> Co, <sup>134</sup> Cs, <sup>137</sup> Cs, <sup>54</sup> Mn, <sup>65</sup> Zn, <sup>60</sup> Co	< 10 Bq/sample
<sup>90</sup> Sr	< 4 Bq/sample		

**MAPEP-10-GrW23 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-10-GrF23 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

**Appendix B (continued).**

**Typical Sample Descriptions**

**MAPEP-10-RdV23 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; and 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. 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 the 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 40-mL sample). Since both samples are identically spiked, either sample may be used if the results are reported in Bq/sample.

**RADIOLOGICAL CONSTITUENT DESCRIPTION**

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

## Appendix C.

### Method Codes for Radionuclides

#### RADIONUCLIDES

#### \*

1. The first pair of digits designates the method of detection (instrument).

00	Alpha Spectrometry
01	Beta Counting - 2 pi gas flow proportional counter
02	Beta Counting - liquid scintillation counter
03	Gamma Spectrometry
04	Gross Alpha/Beta - 2 pi gas flow proportional counter
05	Thermal Ionization Mass Spectrometry
06	PEARLES
07	Kinetic Phosphorescence Analyzer (KPA)
08	Inductively Coupled Plasma Mass Spectrometry
99	Other

#### \*

2. The second pair of digits designates the sample preparation technique.

00	No preparation - analyzed as received
01	Evaporation, straight
02	Evaporation, acidified
03	Coprecipitation, straight
04	Coprecipitation, acidified
05	Distillation
06	Acid leaching without hydrofluoric acid
07	Wet ash - Acid digestion - the use of oxidizers to destroy organics
08	Acid dissolution by strong Aqua Regia, hydrofluoric acid, etc.
09	Total dissolution by fusion
10	Ion Exchange Chromatography / Ion Chromatography
11	EPA 900, Radioactivity, Gross Alpha/Beta Screening, 600/4-80-032
12	EPA 901, Radioactive Cesium, 600/4-80-032
13	EPA 901.1, Gamma Emitting, 600/4-80-032
14	EPA 905, Radioactive Strontium, 600/4-80-032
15	EPA 906, Tritium, 600/4/80-032
16	EPA 907, Actinide Elements, 600/4/80-032
17	EPA 908, Uranium-Radiochemical Method, 600/4/80-032
18	EPA 908.1, Uranium-Fluorometric Method, 600/4-80-032
99	Other

#### \*

3. The \* is a letter (A through G) indicating sample size (see Appendix F).

## Appendix D.

### Method Codes for Inorganic Metals (see Appendix C for code positioning)

#### INORGANIC METALS

1. The first pair of digits designates the method of detection (instrument).

00	Flame Atomic Absorption Spectrometry
01	Furnace Atomic Absorption Spectrometry (Zeeman Background Correction)
02	Radial - Inductively Coupled Plasma Emission Spectrometry
03	Axial - Inductively Coupled Plasma Emission Spectrometry
04	Inductively Coupled Plasma Mass Spectrometry
05	Cold Vapor Atomic Absorption Spectrometry
06	Hydride Generation (AAS, ICP/OES, ICP-MS)
07	DC Plasma Emission
08	Furnace Atomic Absorption Spectrometry (Deuterium Continuum Background)
09	Ion Chromatography - EPA Method
10	Flame Emission Spectrophotometry
11	Thermal Ionization Mass Spectrometry
12	Neutron Activation Analysis
13	X-ray Fluorescence
14	Hg per SW846 Method 7473 (AAS)
15	Kinetic Phosphorescence Analyzer (KPA)
99	Other

2. The second pair of digits designates the sample preparation technique.

00	No preparation - analyzed as received
01	SW846 Methods 3005, 3010, 3020, 3050 or CLP ILM03.0
02	SW846 Methods 3015, 3051 (Microwave assisted)
05	Total Metals Analysis (i.e. XRF, Fusion, neutron activation)
06	SW846 Method 3050B, Section 7.5, Increased Solubility
07	Mercury per SW846 Method 7470 or 7471
08	Mercury per SW846 Method 7473 (Thermal Decomp/AAS)
09	Mercury per SW846 Method 7474
10	EPA Method 200.2 Sample Preparation Methods
11	EPA Method 200.7 Trace Metals in Waters & Wastes
12	EPA Method 200.8 Trace Metals in Waters & Wastes
13	EPA Method 200.9 Trace Elements
99	Other

3. The \* is a letter (A through G) indicating sample size (see Appendix F).

## Appendix E.

### Method Codes for Organic Analytes (see Appendix C for code positioning)

#### ORGANIC ANALYTES

1. The first pair of digits designates the method of analysis.

00	USEPA Method 601 - Purgable Halocarbons
01	USEPA Method 602 - Purgable Aromatics
02	USEPA Method 608 - Organochlorine Pesticides and PCB's
03	USEPA Method 624 - Purgables
04	USEPA Method 625 - Base/Neutrals and Acids
05	SW-846 8021 Aromatic and Halogenated Volatiles by GC using PID and/or EC
06	SW-846 8041 Phenols by Gas Chromatography
07	SW-846 8061 Phthalate Esters by GC/ECD
08	SW-846 8081 Organochlorine Pesticides by Gas Chromatography
09	SW-846 8082 Polychlorinated Biphenyls by Gas Chromatography
10	SW-846 8091 Nitroaromatics and Cyclic Ketones by Gas Chromatography
11	SW-846 8100 Polynuclear Aromatic Hydrocarbons
12	SW-846 8121 Chlorinated Hydrocarbons by Gas Chromatography: Capillary
13	SW-846 8260 Volatile Organics Compounds by GC/MS
14	SW-846 8270 Semivolatile Organic Compounds by GC/MS
15	SW-846 8275 Semivolatile Organic Compounds (PAHs and PCBs) TE/GC/MS
16	SW-846 8310 Polynuclear Aromatic Hydrocarbons
17	SW-846 GC/GTIR for Semivolatile Organics: Capillary Column
99	Other

2. The second pair of digits designates the sample preparation technique/method.

00	No Preparation - analyzed as received
01	Separatory Funnel Liquid-Liquid Extraction (Method 3510C)
02	Continuous Liquid-Liquid Extraction (Method 3520C)
03	Soxhlet Extraction (Method 3540C)
04	Automated Soxhlet Extraction (Method 3541)
05	Supercritical Fluid Extraction (Method 3560)
06	Ultrasonic Extraction (Method 3550B)
07	Supercritical Fluid Extraction of PAHs (Method 3561)
08	Waste Dilution for Volatile Organics (Method 3585)
09	Purge-and-Trap for Aqueous Samples (Method 5030B)
10	Closed-System-Purge-and-Trap and Extraction for Volatiles (Method 5035)
11	Pressurized Fluid Extraction (Method 3545A)
99	Other

3. The \* is a letter (A through G) indicating sample size (see Appendix F).

## Appendix F.

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### Sample Size Table

For all analyte types, the '\*' in the Method Code corresponds to values in the following table:

- A less than 1 gram or 1 milliliter
- B 1 to 5 grams or 1 to 5 milliliters
- C 6 to 10 grams or 6 to 10 milliliters
- D 11 to 30 grams or 11 to 30 milliliters
- E 31 to 75 grams or 31 to 75 milliliters
- F 76 to 100 grams or 76 to 100 milliliters
- G 101+ grams or 101+ milliliters

## Appendix G.

### Mixed Analyte Performance Evaluation Program (MAPEP) Data Entry Instructions

#### PRELIMINARY CONSIDERATIONS:

The data entry software has been tested primarily with Microsoft's Internet Explorer and Netscape. Due to the multiplicity of potential Internet web browsers, products other than Microsoft's Internet Explorer or Netscape may operate the reporting software with or without issues. Laboratory personnel using other products should test their browser with the reporting software to ascertain if any issues arise.

