

# 7 EVALUATING METHODS AND LABORATORIES

## 7.1 Introduction

This chapter provides guidance for the initial and ongoing evaluation of radioanalytical laboratories and methods proposed by laboratories. Appendix E, *Contracting Laboratory Services*, provides additional guidance on the initial laboratory evaluation. More details about evaluating and overseeing a laboratory's performance can be found in ASTM E1691 and ASTM E548.

The performance-based approach to method selection allows a laboratory the freedom to propose one or several methods for a specific analyte/matrix combination that will meet the needs of the Analytical Protocol Specifications (APSs) and measurement quality objectives (MQOs) delineated in the Statement of Work (SOW). However, the laboratory should demonstrate, through a method validation process, that the method is capable of producing analytical results of quality that meet the needs of the SOW (Chapter 5). Guidance and recommendations on the selection of an analytical method based on the performance-based approach were presented in Chapter 6. Section 7.2 of this chapter provides guidance on how to evaluate the methods proposed by a laboratory. Section 7.3 provides guidance on the initial evaluation of a laboratory, and Section 7.4 discusses the continual evaluation of the quantitative measures of quality and operational aspects of the laboratory once sample processing has commenced.

Method applicability and performance compliance should be demonstrated prior to the initiation of the sample analyses, as well as during the project period. A defined logical process for demonstrating and documenting that the analytical method selected meets the project's data needs and requirements may involve, for example, a review of the method validation documentation, an evaluation of past performance data from other projects (if available), the analysis of external performance evaluation (PE) program results, the analysis of matrix-specific standard reference materials (or method validation reference materials) sent during the initial work period and throughout the project, and the final evaluation of the protocol's performance during the data verification and validation process. Chapter 8, *Radiochemical Data Verification and Validation*, covers the final evaluation of the protocol's performance.

In addition to the evaluation of the analytical methods, the capability of the laboratory to meet all SOW requirements needs to be reviewed and evaluated. Supporting information, such as method validation documentation, safety manuals, licenses and certificates, and quality manual are typically submitted with the response to the Request for Proposals (RFP). A generic evaluation of the laboratory operation may be conducted during the initial laboratory audit or assessment. This may be an initial onsite audit. This first evaluation covers those generic SOW requirements dealing with the laboratory's capability and operation, including verification of adequate

35 facilities, instrumentation, and staffing and staff training and qualifications. Following the first  
36 audit, emphasis should be on ensuring the laboratory continues to meet the APSs through a  
37 continuous or ongoing evaluation effort.

## 38 **7.2 Evaluation of Proposed Analytical Methods**

39 A laboratory may submit several methods for a particular APS contained in the SOW, but each  
40 method should be evaluated separately and, if appropriate, approved by the project manager or  
41 designee. The method should be evaluated to be consistent with the overall analytical process  
42 that includes the proposed field sampling and preservation protocols (Chapter 1). The project  
43 manager may delegate the method review process to a technical evaluation committee (TEC) that  
44 has a radioanalytical specialist. MARLAP recommends that a radioanalytical specialist review  
45 the methods for technical adequacy. The acceptance, especially of a new method, may be the  
46 most critical aspect of the performance-based approach for method selection. Acceptance of the  
47 method requires the project manager to verify that the method is scientifically sound.

48 Each step of the method should be evaluated by a radioanalytical specialist in order to understand  
49 how the results are derived. These steps may involve sample digestion, analyte purification and  
50 decontamination steps that use ion exchange, solvent extraction, precipitation or oxidation/  
51 reduction applications. Once these steps have been reviewed, and the method evaluation data  
52 (e.g., from method validation documentation or various performance evaluation results) confirm  
53 that the proposed method is acceptable, the project manager should have the confidence  
54 necessary to endorse and verify the use of the method in the analysis of the routine samples.

55 As discussed in Chapter 6, the laboratory should provide method validation and analytical data  
56 that demonstrates method performance. The data should show conclusively that the proposed  
57 method meets the requirements as defined by the APSs. If method performance is questionable,  
58 additional data may be required. For such cases, the project manager may decide to send per-  
59 formance testing (PT) materials to the laboratory in order to evaluate or validate the method. The  
60 preparation of the PT material used to evaluate the method should be based on sound scientific  
61 principles and representative of the expected sample matrix (see Chapter 6 on method validation  
62 options using site-specific materials). If there is sufficient reason to believe that the PT material  
63 is an adequate substitute for the sample matrix and that the laboratory will follow the same  
64 method, then the need to justify each step in the method may be drastically reduced.

### 65 **7.2.1 Documentation of Required Method Performance**

66 Certain documentation submitted by the laboratory with the proposed methods, as well as

67 available external information on the laboratory's analytical performance, should be reviewed  
68 and evaluated by the radioanalytical specialist. Table 7.1 outlines where such information can be  
69 typically found by the TEC. This section will discuss various information categories that may be  
70 available during the method evaluation process.

#### 71 7.2.1.1 Method Validation Documentation

72 Chapter 6 outlines the various method validation options that can be specified by the project  
73 manager. In the MARLAP process, the method validation requirements will be contained in the  
74 SOW. The laboratory must submit the necessary method validation documentation consistent  
75 with the SOW specification. The laboratory may choose to validate a method to a higher degree  
76 of validation or to submit method validation documentation for a higher degree of validation than  
77 that specified by the SOW. The radioanalytical specialist or project manager should review the  
78 documentation to ensure that validation criteria for the number of analyte concentration levels  
79 and replicates meet or exceed the required validation criteria (Chapter 6, Table 6.1). Although  
80 not specified in the method validation protocol, some laboratories may include chemical and  
81 analytical interferences in their method validation plan to gain a perspective on the method's  
82 specificity and ruggedness. However, it should be noted that the graded approach to method  
83 validation presented in Chapter 6 does inherently increase the degree of ruggedness in terms of  
84 having the method address site-specific materials which may include chemical and radionuclide  
85 interferences.

86 In addition to reviewing the documentation for compliance with the method validation protocol,  
87 the results of the method validation process should be evaluated to determine if the project  
88 specific MQOs will be met. The method validation may or may not have been specifically  
89 conducted for the project at hand. When the method has been validated (Chapter 6, Section 6.6)  
90 to the SOW specifications (validation level and MQOs), then evaluation of the documentation  
91 can be straight forward. If the method has been previously validated for the MQOs of other  
92 projects, then the laboratory should provide a justification and calculations to show that the  
93 method validation results will meet the MQOs for the new project. The TEC should verify these  
94 calculations and review the assumptions and justifications for reasonableness and technical  
95 correctness.

#### 96 7.2.1.2 Internal Quality Control or External PE Program Reports

97 The documentation of internal QC and external PE program results should be reviewed relative  
98 to the MQOs. Method uncertainty and internal biases can be estimated from the information  
99 available in the laboratory's internal quality control reports, summaries of batch QC results that

TABLE 7.1 — Cross reference of information available for method evaluation

Evaluation Element Addressed	Method Validation	Internal and External QC Reports	External PE Programs	Internal/External QA Assessments	Information from RFP and Other Sources
Analyte/Matrix	●				●
Process Knowledge					●
Previous Experience					●
Radiological Holding Time	●	○	○	●	●
Turnaround Time		○	○	●	●
Unique Process Specifications	●				●
Bias	●	●	●	○	●
Method Uncertainty (MQC)/MDC	●	●	●	○	●
Analyte/Interference Range	●	●	●		●
Method Ruggedness	●	○	●	●	●
Method Specificity	●	○	●	●	●

● Denotes that the information relevant to method evaluation should be present.

○ Denotes that the information relevant to method evaluation may be present.

100 may be submitted with the RFP response and external PE program reports. The TEC should  
101 review these documents and, when possible, estimate the method uncertainty and bias for various  
102 analyte concentration levels. However, it is imperative that no confusion exists in terms of what  
103 method produced the results: the proposed method or another method available to the laboratory.  
104 This is especially important when reviewing external PE program results. It should also be noted  
105 that although a laboratory may meet performance acceptance criteria for an external PE program,  
106 this fact may have no bearing on whether the method will meet the MQOs of the SOW.

107 Review of the internal batch QC data can provide additional information on typical sample  
108 analysis times and rates of blank contamination and sample reanalysis. This information is  
109 important when comparing methods (from the same or between laboratories) in terms of APS  
110 characteristics. The frequency of blank contamination would be very important to national char-  
111 acterization studies (groundwater or soil analyses) for the determination of ambient analyte  
112 levels. Method evaluation for these projects may weight the blank contamination rate more  
113 heavily than other SOW parameters. The rate of sample re-analysis would be important to  
114 projects having pending operations that are conducted based on a short sample processing turn-  
115 around time (TAT). In some site remediation projects, the contractor may remain onsite pending  
116 analytical results. A delay in reporting data or not meeting a TAT due to sample re-analysis may  
117 be costly. Projects of this nature may weight TAT and low sample re-analyses more heavily than  
118 other SOW parameters.

#### 119 7.2.1.4 Method Experience, Previous Projects, and Clients

120 When permitted by former clients, the laboratory may submit information relative to the previous  
121 or ongoing clients and projects for which the proposed method has been used. The TEC should  
122 verify with the laboratory's clients that the laboratory has previous experience using the method.  
123 When available and allowed, the information should also include the analyte(s) and interferences  
124 and their applicable concentration range, matrix type, and project size in terms of the number of  
125 samples per week or other time periods. From this information, the TEC can evaluate whether or  
126 not to contact the laboratory's client for further information on the operational adequacy of the  
127 method. The client may offer some information on the quality of the results based on their  
128 external single- or double-blind QC program, percent completion of reports, TAT, and sample re-  
129 analysis frequency. The sharing of laboratory assessment reports may be advantageous when  
130 reviewing the performance of the laboratory during its employment of the method.

