Standard for a Power Plant Analytical Chemistry Quality Management System
Revision 7

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Power Plant Chemistry Advisory Group
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1.0 Introduction

1.1. The Power Plant Chemistry Advisory Group, established in 2004, is a voluntary assembly of individuals associated with the laboratory operations of organizations involved in the production of electricity from fossil and nuclear facilities. Part of the mandate of this group includes the exchange of information and best practices with regard to the proficient operation of power plant chemistry laboratories. This Standard has been produced to provide relevant information for power plant laboratory quality assurance (QA) and quality control (QC) programs. Individual plants may require higher standards in certain areas to meet their corporate objectives.

1.2. This Standard has been produced as a result of the Power Plant Chemistry Advisory Group determining that it would use ISO/IEC 17025:1999, General Requirements for the Competence of Calibration and Testing Laboratories, as a basis. Each clause of this referenced document was reviewed at the August 2004 General Membership meeting and either accepted, modified or deleted in order to customize this Standard’s requirements to the power plant chemistry laboratory environment. It contains all the requirements power plant laboratories have to meet if they wish to demonstrate that they operate a Quality Management System (QMS), are technically competent, and are able to generate technically valid results. If, due to business needs, a laboratory opts out of a requirement, the rationale, approval and circumstances shall be documented.

1.3. Detailed standards or “implementing tactics” are included in the appendices. These standards provide a practical interpretation of the requirements for a successful power plant chemistry QA/QC program. The order of the items covered in the appendices has significance. The appendices are ordered in a logical QMS program implementation sequence such that if an end-user wishes to start a QMS, or for that matter improve on an existing QMS, they should (since the preceding statement is “if you wish”) evaluate their existing QMS practices to the standards in the appendices in the order shown. In other words, one Standard builds on another. For example, the end-user should first establish the Valid Requirements, and then conduct Method Validation. In essence, determining the Valid Requirements for an analytical method must be done before an analytical method can be "Validated" to meet those requirements.

1.4. References noted in this Standard shall be reviewed on a two year cycle.

2.0 Scope

2.1. This Standard specifies the general requirements for the competence of power plant laboratories to carry out testing and sampling. Although non-standard methods are covered, laboratories are strongly encouraged in the use of standard methods.

2.2. This Standard is applicable to all power plant laboratories regardless of the number of personnel. When a laboratory does not undertake one or more of the activities covered by this Standard, those specific requirements do not apply.

2.3. Regulatory or local safety requirements that may be in conflict with this Standard, supersede this Standard.

2.4. This Standard is applicable to all work carried out, whether in central or in satellite laboratory facilities, including plant on-line instrumentation for which the laboratory is charged with assuring the production of competent data.
3.0 Organization

3.1. In order for the laboratory to achieve its goal of competent results, management support is required. The important commitment to meeting the requirements of this Standard is normally (although not necessarily) stated in the laboratory’s quality manual.

3.2. Part of this management commitment is the recognition that production is always to be considered as secondary to public safety, plant safety, or plant material condition. By predefining the responsibilities of key laboratory personnel, any analytically derived data that indicate a possible adverse effect concerning these items is to be reported to the appropriate authorities in a timely manner.

3.3. A policy, or the like, must be in place to specify management’s expectations regarding the pressures of production, versus the requirements of the laboratory’s Quality Management System (QMS), as well as its expectations concerning the professional conduct of laboratory personnel.

3.4. The laboratory organizational structure should be such that the responsibilities of technical management and quality management are distinct. It is the responsibility of the technical management to provide all resources necessary for the QMS. One member of staff shall be designated as the Quality Manager (however named) to ensure the QMS is effectively implemented and followed. As stated above, the roles of technical management and Quality Manager are preferably separate, however if the same individual must perform in both roles (as may be a business case requirement), how the person is to effectively separate both functions shall be documented. The Quality Manager should have direct access to the highest level of laboratory management responsible for the budgeting of all necessary resources.

3.5. Current authorized organizational charts shall be in place, defining both the reporting relationships between the laboratory and other parts of the organization, as well as the reporting structure for all positions within the laboratory organization. Up-to-date job descriptions should exist for all laboratory positions appearing on the charts.

3.6. In order to provide effective guidance to laboratory personnel, supervisory staff should have a thorough understanding of the requirements for the production of competent results (i.e., a thorough knowledge of this Standard) prior to the performance of their supervisory duties. This understanding may be demonstrated either through initial selection against a predefined criterion or through subsequent training.

3.7. Alternates for each key management position are to be pre-designated in order to accommodate unexpected circumstances.

3.8. Laboratory personnel must have the necessary training, resources and authority to effectively carry out their duties as well as to identify and act to mitigate the effects of departures from the QMS. If the authority to suspend activities, deemed to be outside the QMS specifications, rests with one authority, then that authority must be present for the activity to take place or an approved process must be in place to contact the pre-designated authority in a timely manner.
3.9. In those situations where the requirements of this Standard are such that they cannot be supported by the business needs of the organization, the details of the deviation, including the authority approving the deviation, must be documented.