While MAPEP is awaiting all laboratory data to be entered, the MAPEP system is read/write. **Users may enter, edit and/or delete any current data until the closing date.** After the MAPEP closing date, the reporting system becomes READ ONLY so users can ONLY review the data they have entered into the system or review previous MAPEP studies. When a new MAPEP standard is distributed, the MAPEP system will once again be ready for data entry for the new sample.

#### DATA ENTRY AND/OR EDITING:

- 1) Start your computer's Web Browser software.  
Type in the URL <http://mapep.inl.gov/>

**WARNING:** You should ***LOG OFF*** the data entry program. Simply closing your browser will not log you off the MAPEP server and additional attempts to LOG IN will fail until the system resets itself (approximately 20 minutes).

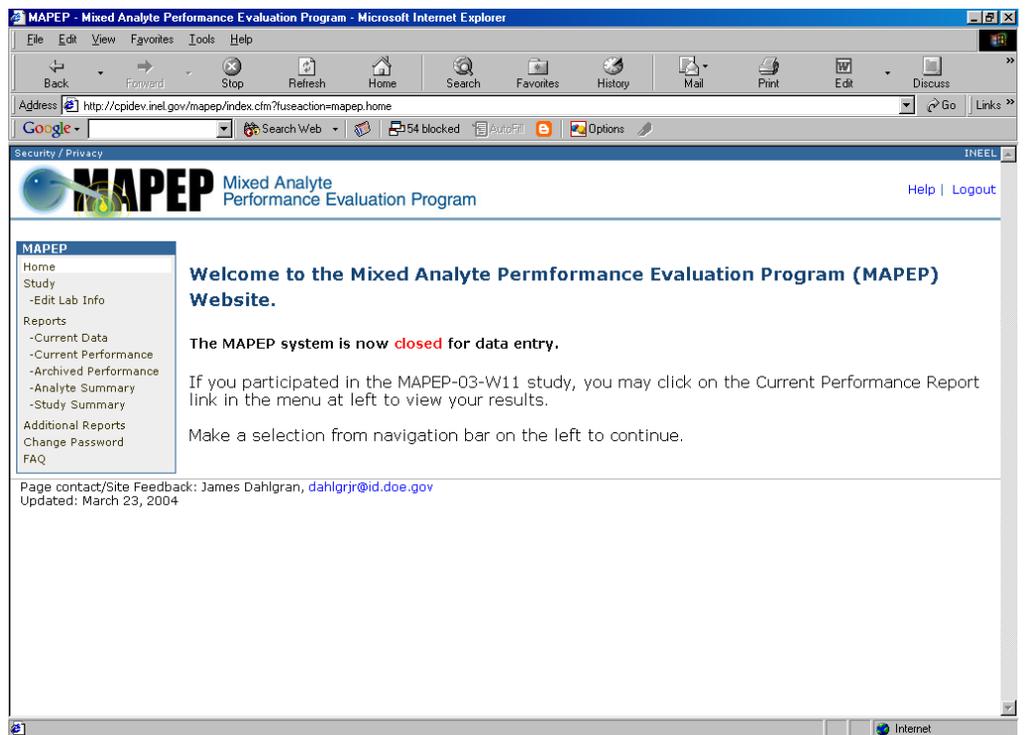
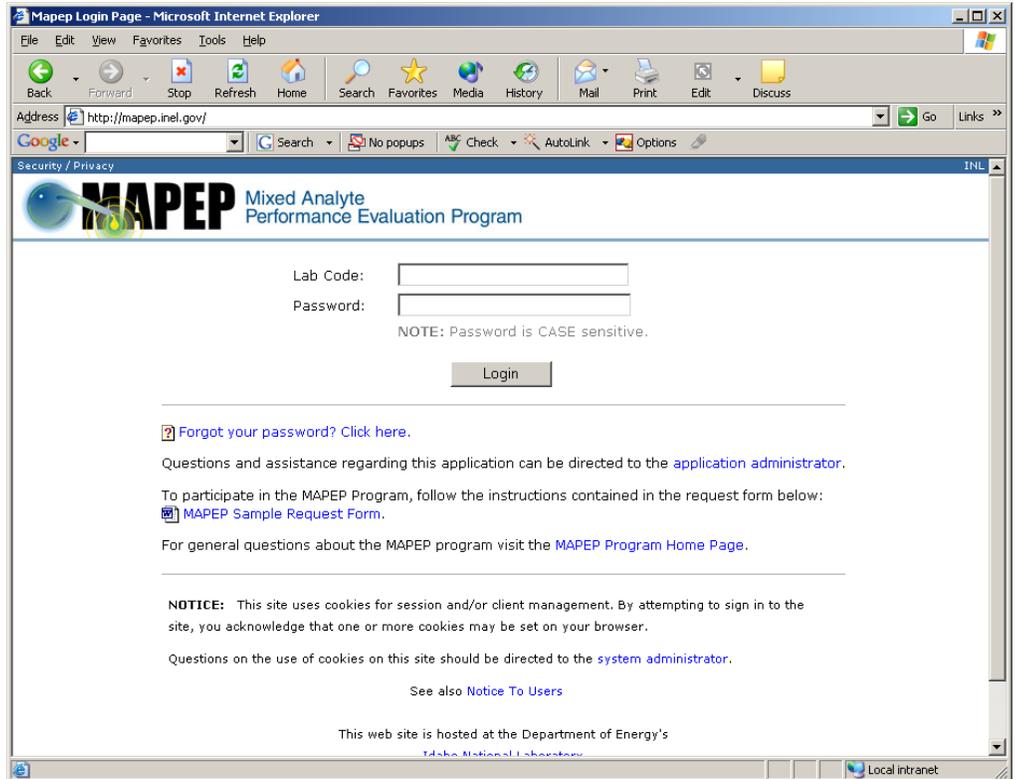
The Following Welcome screen appears:

1) Enter your Lab Code and password and then Click on the Login Button.

If you forget your password, click on that link to have the password emailed to the MAPEP point of contact.

NOTE: Laboratories now have control of their passwords and change them as they desire, HOWEVER they must meet certain security criteria (see below).

2) The Welcome Screen is displayed that will tell you the status of each of the MAPEP studies currently distributed or being evaluated.