#### 131 7.2.1.5 Internal and External Quality Assurance Assessments

132 When available, internal and external quality assurance assessment reports should be evaluated to

133 determine the adequacy of the method performance based on previous projects. Problems with  
134 the conduct of the method due to procedural and technical issues may be readily evident. These  
135 issues may include an ineffective corrective action program creating delayed remedies to  
136 problems, insufficient understanding of the method, inadequate training of staff, internal and  
137 project-specific QC issues, and higher-than-expected failure rates for sample TATs and re-  
138 analyses. Information in these reports may disclose problems with a particular method that are  
139 not common to another proposed method. As such, the TEC may give one method a higher  
140 weighting factor than another method.

## 141 **7.2.2 Performance Requirements of the SOW—Analytical Protocol Specifications**

142 Under the performance-based approach to method selection, a laboratory will propose one or  
143 several analytical methods that can meet the stated APSs and MQOs in the SOW for a given  
144 analyte and matrix combination. Chapters 3, 5, and 6 discuss the APSs and MQOs in detail in  
145 terms of their basic description, their inclusion in a SOW, and as key considerations for  
146 identifying existing validated methods or developing new methods. The purpose of this section is  
147 to provide guidance on what available information should be evaluated in order to approve the  
148 various proposed methods.

149 The following subsections cover key aspects of the SOW that should be addressed during the  
150 method evaluation and approval process.

### 151 7.2.2.1 Matrix and Analyte Identification

152 The TEC should review the method(s) proposed by the laboratory to determine if the method  
153 under evaluation is applicable for the analyte/matrix combination specified in the SOW. In some  
154 cases, several methods may be proposed, including gross screening methods and specific  
155 radionuclide or isotopic methods having high specificity and ruggedness (Section 6.5.1.1 has  
156 additional guidance). Each method should be evaluated on its own application and merit. When  
157 methods are proposed by the laboratory that use alternative nuclides (such as decay products) to  
158 determine the analyte of interest, the TEC should carefully review the objective or summary of  
159 the method to determine if the proposed method is truly applicable for the analyte of interest  
160 given the radiological holding time and MQOs (i.e., can it properly quantify the analyte of  
161 interest through decay progeny measurements?). For gross screening techniques, the TEC should  
162 evaluate the analyte's decay scheme to determine the underlying gross radiation category (beta,  
163 alpha, X-ray, or gamma-ray emitting) and the applicability of the proposed method's radiation  
164 detection methodology.

165 Each proposed method should be evaluated to determine if the method can analyze the sample  
166 matrix identified in the SOW. A method validated for water cannot be applied to soil samples  
167 without modification and validation (Section 6.5). The planning team should have made—  
168 through historical process knowledge, previous matrix characterization studies or common  
169 experience—a determination on the uniqueness of the site-specific matrices compared to typical  
170 matrices and provided guidance in the SOW as to the level of method validation. In addition, if  
171 the radioanalytical specialist of the project planing team is concerned that the physiochemical  
172 form of the analyte or the sample matrix substrate may present special problems to the radio-  
173 analytical process, a detailed description of the analyte and matrix should have been included in  
174 the SOW. Chapters 12 and 13 discuss possible sample matrix problems and Section 6.5 provides  
175 guidance on the need for method validation. The radioanalytical specialist should carefully  
176 review the summary of the method to determine if the proposed method is applicable for the  
177 sample matrix.

178 At this point, if it is determined that the proposed method(s) is not applicable and cannot meet  
179 the SOW specifications, there is no need to continue the method evaluation process.

#### 180 7.2.2.2 Process Knowledge

181 The radioanalytical specialist should review the process knowledge information and determine if  
182 the proposed method is capable of addressing these issues by virtue of its specificity, ruggedness  
183 and applicability. Discussions on method specificity and ruggedness may be found on in  
184 subsections on pages 7-13 and 7-15, respectively.

185 As discussed in Section 6.5.2 and above, process knowledge is extremely important for identify-  
186 ing potential radioanalytical problems on some projects. Historical information or process  
187 knowledge may identify chemical and radionuclide interferences, expected analyte and inter-  
188 fering radionuclide concentration ranges, sample analyte heterogeneity issues, and the physio-  
189 chemical form of the analyte, and the sample matrix substrate. In some special cases, it may be  
190 necessary to determine if the radiological holding time will be an issue if the laboratory must  
191 analyze an alternative nuclide to determine supported and unsupported radionuclides (decay  
192 progeny nuclides) in the matrix.

#### 193 7.2.2.3 Radiological Holding and Turnaround Times

194 The radioanalytical specialist should review the proposed method in light of the radiological  
195 holding time, analyte's half-life and typical sample delivery options and determine if the method  
196 is capable of meeting the MQOs in a reasonable counting period given the typical method param-

197 eters (such as sample weight processed, chemical yields, radiation detection efficiency, branching  
198 ratio and background, ingrowth periods for decay progeny analysis, etc.). Radiological holding  
199 time is defined as the time between the sample collection and the end of the sample analysis (end  
200 of final measurement), while sample processing TAT refers to the time between sample receipt at  
201 the laboratory and the issuance of an analytical report. The physical (analyte's half-life) and  
202 chemical (stability or preservation concerns) characteristics of the analyte, as well as biological  
203 degradation for some matrices, usually will dictate the radiological holding time. Project-specific  
204 schedules and practicalities related to project and laboratory processing capacities normally enter  
205 into establishing TATs. If the radiological holding time appears to be a critical issue, then the  
206 laboratory should submit information on the typical batch size being processed by the method.  
207 This information is needed in the method evaluation and review process. Without special  
208 problems (e.g., inadvertent delay of sample delivery), the laboratory should be able to meet the  
209 MQOs with a good margin of error for the majority of the samples processed. For very short-  
210 lived analytes, too large a batch size may result in the last samples in the batch having difficulty  
211 in meeting the radiological holding time. For short-lived analytes, counting the sample (or final  
212 processing products) longer typically is not practical because the analyte is decaying too rapidly  
213 to make any gain counting the sample longer.

214 In some cases, the laboratory may want to propose two methods for a short-lived analyte: one for  
215 normal delivery and processing schedules and another method for situations when lower detec-  
216 tion limits are needed. An example of such a situation is the analysis of <sup>131</sup>I in environmental  
217 media. A method with adequate detection limits for reasonable radiological holding times is  
218 gamma spectrometry. Another method that can be applied for lower detection limits or longer  
219 radiological holding times is radiochemical separation followed by beta-gamma coincidence  
220 counting.

221 Certain projects may be concerned with the chemical speciation of the analyte in the sample. For  
222 these projects, the radiological holding time should have been specified to ensure that the chem-  
223 ical species are not altered prior to processing. The project normally should specify chemical  
224 preservation specifications applicable at the time of sample collection.

225 In the case of biological media, sample deterioration (Chapter 10) may become a problem, and  
226 biological preservatives should be added to the sample to retard degradation. However, the  
227 radiological holding time should be specified to limit problems with sample degradation. The  
228 radioanalytical specialist should evaluate the method in light of the foregoing information and  
229 determine its adequacy to meet the radiological holding time and the pertinent MQOs

230 A laboratory's sample (processing) TAT for a method typically is not related to the method's

231 technical basis unless the radiological holding time and the TAT are nearly equal for a short-  
232 lived analyte. However, sufficient time should be available between the completion of sample  
233 analysis and the delivery of the analytical report. Meeting the radiological holding time but  
234 failure to meet the TAT will not affect the quality of the analytical results but may place a  
235 hardship on the project to meet schedules. The TEC should review the proposed method, the  
236 radiological holding time and the TAT to determine if the method can process the samples in a  
237 reasonable time period to meet the TAT. The sample delivery rate, sample batch size, level of  
238 data automation and the laboratory's existing sample processing capacity will affect the  
239 laboratory's ability to meet the TAT requirement.

#### 240 7.2.2.4 Unique Processing Specifications

241 The TEC should review the proposed methods for compliance or applicability to unique sample  
242 processing specifications stated in the SOW. Chapter 6 provides a limited discussion on what a  
243 project may identify as unique or special sample process specifications. Examples may include  
244 chemical speciation, analyte depth profiles, analyte particle size distribution, analyte hetero-  
245 geneity within the sample, wet-to-dry analyte concentration ratios in biologicals, and possible  
246 scaling factors between radionuclides in the sample. In some cases, the proposed method(s) for  
247 the analyte(s) may have to be evaluated with respect to all analytes or other sample preparation  
248 specifications in order to determine method applicability and adequacy.

#### 249 7.2.2.5 Measurement Quality Objectives

250 Method performance characteristics (Method Uncertainty, Quantification Capability, Detection  
251 Capability, Applicable Analyte Concentration Range, Method Specificity, and Method  
252 Ruggedness) will be discussed in the following subsections. For a particular project, MQOs  
253 normally will be developed for several (but not all) of the performance characteristics discussed  
254 below.

#### 255 METHOD UNCERTAINTY

256 The SOW should specify the required method uncertainty at a stated analyte concentration (or  
257 activity level) for each sample matrix and the level of method validation (Section 6.6) needed to  
258 qualify the method at the stated analyte concentration.

259 MARLAP uses the term "method uncertainty" to refer to the predicted uncertainty of a result that  
260 would be measured if a method were applied to a hypothetical laboratory sample with a specified  
261 analyte concentration. As presented in Chapter 6 and formulated in Chapter 19, the method

262 uncertainty of the analyte concentration for a given method is determined by mathematically  
263 combining the standard uncertainties of the many input quantities (parameters), involved in the  
264 entire radioanalytical process. This will involve making some assumptions and normally involve  
265 using typical or worst case values for a conservative estimate of the method uncertainty. Some of  
266 these input quantities, and thus the method uncertainty, vary according to analyte level or concen-  
267 tration in the final measured product; others do not. In some cases, the magnitude of the method  
268 uncertainty for an analyte may increase in proportion to the magnitude (concentration/activity) of  
269 any interfering radionuclide present in the final measurement product. Therefore, it is imperative  
270 that the TEC evaluate the laboratory's submitted documentation relative to this requirement,  
271 especially the information provided on method specificity, given the historical or expected inter-  
272 fering nuclides and the needed decontamination factors (chemical separation factors) to render a  
273 good measurement for the analyte of interest.