4.0  **Quality Management System**

4.1. The laboratory shall document its policies, processes, programs and procedures.

4.2. It is recommended that the laboratory should maintain a Quality Manual (QM) that summarizes the goals of its Quality Management System (QMS). The content of the QM defines the laboratory’s standard of service and is, to a large extent, the laboratory’s response to meeting the management requirements of this Standard. This policy document should be authorized by the highest level of laboratory management that is responsible for the budgeting of all necessary resources. Items that should be addressed in the QM include:

- Management’s commitment to good professional practice and to the quality of its testing
- Management’s statement of the laboratory’s standard of service
- Objectives of the QMS
- Requirements that all personnel concerned with testing activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work
- Management’s commitment to comply with this Standard

4.3. The Quality Manual may include, but normally would reference, all other types of procedures included in the laboratory’s QMS (analytical, sampling, maintenance, procurement, etc. procedures) as well as define the technical management’s and the Quality Manager’s roles and responsibilities concerning compliance.

4.4. Other work instructions (e.g., ‘job aides’) shall be authorized for use and a process should be in place to ensure their ongoing validity.

4.5. Affected personnel should have ready access to, have read, and have an understanding of the appropriate QMS documentation.

4.6. For electronic systems that don’t allow for document signing, management’s endorsement and control of its policies and procedures should be ensured through other means (password protection, directory access, etc.).

5.0  **QMS Non-conformances, Corrective and Preventative Actions**

5.1. The laboratory shall have a process in place to address non-conformances in the QMS as well as being able to detect potential non-conformances in the QMS in order to prevent problems from occurring (i.e., being proactive in detecting adverse trends, promoting continuous improvement, etc.).
5.2. This process is to ensure prompt notification of detected non-conformances is made to the position within the laboratory that is authorized to halt work. Other mitigating steps defined in this process may include:

- Confirmation of the non-conformance
- Cessation of use of the method until the problem has been resolved
- Investigation as to whether other like processes are affected (e.g., did the non-conformance affect similar instrumentation?)
- Performing remedial actions to correct the problem
- Monitoring to ensure the non-conformance is corrected
- Evaluating the affect on laboratory output to determine if retesting should be performed

5.3. Timely notification to the appropriate authority is to be made if the non-conformance is determined to have had a significant influence. The cause, cure and effect of the non-conformance are to be documented. The position within the laboratory authorized to permit the resumption of work must be specified.

5.4. If it is determined that the non-conformance may reoccur, the laboratory’s corrective action process is to be promptly followed. This process shall address both unique and repetitive non-conforming work. The non-conformance may be associated with laboratory output or deviations from policies and procedures. The process identifies those positions within the organization that are authorized to deal with the required corrective actions. The extent of this evaluation is commensurate with the significance of the non-conformance.

5.5. Once the source of the non-conformance has been defined, an evaluation is performed to identify potential appropriate corrective actions. The laboratory then selects a corrective action that is most likely to eliminate the problem, prevent its reoccurrence (or occurrence in the case of potential non-conformance) and is appropriate for the magnitude of the problem. All changes are documented.

5.6. The laboratory’s program shall also ensure the continuous improvement of its QMS. The program should be proactive in highlighting potential non-conformances and areas for improvement. Error-likely situations are to be sought out, identified and corrected through preventive actions.

5.7. Once the corrective action(s) have been implemented, they are monitored to evaluate their effectiveness in correcting the non-conformance or area for potential non-conformance. Results of these measures shall be documented.

5.8. In those situations where the laboratory determines the selected corrective action is not appropriate for the magnitude of the problem, the circumstances are to be documented and the original acceptance requirements shall be reevaluated.

6.0 Valid Requirements

6.1. In order for a laboratory to develop a suitable QMS, the Valid Requirements (i.e., required accuracy and precision) of each technique shall be determined. Once these Valid Requirements are established, the items indicated below may be taken into account in order to select the correct methods, equipment, training, etc.
6.2. The ability of a laboratory to produce competent results (i.e., results capable of meeting the Valid Requirements) is determined by:

- Test methods and method validation
- Equipment
- Human factors
- Measurement traceability
- Accommodation and environmental conditions
- Sampling and sample handling

7.0 Document Control

7.1. The laboratory shall have a process in place to control all documentation related to its ability to produce competent results. All procedures of a given type (sampling, analytical, maintenance, etc.) are to have their format specified and all necessary successive steps are to be adequately documented.

7.2. The document control process shall ensure that:

- Copies of required documentation are easily retrievable by staff requiring them to perform the function.
- The documents are reviewed at a predetermined frequency.
- A specified process is used to ensure obsolete documents are removed from use promptly.
- Obsolete documents, retained for legal or knowledge preservation, are segregated and marked in such a way so as to ensure they are not used inadvertently.

7.3. All documents (procedures, manuals, wall charts, drawings, etc.) shall be approved for use by a designated authority. An easily retrievable master list of current approved documentation should be available. If hard copy documentation is used, its distribution shall be controlled. If electronic copy documentation is used, write access should be restricted to designated personnel.

7.4. All documentation should be uniquely identified. All procedures are to be reviewed and approved prior to their use (signature of approving authority for hard copy procedures, password or the like for electronic procedures).

7.5. The document control process shall also specify:

- The process to be used to make document revisions. The approval authority for the revision is to be the same function as that which performed the original review or by a function listed in the document procedure.
- Under what conditions and who may make amendments to documentation by hand. The process should indicate for what length of time the amended procedure may be in effect before a revised procedure must be issued.
- The functions authorized to make changes to documents maintained in electronic systems. If another department (e.g., word processing, etc.) controls part of the process, then that department’s applicable procedures should be referenced.
7.6. In order to make certain that the laboratory’s documentation is maintained up-to-date, a process shall be in place to ensure the laboratory is made aware of changes by external groups. These groups may either be within the parent organization or independent organizations (regulator, etc.). The process shall determine if any actions are needed within the laboratory as a result of changes to such external body’s documents or regulations.

