

8 RADIOCHEMICAL DATA VERIFICATION AND VALIDATION

8.1 Introduction

The goal of the data collection process is to produce credible and cost-effective data to meet the needs of a particular project. The process can be divided into several stages, as illustrated in the data life cycle (Chapter 1). This chapter is the first of two chapters that address the assessment phase of the project. Because the efficiency and success of these assessment activities are heavily dependent on the completion of the preceding steps in the data collection process, especially the initial planning activity (Chapter 2), the integration of planning and assessment is discussed in Section 8.2 prior to presenting material on data verification and validation.

Data verification compares the material delivered by the laboratory to the requirements in the statement of work (SOW) and identifies problems, if present, that should be investigated during data validation. Data validation compares the data produced with the measurement quality objectives (MQOs) and any other analytical process requirements contained in the analytical protocol specifications (APSS) developed in the planning process. It may not be necessary in all instances to validate all project data. This chapter outlines a validation plan that specifies the data deliverables and data qualifiers to be assigned that will facilitate the data quality assessment. The project-specific data validation plan should establish a protocol that prioritizes the data to be validated. This is to eliminate unnecessarily strict requirements that commit scarce resources to the in-depth evaluation of data points with high levels of acceptable uncertainty. For example, results very much above or below an action level may not require rigorous validation, since relatively large measurement uncertainty would not affect the ultimate decision or action. Planners should also identify those samples or data sets that have less rigorous standards for data quality and defensibility.

This chapter presents suggested criteria to evaluate data and addresses the appropriate function and limits of radiochemical techniques and measurements. Since calibration is more efficiently evaluated as part of an audit, this chapter does not recommend that the complete calibration-support documentation be included as part of the data package. MARLAP recommends that calibration be addressed in a Quality System and through an audit (Chapter 18), although demonstration of calibration may be required as part of a project's deliverables. Detector calibration, self absorption curves and efficiencies should be addressed as part of the evaluation of laboratories during the procurement process and continued during subsequent assessments (Chapter 7). Availability and retention of calibration records are decisions that are project-specific, but should be clearly identified for contract clarity and to assure project completeness

35 (i.e., customer needs met). External sources of information, such as performance evaluation
36 sample results and internal laboratory control samples, provide useful interim information on
37 calibration status and accuracy.

38 **8.2 Data Assessment Process**

39 Figure 1.1 of Chapter 1 graphically depicts the three phases—planning, implementation, and
40 assessment—of the data life cycle, and the associated activities and products of each phase.
41 *While these activities are addressed in separate chapters in MARLAP, it should be emphasized*
42 *that integration of planning, sampling, and analysis with subsequent data verification, data*
43 *validation, and data quality assessment (DQA) is essential.*

44 This section reviews the data life cycle from the perspective of the assessment phase and focuses
45 on those issues that have the potential to impact the quality and usability of the data. Section
46 8.2.1 addresses the development of the assessment procedures during project planning. Section
47 8.2.2 considers assessment needs for documentation and a quality system during implemen-
48 tation. Section 8.2.3 focuses on the assessment phase and addresses the interrelationship of the
49 three assessment processes. This introduction to the data life cycle process emphasizes the
50 importance of linkages among planning, implementation, and assessment.

51 **8.2.1 Planning Phase of the Data Life Cycle**

52 Directed project planning and the development of the associated DQOs, MQOs, and other
53 specifications for the project were reviewed in Chapters 2 and 3. *These chapters emphasize the*
54 *need for planners to thoroughly define the assessment processes (i.e., verification, validation and*
55 *data quality assessment) in sufficient detail that success or failure in meeting goals can be*
56 *determined upon project completion.* MARLAP recommends that the assessment phase of a
57 project (verification, validation, and DQA processes) be designed during the directed planning
58 process and documented in the respective plans as part of the project plan documents. This
59 requires the project planning team to develop detailed procedures for data verification, data
60 validation, and data quality assessment, as well as identify the actual personnel who will perform
61 assessment or the required qualifications and expertise of the assessors.

62 The development of these procedures during the directed planning process will increase the
63 likelihood that the appropriate documentation will be available for assessment, and that those
64 generating and assessing data will be aware of how the data will be assessed. A secondary
65 advantage, which assessment plans have, is that prior to their completion, they often result in the
66 detection of design flaws (e.g., lack of proper quality control [QC] samples, lack of a field audit)

67 that upon correction will result in the complete information necessary for the proper assessment
68 of data usability.

69 The culmination of the planning process is documentation of the outputs of the directed planning
70 process in the project plan documents. The project plan documents should capture the DQOs,
71 MQOs, and the optimized data collection design (i.e., Analytical Protocol Specifications,
72 sampling and analysis plans, and SOPs). The project plans should also include the assessment
73 plans as discussed above, and describe the field, lab, safety, and QA activities in sufficient detail
74 that the project can be implemented as designed. Chapter 4 discusses guidance for the authoring
75 and content of project plan documents.

76 If the directed planning process, its outputs (DQOs, MQOs, optimized sampling and analysis
77 designs), and associated assumptions are not documented well in project plan documents, the
78 assessment phase will have difficulties evaluating the resulting data in terms of the project's
79 objectives.

80 **8.2.2 Implementation Phase of the Data Life Cycle**

81 The project plans are executed during the implementation phase. Ideally, the plans would be
82 implemented as designed, but due to errors, misunderstandings, the uncontrolled environments
83 under which sampling is implemented, and matrix-specific issues that complicate sample
84 handling and analysis, most project plans are not implemented without some deviation.

85 Understanding the realities of implementation, the assessment process, in particular the DQA
86 process, will evaluate the project's implementation by considering: (a) if the plans were adequate
87 to meet the project's DQOs, (b) if the plans were implemented as designed, and (c) if the plans as
88 implemented were adequate to meet the project DQOs. MARLAP recommends that project
89 objectives, implementation activities and QA/QC data be well documented in project plans,
90 reports, and records, since the success of the assessment phase is highly dependent upon the
91 availability of such information.

92 Documentation and record keeping during the planning and implementation phase of the data life
93 cycle are essential to subsequent data verification, data validation, and data quality assessment.
94 Thorough documentation will allow for a determination of data quality and data usability.
95 *Missing documentation can result in uncertainty, and a lack of critical documentation (e.g.,*
96 *critical quality control results) can result in unusable data. The quality and usability of data can*
97 *not be assessed if the supporting documentation is not available.*

98 8.2.2.1 Project Objectives

99 The DQOs, MQOs, and other specifications, requirements, and assumptions developed during
100 the planning phase will influence the outcomes during the subsequent implementation and
101 assessment phases of the data life cycle. It is important that these objectives, specifications,
102 requirements, and assumptions are well documented and available to those implementing the
103 program so they can make informed decisions. This documentation is reviewed during the DQA
104 process (see discussion of the review of DQOs in Section 9.6.1.1, sampling plan in
105 Section 9.6.2.1, and analysis plan in Section 9.6.3.1).

106 8.2.2.2 Documenting Project Activities

107 The assessment of data in terms of sampling and analytical MQOs requires an accurate record of
108 QC sample data and compliance with specifications and requirements. If these records are
109 missing or inadequate, then compliance with APSSs, including the MQOs that were identified
110 during the planning phase, will not be ascertainable and will raise questions regarding quality.

111 Additional documentation is required to assess compliance with plans and contracts, and to
112 assess field and lab activities (e.g., compliance with SOPs) and the associated organizational
113 systems (e.g., laboratory Quality Manual). This information is gleaned from the review of field
114 and laboratory notebooks, deviation reports, chain-of-custody forms, verification reports, audit
115 reports, surveillance reports, performance evaluation sample analyses, corrective action reports
116 and reports to management that may identify deviations, contingencies, and quality problems.
117 Assessment of these types of contemporaneous records allow for the assessment of data in the
118 context of pertinent issues that may have arisen during project implementation.