274  
275 In evaluating the documentation relevant to meeting the method uncertainty requirement, it is  
276 important to determine if the method validation requirements stated in the SOW have been met.  
277 The TEC should review the submitted method validation documentation and verify that the  
278 method's performance meets the requirements of Table 6.1 (Chapter 6) for the specified valida-  
279 tion level. It is important that the laboratory submit definitive documentation of method  
280 validation compliance for the method uncertainty requirement.

281 The method performance documentation may include documentation or data from method  
282 validation, internal or external (organization sending QC samples) QC data, external PE program  
283 data, and results of pre-qualifying laboratories by sample analyses. By evaluating the actual QC  
284 and PE program performance data, it can be determined if the quoted measurement uncertainty  
285 for a reported QC sample result (calculated by the laboratory) truly reflects the method uncer-  
286 tainty under routine processing of samples. The required method uncertainty can be viewed as a  
287 target value for the overall average measurement uncertainty for the samples at a specified  
288 analyte concentration. It is important that the precision, as calculated from repeated measure-  
289 ments, is consistent with the laboratory's stated measurement uncertainty for a given sample  
290 result whose analyte concentration is near the specified concentration. If the quoted measurement  
291 uncertainty of a QC or test measurement is quoted to be  $\pm 10$  percent and QC or PE program data  
292 indicates a data set standard deviation of  $\pm 20$  percent, then the laboratory may not have  
293 identified all possible uncertainty components or may have underestimated the magnitude of a  
294 component.

## 295 QUANTIFICATION CAPABILITY

296 A requirement for the quantification capability of a method and the required method validation

297 criteria may be specified in a SOW. The quantification capability, expressed as the minimum  
298 quantifiable concentration (MQC), is the smallest concentration of the analyte that ensures a  
299 result whose relative standard deviation is not greater than a specified value, usually 10 percent.

300 The project manager or TEC should review available documentation on the method to determine  
301 if the laboratory can meet the method quantification requirement. Method validation documen-  
302 tation sent by the laboratory should demonstrate explicitly, or by extrapolation, that the method,  
303 using certain input quantities and their uncertainties, can meet the quantification requirement.  
304 The method validation acceptance criteria presented in Section 6.6 have been formulated to eval-  
305 uate the MQC requirement at the proper analyte concentration level, i.e., action level or other  
306 specified analyte concentration.

307 Some projects may send performance testing material spiked at the MQC level as a more in-  
308 depth verification of the compliance with this requirement. Laboratories may also submit docu-  
309 mentation for internal QC or external PE program results that cover the MQC value. The TEC  
310 should evaluate the reported results to determine if the MQC requirement can be met.

#### 311 DETECTION CAPABILITY

312 A radiochemical method's detection capability for an analyte is usually expressed in terms of  
313 minimum detectable concentration (MDC) or activity (MDA). Chapter 19 provides the definition  
314 and mathematical equations for the MDC<sup>1</sup> and MDA. A MDC requirement for each analyte/  
315 matrix combination may be stated in a SOW. Any proposed method should document the basis  
316 and equation for calculating the MDC. The supporting documentation on the method should  
317 contain the input quantity values that may be entered into the MDC equation to calculate the  
318 detection capability under a variety of assumptions. The TEC should evaluate the assumptions  
319 and parameter values for reasonableness and practicality. This evaluation is especially important  
320 for recently validated methods that have a limited routine processing history. It is recommended  
321 that the TEC perform an independent calculation of the method's MDC using laboratory-stated  
322 typical or sample-specific parameters.

323 When the proposed method has been validated recently or previously used on similar projects,  
324 sufficient data should exist that either are directly related to testing the method's detection capa-  
325 bility or can be used to estimate the method's detection capability. Any data submitted that  
326 document direct testing of the method's detection capability should be reviewed for appropri-  
327 ateness or applicability, reasonableness, and accuracy. If method detection testing is performed, it

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<sup>1</sup>The MDC should not be confused with the concept of the critical value (Chapter 19).

328 normally will be for one analyte concentration level or value. It should not be expected that the  
329 MDC testing process included varying the magnitude of the method's many parameters over a  
330 wide range.

331 The reported quantitative results of the blanks can be used to estimate the MDC to within a  
332 certain degree of confidence (for most methods). At or below the MDC value, the majority of the  
333 measurement uncertainty typically is due to the Poisson counting uncertainty. For well-controlled  
334 methods, the uncertainties of the other method parameters (input quantities), such as sample  
335 weight, detection efficiency, and chemical yield, may range up to 10 percent. Therefore, a simple  
336 rule of thumb to estimate the MDC for most methods involves reviewing the measurement  
337 uncertainty for the reported blank results. If the blanks were analyzed to meet the MDC  
338 requirement, then the reported MDC (based on blank and sample paired observations) for most  
339 methods should be between 3 and 4 times the measurement uncertainty of the blank when the  
340 background counts (per measurement interval) are greater than 10. It is more complicated to  
341 estimate the MDC for methods that use low background detectors (such as alpha spectrometry)  
342 having background counts less than 10 per counting interval. The TEC should evaluate the blank  
343 data to determine the reasonableness of the quoted MDC values. These rules of thumb can be  
344 applied to actual samples when the quoted analyte concentration value is less than two times its  
345 associated combined standard uncertainty value.

#### 346 APPLICABLE ANALYTE CONCENTRATION RANGE

347 The applicable analyte concentration range can vary substantially depending on whether the  
348 project deals with process waste streams, environmental remediation or monitoring, or environ-  
349 mental or waste tank characterization research. The proposed method being evaluated should  
350 provide accurate results over the analyte concentration range stated in the SOW. Acceptable  
351 analytical results used in this context means consistent method uncertainty (at a given analyte  
352 concentration) and without significant bias. The range may be over several decades, from a  
353 minimum value (the MDC for some projects) to 100 times the action level or MQC.

354 Due to the effects of the Poisson counting uncertainty, most methods will provide more precise  
355 results at higher analyte concentration levels compared to those concentration levels near zero. At  
356 concentration levels near zero, background effects will render the results less precise. If the  
357 background (instrument or ambient levels of analyte in the matrix) is not well characterized, a  
358 bias may also exist. For projects or programs (environmental characterization research) that have  
359 no action level requirement, the lower portion of the required concentration range or the MDC  
360 requirement may be most important. For those situations, particular emphasis should be placed  
361 on evaluating method and reagent blank data (i.e., net results that take into account inherent

362 analyte content in the reagents or tracers) to ensure that a bias does not exist. Refer to Section  
363 7.2.2.6, “Bias Considerations,” on page 7-15 for additional guidance.

364 Typically, radiation detection systems are linear in signal response over a very large range of  
365 count rates. However, depending on the magnitude of the chemical or radionuclide interferences  
366 in the sample, the method may not produce linear results over the entire application range.  
367 Therefore, it is critical that when a mixture of radionuclides is present in a sample, the method  
368 must provide sufficient “analyte selectivity/isolation or impurity decontamination” to ensure  
369 valid results and “method linearity.” In some cases, such as that for pure beta-emitting analytes,  
370 the degree of needed decontamination from other interfering nuclides may be as much as six  
371 orders of magnitude.

372 There are several sources of information available from the laboratory that should be reviewed  
373 and possibly evaluated to ensure the method is capable of meeting this MQO. These include  
374 method validation documentation, previous projects or experience using the method, PE program  
375 results, internal and external QC sample results, and pre-qualifying test samples. When evalua-  
376 ting the data, the TEC should evaluate the method’s performance as a function of analyte concen-  
377 tration with and without interferences. However, this evaluation would be most valid when the  
378 samples were processed to the same MQO (especially MDC or MQC), a situation that may not  
379 be realistic for different projects. If the MDC requirement results in a longer counting time from  
380 one project to another, there may be an impact on the method’s uncertainty for a given analyte  
381 concentration due to difference in the Poisson counting uncertainty. Bias typically is not affected  
382 by increasing the counting time. A graphical plot of this data would be visually helpful and may  
383 be used to determine if the method uncertainty requirement would be met at the action level  
384 (extrapolation may be necessary).

#### 385 METHOD SPECIFICITY

386 Method specificity refers to the ability of the method to measure the analyte of concern in the  
387 presence of other radionuclide or chemical interferences. The need for or degree of method  
388 specificity depends on the degree or magnitude of the interferences and their effect on the ability  
389 to measure the analyte of interest. Gross alpha, beta, and gamma-ray methods are considered to  
390 be methods of low specificity and are used when individual nuclide specificity is not possible or  
391 needed. Radiochemical methods involving sample digestion, purification and decontamination  
392 steps followed by alpha spectrometry, such as for <sup>239</sup>Pu in soil, are considered methods of high  
393 specificity. However, the relative degree of specificity of these nuclide specific methods depends  
394 on the number of analyte isolation and interference decontamination steps. High resolution  
395 gamma-ray spectrometry employing a germanium (Ge) detector is considered to have better

396 specificity than the lower resolution sodium iodide (NaI) gamma-ray spectrometry.