8.0 **Test Methods and Method Verification/Validation**

8.1. The laboratory must have approved processes in place to ensure Valid Requirements are met. **All methods (procedures) used within a laboratory shall state that the method does not exceed the valid requirements**

8.2. Whenever possible the test methods and procedures used are based on one of the following:

- Internationally or nationally accepted standards
- Those published in reputable scientific journals
- Those specified by the manufacturer of the equipment
- As specified by the regulating authority

8.3. If the laboratory’s procedure closely follows the published procedure, the laboratory must only verify its ability to properly perform the test and obtain the published outputs (e.g. method detection limit, linear range, precision, etc.). All verification data must be documented.

8.4. The laboratory will validate all methods that do not closely follow published methods as described above. Validation is required to ensure, through the provision of objective evidence, that the method is adequate for its intended use. It is a measure of the performance parameters of the method versus the Valid Requirements for the analytical data produced. A valid method should be capable of producing data that is capable of attaining statistical control. Qualified personnel equipped with adequate resources and following approved procedures, are to carry out the validation. The personnel carrying out the validation should be independent of the method developer.

8.5. The validation is to be as extensive as required to meet the needs of the samples to be analyzed (e.g., if only feedwater samples are to be analyzed, then the validation must demonstrate the method’s ability to perform the analysis in the feedwater matrix). Documented evidence of the validation and approval for use for specific applications must be recorded.

8.6. Some validation methods that may be used include:

- Analysis of Standard Reference Materials (SRM) having the same or very nearly the same matrix as the test samples
- In the absence of suitable SRMs, the analysis of reference materials that are similar in all respects to the test samples. The validity of the reference material used must be documented.
- The use of an alternative method
- The use of Recovery Studies (i.e., Standard Addition)
8.7. Apparent differences in results obtained between the non-standard method being validated and the method(s) above are statistically analyzed to determine their significance.

8.8. Validation requirements depend in large part on the extent of the deviation from the original method. Some parameters that may be determined include:

- The scope of the method and any known interference
- The method detection limit
- The range of concentration over which the method is valid
- The number of standards required for calibration
- Precision and bias
- Intra-laboratory and Inter-laboratory variations

8.9. Whether the method is verified or validated, the laboratory has a process in place to ensure sample results meet predetermined Valid Requirements. Control charts are developed, implemented and used to ensure the analytical system is maintained within acceptable limits. The laboratory is to have a process in place to provide, upon request from the appropriate authorities, an estimation of uncertainty for all results generated (it is not necessary to provide an estimate of uncertainty with each result generated). The laboratory participates in proficiency testing programs or round robins at a regular frequency to evaluate the laboratory’s performance against predetermined conditions.

9.0 **Equipment and Instrumentation**

9.1. Technical management is responsible for furnishing the laboratory with the necessary equipment for it to meet the Valid Requirements. Any equipment used on a temporary basis (e.g., during repairs to permanent equipment, short-term projects, etc.) and used in such a way so as to monitor these requirements, must achieve the same level of performance as the permanent equipment. Associated labware is cleaned in a manner appropriate for the test.

9.2. All new equipment and equipment that has undergone significant repair or modification must be checked prior to being placed in service in order to ensure it meets the Valid Requirements. Key instrument parameters shall be monitored on a routine basis.

9.3. Only authorized personnel operate the equipment. Job performance measure (or the like) documentation is maintained current. The training program identifies those tasks requiring retraining and the retraining shall take place on a scheduled basis. Routine checks are in place to ensure the ongoing competence of the authorized staff to perform these operations. All equipment documentation (e.g., procedures, manufacturer’s manuals, etc.) is available for routine use.

9.4. All equipment is uniquely identifiable to allow for analyses to be easily traced back to the equipment used. Single pieces of equipment (e.g., if the laboratory only has one AA spectrometer) are by their nature unique, however multiple instruments of a given type (e.g., if the laboratory has two pH meters) are to be uniquely identified (e.g., pH meter ‘A’, pH meter ‘B’).
9.5. Records are maintained to indicate the ongoing ability of the instrument to meet Valid Requirements. These records date from the initial indication of compliance with specification (including calibration certificates), through a planned and implemented maintenance schedule, to all maintenance and repairs performed.

9.6. Equipment that is, or is suspected to be, defective is taken out of service and clearly marked so as to ensure that it is not used inadvertently. A review is undertaken to determine any possible negative effect on previously produced data. If suspect results have been generated, the laboratory’s process to address non-conforming data is instituted.

9.7. Instruments requiring calibration are to be calibrated prior to initial use. Calibration expiry dates are readily available (this does not apply to instruments requiring calibration prior to each use). Calibrations are protected by appropriate means (procedural, passwords, locking devices, etc.) from changes that would invalidate the results.

9.8. For instrumentation not requiring calibration prior to each use (e.g., liquid scintillation counters, gamma spectrometers, conductivity meters, etc.) interim checks are made to ensure the instrument is operating within predetermined acceptance limits. The requirement for these interim checks is documented.

9.9. A process is in place to ensure instruments that leave the direct control of the laboratory (off site for repair, etc.) are checked prior to return to service to demonstrate their continued satisfactory operation.