3) Users are **REQUIRED** to maintain the Laboratory Information up to date, as this is the contact information MAPEP will use for communicating with the participants.

For each new study, the MAPEP users **MUST** validate the laboratory information before they are allowed to enter data.

To change data in a cell, click in that cell.

**DO NOT ENTER POST OFFICE BOX INFORMATION IN THE SHIPPING INFORMATION AREA.**

The participant’s NRC license or state license number, and the expiration date, must be provided for ALL United States Laboratories. If a license exemption applies, the user must enter the appropriate DOE contract number and expiration date. A U.S. Federal Laboratory (owned and operated by the federal government, i.e., the laboratory must have federal employees, not an M & O contractor) may enter any appropriate license information or select the federal laboratory option. A foreign laboratory (outside U.S. jurisdiction) will not see the NRC License request, as this option does not apply.

When users get to the shipping information, they may elect to check the “Same as Mailing Info” and/or “Same as Contact Info” to help provide information for shipping.

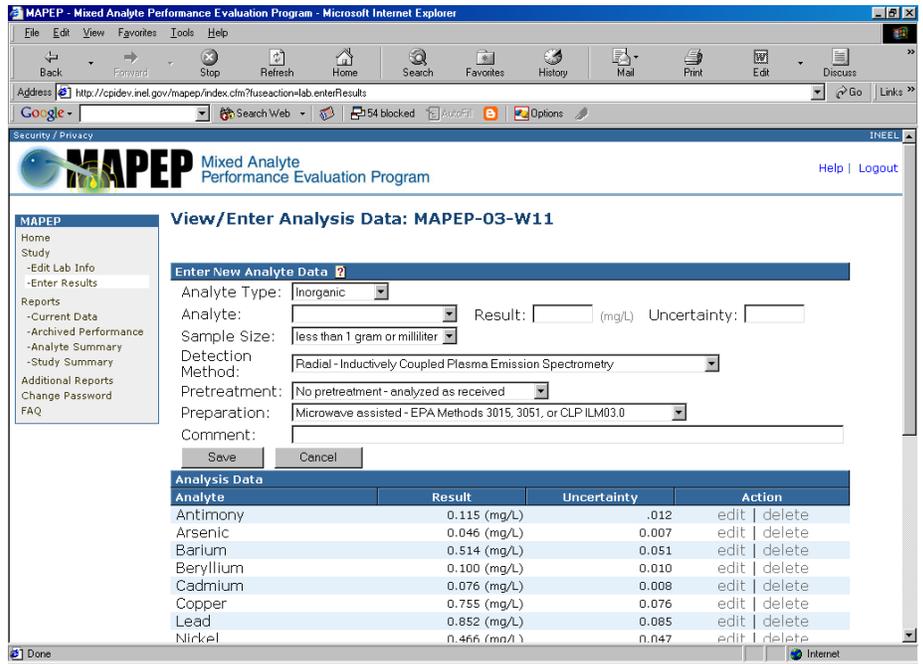
Once the user has updated their laboratory information, at the **bottom** of the screen click the **SAVE** button.

4) If your laboratory requests your MAPEP reports to be sent to other facilities, you may specify these facilities by clicking on **ADDITIONAL REPORTS** link in the menu window on the left of the screen display. Please strive to keep this information current. Reports returned to RESL due to incorrect address and/or email will result in the contact being deleted.

Users may now enter their analytical data:

5) As long as the data session is open, you may click on ENTER RESULTS on the menu bar to input or edit your results. When the data session has closed, this menu option will disappear.

Select the appropriate analyte type (radiological, inorganic, volatile, semi-volatile or pesticides) to start reporting data. The appropriate analyte list, units, and potential method codes are presented based upon the analyte type selected. The Web reporting system for MAPEP is very similar to the previous version.

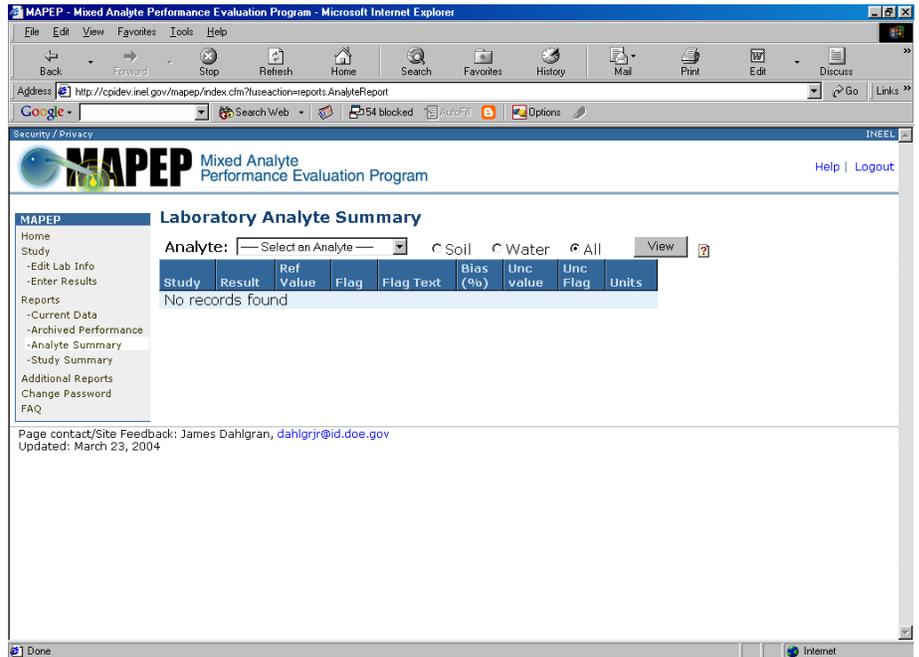


After each data point has been entered, the user must click the SAVE button at the bottom of the data entry area to save the data. The list of data entered appears below the data entry area. You will notice that to the far right of each of the analytes entered there is an “edit | delete” action button. This allows users to edit the data entered for the analyte chosen or you may delete that analyte as necessary. When the mouse pointer hovers over the name of the analyte, a small pop-up window appears that gives you details of the data you have entered.

6) From the data entry screen, you may elect to go to the REPORTS section of the Website. The user can view and/or printout reports of the currently entered data (CURRENT DATA) or historical data (ARCHIVED PERFORMANCE) reported under previous studies.

7) With this version of the MAPEP Website, users can ascertain their historical performance for any analyte they have reported earlier.

Click on the menu item



ANALYTE SUMMARY to access this function.

Clicking on the Series Identifier rather than a particular matrix will retrieve all results for that Series.

From the dropdown menu window, select an analyte you wish to review. Then select whether you wish to review this performance in water, soil or all. Finally, click the VIEW button to retrieve the analyte specific performance data.