397 The TEC should evaluate the proposed methods for adequacy to meet the specificity require-  
398 ments stated in the SOW. As mentioned in Chapter 6, methods of low specificity, such as gross  
399 radiation detection methods, may be proposed if the methods meet the MQOs. For example,  
400 when a single analyte having a relatively elevated action level needs to be evaluated, such as <sup>60</sup>Co  
401 in soil at an action level of 26 Bq/kg (0.7 pCi/g), then a method with less specificity (gross  
402 counting methods for gamma-ray or beta emitting nuclides) may be sufficient to meet the MQOs.  
403 For this example, a less expensive NaI gamma-ray spectrometric analysis with a lower resolution  
404 capability may be more desirable compared to a more costly high resolution germanium gamma-  
405 ray spectrometric analysis. If greater method specificity for a certain analyte/matrix combination  
406 has been required in the SOW, then a high resolution non-destructive sample analysis method  
407 (such as high resolution gamma-ray spectrometry) or a destructive sample analysis by a detailed  
408 radiochemical method would be appropriate. For proposed methods of high specificity, it is  
409 important that the TEC review and evaluate the basic purification and decontamination steps of  
410 the method, or the resolution of the radiation detection system, for adequacy in relation to the  
411 expected mixture of analytes and interferences. For radiochemical methods, the TEC may be able  
412 to estimate the needed distribution/partition coefficients, extraction and solubility factors, etc., of  
413 the various purification steps and compare the values against the needed decontamination factors  
414 for the interfering chemical or radionuclide interferences.

415 The adequacy of method specificity can be evaluated by the analytical results from the analysis of  
416 site-specific PT materials during method validation and/or laboratory pre-qualifying tests. A  
417 further discussion on the use of these materials is presented below.

#### 418 METHOD RUGGEDNESS

419 Method ruggedness refers to the ability of the method to produce accurate results over wide  
420 variations in sample matrix composition and chemical and radionuclide interferences, as well as  
421 when steps (such as pH adjustments) in the method are varied slightly by the analyst. For some  
422 projects, the matrix composition and level of analyte or interferences may vary dramatically in a  
423 given project.

424 Ruggedness studies have been defined by EPA (1998). A testing protocol for method ruggedness  
425 has been outlined by the American Public Health Association (APHA). Some laboratories may  
426 have developed methods according to the APHA protocol for method ruggedness or are using  
427 methods contained in standards methods (APHA, 1989). Documentation on any internal  
428 ruggedness study may be available from the laboratory.

429 As mentioned in Chapter 5 and 6, the use of site-specific PT materials is a means of testing the  
430 ruggedness of a method for a defined project. If ruggedness and method specificity are concerns  
431 due to the sample matrix of a defined project, then a variety of site-specific performance testing  
432 materials should be sent to the laboratory as part of the pre-qualification process or as a method  
433 validation requirement. National PE programs, such as DOE's Multiple Analyte Performance  
434 Evaluation Program (MAPEP) and Quality Assessment Program (QAP), use generic PT  
435 materials and may not be applicable or representative of the matrices for a defined project. The  
436 results of the pre-qualifying or method validation processes using site-specific PT materials  
437 should be evaluated by the TEC to determine the adequacy of the method to meet this MQO  
438 parameter. If the sample matrix and analytes are fairly standard, then no other evaluation of the  
439 available information may be necessary.

#### 440 7.2.2.6 Bias Considerations

441 The method proposed by the laboratory should produce analytical results that are unbiased.  
442 MARLAP considers bias to be a persistent difference of the measured result from the true value  
443 of the quantity being measured, which does not vary if the measurement is repeated. Normally,  
444 bias cannot be determined from a single result or a few results (unless the bias is large). Bias may  
445 be expressed as the percent deviation in (or deviation from) the "known" analyte concentration.  
446 Since bias is estimated by repeated measurements, there will be an uncertainty in the calculated  
447 value. It is incumbent upon the project manager or TEC to evaluate the proposed methods for  
448 possible bias over the applicable analyte concentration range. A laboratory should eliminate all  
449 known biases before using a method. However, there may be circumstances, such as the  
450 processing of site-specific sample matrices, that may produce some inherent bias that is difficult  
451 to assess or correct in a reasonable time or economical fashion. For the methods proposed, the  
452 project manager must determine if the magnitude of the bias will significantly affect the data  
453 quality.

454 A bias can be positive or negative. Methods may have a bias at all analyte concentration levels  
455 due to the improper determinations of chemical yield, detector efficiency or resolution, subtrac-  
456 tion of interferences, and improper assumptions for the analyte's half-life or an emission  
457 branching ratio. When reporting an analyte concentration based on a decay progeny analysis,  
458 improper ingrowth assumptions may lead to a bias.

459 It is recommended that the project manager or TEC evaluate the available data provided by the  
460 laboratory or from performance evaluations for bias, based on multiple analyses covering the  
461 applicable analyte concentration range. One means of estimating a bias is through the evaluation

462 of external PE program data.<sup>2</sup> For proper evaluation of the PE program sample results, it is  
463 essential that the PE program provider use sample preparation techniques that will produce  
464 performance testing (PT) samples (or a sample distribution) having insignificant “within or  
465 between” sample analyte heterogeneity and whose analyte concentrations are accurately known.

466 For the purpose of evaluating whether a laboratory method has an observable bias based on  
467 multiple laboratory internal QC samples (matrix or method spikes) or external PE program  
468 samples, the following equations can be used:

$$D_i = 100 * \left( \frac{X_i - Y_{i \text{ known}}}{Y_{i \text{ known}}} \right) \quad (7.1)$$

469 where  $D_i$  is the percent deviation,  $X_i$  is an individual analytical result and  $Y_{i \text{ known}}$  is the “known”  
470 value for the sample analyzed. The  $D_i$  should be determined for each test sample in the data set.  
471 The mean percent deviation for the method for a series of analyses in the data set can be  
472 estimated by the equation:

$$\bar{D} = \frac{\sum_{i=1}^N (D_i)}{N} \quad (7.2)$$

473 Refer to various references (ASTM D2777, NBS 1963, Taylor 1990) for applicable tests that may  
474 be performed to determine if there is a statistical difference at a given significance level.

475 There may be a negative or positive bias at low analyte concentrations due to the improper  
476 determination of the appropriate detector background or analytical blank value. For an individual  
477 blank result, the result (net activity or concentration value) would be considered to be a  
478 statistically positive value if the magnitude of its value is greater than 1.65 times the quoted  
479 measurement uncertainty. An older, much more conservative approach was to consider a reported  
480 value as a positive value when the magnitude of a result was greater than 3 times the measure-  
481 ment uncertainty.

482 Since the measurement process is statistical in nature and involves the subtraction of an  
483 appropriate background or blank which also has an uncertainty, there is a 50 percent probability

---

<sup>2</sup> In order to standardize against the national standard (NIST), an external performance evaluation program should be implemented by a well-qualified provider that has standardized its reference materials to NIST or is participating in a NIST traceability program

484 (half of the results) that the analytical result for a blank sample will have a negative magnitude,  
485 e.g.,  $-1.5 \pm 2.0$ . For an individual blank measurement, the measurement may be considered to be  
486 problematic when the negative magnitude is greater than 2 or 3 times the measurement  
487 uncertainty.

488 For most radionuclides, other than those that are naturally occurring, the major source of a  
489 positive blank is from contamination, either cross-contamination from other samples or dirty  
490 glassware during sample processing or from tracer impurities. A poor estimate of the instrument  
491 background or ambient analyte levels in the matrix/reagent can lead to results being too negative  
492 in magnitude. A statistical test should be performed on a series of the data results to determine if  
493 there is a negative bias. The relative importance of the negative bias depends on the magnitude of  
494 the negative bias, magnitude of the action level and type of project.

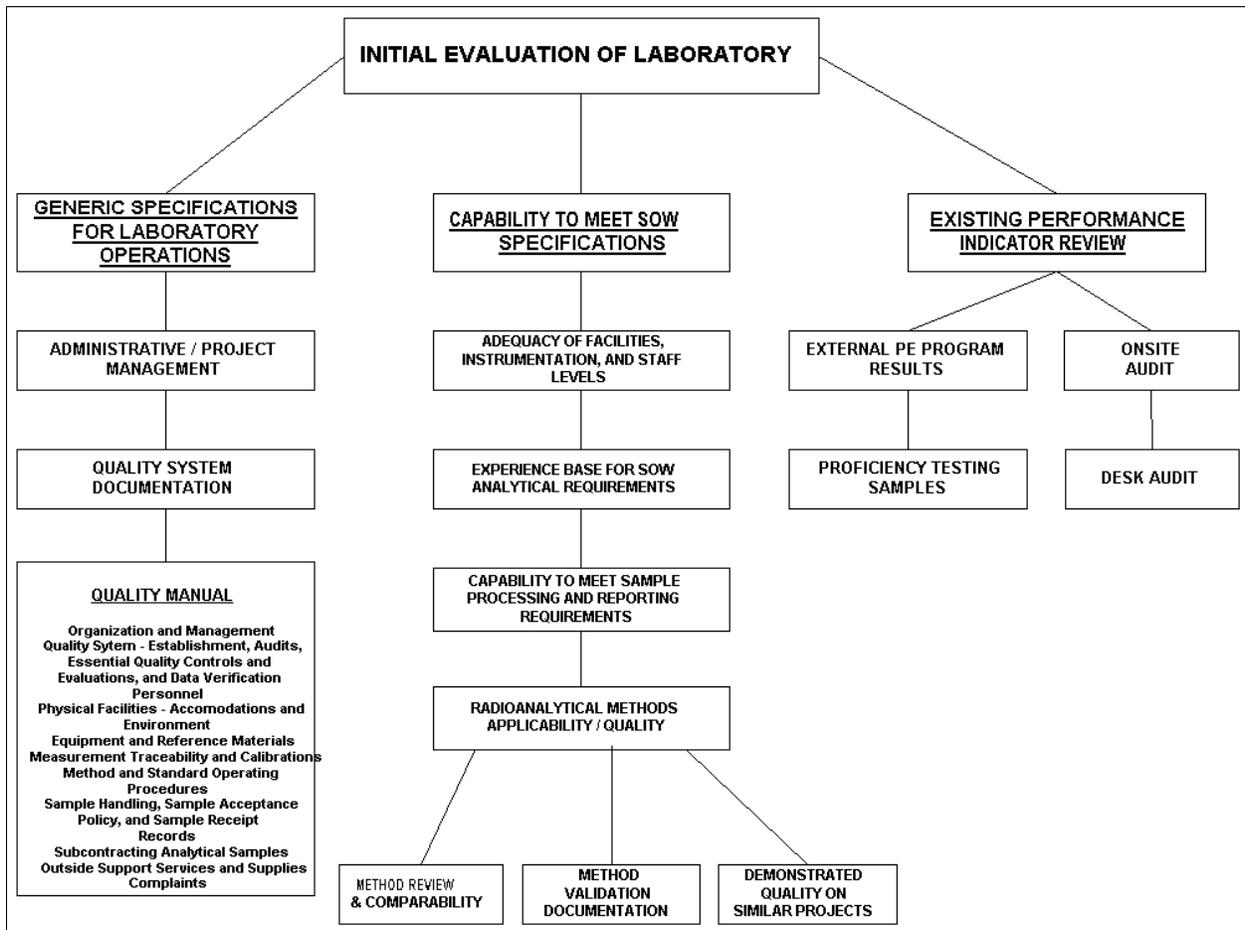
### 495 **7.3 Initial Evaluation of a Laboratory**