119 Project records should be maintained for an agreed upon period of time, which should be
120 specified in project plan documents. Record maintenance should comply with all regulatory
121 requirements and parallel the useful life of the data for purposes of re-assessment as questions
122 arise or for purposes of secondary data uses that were not originally anticipated.

123 8.2.2.3 QA/QC

124 To ensure that the data collection activity generates data of known quality, it is essential that the
125 project plan documents specify the requirements for an appropriate quality system that is capable
126 of implementing the quality controls and the quality assurance necessary for success.

127 The quality system will oversee the implementation of QC samples, documentation of QC
128 sample compliance or non-compliance with MQOs, audits, surveillances, performance evaluation
129 sample analyses, corrective actions, quality improvement and reports to management. The
130 documentation generated by these quality assurance activities and their outputs during project
131 implementation will be a key basis for subsequent assessments and data usability decisions.

132 **8.2.3 Assessment Phase of the Data Life Cycle**

133 Assessment of environmental data currently consists of three separate and identifiable phases:
134 data verification, data validation, and DQA. Verification and validation pertain to evaluation of
135 analytical data. *Verification and validation are considered as two separate processes, but as the*
136 *MARLAP recommended planning process is implemented, they may be combined—with the*
137 *verification activities constituting the bulk of the review.* DQA considers all sampling, analytical,
138 and data handling details, external QA assessments, and other historical project data to determine
139 the usability of data for decision-making.

140 Figure 8.1 is a graphical depiction of the assessment phase. Although, it portrays a linear
141 progression through the various steps, and from verification and validation to data quality
142 assessment, this linear advancement is not entirely necessary. It is possible for parallel progress
143 within an assessment process (e.g., existing documents are verified while waiting for the
144 production of others) and between assessment processes (e.g., analysis of the DQOs for data
145 quality assessment while data validation is being completed). *Typically, the focus of verification*
146 *and validation is on the analytical process and on a data point by data point review, while data*
147 *quality assessment considers the entire data collection process and the entire data set as it*
148 *assesses data quality.*

149 Analytical data *verification* assures laboratory conditions and operations were compliant with the
150 SOW based on project plan documents. The updated project plan documents specify the
151 analytical protocols the laboratory should use to produce data of acceptable quality and the
152 content of the analytical data package (see MARLAP Process in Chapter 1). Verification
153 compares the analytical data package delivered by the laboratory to these requirements
154 (compliance), and checks for consistency and comparability of the data throughout the data
155 package, correctness of basic calculations, data for basic calculations, and completeness of the
156 results to ensure all necessary documentation is available. Verification can be accomplished
157 through use of a plan or simply a check list. The verification process produces a report
158 identifying which requirements are not met (i.e., exceptions qualified with an “E” to alert the
159 validator). The verification report is used to confirm laboratory compliance with the SOW and to
160 identify problems that should be investigated during data validation. Verification works

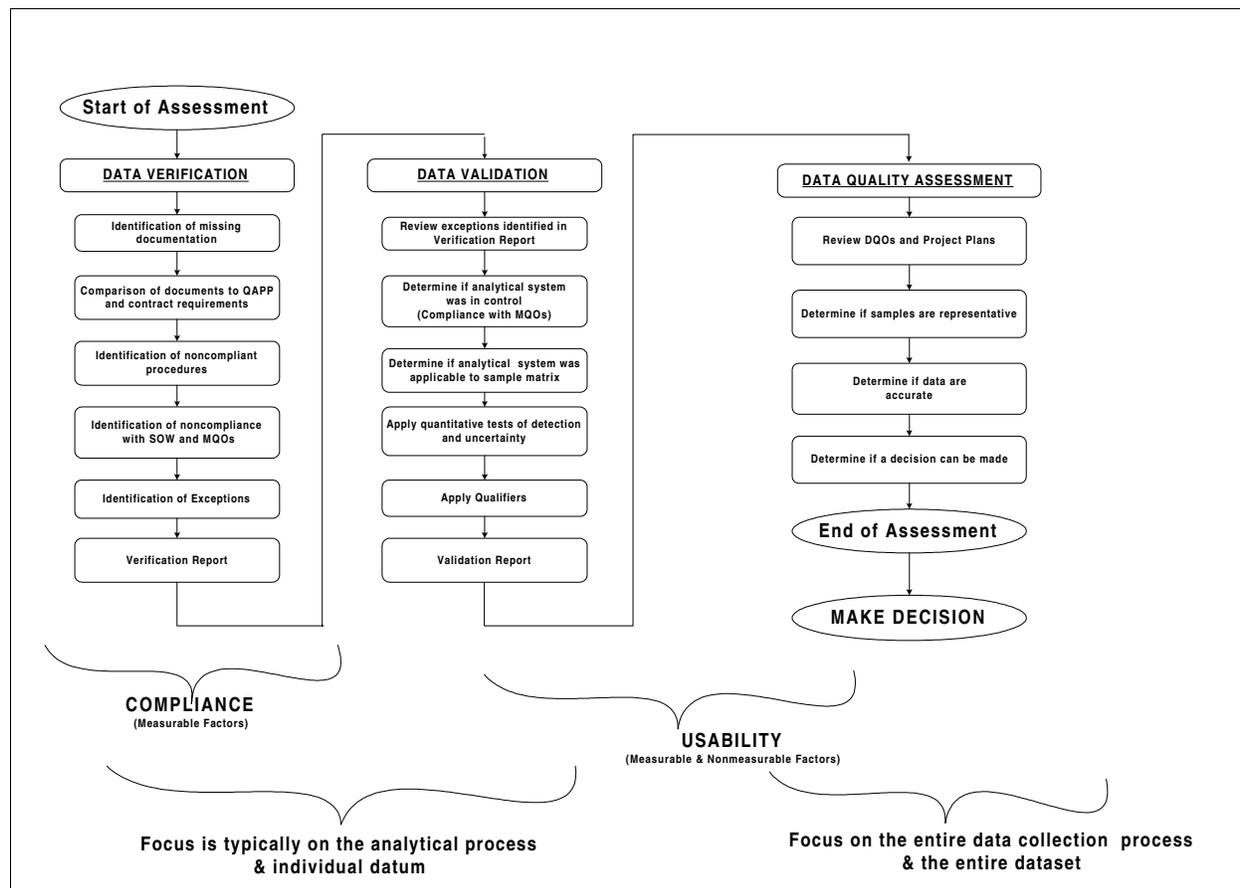


FIGURE 8.1 — The Assessment Process

161 iteratively and interactively with the generator (i.e., laboratory) to assure receipt of all necessary
 162 data. Although the verification process identifies specific problems, the primary function should
 163 be to apply appropriate feedback to the lab resulting in corrective action improving the analytical
 164 services before the project is completed.

165 *Validation* addresses the reliability of the data. The validation process begins with a review of the
 166 verification report and laboratory data package to identify its areas of strength and weakness.
 167 This process involves the application of qualifiers that reflect the impact of not meeting the
 168 MQOs. Validation then evaluates the data to determine the presence or absence of an analyte,
 169 and the uncertainty of the measurement process. During validation, the technical reliability and
 170 the degree of confidence in reported analytical data are considered. The data validator should be
 171 a scientist with radiochemistry experience.

172 Validation flags (i.e., qualifiers) are applied to data that do not meet the performance acceptance
173 criteria established in the SOW and the project plan documents. The products of the validation
174 process are validated data and a validation report stating which data are acceptable, which data
175 are sufficiently inconsistent with the validation acceptance criteria in the expert opinion of the
176 validator, and a summary of the QC sample performance. The appropriate data validation tests
177 should be established during the project planning phase. The point of validation is to perform a
178 systematic check on a set of data being used to meet the project MQOs and any other analytical
179 process requirements. Documenting that such a check cannot be done is an appropriate and
180 essential validation activity. (For example, applying numerical tests to data already determined to
181 be unreliable data are of no value.)