10.0 Measurement Traceability and Quality Control Checks

10.1. All laboratory equipment used that has a significant effect on the test result must be calibrated. Whenever possible, these calibrations must be traceable back to a national or international standard. The laboratory must have a process in place that describes:

- The selection of approved calibration solutions (reference materials, certified reference materials, etc.) and devices (certified thermometers, primary weights, etc.)
- The procedure for use of these items (or references associated procedures)
- The conditions under which these items are to be stored (temperature, etc.) and secured (locked cabinet, etc.)
- The method to be used to ensure their transportation does not adversely affect performance

10.2. For those instances when a standard or device is not traceable, the laboratory shall have a process in place to describe an acceptable alternative (e.g., the preparation of chemical standards from pure salts, etc.). The degree of rigor required to verify the accuracy of the standard or device (analysis by an independent laboratory, etc.), is dictated by the significance of the test.

10.3. Standards (Certified Reference Materials, etc.) and devices (primary thermometers, etc.) supplied to the laboratory by a competent calibration source provide the required traceability. The calibration certificates issued by the calibration laboratory must contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification.
10.4. Standards produced internally are checked against traceable standards whenever practical.

10.5. Laboratory procedures dictate the frequency of QC checks to monitor overall performance. These intermediate checks are whenever possible not produced from the same source of material as the standard used to calibrate the analytical system.

11.0 **Personnel**

11.1. Management is to ensure that all staff that is authorized to perform tasks independently in the laboratory have demonstrated their capabilities through appropriate job performance measures.

11.2. A training program, based on the Systematic Approach to Training (SAT), is developed to ensure laboratory staff has the necessary skills and knowledge to competently perform their duties. All training shall be documented, including any in-house training provided by the laboratory. The SAT program shall also identify those tasks requiring retraining in order to ensure the ongoing competence of individuals. Personnel still undergoing training, perform these tasks under close supervision. In any case, adequate competent supervision is provided for all personnel.

11.3. The laboratory shall maintain a current listing of which staff are authorized to perform particular types of tasks such as sampling, conducting tests, approving results, operating equipment, evaluating quality control data and trends, conducting internal audits and authorizing procedures. This information should be readily accessible.

11.4. Current job descriptions shall be maintained for all positions appearing on the Laboratory Organization Chart. There shall be a process in place to ensure the incumbent(s) have read and comply with the contents.

11.5. There is an intra-laboratory program in place to regularly evaluate the laboratory staff’s proficiency in performing analytical determinations against predetermined conditions.

11.6. If sub-contracted personnel are used, they are to:

- Be supervised by a competent individual
- Have the required knowledge and skills
- Be deemed competent in the designated tasks
- Perform to the laboratory’s QMS standards

12.0 **Accommodation and Environmental Conditions**

12.1. The laboratory’s environmental conditions shall be adequate to allow for Valid Requirements to be achieved. The necessary environmental conditions shall be documented for all laboratory facilities.

12.2. Acceptance limits are set for those environmental conditions that could adversely affect the quality of results. Actions to restore the environmental conditions (e.g., supplemental heating/cooling, ventilation) and changes to the quality assurance requirements (e.g., an increase in the frequency of quality control checks) are taken to mitigate the adverse effect when acceptance limits are exceeded.
12.3. Laboratory activities are segregated to minimize the possibility of both chemical and radiological cross contamination. Key items that have a direct effect on the quality of data (certified standards, primary thermometers, primary weights, etc.) are kept secure and stored under appropriate conditions.

12.4. Standards of housekeeping are identified and routine checks are made to ensure standards are being met. A safety committee or other similar process is in place, with routine feedback to management on safety issues. Safety issues are documented with required actions tracked to ensure timely resolution.

13.0 Sampling and Sample Handling

13.1. Obtaining representative samples is critical to the production of competent results. For samples obtained by laboratory personnel, the laboratory shall have approved sampling procedures in place. These procedures are part of the laboratory’s Document Control process and shall specify or reference, as appropriate:

- Sample location (room number, valve number, etc.)
- Flow rate, flush time
- Operating state for which the sample is valid
- Approved sample container
- Approved alternate sample point
- Sequential steps to be taken to obtain the sample
- Sample preservation requirements and holding time limitations
- Personal protective equipment

13.2. For plant systems, the laboratory shall have a process in place to allow for the determination of the status of the unit and the system being sampled at the time the sample was obtained.

13.3. Regardless of whether or not laboratory staff obtain the sample, the laboratory has a process in place to ensure:

- Samples are received, stored and handled in a manner that protects their integrity. Each sample is uniquely identified. If it is necessary to sub-divide a sample, the process ensures that each of the parts is identified as part of the original.
- Each sample is examined on receipt to ensure it is fit for purpose. If there is doubt as to the analytical requirements for samples obtained by non-laboratory personnel, the external group is contacted for further instructions prior to proceeding with analysis.
- Samples are maintained in such a way so as to avoid deterioration and damage. The process must ensure this takes place, whether the sample is in direct control of the laboratory or not.

14.0 Control of Data

14.1. Calculations involved with the production of analytical data (corrections for dilution, the use of certain factors, etc.) are a potential source of error. In order to minimize this error, the correctness of calculations and data transfers is assured by independent verification and rationality checks. When factors embedded in spreadsheets, etc. require updating, a process is available to ensure the update’s correctness. Updates are made and verified by authorized personnel.
14.2. Commercial off-the-shelf software, used to acquire, process, record, report, store or retrieve data, is considered sufficiently validated, however the laboratory shall have a process in place to validate user-developed software or off-the-shelf software that has been modified by the user.