496 The basic information to be considered in the initial evaluation of a laboratory has been  
497 summarized according to major categories in Figure 7.1. Not all categories will be discussed in  
498 detail as subsections. Some categories may be grouped and discussed under a single generic  
499 subsection heading. In order to allow for flexibility, no definitive guidance or detailed acceptance  
500 criteria for the parameters under discussion will be provided.

#### 501 **7.3.1 Review of Quality System Documents**

502 A radiochemical laboratory providing usable analytical data should have a quality manual. A  
503 review of this document by a knowledgeable evaluator can reveal a great deal about the quality  
504 and acceptability of the laboratory relative to the work to be performed. A well-developed quality  
505 manual contains a description of the quality system and descriptive material covering most other  
506 aspects of a laboratory's operation. The standard operating procedures, method documentation,  
507 list of instrumentation, and personnel resumes should be reviewed. For some projects, the project  
508 manager may require the laboratory to develop a specific project quality plan, system, and  
509 manual. The following items, taken from the NELAC *Quality Systems* (NELAC 2000), should be  
510 discussed at a minimum:

- 511 • Organization and management
- 512 • Quality system establishment, audits, essential quality controls and evaluation, and data  
513 verification
- 514 • Personnel (qualifications and resumes)
- 515 • Physical facilities (accommodations and environment)



**FIGURE 7.1 — Considerations for the initial evaluation of a laboratory**

- 516 • Equipment and reference materials
- 517 • Measurement traceability and calibration
- 518 • Test methods and standard operating procedures (methods)
- 519 • Sample handling, sample acceptance policy and sample receipt
- 520 • Records
- 521 • Subcontracting analytical samples
- 522 • Outside support services and supplies
- 523 • Complaints

524 The laboratory evaluation should involve a review of the quality system documents for  
 525 completeness, thoroughness, and clarity.

526 **7.3.2 Adequacy of Facilities, Instrumentation, and Staff Levels**

527 Many factors enter into a laboratory's ability to meet the analytical requirements of a SOW. The  
528 resources and facilities of a laboratory may become stretched depending on the number of clients,  
529 the analytical services needed, and the deadlines of the committed work activities. Some SOWs  
530 may request information about the current workload of the laboratory and available facilities,  
531 staff and nuclear instrumentation for the specified work scope. The resources needed will vary  
532 considerably depending on the analysis and number of samples: from minimal bench space,  
533 hoods, and nuclear instrumentation for fairly simple gross analyses to maximum bench space,  
534 hoods, staff, and nuclear instrumentation for low-level analyses of soil. In addition, the laboratory  
535 capacity also depends on the number of samples that are routinely processed in a batch. Various  
536 factors may control the batch size, including the hood processing area, bench space, and  
537 equipment setup, available number of radiation detectors, counting time, and half-life of  
538 radionuclide, among others.

539 The adequacy of the facilities, instrumentation, and staff levels can be estimated by two general  
540 mechanisms: detailed supporting information in the SOW and an initial onsite audit. Information  
541 received from the prospective laboratory may provide an estimate of the laboratory's resources,  
542 but an initial onsite audit goes verifies the actual existence and maintenance of the resources.

543 **7.3.3 Review of Applicable Prior Work**

544 If required in a SOW, a laboratory will provide a list of clients for whom radioanalytical services  
545 had been performed that are considered comparable in terms of work scope, DQOs, MQOs,  
546 APSs, and project type. A written or oral verification of the client list should be performed. As  
547 part of the verification process, the following items related to adherence to contract or project  
548 requirements should be discussed and documented:

- 549 • Radionuclides analyzed;
- 550 • Sample matrices types;
- 551 • Laboratory capacity (number of samples per week or another time period);
- 552 • MQO for method uncertainty, detection and quantification capability;
- 553 • Radiological holding times;
- 554 • Sample turnaround times;
- 555 • Corrective actions; and
- 556 • Communications related to schedule, capacity, or quality issues.

557 It should be noted that under performance-based contracting, a laboratory's prior work for an

558 agency should be considered, either as a positive or negative performance weighting factor, when  
559 scoring a laboratory's performance during the technical evaluation process.

#### 560 **7.3.4 Review of Performance Indicators**

561 Some laboratories compile a semiannual or annual QA report summarizing the internal QC  
562 sample results for the methods used during a given time period, as well as an internal quality  
563 assessment report summarizing the internal and external audit findings and corrective actions  
564 taken. Although the laboratory's internal quality criteria for a given radionuclide/matrix may be  
565 different from the project MQOs, the internal QC sample results can be used to gauge the  
566 laboratory's performance capabilities. If these documents are available, they should be reviewed  
567 for documentation of process control and pertinent quality parameters such as bias, precision,  
568 unusually high number of positive blank detection, chemical recoveries, turnaround times,  
569 number of recurring deficiencies or findings, and corrective action effectiveness.

##### 570 7.3.4.1 Review of Internal QC Results

571 A quality assessment report may contain a summary of various QA-related activities, including  
572 internal audits and surveillance, report of conditions adverse to quality, investigation requests,  
573 corrective actions, and the results of external PE programs and internal QC samples. The content  
574 and frequency of the reports normally are outlined in the laboratory's quality manual. Frequently,  
575 this type of quality assessment report may be submitted with the laboratory's response to the RFP  
576 without request. The TEC may want to specifically request such a report when available.

577 When the laboratory's quality system is effectively implemented, the information contained in  
578 these QA reports can be used not only to gauge the quality of the analyses but also the effective-  
579 ness and timeliness of such quality system activities as identifying conditions adverse to quality,  
580 controlling and monitoring the radioanalytical quality using internal QC samples, and corrective  
581 actions. The internal QC sample results can be used to gauge the laboratory's performance  
582 capability. Results of the QC samples for a radionuclide and sample matrix should be reviewed  
583 for both the batch QC samples and single- or double-blind samples submitted by the QA officer.  
584 Batch QC samples typical include laboratory control samples, method blanks, matrix spikes, and  
585 duplicates. Such parameters as acceptable percent deviation for spiked samples, acceptable  
586 precision as measured by duplicate sample analyses, false nuclide detection, positive blanks, and  
587 compliance to internal quality requirements should be reviewed, depending on the type of QC  
588 sample. The single- and double-blind samples submitted independently by the QA officer are  
589 considered more operationally independent than the batch QC samples.

590 When quality problems are observed by the reviewer, it is important to check if the laboratory's  
591 quality system also has found and reported the same problem and whether an investigation or  
592 corrective action has been undertaken.

593 Additional specific guidance is provided in Chapter 18 on evaluating internal QC samples to  
594 meet internal laboratory QC performance criteria. It is recommended that the project managers  
595 review this chapter to gain a perspective on how to use reported internal QC results to gauge a  
596 laboratory's potential to meet project MQOs.

#### 597 7.3.4.2 External PE Program Results

598 Typically, a laboratory's performance or capability to perform high quality radioanalyses can be  
599 evaluated through two external PE program mechanisms. The first mechanism, which may not be  
600 available for all projects, is the submittal, as an initial laboratory evaluation process, of project-  
601 specific PT samples prepared by the organization or a contracted source manufacturer. When  
602 previous knowledge or experience exists, well-characterized site-specific matrix samples  
603 containing the nuclides of interest can be used. This approach can use site-specific matrix  
604 materials for background samples or for samples spiked with target analytes. For this evaluation  
605 mechanism, and depending on the number and type of samples, the laboratory's capability to  
606 meet all proposed project MQOs and quality performance specifications may be evaluated.

607 The second mechanism, available to most projects, is the laboratory's participation in  
608 government or commercial PE programs for radioanalyses. Each PE program has its own  
609 acceptable performance criteria related to a laboratory's bias with respect to the PE program's  
610 "known" analyte concentration value. Acceptable performance criteria are established for each  
611 nuclide/matrix combination. A PE program may also evaluate a laboratory based on a false  
612 positive analyte detection criterion. Typically, the laboratory's performance data in government  
613 PE programs are provided in reports available to the public.