182 *Data Quality Assessment* is the last phase of the data collection process, and consists of a
183 scientific and statistical evaluation of project-wide knowledge to assess the usability of data sets.
184 To assess and document overall data quality and usability, the data quality assessor integrates the
185 data validation report, field information, assessment reports, and historical project data, and
186 compares the findings to the original project DQOs. The DQA process uses the combined
187 findings of these multi-disciplinary assessments to determine data usability for the intended
188 decisions, and to generate a report documenting that usability and the causes of any deficiencies.
189 It may be useful for a validator to work with the assessor to assure the value of the validation
190 process (e.g., appropriateness of rejection decision), and to make the process more efficient.
191 DQA will be covered in Chapter 9.

192 **8.3 Validation Plan**

193 The validation plan should integrate the contributions and requirements of all stakeholders and
194 present this information in a clear, concise format. To achieve this goal, validation planning
195 should be part of initial planning (e.g., directed planning process) to assure that the data will be
196 validated efficiently to determine its reliability and technical defensibility in an appropriate
197 context and to an appropriate degree.

198 The validation plan is an integral part of the project plan documents (Chapter 4), and should be
199 included as either a section within the plan or as a stand-alone document attached as an appendix.
200 The validation plan should be approved by an authorized representative of the project, the
201 validation group performing the validation, and any other stakeholder whose agreement is
202 needed.

203 The information and documentation identified in the validation plans should be communicated to
204 the laboratory as part of the SOW. Integration of validation plan specifications, contractual

205 requirements, and validator instructions/contracts is essential to ensure data collection process
206 efficiency. Implementation of the data validation plan will ensure that proper laboratory
207 procedures are followed and data are reported in a format useful for validation and assessment,
208 and will improve cost-effectiveness of the data collection process.

209 The data validation plan should contain the following information:

- 210 • Summarize the project that provides sufficient detail about the project technical and quality
211 objectives in terms of sample and analyte lists, required measurement uncertainty, and
212 required detection limit and action level on a sample/analyte-specific basis. Specify the scope
213 of validation, e.g., whether all the raw data will be reviewed and in what detail (see Section
214 8.3.1).
- 215 • Specify the necessary validation criteria, as derived from the MQOs, and performance
216 objectives deemed appropriate for achieving project objectives (see Section 8.3.2).
- 217 • Direction to the validator on what qualifiers are to be used and how final qualifiers are
218 assigned (see Section 8.3.3).
- 219 • Direction to the validator on the content of the validation report (see Section 8.3.4).

220 **8.3.1 Technical and Quality Objectives of the Project**

221 The identity of key analytes and how the sample results drive project decisions should be
222 specified in the validation plan. In addition, the plan should define the association of required
223 quality control samples with project environmental samples.

224 This section of the validation plan should specify the following:

- 225 • Quality control (QC) acceptance criteria;
- 226 • Level of measurement uncertainty considered unusually high and unacceptable (tests of
227 unusual uncertainty and rejection); and
- 228 • Action level and MQOs for detection and quantification capability (e.g., required detection
229 and quantification limit) (tests of detection).

230 The *quality control acceptance criteria* serve two purposes: (1) to establish if the analytical
231 process was in control; and (2) to determine if project requirements were met. If the analytical
232 process is in control, the assumption was that the analysis was performing within established
233 limits and indicates a reasonable match among matrix/analyte/method. Generally this means that
234 routine data quality expectations are appropriate. The *tests of unusual* (i.e., analysis not in
235 control) *uncertainty* should verify the data meet the statistical confidence limits for uncertainty
236 associated with the planning process. During validation, the uncertainty associated with sampling
237 cannot be estimated. The *tests of detection* determine the presence or absence of analytes.

238 **8.3.2 Validation Tests**

239 Validating data requires three specific decisions that will allow the validator to qualify the data.
240 The project planning team should determine:

- 241 • Which QC samples should be employed and how do they relate to the environmental
242 samples?
- 243 • Which validation tests are appropriate?
- 244 • What validation limits should be used for the specific tests?

245 The answers to these questions are driven by the need to know whether the data meets the MQOs
246 for the project, and the allocation of resources between planning and implementation (i.e.,
247 conservative review may be more costly than real or perceived value in the decision). This
248 section of the validation plan should address the following:

- 249 • QC sample validation criteria;
- 250 • Specific validation tests to be used; and
- 251 • Statistical confidence intervals or fixed limit intervals applied to each of the validation tests
252 and criteria based on the MQOs for the project (Appendix C).

253 **8.3.3 Data Qualifiers**

254 Data qualifiers are codes placed on an analytical result that alert data users to the validator's or
255 verifier's concern about the result. This section of the validation plan should outline:

- 256 • The basis for rejection or qualification of data; and
257 • The qualification codes that will be assigned.

258 These issues are discussed in detail in Section 8.5, which provides guidance for assigning data
259 qualifiers.

260 The verification process uses a qualifier (E) to alert the validator to non-compliance, including
261 missing documentation, contract compliance, etc. This qualifier may be removed or replaced
262 during validation, based on the validator's interpretation of the effect of the non-compliance on
263 the data's integrity.

264 **E** A notice to the validator that something was noncompliant.

265 The validation process uses the qualifiers listed below to identify data points that do not meet the
266 project MQOs or other analytical process requirements listed in the SOW or appropriate project
267 plan document. The assignment of the J and R qualifiers relies heavily on the judgement and
268 expertise of the reviewer and therefore, these qualifiers should be assigned as appropriate at the
269 end of data validation.

270 **U** A normal, not detected (< critical value) result.

271
272 **Q** A reported combined standard uncertainty, which exceeds the project's required method
273 uncertainty.

274 **J** An unusually uncertain or estimated result.

275 **R** A rejected result: the problems (quantitative and/or qualitative) are so severe that the data
276 can not be used.

277 The data validator should be aware that a data qualifier or a set of qualifiers does not apply to all
278 similar data. The data validator should incorporate the project MQOs into the testing and
279 qualifying decision-making process. During the data validation process the data validator may
280 use additional qualifiers based on QC sample results and acceptance criteria. These qualifiers
281 may be summarized as U, J, R or Q in the final validation report. The final validation reports
282 should also include a summary of QC sample performance for use by the data assessor.

283 **S** A result with a related spike result (laboratory control sample (LCS), matrix spike (MS) or
284 matrix spike duplicate MSD), which is outside the control limit for recovery (%R), S+ or
285 S- used to indicate high or low recovery.

286 **P** A result with an associated replicate result that exceeds the control limit.

287 **B** A result with associated blank result, which is outside the control limit, B+ or B-.

288 **8.3.4 Reporting and Documentation**

289 The purpose of this section is to define the format and program needs for validation reports and
290 supporting documentation. This section should include:

- 291 • Documentation and records that should be included in a validation report;
- 292 • Disposition requirements for records and documents from the project;
- 293 • Report format, i.e., a summary table with results, uncertainties and qualifiers; and
- 294 • Procedures for non-conformance reporting, which detail the means by which the laboratory
295 communicates non-conformances against the validation plan. The procedures should include
296 all instances where the analytical data requirements and validation requirements established
297 by the planning process and validation plan, respectively, cannot be met due to sample matrix
298 problems and/or unanticipated laboratory issues (loss of critical personnel or equipment).

299 Detailed information about the Validation Report is presented in Section 8.6.

300 **8.4 Other Essential Elements**

301 Effective data validation is dependent on:

- 302 • A SOW and project plan documents that clearly define the data needs and the data quality
303 requirements (i.e., MQOs); and
- 304 • A data package that has been verified for completeness, consistency, compliance, and
305 correctness.

306 **8.4.1 Statement of Work**

307 The analytical services procurement options should be considered during the planning process.
308 The SOW should specify the QC requirements that will be evaluated by the validator (see
309 Chapter 5). The elements that should be specified include, but are not limited to:

- 310 • External performance evaluation (PE) participation and acceptance criteria;
- 311 • Replicate sample frequency and acceptance criteria;
- 312 • LCS and acceptance criteria;
- 313 • Blank requirements and acceptance criteria;
- 314 • MS and MSD samples and acceptance criteria;
- 315 • Uncertainty calculations; and
- 316 • Sample result equations and calculations including corrections for yield, percent moisture,
317 efficiencies and blank, if applied.