15.0 **Reports**

15.1. Power plant (PP) laboratories traditionally report the vast majority of their generated data to internal customers only (i.e., other departments within the plant or within the parent organization). Results are reported in the way that best meets the business needs of the organization. Although a simplified reporting format is used, results, either on paper or electronically are presented in a clear, unambiguous manner, which meets the needs of these customers.

15.2. Besides the results themselves, information such as the sample description, date and time of sampling and analytical units (if appropriate) are made evident to the user. Other background information such as analyst, unique sample identifier, method used, date and time of analysis, equipment used, quality assurance results, traceability, etc. should also be readily available.

15.3. For those situations in which a deviation from the normal protocol was used to generate the result (e.g., the use of a temporarily modified procedure or abnormal laboratory environmental conditions), a notation shall be included with the result to alert the end user to the possible adverse effect on the validity of the data.

15.4. PP laboratory personnel do not normally offer opinions and interpretations of the results, however pass-fail statements against predetermined specifications may be included with the reported value.

15.5. When a subcontracted laboratory is used to generate results that are reported, the identity of the subcontracted laboratory need not be included with the PP laboratory’s data, however it is readily available.

15.6. If it becomes apparent that an invalid result has been reported, the laboratory must, when required, ensure that the proper authorities are notified so as to help minimize the effect of the error. Corrected results shall be made available to those who may have acted on the original incorrect result. Amended results are traceable to the original result and reported according to this Standard.

16.0 **Control of Records**

16.1. The laboratory shall have a process in place to control all records related to its ability to produce competent results. The process shall ensure that records are stored in such a way so as to ensure legibility and to prevent damage or loss. Retention times of record types are established so as to ensure both Quality Assurance and regulatory requirements are met.

16.2. The laboratory’s Control of Records process shall ensure the prevention of alteration of stored records and the backup of electronic records. In those instances when the backup is the responsibility of another department, that department’s process is referenced.
16.3. Records shall be maintained so as to allow for the determination of factors affecting uncertainty and to allow the test to be repeated under conditions as close as possible to the original ones. Recorded data shall include such items as personnel involved in the sampling, analysis, derivation and verification of results, as well as their authorization to do so. In addition, records of dates and times, sample point, instrument parameters, instrument identification, quality control results, procedure(s) used, calibration certificates, etc. shall be readily retrievable. Reportable data shall be recorded in a permanent medium (e.g., no pencils). Any standardized forms used to record raw data shall be authorized. Raw data, as well as the data recorder, should be noted at the time it is made and be easily identifiable to the specific task.

16.4. The process used to make changes to correct originally recorded raw data, whether on paper or electronically, should be approved and ensure that the original data is not obscured or lost. The identity of the person making the change, as well as the date of the change, is evident.

17.0 Purchasing Services and Supplies

17.1. The laboratory shall have an acquisition process in place to ensure that the subcontracting of tests and the purchasing of supplies or services does not adversely affect its ability to produce competent results.

17.2. If the laboratory subcontracts work to another laboratory, the subcontracted laboratory shall meet the requirements of this Standard for the work being done. This requirement is not applicable if a regulator mandates use of the subcontracted laboratory. The laboratory is responsible for the competence of the subcontracted laboratory’s work.

17.3. If the subcontracted laboratory is not compliant with this Standard, then appropriate corporate personnel shall conduct an onsite assessment of the subcontracted laboratory’s facilities to evaluate its ability to produce adequate results. If the work is to be carried out over a protracted period of time, regular reassessments should be completed to ensure the ongoing competence of the subcontracted laboratory. Documentation must be maintained for those laboratories that are deemed to be competent subcontractors.

17.4. In those situations where part of the acquisition process is performed by another department in the parent organization, the laboratory’s process will address that portion under its control and reference the other department’s practices for their part of the process.

17.5. Storage of reagents and other consumables are to meet manufacturer’s recommendations or other good laboratory practices, whether the storage is within the laboratory or in a central storage area.

17.6. Prior to use, reagent and consumable materials that may affect the quality of tests will, as a minimum, be inspected. Deviations (e.g., past expiry date, seals not intact and in place, signs of precipitation, etc.) should be documented and investigated as to cause.

17.7. Calibration sources and services are to be purchased from accredited organizations.

17.8. Regardless of whether the laboratory is solely, or partially responsible for purchasing of services and supplies, the process should allow for the laboratory to specify the technical content of the material or service. Types of material (class, grade, etc.) are to be specified as required in order to maintain the quality of the laboratory’s output.
17.9. The process shall ensure that substitution of laboratory specified services and supplies do not take place without the concurrence of the laboratory. A list of acceptable consumables, suppliers and services is maintained.

18.0 Internal Audits

18.1. An independent internal assessment of the laboratory's performance, against the requirements of this Standard, provides an effective measure of the current state of the QMS.

18.2. These internal audits are to be conducted periodically by trained and qualified personnel who are, whenever possible, independent of the activities being audited. In those circumstances when it is not practical for the personnel to be independent of the activities being audited, the audit results should be reviewed by qualified personnel external to the laboratory (i.e., from another department) to ensure the process used and the interpretation of the results was completed in a competent manner.