**MAPEP - Mixed Analyte Performance Evaluation Program - Microsoft Internet Explorer**

Address: <http://cpidev.inel.gov/mapep/index.cfm>

**MAPEP Mixed Analyte Performance Evaluation Program** [Help](#) [Logout](#)

**MAPEP Laboratory Analyte Summary**

Analyte:   Soil  Water  All

Study	Result	Ref Value	Flag	Flag Text	Bias (%)	Unc Value	Unc Flag	Units
MAPEP-03-W11	290	322	A			14		(Bq/L)
MAPEP-03-S10	236	238	A			10		(Bq/kg)
MAPEP-02-W10	400	421	A			20		(Bq/L)
MAPEP-02-S9	855	862	A			30		(Bq/kg)
MAPEP-01-W9	28	28.50	A			4		(Bq/L)
MAPEP-01-S8	68.8	91.10	W			6.68		(Bq/kg)
MAPEP-00-W8	3.02	283	N			2	H	(Bq/L)

Page contact/Site Feedback: James Dahlgran, [dahlgrjr@id.doe.gov](mailto:dahlgrjr@id.doe.gov)  
Updated: March 23, 2004

An additional report STUDY SUMMARY allows users to review the historical performance of past studies. Click on this menu item to generate a report like that to the right.

**MAPEP - Mixed Analyte Performance Evaluation Program - Microsoft Internet Explorer**

Address: <http://cpidev.inel.gov/mapep/index.cfm>

**MAPEP Mixed Analyte Performance Evaluation Program** [Help](#) [Logout](#)

**MAPEP Sample Statistical Summary**

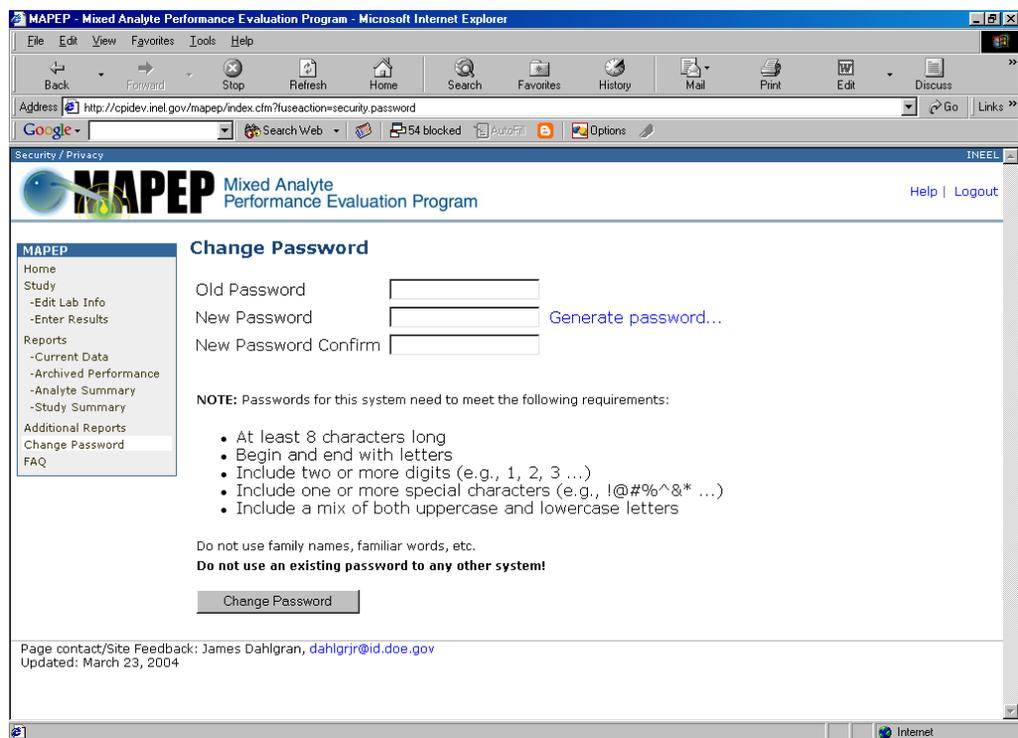
Sample ID:

Analyte	T(1)	A(2)	Grand Mean	Standard Deviation	Ref Value	Analyte Text	Acceptance Range	Units
Antimony	34	33	0.25	0.02	0.241		0.17 - 0.31	(mg/L)
Arsenic	17	17				False Positive Test		(mg/L)
Barium	36	34	0.76	0.03	0.756		0.53 - 0.98	(mg/L)
Beryllium	33	33	0.79	0.04	0.802		0.56 - 1.04	(mg/L)
Cadmium	17	17				False Positive Test		(mg/L)
Chromium	38	38	2.53	0.20	2.51		1.76 - 3.26	(mg/L)
Copper	37	37	1.93	0.13	1.95		1.37 - 2.54	(mg/L)
Lead	37	37	3.02	0.18	3.11		2.18 - 4.04	(mg/L)
Nickel	37	37	2.12	0.13	2.16		1.51 - 2.81	(mg/L)
Selenium	35	34	0.63	0.05	0.652		0.46 - 0.85	(mg/L)
Silver	32		0.06	0.03		Not Evaluated		(mg/L)
Thallium	33	33	1.40	0.09	1.4		0.98 - 1.82	(mg/L)
Uranium-Total	5	5			0.13			(mg/L)
Uranium-235	2	2			9.19E-4			(mg/L)
Uranium-238	2	2			0.129			(mg/L)
Vanadium	16	15				False Positive Test		(mg/L)
Zinc	37	37	2.46	0.16	2.46		1.72 - 3.20	(mg/L)
Americium-241	37	33	0.56	0.04	0.578		0.40 - 0.75	(Bq/L)
Cesium-134	52	51	382.30	22.36	421		294.70 - 447.30	(Bq/L)
Cesium-137	53	52	326.29	16.26	329		230.30 - 427.70	(Bq/L)
Cobalt-57	53	52	57.23	3.37	57		39.90 - 74.10	(Bq/L)
Cobalt-60	53	52	39.44	2.15	38.2		26.74 - 49.66	(Bq/L)

The MAPEP reporting system allows users to control their own passwords (within limits). Passwords must be changed or updated every six months. Passwords must meet the following criteria for security reasons:

- At least 8 characters long
- Begin and end with letters
- Include two or more digits (e.g., 1, 2, 3 ...)
- Include one or more special characters (e.g., ! @ # % ^ & \* ...)
- Include a mix of both uppercase and lowercase letters.

To change your password, click the CHANGE PASSWORD menu item and the password screen will appear. There is a Generate Password tool incorporated into this screen that will allow you to generate a compliant password if you desire. Just click on this link and a pop-up window will appear with a suggested password.



## **DATA MODIFICATION OR DELETION**

1) If it is desirable to modify or delete data entries from the data entered, Click on the “ENTER RESULTS” menu item while the study is open. The list of analytes entered will appear below the data entry area. To the far right of each of the analytes you will notice the “edit | delete” selection. Selecting the “edit” function will allow you to edit the data entered for this analyte. Selecting the “delete” function will delete the analyte from the list of analytes reported and from the database.

## **LOG OFF**

To exit the MAPEP data entry program, select LOG OFF from upper right menu bar. Your data and information will be saved for your update and/or review at anytime.