318 Section 8.5.2 provides guidance on evaluating QC sample results based on the project's MQO for
319 measurement uncertainty.

320 **8.4.2 Verified Data Deliverables**

321 Verification compares the sample receipt information and the sample report delivered by the
322 laboratory against the SOW and produces a report that identifies those requirements that were not
323 met (called exceptions). Verification can be accomplished using a plan or checklist, which
324 doesn't necessarily need to be project-specific. Verification exceptions normally identify:

- 325 • Required steps not carried out by the laboratory (i.e., correction for yield, proper signatures);
- 326 • Method QC not conducted at the required frequency (i.e., blanks, duplicates); and
- 327 • Method QC not meeting pre-set acceptance criteria (i.e., non-compliant laboratory control
328 sample analysis).

329 The verifier checks the data package (paper or electronic) for completeness, consistency,
330 correctness, and compliance. Completeness means all required information is present.
331 Consistency means values are the same when reported redundantly on different reports, or
332 transcribed from one report to another. Correctness means the reported results are based on
333 properly documented and correctly applied algorithms. Compliance means the data pass
334 numerical QC tests based on parameters or limits derived from the MQOs specified in the SOW.

335 The verifier should provide, within the verification package, checklists for contract or SOW
336 specifications, noted deficiencies related to contract compliance, noted discrepancies or obvious
337 quality related problems, and pertinent external QC results. *The verification package notes the*
338 *deficiencies, discrepancies, and quality-related problems that could not be resolved with the*
339 *laboratory.* The validator should take this information into consideration during the data
340 validation process.

341 **8.5 Data Verification and Validation Process**

342 In its most basic form, data validation focuses on the reliability of each data point. After each
343 point is evaluated, summary conclusions concerning the validity of groups of data (sets) are
344 drawn and finally, after the reliability of all data sets has been established, an overall conclusion
345 about the quality and defensibility of a project's analytical database is reached (DQA).

346 The first step in establishing the reliability of an analytical measurement is to determine that the
347 measurement analytical process used in making the measurement is in control. That is, the
348 sample handling and analysis system is performing within an accepted operating range
349 (established by instrument manufacturer, method, or contract specifications and/or long-term
350 historical laboratory performance). After it has been determined that the measurement analytical
351 process is in control, it is necessary to demonstrate that the sample is responding as expected
352 when introduced into the measurement system.

353 The measurement process includes devices such as detectors for measuring radioactive decay and
354 balances for determining the mass of materials. The measurement process also includes the
355 software that takes the output from the measurement device and calculates the result as a quantity
356 of target radionuclide (activity/mass activity/volume). The measurement process performance
357 normally is specified by the SOW and appropriate project plan documents, and monitored by
358 routine laboratory quality control procedures. Laboratory performance against these requirements
359 is determined by the verification process uses these requirements to determine laboratory
360 performance.

361 When an environmental sample is analyzed, new sources of variability are encountered in
362 addition to those associated with the measurement process. These sources include laboratory
363 subsampling, sample preparation (e.g., digestion, leaching, etc.), sample matrix effects, and data
364 transcription, to list a few. These processes, taken together with the previously discussed
365 measurement process, comprise the analytical process.

366 The performance of the analysis can be predicted based on previous experience with similar
367 materials. Analysis performance is monitored by laboratory quality control procedures specified
368 in the SOW and appropriate project plan documents. Unlike the analytical process performance,
369 the overall performance of the analysis is not amenable to assessment by the data verification
370 process. Since each sample matrix, analyte, and method set is unique, the evaluation of overall
371 analysis performance and resulting data is the role of a knowledgeable validator.

372 Using the validation plan, which specifies QC samples, validation tests, and validation limits,
373 validation occurs in four stages:

- 374 • Determine whether the sample handling and analysis system is in control (Section 8.5.1);
- 375 • Determine whether quality control sample analyses meet specified MQOs (Section 8.5.2);
- 376 • Apply validation tests of detection and unusual uncertainty (Section 8.5.3); and
- 377 • Determine final data qualifiers and document the results (Section 8.5.4).

378 For other chemistry methods, identification of the analyte is also a primary decision. Except for
379 gamma spectroscopy, this is rarely an issue in radiochemistry. For radiochemistry, the
380 laboratory's ability to reliably identify analytes do reliable identifications is best checked by
381 auditors and verified by checking the calibration check samples.

382 **8.5.1 The Sample Handling and Analysis System**

383 As described in earlier sections of this guidance, it is necessary to know the extent to which the
384 data delivered for validation meet the requirements of the SOW and appropriate project plan
385 documents. These documents normally specify the minimum acceptable performance of the
386 analytical process. These specifications are the basis of the tests of quality control (QC tests) that
387 establish that the sample handling and analysis system is in control at the time the analyses were
388 performed. It is also necessary to know that all reporting requirements are complete. Normally,
389 this evaluation against the requirements is made during the data verification process. If the data
390 do not conform to the requirements, notification should be provided in the verification report.

391 The review of the verification package (and data package) by the validator determines if
392 sufficient information is provided to proceed with data validation. The outcome of the
393 verification process is the designation of exceptions to the quality control tests. These exceptions
394 should be flagged with a qualifier (re-evaluated by the validator), which is appended to a data or
395 report requirement that does not meet specifications to alert the validator of potential problems.
396 The validator should then determine if sufficient reliable data are available to proceed with
397 validation. The validator should use the data requirements and criteria developed in the

398 validation plan to determine if the quality control exceptions have an adverse impact on one or
399 more of the data points being validated.

400 Rarely, if ever, should quality control exceptions result in the decision to reject a complete data
401 set. Those types of situations should have been detected by the laboratory during the analytical
402 process and the samples reanalyzed. The validator should not reject (assign an “R” code) single
403 data points based on a single QC test exception. Normally, only numerous QC exceptions *and*
404 failures in one or more of the tests of detection and uncertainty are sufficient reason to reject
405 data. The validation report should fully explain the assignment of all qualifiers as previously
406 discussed.

407 The following paragraphs discuss some of the more important evaluations that should be applied
408 to the sample handling and analysis system. Limited guidance is provided on how the QC test
409 may impact data quality and defensibility.

410 8.5.1.1 Sample Descriptors

411 Sample descriptors include sample identification number, analytical method, analyte, and matrix,
412 among others.

413 **Criteria.** Each sample should have a unique identifier code that can be cross-referenced to a
414 unique field sample or an internally generated laboratory sample. This unique identifier and
415 associated sample descriptors should be included in all analytical reports to properly document
416 the sample and requested analysis (Chapters 10 and 11).

417 The matrix and other characteristics of the sample that affect method selection and performance
418 should be clearly identified. The method(s) used in sample preparation and analysis should be
419 identified.

420 If laboratory replicate analyses are reported for a sample, they should be distinguishable by a
421 laboratory-assigned code.

422 **Verification.** Each of the criteria related to describing the sample should be checked for and
423 found in the analytical data package. If any of the criteria are missing, they should be flagged
424 with an “E” code.

425 **Validation.** Missing information will increase the uncertainty on any result reported on a
426 sample(s) and justify the assignment of a “J” code. Missing information may be inferred from

427 other information in the data package and eliminate the added uncertainty. For example, if the
428 sample matrix is not provided, it may be inferred from:

- 429 • The aliquant units are expressed in units of mass or volume;
- 430 • The sample preparation method is specific for soils;
- 431 • The final results are expressed in units of mass; and
- 432 • The sampling report describes sampling soil.

433 The majority of related information should support the decision that the exception does not
434 increase the uncertainty of the result. If the supporting information is incomplete or conflicting,
435 the assignment of a “J” code to data points is warranted. If documentation is inadequate to
436 support the reporting of a data point, the data point should be qualified with an “R” code.

437 8.5.1.2 Aliquant Size

438 **Criteria.** The aliquant or sample size used for analysis should be documented so that it can be
439 checked when reviewing calculations, examining dilution factors or analyzing any data that
440 requires aliquant as an input. It is also imperative that the appropriate unit (liter, kilogram, etc.) is
441 assigned to the aliquant.