18.3. If the results of the audit cast doubt on the ability, or previous ability, of the laboratory to produce competent results, the laboratory will notify all authorities that may have acted on the potentially invalid data. The laboratory will take timely corrective actions to eliminate the cause and prevent the reoccurrence of the non-conformity.

18.4. Records are kept of the audited activity, the findings and corrective actions. An overview of the audit’s findings is made available, upon request, to all authorities that utilize the laboratory’s output.

18.5. Any corrective action put in place is monitored, with its effectiveness recorded. The degree of monitoring is commensurate with the magnitude of the problem.

18.6. Although the quality system is to be audited annually, it is not necessary to audit every process or aspect at one time.

18.7. A QMS Assessment (Appendix A-12) can be used to meet the requirements of an internal audit.

19.0 Management Reviews

19.1. The laboratory management team, including but not limited to, the highest level of laboratory management responsible for the budgeting of all necessary resources, the technical management, the Quality Manager as well as others deemed appropriate, meet periodically to ensure the QMS continues to be effective.

19.2. The process is documented and subjects discussed typically include the following:

- A review of the laboratory’s policies and procedures to ensure both their continued adequacy and that they are being reviewed at the predetermined frequency. Document change turnaround times are monitored to ensure they achieve the desired goals, both for routine reviews and for ad hoc changes.
- The effectiveness of corrective actions put in place for those non-conformances identified in recent internal audits are reviewed.
- The effectiveness of corrective or preventative measures put in place for actual or possible non-conformances are reviewed.
• The results of assessments performed by external bodies and any actions arising from the assessments are reviewed.
• Changes to the volume and type of work, both present and projected, are reviewed to ensure adequate resources are available to maintain the QMS. If resources cannot meet increased demand, then priorities are adjusted accordingly and any changes are communicated to laboratory personnel.
• The results of the laboratory’s program, designed to actively encourage customer feedback of laboratory operations, are reviewed; both to determine trends and to ensure the adequacy of actions taken in response to previous comments.
• The training programs are reviewed to ensure the ongoing competence of laboratory staff. The review is to ensure adequate training is taking place, targets are being met and the quality of the training is adequate.

19.3. Minutes of management review meetings are kept. Actions arising from the meeting should (the minutes track the action items) be placed in an action-tracking program. Meetings are typically held once per year and cover all topics however, if it better meets the business needs of the organization, the suggested topics may be discussed at other management meetings during the year. In any case, all topics must be covered and minutes shall be kept.
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Change History

1. Revision 0, December, 2004
   a. Original revision

2. Revision 1, December, 2005
   a. Added Appendices
      - Standard for Valid Requirements
      - Standard for Method Validation
      - Standard for LOD/LOQ/UQL
      - Standard for Determining QC Check Concentration
      - Standard for Control Charting
      - Standard for Inter- and Intra-laboratory Programs
      - Standard for Significant Digits
      - Standard for Stating Analytical Uncertainty

3. Revision 2, February, 2008
   a. Edited should to shall where appropriate throughout
   b. Added references to on-line instrumentation throughout
   c. Added Section 19 “On-line Instrumentation” pg 13 of 13
   d. Added clarification on using correlation coefficient to the Standard for Method Validation pg 1 of 3
   e. Added clarification for determining detection limits to the Standard for LOD/LOQ/UQL pg 2 of 2
   f. Added considerations for response time for on-line instruments to the Standard for Determining QC Check Concentration
   g. Added considerations for the frequency of QC Standard analysis for on-line instrumentation to the Standard for Control Charting
   h. Added clarification that Appendix A6 – “Standard for Inter- and Intra-laboratory Programs” does not apply to on-line instrumentation

4. Revision 3, April, 2008
   a. Added Appendix A9 – Standard for On-line Instrumentation

5. Revision 4, August 2008
   a. Added a note to the ‘Scope’ to apply the requirements of the Standard to whomever performs the analyses
   b. Added a note to Appendix 6 to indicate that labs are to participate in interlaboratory programs per category of radioanalytical analysis
   c. Added Section 20
   d. Added Appendix A10 – Standard for Analytical Radiation Detection Instrumentation

6. Revision 5, August 2009
   a. Added appendix for conducting a QMS Assessment
   b. Changed wording for calibration frequency in appendix 10, radiochemistry
   c. Rename chapter eight to Equipment and Instrumentation
   d. Moved Chapter 19 and 20 into Appendices A9 and A10 respectively

7. Revision 6, August 2010
   a. Changed “GAP Analysis” to “QMS Assessment”
   b. Changed bulleted format to numbered format
   d. Added Appendix A11 – Standard for Bench Top Instrumentation

8. Revision 7, April 2012
   a. Formatted tabs, numbering, and punctuation to be more consistent with generic nuclear procedure guidelines
   b. Corrected typographical errors
   c. LOD/LOQ reworded to make it clearer
   d. Table 1 in A11 updated
A1 - Standard for Valid Requirements

VALID REQUIREMENT

1.0 General

1.1. A successful power plant chemistry Quality Management System defines the resources necessary to ensure the laboratory has the capability to produce data appropriate for meeting its business needs. Being appropriate for meeting its business needs means the data produced has an adequate level of accuracy (bias) and precision (reproducibility) to fulfill its intended use in support of plant operation. Some data may require significant resources to provide a high degree of accuracy and precision, while other data may require fewer resources to meet a lower degree of accuracy and precision. For the purposes of this standard, an adequate level of accuracy and precision for meeting the business needs for an analytical method will be referred to as a Valid Requirement (VR).