**DO NOT CLOSE YOUR BROWSER PROGRAM (WINDOW) UNTIL YOU HAVE LOGGED OFF. DOING SO MAY LOCK YOU OUT OF ADDITIONAL SESSIONS FOR 20 MINUTES UNTIL THE SERVER RESETS ACCESS.**

Keep the password, instructions, and any hard copy in a secure location. If you have problems or questions, please call Guy M. Marlette at (208) 526-2532 or e-mail [MAPEP@INEL.GOV](mailto:MAPEP@INEL.GOV). Include your lab code/user id with all communications.

## Appendix H.

### Mixed Analyte Performance Evaluation Program (MAPEP) Performance Evaluation (PE) Material Production and Verification

**MAPEP PE samples shall meet these requirements and general characteristics for each MAPEP test session:**

#### **Preparation and Production of MAPEP samples:**

Whole-volume PE samples for each sample matrix shall be prepared in sufficient quantities to provide all the participating laboratories the PE samples needed for the test session. Additional PE samples shall be required for homogeneity, verification, and stability testing and extra PE samples shall be available in storage for additional sample requests. The whole-volume MAPEP PE material shall be traceable to NIST.

- MAPEP PE samples shall use radiological, stable inorganic, and organic analytes in the same soil PE sample; water PE samples shall use radiological and stable inorganic analytes in the same PE sample. This not only ensures a more representative real-world mixed analyte sample, but also provides an efficient means for laboratories to demonstrate their analytical proficiencies. Organics in water are prepared separately (see below).
- MAPEP is performance based and shall not dictate the analytical methods used. MAPEP shall not prescribe the sample size used, count time, or other analytical parameters.
- MAPEPP participants shall use their routine analytical procedures for the analysis of MAPEP PE samples. Participants shall, however, also be allowed to use MAPEP samples to test and develop new analytical methods.
- MAPEP PE samples shall use only whole-volume PE material. Participants shall not receive a concentrated volume of PE material that requires subsequent dilution to achieve some specified final volume or concentration. For example, if participants are sent a 5-mL ampoule of concentrated material and directed to dilute the ampoule to a final 1-L volume, the participant can analyze the concentrated portion as well as the diluted portion and compare results. Whole-volume PE material prevents this possibility and ensures that the PE material is treated the same as a real-world sample.
- MAPEP PE samples shall use real-world natural ground or surface water and soil samples spiked with mixed-analytes (radiological and stable inorganic) that are traceable to the National Institute of Standards & Technology (NIST). MAPEP PE samples shall also use real-world air filters and vegetation spiked with radionuclides that are traceable to NIST. Organic analytes shall use vendor certified standards if NIST traceable standards are unavailable.
- MAPEP PE samples shall use multi-analyte mixtures for radiological, stable inorganic, and organic PE material preparation. MAPEP shall not use single-analyte PE material for any PE sample matrix.
- MAPEP PE samples shall be homogeneous, reproducible, and stable for the time required to conduct the MAPEP test session (at a minimum). Specific information about homogeneity testing is given below.
- MAPEP PE samples shall use a representative number of target analytes from those found in Appendix A (see below). The total number of targeted analytes for each analyte category

(i.e., radiological, stable inorganic, and organic) and for each sample matrix may exceed, but shall not be significantly less than, the number of targeted analytes listed in the Sample Descriptions in Appendix B.

- MAPEP PE samples shall use constituents that cause known analytical and preparatory interferences in addition to the target analytes. Participants shall be tested in the application of any necessary interference corrections.
- MAPEP samples shall test for coincident summing corrections in gamma-ray spectrometry.
- MAPEP PE material shall be verified with the same gamma-ray detectors and counting geometries that are used to demonstrate NIST traceability.
- MAPEP mixed analyte soil (MaS) PE samples shall demonstrate homogeneity with selected radionuclides such that individual 1-g aliquots of soil from each batch of mixed analyte PE material of about 50,000 grams do not vary by more than 5% from the known NIST reference values.
- Radioactivity shall be homogeneously distributed over the entire area of each MAPEP PE air filter.
- The radioactivity of each individual radionuclide shall not vary by more than 1.0% among the MAPEP air filter PE samples. Radioactivity among the vegetation PE samples also shall not vary by more than 1.0%.
- MAPEP PE material shall challenge the routine analytical capability of participants in the areas of chemical and radiochemical interferences, measurement accuracy and precision, measurement sensitivity, and false positive/negative results (see below).
- MAPEP PE samples shall use low-energy beta emitters, including Ni-63 and Fe-55, in both the water and soil matrices. Both of these radionuclides are of interest to DOE for testing low-energy beta analytical methods.
- MAPEP PE samples shall use Tc-99 in the water and soil matrices. The Tc-99 must be homogeneously distributed in addition to the other radionuclides of interest and remain chemically stable, non-volatile, and have a NIST traceable reference value. Tc-99 is an important radionuclide of interest for DOE and shall be included in the performance evaluations for these matrices.
- MAPEP PE samples shall use refractory plutonium from time to time among the various test sessions and PE sample matrices. Refractory plutonium and its analysis is an important quality issue for DOE environmental programs and analytical performance.
- MAPEP PE samples shall use antimony-125 routinely in soil and shall test to ensure participants use analytical methods for increased solubility during sample preparation, such as digestion with hydrochloric acid and nitric acid. EPA-HQ states in a letter to MAPEP that inorganic methods for the determination of antimony in soil must use increased solubility techniques and that the failure to do so is unacceptable.
- MAPEP PE samples shall test for specific analytical capabilities that are of importance for DOE analytical services. Participants that fail to meet the MAPEP acceptance criteria shall not be excused for poor performance, even if the majority of other participants also choose a poor methodology and fail. This is especially true for refractory plutonium, antimony-125 in soil, and other problem analytes where poor analytical performance is associated with inappropriate methodology.
- The MAPEP PE samples shall be verified with radiochemical sample dissolution techniques that shall guarantee total dissolution of the PE sample and the dissolution of any refractory

constituents contained in the sample. Sample dissolution techniques that use acid leaching shall not be used. Total dissolution techniques are required to ensure accurate verification of the reference values.

- The MAPEP PE samples shall be verified with radiochemical procedures that use sequential chemical separation procedures for the determination of the actinides. Sequential separation procedures are required to ensure that consistent analytical results are obtained from the same sample aliquot.
- The MAPEP PE samples shall be verified with radiochemical procedures that use perchloric acid to ensure the complete wet oxidation of organic material. Other analytical methods cannot perform the wet oxidation as completely or as quickly as perchloric acid, and both factors are important to the quality of the verification process.
- MAPEP PE samples shall be prepared for false positive/negative testing and sensitivity evaluations in each test session.
- MAPEP PE samples shall ensure that MAPEP test sessions vary in complexity over time. Each test session shall be unique with varying PE sample parameters. PE samples shall vary in the choice of target analytes, specific analyte concentrations, interferences, isotopic ratios, refractory PE material, natural/depleted/enriched uranium, analytes targeted for false positive/negative testing or sensitivity evaluations, choice of matrix material, and other sample parameters.
- MAPEP PE samples shall rotate the radiological, stable inorganic, and organic analytes of interest for accuracy, sensitivity, and false positive/negative testing in the PE sample matrices for each PE test session to ensure complexity and variability among test sessions.
- A radiological, stable inorganic, or organic master spiking solution that contains all targeted analytes for a given PE sample matrix shall not be diluted or concentrated and used in a subsequent PE sample matrix.
- The variation in MAPEP PE sample complexity shall ensure that MAPEP test sessions are not substantially duplicated and reference values cannot be derived from previous test sessions, or from a ratio of the reference values used in a previous test session for any of the PE sample matrices.
- MAPEP PE samples shall use target analyte concentrations that are typically well above detection limits, but specific analytes shall be tested at relatively low concentrations from time to time among test sessions to provide variety and complexity in the PE material.
- MAPEP PE samples for gross alpha/beta measurements in water and air filter matrices shall use Th-230 and Sr-90 or other equivalent radionuclides that will ensure that only alpha and beta measurements can be performed. For example, Am-241 and Cs-137 shall not be used for gross alpha/beta PE samples because they emit gamma rays that can be used by gamma-ray spectrometry to make the measurement.