442 **Verification.** The criteria related to describing the sample aliquant should be checked for and
443 found in the analytical data package. If the aliquant size is missing, it should be flagged with an
444 “E” code.

445 **Validation.** The missing information will increase the uncertainty on any result reported on a
446 sample(s) and justify the assignment of a “J” code.

447 8.5.1.3 Dates of Sample Collection, Preparation, and Analysis

448 **Criteria.** The analytical data package should report date of sampling, preparation, and analysis.
449 These data are used to calculate radiological holding times, some of which may be specified in
450 the sampling and analysis plan.

451 *There are few circumstances where radiological holding times are significant for radionuclides.*
452 The best approach to minimize the impact of holding time on analysis is to analyze the samples
453 as quickly as possible. Holding times may be applied to environmental samples that contain
454 radionuclides with short half lives. Holding times would apply to these radionuclides to prevent

455 reporting of high measurement uncertainties and MDCs, and to detect the radionuclide, if present
456 at low concentration, before it decays to undetectable levels.

457 **Verification.** Each of the criteria related to sample holding time should be checked for and found
458 in the analytical data package. If any of the objectives are missing, they should be flagged with
459 an “E” code.

460 If a holding time is specified in the project plan documents or validation plan, the reported values
461 should be compared to this specification. If the holding time is exceeded, the affected criteria
462 (holding time) should be flagged with an “E” code.

463 **Validation.** The data points impacted by the missed holding time should be flagged with a “J”
464 code by the validator or the justification for discounting the holding time impact described in the
465 narrative section of the validation report.

466 8.5.1.4 Preservation

467 **Criteria.** Appropriate preservation is dependent upon analyte and matrix, and should be defined
468 in sampling and analysis documentation. Generally, preservation is applied to samples being
469 analyzed for radionuclides to prevent precipitation, adsorption to container walls, etc. The criteria
470 (required presence or absence) for this QC process should be provided in the sampling and
471 analysis plan (see Chapter 10).

472 **Verification.** The criteria related to preservation should be checked for and found in the
473 analytical data package. If any of the criteria are missing, they should be flagged with an “E”
474 code.

475 **Validation.** If exceptions to the preservation criteria are noted, the validator should decide if a
476 “J” code should be assigned to data points because the improper preservation increased the
477 overall uncertainty in the data point(s). In some cases where improper preservation severely
478 impacts data quality or defensibility (e.g., the use of acid preservation in water samples being
479 analyzed for ¹⁴C), the validator should assign an “R” qualifier. The assessor may elect to use the
480 data, but they have the responsibility of addressing the data quality and defensibility in the
481 assessment report.

482 8.5.1.5 Tracking

483 **Criteria.** Each analytical result should be traceable to the instrument or detector on which it was
484 counted. The requirement for this traceability normally is found in the project plan documents.
485 The analytical sequence log (or some other suitable record) should be available in the data
486 package submitted by the laboratory.

487 **Verification.** If any of the analytical data are not traceable to the instrument or detector, it should
488 be flagged with an “E” code.

489 **Validation.** The validator may factor the absence of the traceability into their evaluation of data
490 quality and usability. At most, this should result in increasing the uncertainty of the
491 determination and the possible assignment of a “J” code to the data. This would not occur
492 normally unless one or more of the detectors used in analyzing the samples was shown to be
493 unreliable. Then, the inability to trace a reliable detector to a sample increases the uncertainty of
494 the data point(s).

495 8.5.1.6 Traceability

496 **Criteria.** The traceability of standards and reference materials to be used during the analysis
497 should be specified in the sampling and analysis plan.

498 **Verification.** The source of the reference materials and standards should be checked for and
499 found or referenced in the analytical data package. If any of the sources are missing they should
500 be flagged with an “E” code.

501 **Validation.** The validator may factor the absence of the traceability into their evaluation of data
502 quality and usability. At most, this should result in increasing the uncertainty of the
503 determination and the possible assignment of a “J” code to the data. This would not occur
504 normally unless one or more of the standards used in analyzing the samples was shown to be
505 unreliable. Then, the inability to trace a reliable standard to a sample increases the uncertainty of
506 the data point(s).

507 8.5.1.7 QC Types and Linkages

508 **Criteria.** The type and quantity of QC samples should be identified and listed in the SOW, and
509 the results provided by the laboratory in a summary report. Replicates and matrix spike results
510 should be linked to the original sample results. The approximate level of matrix spike

511 concentrations should be specified in the SOW, but the actual levels should be reported by the
512 laboratory. The QC analyses should be traceable to the original field sample.

513 **Verification.** Each of the criteria related to the QC samples should be checked for and found in
514 the analytical data package. If any of the objectives are missing, they should be flagged with
515 an “E” code.

516 **Validation.** The validator should compare any QC sample exceptions to similar ones that
517 precede and follow the non-conforming QC sample. If these are in control, the validator can
518 discount the impact of the single QC sample exception on the data results (i.e., analytical
519 blunder). If a trend of failing values is found, the validator should consider if they affected a
520 group of data points to the extent that the level of uncertainty was increased. This may warrant
521 the assignment of a “J” code to the data.

522 8.5.1.8 Chemical Separation (Yield)

523 **Criteria.** Yield assesses the effects of the sample matrix and the chemical separation steps on the
524 analytical result and estimates the analyte loss throughout the total analytical process. Yield is
525 typically measured gravimetrically (via a carrier) or radiometrically (via a tracer). All the
526 components in the calculation of the yield should be identified in a defined sequence. These
527 specifications are found in the project plan documents.

528 Criteria for both analytical process and sample analysis may be given in the project plan
529 documents. The criteria should be based on historical data for the method and matrix. In that
530 case, yield is determined on both quality control samples and actual field samples.

531 The most important yield-related question is whether the yield has been determined accurately.
532 Typically, a yield estimate that is much greater than 100 percent cannot be accurate, but the
533 estimate may also be questionable if the yield is far outside its historical range. Extremely low
534 yields also tend to have large measurement uncertainties, which increase the uncertainties of the
535 results. The uncertainties of factors such as the yield, counting efficiency, and aliquant volume,
536 which affect the sensitivity of the measurement, should be kept relatively small.

537 **Verification.** Each of the yield-related criteria pertaining to the sample should be checked for
538 and found in the analytical data package. If missing, the data should be returned to the lab to
539 correct for yield.

540 **Validation.** The experimentally determined yield is used to normalize the observed sample
541 results to 100% yield. Exceptions to the yield value outside the range specified in the project plan
542 documents may result in the validator assigning a “J” qualifier to otherwise acceptable data.

543 8.5.1.9 Self-Absorption (Residue)

544 **Criteria.** For some radiochemical analytical methods, the SOW may specify the generation of a
545 self-absorption curve, which correlates mass of sample deposited in a known geometry to
546 efficiency.

547 **Verification.** Each self-absorption curve called for in the SOW should be checked for and found
548 in the analytical data package. If missing, they should be flagged with an “E” code.

549 **Validation.** If required self-absorption curves are missing, the validator may select to qualify
550 affected data with a “J” qualifier to signify an increased level of uncertainty in the measurement
551 because of the inability to correct the measured value for self-absorption.

552 8.5.1.10 Efficiency, Calibration Curves, and Instrument Background

553 **Criteria.** For some methods based on decay emission counting, efficiency is reported as count
554 rate divided by disintegration rate. Methods employing radiotracers determine a sample-specific
555 effective efficiency factor that is a product of the chemical yield and the detector efficiency. This
556 criteria may be specified in the SOW. Instrument background count rate is determined for each
557 detector for each region of interest and subtracted from the sample count rate.

558 **Verification.** Each efficiency determination, efficiency calibration curve, and instrument
559 background called for in the project plan documents should be checked for and found in the
560 analytical data package. If missing, they should be flagged with an “E” code.