614  
615 The project manager should be aware that the acceptable performance criteria used by the PE  
616 programs may be inconsistent with or more lenient than the MQOs of the project. The  
617 laboratory's performance should be evaluated in terms of the established MQOs of the project  
618 rather than a PE program's acceptable performance criteria. In some cases, the laboratories could  
619 be ranked as to their level of performance in these programs.

#### 620 7.3.4.3 Internal and External Quality Assessment Reports

621 Most laboratories undergo several external and internal QA audits per year, with resultant audit

622 reports. Typically, a summary of the findings and commitments of internal and external quality  
623 audits or assessments are tracked on some type of QA database as part of the laboratory's  
624 corrective action process. Access to the audit reports or database information may be limited.  
625 This information is not normally requested as part of the RFP process, nor do most laboratories  
626 submit such information with their response to an RFP. Therefore, obtaining previous QA audit  
627 information from a laboratory outside a formal, external, onsite audit process may be limited.

### 628 **7.3.5 Initial Audit**

629 An initial assessment or audit may be performed to provide assurance that a potentially selected  
630 laboratory is capable of fulfilling the project requirements in accordance with the SOW.  
631 Essentially, the objectives of an initial audit are twofold. The first objective is to verify that what  
632 the laboratory claims in response to the SOW or RFP, such as the various quality and safety  
633 programs, are being correctly and fully implemented, and when used during the project period,  
634 will ensure that stipulated requirements will be met. The second objective is to determine if the  
635 laboratory has the instruments, facilities, staffing levels and other operational requirements  
636 available to handle the anticipated volume of work. In other words, is the laboratory's proposal  
637 realistic when compared to the actual facilities? To answer this question, auditors will be looking  
638 to see whether a candidate laboratory has all the required elements to meet the project needs.

639 Detailed guidance and information on what should be evaluated in an initial audit has been  
640 provided in Appendix E, Section E5.5 and Table E7. This section also contains recommendations  
641 on the key items or parameters that should be reviewed during the initial audit. Depending on the  
642 project, other quality or operational parameters/requirements (such as requirements related to  
643 chemical speciation or subsampling at the laboratory) not covered in Appendix E should be  
644 included in the initial audit plan.

## 645 **7.4 Ongoing Evaluation of the Laboratory's Performance**

646 The evaluation framework presented here is intended to be sufficiently generic to cover the  
647 operations of a laboratory performing work according to a SOW as recommended in Chapter 5.  
648 As described in MARLAP, MQOs are a key component of the SOW. Therefore, the sample  
649 schedule, analyses to be performed, MQOs, and other analytical requirements have been defined.  
650 The methods selected by the laboratory have been demonstrated to meet the MQOs and have  
651 been approved by the project manager. In addition, the laboratory and its programs should have  
652 undergone an initial audit to ensure that the laboratory has met or is capable of meeting project  
653 requirements, including sample processing capacity, sample TATs, deliverables for analytical  
654 reports, etc. This would include maintaining a satisfactory quality system that includes

655 monitoring and controlling the radioanalytical processes through an instrument and internal  
656 sample QC program and the acceptable performance in an external PE program.

657 The ongoing evaluation of a laboratory's performance includes the evaluation of the method  
658 applicability or the quality of the data produced, and assessing the laboratory's quality system  
659 and operations through onsite or desk audits or assessments. The continued method performance  
660 can be evaluated through the laboratory's internal sample QC program, a possible external QC  
661 program maintained by the project manager, or an external PE program. It should be noted that  
662 samples used to control and monitor the quality of laboratory analyses have been defined  
663 according to their use. For example, batch or external QC samples are used to control as well as  
664 monitor the quality of the analytical process (the process can be stopped immediately if the QC  
665 sample results indicate that the process is outside appropriate SOW specifications or laboratory  
666 control limits). As defined previously, PT samples are used to compare the performance of the  
667 radioanalytical processing to some acceptance criteria but are not used to control the process.

668 The ongoing evaluation of the laboratory quality system and operations is accomplished through  
669 a visit to the laboratory or by a desk audit (the review of records and data from the laboratory).  
670 These audits or assessments are more focused on whether the laboratory is meeting project  
671 specifications rather than whether the laboratory has the capability to meet project or SOW  
672 requirements.

673 Once a laboratory has initiated work on a project, the laboratory's performance should be  
674 evaluated for the duration of the project. The quality of the radioanalytical measurements, as well  
675 as the pertinent key operational aspects of the laboratory, should be evaluated against the  
676 requirements of the MQOs and SOW. Both the quantitative and qualitative measures of  
677 laboratory performance should be evaluated on a continual basis. In addition, the operational  
678 aspects of the laboratory germane to the effective implementation of the project requirements  
679 should be evaluated/monitored on a continual basis.

#### 680 **7.4.1 Quantitative Measures of Quality**

681 The laboratory's ongoing demonstrated ability to meet the MQOs and other APS requirements  
682 can be evaluated through various quantitative measures using internal QC data and external PE  
683 program QC data. From these data, quantitative tests, as outlined in Appendix C can be used to  
684 measure and monitor the MQO parameters on a short-term basis. Also, the QC and PE program  
685 data can be used to evaluate the laboratory's performance, on a long-term trending basis, in  
686 meeting other quality related parameters such as bias and precision, unusually high number of  
687 positive blank detection, false nuclide detection, MDC or MQC adherence, radiological holding

688 times, etc. The following subsections will discuss the use of data from these samples to evaluate  
689 the laboratory's radioanalytical quality with respect to the requirements.

#### 690 7.4.1.1 MQO Compliance

691 MARLAP recommends that project specific MQOs be established and incorporated into the  
692 SOW for laboratory radioanalytical services. Appendix C provides guidance on developing the  
693 MQOs for method uncertainty, detection capability, and quantification capability. Establishing a  
694 gray region and action level are important to the development of the MQOs. For certain research  
695 programs and characterization studies, the concept of an action level may not be applicable. For  
696 these studies or programs, the MDC requirement and restrictions on the frequency of false  
697 positive detections may be more important. As such, the project planning team for these  
698 programs should establish the basis for their own MQOs and develop tests to evaluate a  
699 laboratory's performance to meet the requirements. These tests may be different from those  
700 presented below.

701 MARLAP recommends that a MQO for method uncertainty be established for each analyte/  
702 matrix combination. The method uncertainty is affected by laboratory sample preparation, sub-  
703 sampling, and the analytical method. In the absence of other information, the required method  
704 uncertainty ( $u_{MR}$ ) at the upper bound of the gray region (UBGR) may be defined as:

$$u_{MR} = \frac{\Delta}{10} \quad (7.3)$$

705 where  $u_{MR}$  is the method uncertainty and  $\Delta$  is the width of the gray region (difference between the  
706 upper and lower bounds of the gray region) as defined in Appendix C. In terms of the relative  
707 fraction of the upper bound of the gray region (action level),  $\phi_{MR}$ , is defined:

$$\phi_{MR} = \frac{u_{MR}}{UBGR} \quad (7.4)$$

708 The following subsections describe methods to quantitatively monitor a laboratory's performance  
709 relative to meeting this principal MQO through the use of internal or external batch QC samples.  
710 In some cases, the laboratory's internal quality program may have more restrictive quality control  
711 limitations for method performance compared to the proposed control limits used by the project  
712 manager to monitor adherence to the MQO for method uncertainty. Evaluation of the labora-  
713 tory's performance in NIST-traceable external PE programs will determine the degree of bias of  
714 the laboratory's method with respect to the national standard, as opposed to the determination of

715 the laboratory’s internal bias through the use internal QC samples. The tests presented assume  
 716 that all known internal (related to QC values and calibrations) and external (calibration differ-  
 717 ences with respect to the national standard) biases have been defined and eliminated and, as such,  
 718 the difference between the measured result and the “expected known” value is a result of the  
 719 method uncertainty only.

720 USE OF INTERNAL QC SAMPLE RESULTS

721 For most projects, the SOW will specify that the laboratory incorporate internal QC samples  
 722 within a defined batch of samples. The QC samples may include a laboratory control sample,  
 723 sample duplicates, a matrix spike sample and a method or reagent blank, or both. Appendix C  
 724 provides examples on the use of the following quantitative tests to measure a laboratory’s  
 725 performance in meeting the MQO for method uncertainty.

726 *Quality Performance Tests and Acceptance Criteria for Quality Control Samples*

727 Laboratory Control Sample (LCS). The analyte concentration of an LCS should be high enough  
 728 so that the resulting Poisson counting uncertainty is small and the relative uncertainty limit  $\phi_{MR}$  is  
 729 appropriate with respect to the action level and the spike concentration chosen. The percent  
 730 deviation (%D) for the LCS analysis is defined as

$$\%D = \frac{SSR - SA}{SA} \times 100\% \quad (7.5)$$

731 where

732 SSR is the measured result (spiked sample result) and  
 733 SA is the spike activity (or concentration) added.

734 It is assumed that the uncertainty of SA is negligible with respect to the uncertainty of SSR.  
 735 Refer to Appendix C for the basic assumption and limitation of this test. For long-term trending,  
 736 the %D results should be plotted graphically in terms of a quality control chart as described in  
 737 Chapter 18. The warning and control limits on %D are summarized below:

**Laboratory Control Samples**

Statistic:	%D
Warning limits:	$(\pm 2\phi_{MR}) \times 100\%$
Control limits:	$(\pm 3\phi_{MR}) \times 100\%$

742 Duplicate Analyses. The acceptance criteria for duplicate analysis results depend on the analyte  
 743 concentration of the sample, which is estimated by the average  $\bar{x}$  of the two measured results  $x_1$   
 744 and  $x_2$ .

$$\bar{x} = \frac{x_1 + x_2}{2} \quad (7.6)$$

745 When  $\bar{x} < UBGR$ , the absolute difference  $|x_1 - x_2|$  of the two measurements is used in the testing  
 746 protocol. For these tests, only upper warning and control limits are used, because the absolute  
 747 value  $|x_1 - x_2|$  is being tested.