#### **Measurement Traceability of PE Samples:**

MAPEP reference values for the target analytes in the PE samples shall be traceable to NIST and proof of the NIST traceability shall be maintained. Uncertainties shall be calculated for all reference values according to the ISO/IEC/OIML/BIPM Guide to the Expression of Uncertainty in Measurement: 1995, NIST Technical Note 1297, 1994, or other authoritative standard references.

- MAPEP PE samples shall use scientifically valid and legally defensible reference values with associated uncertainties and documented verification data according to ILACG13:2000

(see below).

- MAPEP PE sample results shall be evaluated with scientifically defensible acceptance criteria.
- The reference value for radiological and stable inorganic analytes shall be calculated from the NIST certified standard value and the standard dilution(s) used. The reference value shall not be determined by the experimental analysis of the sample. Rarely, a radiological or stable inorganic reference value is derived from sample characterizations in accordance with ISO Guide 43. Reference values for organic analytes are derived from vendor certified standards, if NIST traceable standards are not available, and procedures that are used in accordance with ISO Guide 43 (see below).
- Total uncertainties for the reference values shall not be determined empirically, but shall be determined by mathematical error propagation of the uncertainty of the NIST certified standard value and the uncertainty associated with the standard dilution(s) used in constructing the sample. Therefore, the total uncertainty for the radiological and stable inorganic reference values shall be minimized because they are based on mathematical calculation and not experimental error.

**MAPEP shall utilize the individual analytes that are listed in Appendix A of the MAPEP Handbook. There are seven major matrix/analyte categories:**

1. Mixed Analyte Soil (MaS) matrix. MAPEP shall use a natural soil characterized for background activities of target radionuclides and background concentrations of target inorganics compounds.
2. Mixed Analyte Water (MaW) matrix. MAPEP shall use natural occurring water (well, sub-surface, surface, spring, river, lake, etc.) that has been characterized for background activities of target radionuclides and background concentrations of target inorganic analytes. The MaW water shall not be prepared from deionized or distilled water.
3. Organic Analyte Water (OrW) matrix. MAPEP shall use water that contains no background organic compounds.
4. Organic Analyte Soil (OrS) matrix. MAPEP shall use soil that contains no background organic compounds.
5. Radiological analytes in a vegetation (RdV) matrix. MAPEP shall use vegetation that is a natural occurring grass-type matrix that has been characterized for background radionuclide activities.
6. Radiological analytes in an air filter (RdF) matrix. MAPEP shall use 47-mm glass fiber filters that have been characterized for background radionuclide activities.
7. Gross alpha/beta radionuclides in water (GrW) matrix. MAPEP shall use natural occurring water that has been characterized for background radionuclide activities.
8. Gross alpha/beta radionuclides in air filter (GrF) matrix. MAPEP shall use 47-mm glass fiber filters that have been characterized for background radionuclide activities.

**Specific Activities and Concentrations for Analytes Listed in Appendix A.** The target analyte specific activity or concentration is typically well above detection limits, but the amount of PE material provided for each participant is, however, limited. Therefore, the specific activity and

concentration ranges indicated in the sample description shall be used to select the optimum quantity of sample for each analysis.

**Guidelines for Radiological Specific Activities:**

- Specific activities for target radionuclides shall be representative of levels expected in the DOE Complex, for DOE-site characterization, remediation, environmental monitoring, and long-term stewardship. Specific activities shall span the range of the radiological methods and instrumentation used in these environmental programs.
- Specific activities shall not exceed Department of Transportation (DOT) shipping regulations for non-radioactive shipments. MAPEP PE samples shall not be shipped as DOT radioactive material.
- Specific activities shall be sufficient for most radionuclides to provide less than 10% (and typically less than 5%) counting uncertainty with a reasonable sample size and count time.

**Guidelines for Inorganic/Organic Analyte Concentrations:**

- Stable inorganic/organic analyte concentrations shall not exceed the Resource Conservation and Recovery Act (RCRA) limits for hazardous material. The unaltered (except for acid neutralization) MAPEP PE sample shall not become a RCRA mixed-waste upon disposal.
- The semi-volatile organic analyte concentrations shall be based on the NELAC Institute Performance Criteria Field of Proficiency Testing tables.
- Lower concentration limits for stable inorganic analytes shall be based on the EPA’s Contract Laboratory Program (CLP) Quantitation Limits (ILM05.3 SOW), but shall not limit the use of false positive/negative testing and sensitivity evaluations for the inorganic analytes. For example, see the inorganic water matrix (MaW) sample description in Appendix B.
- Stable inorganic analyte concentrations shall also be dependent on the target analytes of interest and the instrument/method of analysis. For example, refer to the target analyte quantitation levels as described in the EPA’s CLP ILM05.3 SOW.
- Analyte concentrations shall be sufficient to allow measurement uncertainties of 5-10% for most stable inorganic/organic analytes.

**False Positive/Negative Testing and Sensitivity Evaluations.** False positive/negative testing and sensitivity evaluations shall be used in radiological, stable inorganic, and organic performance evaluations. The specific analytes used for testing shall vary among PE test sessions.

**Radiological Analytes:**

The radiological false positive/negative and sensitivity evaluation tests are based in part on information found in ANSI N42.23 and several measurement uncertainty articles by Lloyd A. Currie.