1.2. Ideally, VRs are determined as early as possible in the laboratory’s design to allow for the selection of:

- Appropriate facilities, equipment and procedures
- Suitable sampling and sample handling techniques
- Proper training of staff
- Specific quality control and traceability practices

1.3. For existing labs, VRs shall be determined in an orderly fashion with the results being used to replace inadequate instruments or provide necessary facilities, training or procedures.

2.0 Determining Valid Requirements

2.1. VRs are determined with consideration of regulatory and operating requirements. These requirements should be determined by review of appropriate documentation and through interviews with appropriate plant staff. In the absence of regulatory requirements, considerations for operational decision making requirements take precedence. If no regulatory-based VRs exist, then the operational needs of the plant determine the required level of accuracy and precision. When determining VRs, the following items may be evaluated:

- Regulatory considerations
- Action limits or Limiting Condition for Operation (LCO)
- Nuclear Systems Supplier or Original Equipment Manufacturer (OEM) requirements
- Other system specifications
- Typical analyte concentration
- Lower limit of detection
- Smallest reportable increment for determination of trends
- Consequences of using erroneous data
- Customer needs
- Instrument response time
3.0 Selecting Suitable Analytical Methods

3.1. Once the required level of accuracy and precision (VR) has been determined, an evaluation for a suitable analytical method is performed. In this evaluation, an appropriate method is sought that will provide the required level of accuracy and precision (i.e., meet the VRs). When selecting an analytical method to be used to meet the VRs, the following may be evaluated:

- Personal safety
- Instrument safety
- Other hazards associated with analysis (e.g., hazardous waste generation)
- Dose
- Complexity of analytical method
- Interferences
- Laboratory analytical capability and environmental limitations
- Frequency and timeliness of analysis
- Analyst training and qualification requirements
- Instrument life-cycle cost
- Overall cost of analysis

3.2. Ultimately, the process of determining VRs and evaluating analytical methods capable of meeting the VRs will yield a list of VRs and appropriate analytical methods that may be used. It may be necessary to iterate several times between identifying the VRs and selecting appropriate methods to finally achieve an appropriate balance between VRs and analytical capabilities.

3.3. Iteration may be necessary if the only methods found are too hazardous, expensive, or otherwise incompatible with the lab. In this case, it may be necessary to reevaluate (iterate) the determination of VRs in light of these incompatibilities.

4.0 Regulatory Considerations

4.1. In those situations when an external body (e.g., regulatory agency or other authority) states a required level of accuracy and precision, then these stated limits become the VRs and the laboratory must take suitable steps to ensure these limits are met.

5.0 Stating Valid Requirements

5.1. How VRs are determined and stated shall be clearly documented. Different VRs may be stated at different concentration ranges or for different systems. VRs can be expressed a number of ways including:

5.1.1. A maximum deviation from a target value (bias):
Statistical evaluation for significant bias is assessed using the Student’s “t” test at 95% confidence coefficient, two-tail test. The analysis can be determined to meet the VR if the Student’s “t” test is passed. A statistically significant bias may be tolerated in cases of extreme precision. In this case, the analysis can be determined to meet the VRs if the data average is within ± 1 standard deviation (SD) of the target. (4a)
5.1.2. A maximum variation at a specified concentration (precision): Statistical evaluation for precision is assessed using RSD %. The VR shall be stated at the 95% confidence interval (CI). The analysis can be determined to meet the VR if the calculated RSDx2 is less than the VR at the 95% CI.

Note: The factor of two (2) above represents the 95% CI.(4b)

5.1.3. A percent or stated value maximum error (combining bias and precision): Various methods exist for combining bias and precision into an “accuracy statement.” Statistical techniques can be used to “pool” errors or errors can be combined in a statement of total error without statistical treatment. In either case, a significant error exists when the observed error, however calculated is greater than the maximum allowable error. The maximum allowable error shall be stated at the 95% CI.(5)

6.0 Example, Determining Valid Requirements

6.1. Consider the hypothetical case in which an analyte is to be controlled between 6 and 8 ppm.

6.2. Upon review it is determined that:

   6.2.1. There are no regulatory or other authority-specified accuracy or precision limits.
   6.2.2. Operations state they want to be able to take corrective actions if the analyte concentration approaches either limit.
   6.2.3. Operations believe a precision of ± 0.1 ppm would provide enough operational flexibility.
   6.2.4. No other more restrictive limits are found.

6.3. Conclusion:

   6.3.1. Chemistry, in collaboration with operations, establishes the VRs at:

       • ± 0.1 ppm at 95% confidence coefficient for precision
       • Passing the Student’s “t” test at 95% confidence coefficient for determination of acceptable bias.

7.0 Example for the Comparison of Valid Requirements to Several Proposed Analytical Methods

7.1. Suppose all analytical methods pass the Student’s “t” test for determination of bias. Therefore, all methods could be acceptable based on accuracy limits alone.