748 When  $\bar{x} \geq UBGR$ , the acceptance criteria may be expressed in terms of the *relative percent*  
 749 *difference* (RPD) defined as

$$RPD = \frac{|x_1 - x_2|}{\bar{x}} \times 100\% \quad (7.7)$$

750 The requirements for duplicate analyses are summarized below.

**Duplicate Analyses**

If  $\bar{x} < UBGR$ :

Statistic:  $|x_1 - x_2|$   
 Warning limit:  $2.83 u_{MR}$   
 Control limit:  $4.24 u_{MR}$

If  $\bar{x} \geq UBGR$ :

Statistic:  $RPD = \frac{|x_1 - x_2|}{\bar{x}} \times 100\%$   
 Warning limit:  $2.83 \phi_{MR} \times 100\%$   
 Control limit:  $4.24 \phi_{MR} \times 100\%$

760 Method Blanks. When an aliquant of a blank material is analyzed, the target value is zero.  
 761 However, the measured value may be either positive or negative. The applicable warning and  
 762 control uncertainty limits for blank samples are defined as:

763

**Method Blanks**

764      Statistic:            Measured Concentration Value

765      Warning limits:     $\pm 2u_{MR}$ 766      Control limits:     $\pm 3u_{MR}$ 

767      *Matrix Spikes.* The acceptance criteria for matrix spikes are more complicated than those  
 768      described above for the other laboratory QC samples because of the pre-existing activity that is  
 769      inherent to the unspiked sample. The pre-existing activity (or concentration) must be measured  
 770      and subtracted from the activity measured after spiking.

771      MARLAP recommends the “Z score,” defined below, as the test for matrix spikes.

772

$$Z = \frac{SSR - SR - SA}{\phi_{AR} \sqrt{SSR^2 + \max(SR, UBGR)^2}} \quad (7.8)$$

773      where:

774      SSR    is the spiked sample result,

775      SR     is the unspiked sample result,

776      SA     is the spike concentration added (total activity divided by aliquant mass), and

777      max(SR,UBGR) denotes the maximum of SR and UBGR.

778      The warning and control limits for Z are set at  $\pm 2$  and  $\pm 3$ , respectively. It is assumed that the  
 779      uncertainty of SA is negligible with respect to the uncertainty of SSR. For long-term trending, the  
 780      Z results should be plotted graphically in terms of a quality control chart, as described in Chapter  
 781      18.

782      The requirements for matrix spikes are summarized below.

783

**Matrix Spikes**784      Statistic:             $Z = \frac{SSR - SR - SA}{\phi_{AR} \sqrt{SSR^2 + \max(SR, UBGR)^2}}$ 785      Warning limits:     $\pm 2$ 786      Control limits:     $\pm 3$

787 USE OF EXTERNAL PE PROGRAM AND QC SAMPLE RESULTS

788 Information on a laboratory's performance in an external PE program or from double-blind QC  
789 samples is very useful in monitoring a laboratory's ability to meet MQOs. A PE program will  
790 provide a snapshot in time whereas external QC samples included with samples submitted to the  
791 laboratory permit a continuous evaluation of the method's performance. When traceable to NIST,  
792 the PE program will elucidate any measurement or instrument calibration biases as related to the  
793 national standard. An external QC program may not have NIST traceability, and thus calibration  
794 biases to the national standard would not be determined.

795 For monitoring the performance of a laboratory using external PE program and QC sample  
796 results, the tests provided in the previous subsection ("Use of Internal QC Sample Results," page  
797 7-25) may be used when there are sufficient data. The test equations assume that the project has  
798 an MQO for method uncertainty at a specific concentration. In addition, it is assumed that the  
799 Poisson counting uncertainty for the radioanalysis of these samples is minimal.

800 *Results from PE Programs*

801 In many SOWs, the laboratory is required to participate in a recognized PE program for the  
802 nuclides and media of interest. In some cases, a certificate of participation may be needed as part  
803 of response to the RFP. However, it also should be noted that although a laboratory may meet  
804 performance acceptance criteria for an external PE program, this fact may have no bearing on  
805 whether the method will meet the MQOs of the SOW.

806 Monitoring ongoing laboratory performance is limited due to the minimum frequency of testing  
807 of the PE program, i.e., usually quarterly or semiannually. Some PE programs require multiple  
808 measurements to estimate precision but most only request a single result be reported. In addition,  
809 the concentration of the analyte typically never approaches an action level value and the media  
810 used are not site specific. For PE program samples, when possible, the laboratory should analyze  
811 a sample to reach a  $1\sigma$  Poisson counting uncertainty that is less than five percent.

812 *Multiple Analyses and Results*

813 When a PE program requires the analysis of multiple samples, the laboratory's measurement  
814 precision and bias (to a "known value") at the analyte concentration may be estimated and  
815 reported by the PE program provider. When only duplicates sample results are reported, then the  
816 tests for laboratory control samples and duplicate analyses given in the previous section should  
817 be used. The duplicate analysis test can be used as is, but the laboratory control sample test

818 should be evaluated based on the mean of the duplicate results. By using the mean of the two  
819 results, the LCS test provides a better estimate of any laboratory measurement bias with respect  
820 to the PE program provider. As discussed in Appendix C, the measurement (combined standard)  
821 uncertainty of each measured result value should be smaller than the required  $u_{MR}$  or  $\phi_{MR}$ .

822 *Results from External QC Samples*

823 The project manager may elect to establish an external QC program wherein QC samples are  
824 submitted to the laboratory with each batch of routine samples for the purpose of “controlling,”  
825 rather than monitoring, the quality of the analytical processes. The types of QC samples may  
826 include matrix spikes, blanks, and possibly duplicates if prepared under controlled and exacting  
827 protocols. An agency may use a qualified reference or monitoring laboratory (ANSI N42.23) to  
828 prepare the performance testing materials. When available, these QC samples may be prepared  
829 from site-specific materials.

830 When acceptance criteria are not met, the organization may issue a stop-work order and request  
831 corrective actions and reanalysis before routine processing can resume. In order to do this, the  
832 SOW must define the performance acceptance criteria and stipulate that the agency or  
833 organization has the right to stop laboratory processing when the performance requirements are  
834 not met. This application is not widespread but may have merit for certain project types. For  
835 example, research or national monitoring programs may monitor groundwater for specific  
836 naturally occurring radionuclides at state-of-art detection levels. For these programs, frequent  
837 false positive results, due to the application of incorrect instrument background or an analytical  
838 blank to the analytical result, would be unacceptable. Rather than permit a high rate of false  
839 positive results to continue, the agency can use the external batch QC samples to detect problems  
840 early and have the laboratory discontinue sample processing until a root cause is discovered and a  
841 corrective action undertaken. Non-conformance of a single analysis to performance criteria  
842 would not warrant the issuance of a stop work order unless a severe blunder has occurred.  
843 Typically, a certain amount of statistical trending of the data is in order to truly elucidate  
844 deficiencies.

845 Since the number of QC samples is similar to the recommendations for the laboratory’s internal  
846 batch QC samples, there should be sufficient data for trending. The statistical tests provided in  
847 the section on “Use of Internal QC Sample Results,” beginning on page 7-25, may be applied to  
848 these QC samples.

849 7.4.1.2 Other Parameters

850 The laboratory's performance in meeting the requirements for the other APSs that are listed in  
851 the SOW should be evaluated quantitatively when possible. In some cases, the information  
852 needed to perform the evaluations may be found in the final analytical results data package. For  
853 certain types of evaluations, a follow-up onsite or desk audit may be needed to complete the  
854 evaluation, e.g., a review of logbooks on unique processes or software algorithms and the  
855 analytical data base for proper spectral resolution.

856 RADIOLOGICAL HOLDING AND TURNAROUND TIMES

857 The data packages or analytical results report should contain the sample collection (reference),  
858 sample analysis, and reporting dates. From this information, the radiological holding and sample  
859 processing TATs can be calculated and compared against requirements. When a method uses a  
860 decay progeny to measure the analyte of interest ( $^{222}\text{Rn}$  to measure  $^{226}\text{Ra}$ ), the decay of the parent  
861 nuclide and ingrowth of the decay progeny are important parameters for evaluation. Unless  
862 requested in the SOW, most laboratories do not report the ingrowth factor as a standard output.  
863 Therefore, the information on the sample specific ingrowth factor may be available in the data  
864 reports or during audits. When required, these time related requirements will be evaluated for  
865 compliance during data verification and validation.

866 CHEMICAL YIELD

867 When appropriate, the SOW may specify limits on the chemical yield for each analyte. For  
868 radionuclides, this requirement typically is related to the provision of robust or rugged methods  
869 so that extreme yields become flags indicating potential problems. Wide swings in the chemical  
870 yield may be indicative of method's difficulty handling matrix or radionuclide interferences. The  
871 data packages or analytical results report should contain the chemical yield for each analyte  
872 listed. This reported value can be compared to the SOW yield limit. When required, these  
873 requirements will be evaluated for compliance during data verification and validation.

874 SPECTRAL RESOLUTION

875 Problems with spectral resolution of gamma-ray and alpha spectra cannot be evaluated through a  
876 review of the analytical results report. If spectral resolution limits have been stated in the SOW,  
877 the evaluator should review and evaluate each sample spectrum against the SOW limit. Spectral  
878 information may be available in data packages when required or may be obtained during audits.

879 During an initial audit, a preliminary evaluation of the method's SOP and review of past  
880 performance data for spectral resolution should be undertaken. The TEC may want to determine  
881 the baseline or typical spectral resolution for the radiation detection systems that will be used in  
882 the analysis of project samples. Trends of the spectral resolution of each detection system during  
883 the conduct of the project may be used to determine compliance with a spectral resolution  
884 specification.