- 1) The MAPEP program shall use false positive testing to identify laboratory results that indicate the presence of a particular radionuclide in a MAPEP sample when, in fact, the actual activity of the radionuclide is far below the detection limit of the measurement. Not Acceptable ("N") performance, and hence a false positive result, is indicated when the range encompassing the result, plus or minus the total uncertainty at three standard deviations, does not include zero (e.g., 2.5 +/- 0.2; range of 1.9 to 3.1). Statistically, the probability that a result can exceed the absolute value of its total uncertainty at three standard deviations by chance alone is less than 1%. MAPEP uses a three standard deviation criterion for the false positive test to ensure confidence about issuing a false positive performance evaluation. A result that is greater than three times the total uncertainty of the measurement represents a statistically positive detection with over 99% confidence.
- 2) Sensitivity evaluations shall also be routinely performed to complement the false positive tests. In a sensitivity evaluation the radionuclide is present at or near the detection limit, and the difference between the reported result and the MAPEP reference value is compared to the propagated combined total uncertainties. Again, the results are evaluated at three standard deviations. If the observed difference is greater than three times the combined total uncertainty, the sensitivity evaluation is "Not Acceptable". The probability that such a difference can occur by chance alone is less than 1%. If the participant did not report a statistically positive result, a "Not-Detected" is noted in the text field of the MAPEP performance report. A non-detect is potentially a false negative result, dependent upon the laboratory's detection limit for the radionuclide.
- 3) False negative tests shall also be performed in combination with the sensitivity evaluations. In this scenario, the sensitivity of the reported measurement indicates that the known specific activity of the targeted radionuclide in the PE sample should have been detected, but was not, and a "Not Acceptable" performance evaluation is issued. The uncertainty of the MAPEP reference value and of the reported result at three standard deviations is used for the false negative test.
- 4) The false positive/negative and sensitivity evaluation tests shall be conducted in a manner that assists the participants with their measurement uncertainty estimates and helps ensure they are not under estimating or over inflating their total uncertainties. If the total uncertainty is over inflated to try to pass a false positive test, it will result in a "Not Detected" if the test is actually a sensitivity evaluation, and vice versa for a false positive test. False negatives and failed sensitivity evaluations can also result from under estimating the total uncertainty. An accurate estimate of measurement uncertainty is required for consistent performance at the acceptable level.

#### **Stable Inorganic/Organic Analytes:**

Total uncertainties are not currently required for stable inorganic/organic results; therefore, the radiological false positive/negative tests that use measurement uncertainty cannot be applied to inorganic/organic analyses. Contamination, spectral interferences, retention time shift, and isomer misidentification are common causes of false positive/negative results for inorganic/organic analyses.

- 1) MAPEP shall perform inorganic false positive tests by requesting results for an analyte listed on the PE sample description, but intentionally not included at detectable levels in the MAPEP PE sample. Participants shall also report results for an analyte only if the analyte's concentration exceeds the lower concentration range listed on the MAPEP sample description. For example, see the note following the inorganic water matrix (MaW) sample description in Appendix B.
- 2) MAPEP shall test for false positive/negative results with common analytical problems associated with reporting errors. For example, MAPEP shall construct PE samples that shall contain isomers of analytes that elute closely and possess certain common mass ions or high levels of transition metals that exhibit potentially interfering spectral lines. Excess phthalate esters or elemental sulfur also interferes with the analysis of organic analytes.
- 3) MAPEP shall perform sensitivity evaluations and false negative tests by spiking inorganic target analyte concentrations three to five times above the instrument detection limit (EPA's CLP ILM05.3 SOW).
- 4) MAPEP shall test for false positive/negative organic results for each PE test session. MAPEP has determined that some analytical laboratories may not calibrate for all the components present in the target analyte list in U.S. EPA SW-846 Method 8270 "Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Technique". For this reason, MAPEP shall use reported "less than" values to represent calibrated target components and empty result fields shall imply that these components are not part of the facilities normal calibration.
- 5) For the organics in water standards: a laboratory that does not report a target analyte whose concentration is known to be greater than the laboratory reported "less than" value shall be issued a "False Negative" and "Not Acceptable" performance evaluation. A laboratory that reports results for a target analyte known not to be present, and other participants substantiate the analyte's absence, shall be issued a "False Positive" and "Not Acceptable" performance evaluation.
- 6) For the organics in soil standards: a laboratory that does not report a target analyte whose concentration is known to be greater than the laboratory reported "less than" value, and other participants substantiate the analyte's presence, shall be issued a "False Negative" and "Not Acceptable" performance evaluation. A laboratory that reports results for a target analyte known not to be present, and other participants substantiate the analyte's absence, shall be issued a "False Positive" and "Not Acceptable" performance evaluation.
- 7) Misidentification of isomers (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene) will be flagged as "Not Acceptable".
- 8) Both high and low concentrations in an analyte category shall be included to evaluate if the participants analyze field samples at project-required dilutions. Excessive interference between closely eluted compounds of substantial concentration difference that requires

unique or non-routine treatments shall not be used.

**The MAPEP Program Coordinator shall hold organizational meetings to determine the following for each test session (Series):**

- Establish the matrix source for the water PE samples: 1) surface, groundwater, spring, stream, etc., 2) assess the collection methods, 3) determine the collection quantity, 4) determine the collection date/time, and 5) assess any preservation and characterization issues.
- Establish the matrix source for the soil PE samples: 1) use the existing source or identify a new source, 2) if a new source is needed, assess the collection options, methods, and time frame 3) determine the PE sample quantity, and 4) assess any characterization issues for the diluent background soil.
- Establish the matrix source for the vegetation PE samples: use the existing source or identify a new source, 2) if a new source is needed, assess the collection options, methods, and time frame, 3) determine the PE sample quantity, and 4) assess any characterization issues for the diluent background vegetation.
- Establish the matrix source for the air filter PE samples: 1) use the current air filters or identify a new source, 2) determine the PE sample quantity, and 3) assess any characterization issues for the air filter matrix.
- Establish the matrix source for the semi-volatile organic water matrix, determine the PE sample quantity, and assess any characterization issues as outlined above.
- Establish the matrix source for the gross alpha/beta water and air filter matrices, the PE sample quantities, and any characterization issues as outlined above.
- Update as needed any changes to the volumes/mass and number of PE samples required for each PE matrix.
- Ensure equipment and laboratory supplies required for PE sample construction and shipping are available (e.g., check soil blender operation, balance calibrations, procurement of NIST traceable standards, ultra-pure acids, sample containers, packing material, shipping boxes, etc.).
- Ensure PE sample parameters are modified to challenge the participants' analytical performance and ensure variation among MAPEP test sessions. For example, identify potential target analytes for false positive/negative testing, incorporate or remove refractory target analytes, alter the plutonium isotopic ratios, vary the amount of natural/depleted/enriched uranium, add chemical interferences, vary analyte concentrations, etc.
- Establish the timetable for PE sample construction and shipping.
- Incorporate any guidance from DOE-HSS, process improvements, or lessons learned from the previous test session(s).
- Discuss feedback from participants and stakeholders with the MAPEP Team, identify areas for improvement, implement program enhancements, and resolve any quality problems.
- Issue work assignments, identify action items, and resolve other issues as needed.

**Radiological, Stable Inorganic and Organic Technical Leads shall be responsible for**

**the following in each test session (Series):**

- Determine the final target analyte list for each PE sample matrix.
- Determine the final concentrations/specific activities of the targeted analytes.
- Identify the analytes/matrices for false positive/negative testing.
- Identify the analytes/matrices for sensitivity evaluations.
- Procure the required NIST traceable standards for radiological/stable inorganic PE material construction. Procure vendor certified standards for organic PE material construction.
- Review the target analyte list and concentration/activity ranges for the MAPEP Sample Descriptions. Correct the Sample Descriptions as necessary for the current MAPEP test session (Series).
- Perform calculations and required standard dilution(s) for spiking PE material. Ensure traceability to NIST and certified standards is maintained.
- Prepare required items for the construction of the final whole-volume MAPEP PE material.
- Perform measurement uncertainty estimates for the MAPEP PE material construction.