561 **Validation.** If required factors are missing, the validator may select to qualify affected data with
562 a “J” qualifier to signify an increased level of uncertainty in the measurement because of the
563 inability to correct the measured value for efficiency.

564 8.5.1.11 Spectrometry Resolution

565 **Criteria.** The measured resolution of alpha, gamma-ray, and liquid scintillation spectrometers, in
566 terms of the full width of a peak at half maximum (FWHM), can be used to assess the adequacy
567 of instrument setup, detector selectivity, and chemical separation technique that may affect the

568 identification and quantification of the analyte. When sufficient peak definition (i.e., sufficient
569 number of counts to provide an adequate Gaussian peak shape) has been reached for a sample,
570 the resolution of the analyte peak should be evaluated to determine if proper peak identification
571 and separation or deconvolution was made. Spectral information should be provided in the data
572 packages to accomplish this evaluation.

573 **Verification.** There are no established acceptance criteria, but should be provided in the package
574 or available in the audit.

575 **Validation.** If required calculations are missing, the validator may select to qualify affected data
576 with a “J” qualifier to signify an increased level of uncertainty in the measurement because of the
577 inability to evaluate instrument setup and separation technique. An “R” code may be applied if
578 there is no separation.

579 8.5.1.12 Dilution and Correction Factors

580 **Criteria.** Samples for radiochemistry are usually not diluted, but a larger sample may be
581 digested, taking an aliquant for analysis to obtain a more representative subsample. The dilution
582 factors are normally used for tracers and carriers. Dilutions of the stock standards are prepared
583 and added to the samples. This dilution normally affects yield calculations, laboratory control
584 samples, and matrix spikes. This data should be provided in the data package so that the final
585 calculations of all data affected by dilution factors can be recalculated and confirmed, if required.

586 Other correction factors that may be applied to the data are dry weight correction, ashed weight
587 correction, and correction for a two-phased sample analyzed as separate phases.

588 **Verification.** Each dilution and correction factor affecting the sample should be checked for and
589 found in the analytical data package. If any of the factors are missing, they should be flagged
590 with an “E” code.

591 **Validation.** Those results impacted by missing dilution factors should be flagged with a “J” or
592 “R” qualifier, reflecting increased uncertainty in the data point(s). “R” may be warranted if the
593 calculation cannot be confirmed due to missing data.

594 8.5.1.13 Counts and Count Time (Duration)

595 **Criteria.** The count time for each sample, QC analysis, and instrument background should be
596 recorded in the data package. The ability to detect radionuclide disintegrations is directly related
597 to the count time. The longer the count time, the lower the detection limit. The project plan
598 documents should specify the MQOs, which will drive the count time for each analyte.

599 **Verification.** Each count time relating to the sample analysis should be checked for and found in
600 the analytical data package. If any of the objectives are missing, they should be flagged with
601 an “E” code.

602 **Validation.** The validator should estimate the impact of the actual count times on the ability to
603 detect the target analyte and the impact on the uncertainty of the measurement. *If the MQOs are*
604 *met, the sample should not be qualified for count time.* It should be noted that preset count
605 determination, rather than preset count time, will result in the same uncertainty for all the
606 samples. The qualifiers should be adjusted accordingly and the justification provided in the
607 validation report.

608 8.5.1.14 Result of Measurement, Uncertainty, Minimum Detectable Concentration, and Units

609 **Criteria.** MARLAP recommends that the result of each measurement, its expanded measurement
610 uncertainty, and the estimated sample- or analyte-specific MDC be reported for each sample in
611 the appropriate units. These values, when compared with each other, provide information about
612 programmatic problems with the calculations, interference of other substances, and bias. The
613 report should state the coverage factor used if calculating expanded measurement uncertainties,
614 and the Type I and Type II error probabilities used to calculate MDCs.

615 **Verification.** The linkage between the result, measurement uncertainties, MDC, and the sample
616 identification should be checked. If linkage is not evident, data should be flagged with an “E”
617 code.

618 **Validation.** The validator should assign data qualifiers to those data points for which they feel
619 sufficient justification exists. Each qualifier should be discussed in the validation report.

620 **8.5.2 Quality Control Samples**

621 Historically, data validation has placed a strong emphasis on review of QC sample data
622 (laboratory control samples, duplicates, etc). The assumption is that if the analytical process was

623 in control and the QC samples responded properly, then the environmental samples (field
624 samples plus the preparation sequences used to prepare the sample for analysis) would respond
625 properly. It is possible to have excellent performance on simple matrices (e.g., quality control
626 samples), but unacceptable performance on complex matrices (i.e., environmental) reported in
627 the same batch as the QC samples. Directly evaluating the environmental sample performance is
628 essential to determine measurement uncertainty and the likelihood of false positive and negative
629 detection of the target analyte.

630 Method blanks and laboratory control samples relate to the analytical batch (a series of similar
631 samples prepared and analyzed together as a group) quality control function. They are required
632 by most analytical service contracts, sampling and analysis plans, and project plan documents.
633 They serve a useful function as monitoring tools that track the continuing analytical process
634 during extended analytical sequences. They are the most ideal samples analyzed as part of a
635 project. Normally, their performance is compared to fixed limits derived from historical
636 performance or additionally project specific limits derived from the MQOs.

637 Laboratory duplicates and matrix spikes are quality control samples that directly monitor sample
638 system performance. The laboratory duplicates (two equal-sized samples of the material being
639 analyzed, prepared, and analyzed separately as part of the same batch) measure the overall
640 precision of the sample measurement process beginning with laboratory sub-sampling of the field
641 sample. Matrix spikes (a known amount of target analyte added to the environmental sample)
642 provide a direct measure of how the target analyte responds when the environmental sample is
643 prepared and measured, thereby estimating the bias introduced by the sample matrix.

644 Other QC tests can be applied to determine how the analytical process performs during the
645 analysis of environmental samples. These are yield/recovery, efficiency, self-absorption,
646 resolution, and drift. They are the same QC tests that were applied to routine QC samples (blanks
647 and laboratory control samples) in the previous discussion of the analytical process, but now are
648 applied to environmental samples. The difference lies in how performance is measured. Fixed
649 limits based on historical performance and/or statistics are usually the basis for evaluating the
650 results of routine QC samples.

651 The following paragraphs discuss how QC tests should be used to determine if the results for QC
652 samples meet the project MQOs. Guidance is provided on how to relate QC sample *and*
653 environmental sample performance to determine environmental sample data quality and
654 defensibility. Direction is also given about how to assign data qualifiers to environmental sample
655 data based on the tests of quality control. Appendix C provides guidance on developing criteria
656 for evaluating QC sample results. Specifically, Appendix C contains equations that allow for the

657 determination of warning and control limits for QC sample results based on the project's MQO
658 for measurement uncertainty.

659 8.5.2.1 Method Blank

660 The method blank (Section 18.4.1) is generated by carrying all reagents and added materials
661 normally used to prepare an environmental sample through the same preparation process. It
662 establishes how much, if any, of the measured analyte is contributed by the reagents and
663 equipment used in the preparation process. For an ideal system, there will be no detected
664 concentration or activity.

665 Since measured results are usually corrected for instrument and reagent background levels, it is
666 possible to obtain final results that are less than zero. A method blank result that is much less
667 than zero may indicate that the correction term is too large and therefore analyte concentrations
668 in actual samples may be underestimated.

669 **Criteria.** The requirement for a method blank is usually established in the SOW and appropriate
670 plan documents. The objective is to establish the target analyte concentration or activity
671 introduced by the sample preparation sequence. Method blanks are normally analyzed once per
672 analytical batch.

673 Other types of blanks, such as field blanks and trip blanks, are used to evaluate aspects of the
674 data collection effort and laboratory operations that are not directly related to the validation of
675 environmental analytical data quality or technical defensibility. They can be important to the
676 overall data assessment effort, but are beyond the scope of this guidance (Chapter 10).

677 See Appendix C for guidance on developing criteria for evaluating blanks based on the project's
678 MQO for method uncertainty.

679 **Verification.** If a method blank was required but not performed, or if the required data is
680 missing, the verifier flags the missing information with an "E" code.