## 885 **7.4.2 Operational Aspects**

886 Once a laboratory begins providing radioanalytical services, certain operational aspects need to  
887 be reviewed and evaluated periodically to determine if the laboratory is maintaining project  
888 requirements or if new problems have occurred. It is also important to ensure that the laboratory  
889 has been properly maintained and is operated and managed in a manner that will not create a  
890 liability to any client. Many of the operational areas that were discussed in Sections 7.3.1 and  
891 7.3.2 for the initial evaluation of a laboratory also should be evaluated periodically to ensure  
892 commitments are being met. The audit frequency varies according to the organization and the  
893 extent of the project or contract. Desk audits can be conducted more frequently than onsite audits  
894 because they require fewer resources. However, not all operational aspects may be reviewed  
895 during desk audits. The operational aspects that may be considered during desk and onsite audits  
896 are presented below.

### 897 **7.4.2.1 Desk Audits**

898 A desk audit is conducted as an off-site activity, usually by a technical representative of the  
899 project manager. A radioanalytical specialist should review all technical aspects of the desk  
900 audit, including method and calculation (data reduction) changes, method performance,  
901 instrument recalibrations, corrective actions, and case narratives. The desk audit is most useful  
902 when performed periodically to monitor certain activities or programs following an extensive  
903 onsite laboratory audit. However, for some smaller projects, the desk audit may be the only  
904 assessment mechanism used to monitor the laboratory's operations. The desk audit may be used  
905 to review or monitor the following operational aspects or items:

906     ◇ Organization and Management

- 907         ○ Changes in key personnel  
908         ○ Reassignments

909     ◇ Quality System

- 910         ○ Internal and external audits conducted, including laboratory certification audits

- 911           ○ Corrective action implementations
- 912           ○ Quality control and performance evaluations
- 913           – Instrument and batch sample QC results
- 914           – External PE program results
- 915           ○ Laboratory data verification (narrative status reports)
- 916           ○ Additional method validation studies
  
- 917          ◇ Certificates, licenses, equipment, and reference materials
- 918           ○ Standard and tracer certificates
- 919           ○ New and updates to instrument calibrations
- 920           ○ Instrument repairs and new instruments put into service
- 921           ○ NRC/State radioactive materials licence updates
- 922           ○ State or EPA drinking water certification status changes
  
- 923          ◇ Personnel
- 924           ○ Updates to staff qualification/proficiency for methods
- 925           ○ Updates to staff training files
- 926           – Radiation and chemical safety
- 927           – Quality assurance
- 928           – Technical principles
- 929           – Hands-on training records
  
- 930          ◇ Radioanalytical Methods and Standard Operating Procedures
- 931           ○ Updates to methods and SOPs
- 932           ○ Technical basis for updates
- 933           ○ Detection limits or method uncertainty studies
  
- 934          ◇ Sample Receipt, Handling and Disposal
- 935           ○ Sample receipt acknowledgment
- 936           ○ Chain-of-custody
- 937           ○ Sample- and waste-disposal tracking logs and manifests

938          Desk audits may also be used to review the data packages provided by the laboratory and,  
939          periodically, to verify certain method results by hand calculations. In addition, verification of  
940          compliance to radiological holding and turnaround times may be performed during the desk  
941          audit. In the absence of a full data verification and validation program (Chapter 8), the desk audit  
942          may be used to periodically evaluate the detailed instrument and data reduction reports of the  
943          data packages for method adherence, technical correctness and valid application.

944 7.4.2.2 Onsite Audits

945 The onsite laboratory audit is more comprehensive and resource intensive than a desk audit. An  
946 onsite audit typically is conducted to assess, periodically and in depth, a laboratory's capability to  
947 meet project requirements. Section E.5.5 of Appendix E provides guidance on the conduct of an  
948 initial onsite audit during a contract award process. EPA (1997) provides limited guidance on the  
949 conduct of an audit for a radiological laboratory. NELAC (2000) provides some generic guidance  
950 on laboratory assessments, although not specifically for a radiological laboratory.

951 Onsite audits usually cover the operational aspects delineated in Section 7.4.2.1 and also provide  
952 an opportunity to evaluate the physical conditions at the laboratory, in terms of adequacy and  
953 upkeep of the facilities, and the full application or conduct of programs and resources. Informa-  
954 tion sent in data packages or submitted for desk audits can be confirmed or verified during an  
955 onsite audit. Furthermore, an onsite audit permits the tracking of a sample from receipt through  
956 processing to sample storage and disposition and can verify the related instrument and batch QC  
957 samples specific to the sample being tracked. During an onsite audit, the auditors may have  
958 interviews with the staff to gauge their technical proficiency and familiarity with methods.

959 For large projects, onsite audits may be formal in nature and have a predefined audit plan, which  
960 has been developed by a designated audit team, for a specific project or program. The audit team  
961 typically is comprised of qualified QA representatives and technical experts. MARLAP  
962 recommends that the audit team include a radioanalytical specialist familiar with the project's or  
963 program's technical aspects and requirements.

964 In addition to the items in Section 7.4.2.1 ("Desk Audits"), the following items and programs  
965 should be assessed during an onsite laboratory audit:

966 ◇ Organization and Management

- 967 ○ Qualifications of assigned laboratory project manager
- 968 ○ Implementation of management's policy on quality
- 969 ○ Timeliness of addressing client complaints
- 970 ○ Timeliness of implementing corrective actions

971 ◇ Physical Facilities

- 972 ○ Adequacy of facilities (sample receipt, processing, instrumentation and storage areas,  
973 waste processing and storage, offices, etc.)
- 974 ○ Physical conditions of facilities including laboratories, hoods, bench tops, floors, offices,  
975 etc.

- 976           ◦ Environmental controls, such as climate control (heating, ventilation, air conditioning)
- 977           ◦ and electrical power regulation
- 978           ◦ Sample processing capacity
- 979           ◦ Sample storage conditions including chain-of-custody lockup areas and cross
- 980           ◦ contamination control (separation of samples by project and from radioactive sources or
- 981           ◦ wastes)
  
- 982           ◇ Instrumentation and Equipment
- 983           ◦ Age of nuclear instrumentation and equipment
- 984           ◦ Functionality of nuclear instrumentation and equipment
- 985           ◦ Calibrations and QC logs
- 986           ◦ Maintenance and repair logs
- 987           ◦ Sample throughput capacity
- 988           ◦ Contamination control for radiation detectors
- 989           ◦ Background spectra of radiation detectors
  
- 990           ◇ Methods and Standard Operating Procedures
- 991           ◦ Use of latest revisions of methods and SOPs (spot check method manuals used by
- 992           ◦ technical staff)
- 993           ◦ Conformance to method application (surveillance of method implementation)
- 994           ◦ Effectiveness of administering the controlled method manual
  
- 995           ◇ Certifications, Licenses and Certificates of Traceability
- 996           ◦ Ensure existence and applicability of, and conformance to, certifications and licenses
- 997           ◦ Noted citations during audits related to certifications and licenses
- 998           ◦ Ensure use of NIST-traceable materials (calibration standards)/review of vendors' report
- 999           ◦ of NIST traceability
  
- 1000          ◇ Waste Management Practices
- 1001          ◦ Adherence to waste management SOPs
- 1002          ◦ Proper packaging, labeling, manifests, etc.
- 1003          ◦ Sample storage and records
- 1004          ◦ Training and qualification records
  
- 1005          ◇ Radiological Controls
- 1006          ◦ Adherence to radiological safety SOPs
- 1007          ◦ Contamination control effectiveness (spill control, survey requirements and adherence,
- 1008          ◦ posted or restricted areas, proper ventilation, cleaning policies, etc.)

- 1009
  - Badging and survey adherence
- 1010
  - ◇ Personnel
- 1011
  - Number and technical depth of processing staff
- 1012
  - Training files
- 1013
  - Testing/qualifications
- 1014
  - Personal interviews to determine familiarity of methods and safety SOPs
- 1015
  - ◇ Quality Systems
- 1016
  - Performance indicator program (feedback from program)Quality assurance reports (QC
- 1017
  - and audits) for all laboratory processing
- 1018
  - Ongoing method evaluations and validations
- 1019
  - Corrective action program (effectiveness and outstanding issues for all processing; spot
- 1020
  - check for implementation of corrective actions)
- 1021
  - Records/reports related to audits of vendors used by laboratory
- 1022
  - Reagent control program (spot check conformance for effectiveness)
- 1023
  - Audits of laboratories that are subcontracted
- 1024
  - Laboratory's data verification and validation processes
- 1025
  - ◇ Software Verification and Validation
- 1026
  - Spot review of key method calculation and data reduction programs that include MDC,
- 1027
  - MQC, and measurement uncertainty; spectral unfolding routines or crosstalk factors;
- 1028
  - application of instrument background and analytical blanks; etc.
- 1029
  - Spot verification of consistency between electronic data deliverable and data packages
- 1030
  - ◇ Radiological Holding and Sample Turnaround Times
- 1031
  - Verification of compliance to radiological holding and sample TAT specifications (spot
- 1032
  - check samples and confirm paperwork)

1033

**Summary of Recommendations**

- 1034
  - MARLAP recommends that a radioanalytical specialist review the methods for technical
- 1035
  - adequacy.
- 1036
  - MARLAP recommends that project specific MQOs be established and incorporated into
- 1037
  - the SOW for laboratory radioanalytical services.
- 1038
  - MARLAP recommends that a MQO for method uncertainty be established for each

1039 analyte/matrix combination.

- 1040 • MARLAP recommends that an audit team include a radioanalytical specialist familiar with  
1041 the project's or program's technical aspects and requirements.

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