The MAPEP Coordinator shall work and consult with the Technical Leads and serve as the primary point of contact for external communications with MAPEP participants, stakeholders, and the DOE-HSS Analytical Services Program Manager. The MAPEP Coordinator shall be the primary interface with RESL management and is responsible for MAPEP Team operations and overall MAPEP program administration at the RESL level. The MAPEP Coordinator shall approve participant applications, MAPEP reports, MAPEP letters, and significant MAPEP program enhancements or modifications. The MAPEP Coordinator shall facilitate and coordinate work among the three analytical sections of the MAPEP Team with the assistance of the three MAPEP Technical Leads. Each Technical Lead shall be the primary point of contact for their area of expertise and shall be the subject matter expert in this technical area for the MAPEP Team. The Technical Leads are responsible for the day-to-day operations of their respective analytical sections.

**PE Sample Verification:**

MAPEP shall verify the reference values for the MAPEP PE samples of each test session (Series) according to the ILAC G13:2000 requirements and the additional following requirements:

- Radiological Reference Value Verification:

Target radionuclides shall be verified by alpha, beta, or gamma analyses. Radiochemical sample dissolution techniques shall guarantee total dissolution of the sample and dissolution of any refractory compounds contained in the sample. Sample dissolution techniques that use acid leaching as the primary method of dissolution shall not be used. Sequential chemical separation procedures shall be used for the determination of the actinides to ensure that consistent analytical results are obtained from the same sample aliquot. Perchloric acid shall be used safely and on a routine basis to ensure the complete wet oxidation of organic material. The analytical results from the chemistry procedure shall verify the NIST traceable reference value if the analytical result +/- the associated total uncertainty includes the reference value at a 95% (two standard deviation) confidence level. Reference values that include the background concentration of analytes shall also include the uncertainty of the measurement process.

- Inorganic Reference Value Verification:

Target analytes shall be verified by standard inorganic analytical methods. Reference values that include the background concentration of analytes shall also include the total uncertainty of the measurement process. The analytical results from the chemistry procedure shall verify the NIST traceable reference value if the analytical result +/- the associated total uncertainty includes the reference value at a 95% confidence level or the analytical result is within 10% of the calculated NIST traceable reference value.

- Organic Reference Value Verification:

The PE sample composition shall be verified before utilization for the creation of PE samples. Initial verifications ensure that there are no gross errors in the PE sample production process and serve as a baseline for evaluation of laboratory performance. MAPEP organics shall use these guidelines to complete verification: verify the composition of the PE sample spiking solution for the water matrix or the prepared final soil standard with a definitive method typically used for proficiency testing (usually gas chromatographic mass spectrometry). Although the composition of the spiking solution for the PE water samples is well known, conduct initial verification to ensure that the mean measured concentrations +/- 10% of prepared values are analytically within acceptance ranges of analytical errors (including preparation and measurement errors).

### **Homogeneity Testing for the MAPEP Mixed-Analyte Water and Mixed-Analyte Soil Samples:**

MAPEP samples shall be homogeneous so that the variability among PE samples shall not contribute significantly to the variability of the results among participant laboratories. MAPEP shall verify the homogeneity of PE material with statistical evaluations of randomly selected PE samples taken from across the range of samples prepared in the PE material production batch. The statistical evaluations shall demonstrate that variability within, and among PE samples, is within acceptable levels. The alpha probability level will be set at 0.05. This means the probability of

Type I error, or rejecting a true null hypothesis (i.e., concluding sample heterogeneity when the observed variability is due to chance alone) will not exceed 5%. Statistical confidence limits shall be set at the 95% level. Radiological results shall be within the statistics of the measurement at two standard deviations. In addition, the specific activity of selected radionuclides shall demonstrate that individual 1-gram aliquots of soil from each batch of mixed analyte PE material do not vary by more than 5% from the known NIST reference values. The statistical methods used for homogeneity testing shall be based on ILAC G13:2000 and ISO Guide 43. For example, see “THE INTERNATIONAL HARMONIZED PROTOCOL FOR THE PROFICIENCY TESTING OF ANALYTICAL CHEMISTRY LABORATORIES”, *Pure Appl. Chem.*, Vol. 78, No. 1, pp. 145–196, 2006.

Indicator analytes, if used, must be carefully selected. Actinides are typically among the most difficult analytes to distribute homogeneously in a soil, and therefore shall be among the indicator analytes of choice. For the semi-volatiles, the phenolic compounds will be monitored for homogeneity, but shall not be a primary indicator for PE sample homogeneity due to their known reactivity and/or poor extraction efficiencies. If the indicator analytes or a majority of the homogeneity data demonstrate excessive variation in the PE material, a second set of PE samples shall be analyzed. If homogeneity is still questionable, the sample shall be re-blended and the homogeneity testing repeated. If necessary, the PE material shall be discarded and a new PE batch created.

#### **Homogeneity Testing for the MAPEP Radiological Vegetation and Air Filter Samples:**

MAPEP air filters and vegetation PE samples are prepared by individually spiking each PE sample with the target analytes of interest. MAPEP air filter and vegetation PE material is not prepared with a batch methodology. Furthermore, participants are instructed to analyze the entire PE sample; the PE sample cannot be subdivided. Since the PE samples are individually prepared and the entire PE sample is analyzed, variability within the PE sample is not a factor that can influence a participant’s results. Therefore, homogeneity testing for within sample variability is not required for the air filter and vegetation PE material. In addition, since the PE samples are individually spiked and not prepared in a batch, any variability among samples cannot be a function of heterogeneity within a batch material or heterogeneity from dispensing the PE material itself. Therefore, homogeneity testing among samples is not required, at least not from a batch standpoint. Variability among samples can only be a factor if the master spiking solution is not homogeneous, or if the spiking quantity is not reliably reproduced. MAPEP shall ensure that the activity on each air filter sample is homogeneously distributed over the entire area of the filter. The MAPEP verification analyses shall also demonstrate the homogeneity of the master spiking solution and the reproducibility of the PE sample spikes. The verification/homogeneity testing shall demonstrate that aliquots from the master spiking solution used for the PE material are statistically identical at the 95% (two standard deviation) confidence level. Furthermore, the variability of the spikes among vegetation and air filter samples shall not exceed 1%.

**Stability testing for radiological and stable inorganic analytes:**

Radiological and stable inorganic PE samples shall have stability testing performed according to the criteria in ILAC G13:2000 and ISO Guide 43. The results of the stability test shall verify the reference value within the statistics of the measurement at the 95% (two standard deviation) confidence level.

**Stability testing for organic analytes:**

Organic compounds are stable for the length of the test session, but analyses must be performed in accordance with holding time requirements. Therefore, stability testing is not applicable for organic analytes.