681 **Validation.** If a blank result does not comply with the established criteria, the associated samples
682 are flagged "B+" to indicate that the blank result is greater than the upper limit, or "B-" to
683 indicate that the blank result is less than the lower limit.

684 8.5.2.2 Laboratory Control Samples

685 The laboratory control sample (LCS) is a QC sample of known composition or an artificial
686 sample (created by spiking a clean material similar in nature to the environmental sample), which
687 is prepared and analyzed in the sample manner as the environmental sample. In an ideal situation,
688 the LCS would give 100 percent of the concentration or activity known to be present in the
689 fortified sample or standard material. Acceptance criteria for the LCS sample are based on the
690 complexity of the matrix and the historical capability of the lab and method to recover the
691 activity. The result normally is expressed as percent recovery. The LCS recovery differs from the
692 recovery of a matrix spike in that the matrix spike is added directly to the environmental sample
693 and the percent recovery is determined by comparing the difference between the original and
694 spiked samples.

695 **Criteria.** The objective of the LCS is to measure the response of the analytical process to a QC
696 sample with a matrix similar to the environmental sample. This will allow inferences to be drawn
697 about the reliability of the analytical process.

698 See Appendix C for guidance on developing control limits for LCS results based on the project's
699 MQO for method uncertainty.

700 **Verification.** If a required LCS is not analyzed, or if required information is missing, the verifier
701 flags the missing information with an "E" code.

702 **Validation.** When the measured result for the LCS is outside the control limits, the associated
703 samples are flagged with the "S" qualifier (S+ or S-).

704 8.5.2.3 Laboratory Replicates

705 Replicates are used to determine the precision of laboratory preparation and analytical
706 procedures. Laboratory replicates are two aliquants selected from the laboratory sample and
707 carried through preparation and analysis as part of the same batch.

708 The discussion of field replicates is beyond the scope of this chapter.

709 **Criteria.** The objective of replicate analyses is to measure laboratory precision based on each
710 sample matrix. The variability of the samples due to field sample heterogeneity is also reflected
711 in the replicate result. The laboratory may not be in control of the precision. Therefore, replicate

712 results are used to evaluate reproducibility of the complete laboratory process that includes
713 subsampling, preparation, and analytical process.

714 See Appendix C for guidance on developing control limits for replicate results based on the
715 project's MQO for method uncertainty.

716 **Verification.** If replicate analyses are required but not performed, or if the required data is not
717 present in the report, the verifier flags the missing information with an "E" code.

718 **Validation.** When the replicate analysis is outside the control limit, the associated samples are
719 flagged with the "P" qualifier.

720 8.5.2.4 Matrix Spikes and Matrix Spike Duplicates

721 The matrix spike is an aliquant of a sample, fortified (spiked) with known quantities of target
722 analytes and subjected to the entire analytical procedure to establish if the method or procedure is
723 appropriate for the analysis of the particular matrix.

724 **Criteria.** Matrix spike samples provide information about the effect of each sample matrix on
725 the preparation and measurement methodology. The test uncovers the possible existence of
726 recovery problems, based on either a statistical test or a specified fixed control limit.

727 See Appendix C for guidance on developing criteria for evaluating matrix spikes based on the
728 project's MQO for method uncertainty.

729 **Verification.** If a required matrix spike analysis was not performed, or if the required
730 information is missing, the missing information should be flagged with an "E" code.

731 **Validation.** If the results of the matrix spike analysis do not meet the established criteria, the
732 samples should be qualified with an "S+" or "S-" indicating unacceptable spike recoveries.

733 8.5.3 Tests of Detection and Unusual Uncertainty

734 8.5.3.1 Detection

735 The purpose of a test of detection is to decide if each result for a regular sample is significantly
736 different from zero. Since most radiochemistry methods always produce a result, even if a very
737 uncertain or negative one, some notion of a non-detected but measured result may be needed for

738 some projects. A non-detected result is generally as valid as any other measured result, but it is
739 too small relative to its measurement uncertainty to give high confidence that a positive amount
740 of analyte was actually present in the sample. Ordinarily, if the material being analyzed is
741 actually analyte-free, most results should be “non-detected.”

742 For some projects, detection may not be an important issue. For example, it may be known that
743 all the samples contain a particular analyte, and the only question to be answered is whether the
744 mean concentration is less than an action level. However, all laboratories should be able to
745 perform a test of detection routinely for each analyte in each sample.

746 **Criteria.** An analyte is considered detected when the measured analyte concentration exceeds its
747 critical value (see Chapter 19). Both values are calculated by the laboratory performing the
748 measurement; so, the detection decision can be made at the laboratory and indicated in its report.
749 If there is no evidence of additional unquantified uncertainty in the result (e.g., lack of statistical
750 control or blank contamination), the laboratory’s decision may be taken to be final.

751 **Verification.** Typically, the role of the verifier is limited to checking that required information,
752 such as the critical value, is present in the report. If information is missing, the result should be
753 flagged with an “E” code.

754 **Validation.** The validator examines the result of the measurement, its critical value, and other
755 information associated with the sample and the batch in which it was analyzed, including method
756 blank results in particular, to make a final determination of whether the analyte has been detected
757 with confidence. If the data indicates the analyte has been detected in both the sample and the
758 method blank, its presence in the sample may be questionable. A quantitative comparison of the
759 total amounts of analyte in the sample and method blank, which takes into account the associated
760 measurement uncertainties, may be needed to resolve the question.

761 8.5.3.2 Detection Capability

762 **Criteria.** If the project requires a certain detection capability, the requirement should be
763 expressed as a required minimum detectable concentration (RMDC). The data report should
764 indicate the RMDC and the sample-specific estimate of the actual minimum detectable
765 concentration (MDC) for each analyte in each sample.

766 In some situations, it may not be necessary or even possible for a laboratory to meet the MDC
767 requirement for all analytes in all samples. In particular, if the analyte is present and quantifiable
768 at a concentration much greater than the action level, a failure to meet a contract-required

769 detection limit is usually not a cause for concern. A failure to meet the RMDC is more often an
770 important issue when the analyte is not detected.

771 **Verification.** The RMDC specified in the contract is compared to the sample-specific MDC
772 achieved by the method. The analytes that do not meet the RMDC are flagged with an “E” code.

773 **Validation.** If the sample-specific MDC estimate exceeds the RMDC, the data user may be
774 unable to make a decision about the sample with the required degree of certainty. A “UJ”
775 qualifier is warranted if the estimated MDC exceeds the RMDC and the analyte was not detected
776 by the analysis. A final decision about the usability of the data should be made during the data
777 assessment phase of the data collection process.

778 An assignment of “R” to the data points affected by this type of exception may be appropriate in
779 some cases, but the narrative report may classify the data as acceptable (no qualifier), “U,” or “J,”
780 based on the results of the tests of detection and uncertainty. This allows the assessor to make an
781 informed judgement about the usability of the data point(s) and allows them the opportunity to
782 provide a rationale of why the data can be used in the decision process.

783 8.5.3.3 Large or Unusual Uncertainty

784 When project planners follow MARLAP’s recommendations for developing MQOs, they
785 determine a required method uncertainty at a specified analyte concentration. The required
786 method uncertainty is normally expressed in concentration units, but it may be expressed as a
787 relative method uncertainty (percent based on the upper bound of the gray region, which is
788 normally the action level). It is reasonable to expect the laboratory’s combined standard
789 uncertainty at concentrations lower than the action level to be no greater than the required
790 method uncertainty (expressed in concentration units) and to expect the laboratory’s relative
791 combined standard uncertainty at concentrations above the action level to be no greater than the
792 required relative method uncertainty (expressed as a percent). Each measured result should be
793 checked against these expectations (see Appendix C).

794 **Criteria.** The reported combined standard uncertainty is compared to the maximum allowable
795 standard uncertainty. Either absolute (in concentration units) or relative uncertainties (expressed
796 as a percent) are used in the comparison, depending on the reported concentration. The result is
797 qualified with a “Q” if the reported uncertainty is larger than the requirement allows.

798 **Verification.** The test for large uncertainty is straightforward enough to be performed during
799 either verification or validation. If there is a contractual requirement for measurement

800 uncertainty, the verifier should perform the test and assign the “E” qualifier to results that do not
801 meet the requirement. Note that it may sometimes happen that circumstances beyond the control
802 of the laboratory make it impossible to meet the requirement.

803 **Validation.** If a “Q” qualifier is assigned, the validator may consider any special circumstances
804 that tend to explain it, such as interferences, small sample sizes, or long decay times, which were
805 beyond the control of the laboratory. He or she may choose to remove the qualifier, particularly if
806 it is apparent that the original uncertainty requirement was too restrictive.

807 **8.5.4 Final Qualification and Reporting**

808 The final step of the validation process is to assign and report final qualifiers for all regular
809 sample results. The basis for assignment of final qualifiers is qualifiers and reasons from all
810 previous tests, patterns of problems in batches of samples, and validator judgement.

811 The difficult issue during final qualifier assignment is rejecting data. What follows summarizes
812 some of the issues to consider when thinking about rejecting data.

813 Rejecting a result is an unconditional statement that it is not useable for the intended purpose. A
814 result should only be rejected when the risks of using it are significant relative to the benefits of
815 using whatever information it carries. If the DQA team or users feel data is being rejected for
816 reasons that don't affect usability, they may disregard all validation conclusions. Rejected results
817 should be discarded and not used in the DQA phase of the data life cycle.

818 There are three bases on which to reject data:

819 1. Insufficient or only incorrect data are available to make fundamental decisions about data
820 quality. For example, if correctly computed uncertainty estimates are not available, it is
821 not possible to do most of the suggested tests. If the intended use depends on a consistent,
822 high level of validation, it may be proper to reject such data.

823 The missing data should be fundamental. For example, missing certificates for standards
824 are unlikely to be fundamental if lab performance on spiked samples is acceptable. In
825 contrast, if no spiked sample data is available, it may be impossible to determine if a
826 method gives even roughly correct results, and rejection may be appropriate.

827 2. Available data indicate that the assumptions underlying the method are not true. For
828 example, QC samples may demonstrate that the lab’s processes are out of control.
829 Method performance data may indicate that the method simply does not work for
830 particular samples. These problems should be so severe that is not possible to make
831 quantitative estimates of their effects.

832 3. A result is “very unusually uncertain.” It is difficult to say what degree of uncertainty
833 makes a result unusable. Whenever possible, uncertain data should be rejected based on
834 multiple problems with one result, patterns in related data, and the validator’s judgement,
835 not the outcome of a single test. This requires radiochemistry expertise and knowledge of
836 the intended use.

837 Based on an evaluation of the tentative qualifiers, final qualifiers are assigned to each regular
838 sample result.

839 After all necessary validation tests have been completed and a series of qualifiers assigned to
840 each data point based on the results of the tests, a final judgment to determine which, if any, final
841 qualifiers will be attached to the data should be made. The individual sample data from the
842 laboratory should retain all the qualifiers. The basic decision making process for each result is
843 always subject to validator judgement:

- 844 • As appropriate, assign a final “R”;
- 845 • If “S”, “P”, or “B” were assigned, determine whether the qualifiers warrant the assignment of
846 an “R”;
- 847 • If “R” is not assigned, but some test assigned a tentative S, P, B, Q, or J, or a pattern exists
848 that makes it appropriate, assign a final S, P, B, Q, or J and summarize QC sample
849 performance;
- 850 • If a final S, B, or J was assigned, + or -, but not both, was tentatively assigned, and the
851 potential bias is not outweighed by other sources of uncertainty, make the + or - final; and
- 852 • For non-R results, if any test assigned a tentative “U,” make it final.

853 The final validation decision should address the fact that the broader purpose of validation is to
854 contribute to the total data collection process, i.e., effectively translate and interpret analytical
855 results for efficient use by an assessor. This means the validator should examine the full range of
856 data available to search for and utilize relationships among the data elements to support the
857 acceptance and use of data that falls outside method or contract specifications and data validation
858 plan guidance.

859 **8.6 Validation Report**

860 The final product of validation is a package that summaries the validation process and its
861 conclusions in an orderly fashion. This package should include:

- 862 • *A narrative or summary table written by the validator that summarizes exceptional*
863 *circumstances:* In particular, it should document anything that prevented executing the
864 planned validation tests. Further, the narrative should include an explicit statement explaining
865 why data has been rejected or qualified based on the findings of the validation tests and the
866 validator’s judgment.

- 867 • *A list of validated samples that provides a cross-reference of laboratory and client sample*
868 *identifiers:* This report should also include other identifiers useful in the context of the
869 project, such as reporting batch, chain of custody, or other sample management system
870 sample information.

- 871 • *A summary of all validated results with associated uncertainty for each regular sample with*
872 *final qualifiers:* Unless specified in the sampling and analysis plan, non-detects are reported
873 as measured, not replaced by a detection limit or other “less than” value.

- 874 • A summary of QC sample performance and the potential effect on the data both qualified and
875 not qualified.

876 Assuming the client wants additional information, the following, more detailed reports can be
877 included in the validation package. Otherwise, they are simply part of the validation process and
878 the verification contract compliance:

- 879 • A detailed report of all tentative qualifiers and associated reasons for their assignment;
- 880 • QC sample reports that document analytical process problems; and
- 881 • Reports that summarize performance by method—these should support looking across related
882 analyses at values such as yields and result ratios.

883 The data in the summary reports should be available in a computer-readable format. If no result
884 was obtained for a particular analyte, the result field should be left blank. The validation report
885 should package analytical results as effectively as possible for application and use by the
886 individual assembling and assessing all project data.

887 The validation report should contain a discussion describing the problem(s) found during the
888 validation process. For the validation codes, the discussion summarizes the performance criteria
889 established in the validation plan. If the validation test performance criteria were changed (e.g.,
890 increased or decreased level of unusual uncertainty) because the nature of the sample matrix or
891 analyte was different than expected, the new criteria should be explained in the report and the
892 qualifiers applied using the new criteria. The approval of the project manager should be obtained
893 (and documented) before the new criteria are applied. The project manager should communicate

894 the changes to the project planning team to maintain the consensus reached and documented
895 during validation planning.

896 Well-planned and executed analytical activities can be expected to meet reasonable expectations
897 for data reliability. This means that for most data points or data sets, the results of the tests of
898 quality control, detection, and unusual uncertainty will show that the data are of sufficient quality
899 and defensibility to be forwarded to the assessor with little or no qualification for final
900 assessment. A small number of points will be rejected because random errors in the analytical
901 process or unanticipated matrix problems resulted in massive failure of several key validation
902 tests.

903 A smaller number of data points will show conflicting results from the validation tests and
904 present the greatest challenge to the validator. The more important the decision and/or the lower
905 the required detection limit, the more common this conflict will become, and the more critical it
906 is that the data validation plan provide guidance to the validator about how to balance the
907 conflicting results. Is the ability to detect the analyte more important than the associated
908 statistical unusual uncertainty, or is the presence of the analyte relatively definite but the unusual
909 uncertainty around the project decision point critical to major decisions? The necessary guidance
910 should be developed during the planning phase to guide the final judgment of the validator.

911 **8.7 Other Sources of Information**

912 American National Standards Institute (ANSI) N13.30. 1996. *Performance Criteria for*
913 *Radiobioassay*.

914 U.S. Environmental Protection Agency (EPA). 1994. Contract Laboratory Program National
915 Functional Guidelines for Inorganic Data Review. EPA-540/R-94-013 (PB94-963502).
916 February. Available from [http://www.epa.gov/oerrpage/superfund/programs/clp/download/](http://www.epa.gov/oerrpage/superfund/programs/clp/download/fginorg.pdf)
917 [fginorg.pdf](http://www.epa.gov/oerrpage/superfund/programs/clp/download/fginorg.pdf).