7.2. Suppose the analytical instruments capable of measuring the analyte in the range of 6-8 ppm have published precision statements, at 95% confidence coefficient of:

       • Pocket Kit (± 2 ppm)
       • ICP-MS (± 0.001 ppm)
       • IC (± 0.08 ppm)
       • SIE (± 0.15 ppm)
7.3. Suppose published prices are:

- Pocket Kit ($100)
- ICP-MS ($150,000)
- IC ($50,000)
- SIE ($4,000)

7.4. Conclusion:

7.4.1. Clearly the Pocket Kit, although the most economical and potentially the easiest method to use, is not a suitable choice since the stated precision is poor compared to the VR precision.

7.4.2. The ICP-MS easily meets the VR, but its high cost would make it an expensive choice.

7.4.3. The IC’s precision is better than the VR and its use should be considered.

7.4.4. Although the SIE’s precision is greater than the VR, it may be considered. A closer evaluation of the initial determination of VRs should be made to determine if the SIE’s use could be adequate. Operations should be asked if the VR of 0.1 is firm or if a VR of 0.15 could be acceptable. If the limit of 0.1 is firm, then the SIE’s use shall be dismissed. If the limit of 0.15 is acceptable, then the cost savings, space savings, etc. make an acceptable, perhaps a better choice than the IC’s.
Note: This document provides a list of relevant references. The references below are in no particular order of importance. The user of these standards is to be cognizant of regulations and commitments that may lend favor to one reference over another.

REFERENCES

Philosophical References


Calculation References

A2 - Standard for Method Validation

METHOD VALIDATION

1.0 General

1.1. Most power plant chemistry analytical methods are derived from existing published methods,(1,2) or from equipment manufacturer instructions. The purpose of method validation is to demonstrate that the published method can be used by the power plant laboratory such that the analytical data produced is capable of meeting the station’s Valid Requirements. (3) This standard’s aim is to provide relevant information for power plant chemists to demonstrate their ability to use published methods at their location. Care should be taken to select appropriate methods and equipment that meet the required accuracy and precision for the data produced.

1.2. Method Validation shall consist of the following key elements:

- Validating the number of standards to be used for method calibration.
- Verification of calibration curve.
- Verifying method limit of detection (LOD) and upper quantification limit (UQL) are determined.
- Determining matrix interference using spiked sample analysis.
- Determining blank interference.
- Comparison to existing methods.
- Method re-validation.

2.0 Validating the Number of Standards to be Used for Method Calibration

2.1. Calibration curves are normally defined as linear or non-linear. For linear calibrations, a minimum of three (3) calibration standards shall be used at concentrations spaced across the range of interest.(4) The minimum acceptable correlation coefficient (r) for a linear curve is recommended as 0.995. Once the instrument’s response data have been attained, a Rationality Check is made.

Example Rationality Check - Suppose a calibration is performed with the following data pairs:

<table>
<thead>
<tr>
<th>Instrument response</th>
<th>Concentration in ppb</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>5</td>
<td>7.5</td>
</tr>
</tbody>
</table>

Using Microsoft® Excel to calculate the correlation coefficient “r” yields an r = 0.995. Performing a Rationality Check by calculating the relative error for each calibration point yields;

<table>
<thead>
<tr>
<th>Instrument response</th>
<th>Concentration in ppb</th>
<th>Rationality Check Using relative error in %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>7.5</td>
<td>50</td>
</tr>
</tbody>
</table>
This example Rationality Check shows a case where even though the correlation coefficient “r” meets the recommended value of 0.995 or greater, the relative error for the last calibration point is 50%. Agreement criteria for Rationality Checks should not exceed the Valid Requirements for that test. For non-linear calibrations a minimum of five (5) standards shall be used at appropriate concentrations to adequately model the characteristics of the calibration curve.\(^\text{(4)}\)

2.2. Unless otherwise specified by the published method, a blank, although often used to zero the instrument and so is part of the curve, is not to be counted as a calibration standard. The lowest standard shall be at least three (3) times LOD (if the range of interest goes this low).

3.0 Verification of Calibration Curve

3.1. Each calibration curve shall be verified by analyses of QC check standard independent of the calibration standards. The QC check standard is prepared from another supplier, or at least from a separate lot number from the calibration standard. Verification of the calibration curve shall be performed using at least one QC check standard.\(^\text{(4)}\) Optimally the concentration of the QC check standards is different than the calibration standard. Acceptance criteria are determined based on the Valid Requirements. If Valid Requirements are not used, the verification results and the basis for the acceptance criteria (e.g., QC limits, 80-120%, etc.) are to be documented.

4.0 Verifying Method Limit of Detection (LOD) and Upper Limit of Quantification (UQL) are Determined

4.1. Each analytical method important to plant operations shall have a stated LOD and UQL.\(^\text{(4)}\)

5.0 Determining Matrix Interference Using Spiked Sample Analysis

5.1. Spiked samples are used to evaluate matrix interference for each analytical method important to plant operations. Each expected sample matrix shall be spiked with analyte and with each major matrix constituent to evaluate possible interferences. If a sample is normally diluted, the sample shall be spiked after dilution. A simulated sample may be prepared for spiking if an actual sample is not available. The concentration of the spiked samples is approximately 50% to 100% above the sample concentration.

5.2. The spike concentration shall be enough to raise sample concentration to at least greater than 10 times LOD but less than the UQL.\(^\text{(4)}\)

5.3. The volume of standard used to spike the sample shall constitute 10% or less of the final spiked sample solution volume. Acceptance criteria are determined based on the method’s Valid Requirements. The matrix spike results and the basis for the acceptance criteria (e.g., QC limits, 80-120%, etc.) if Valid Requirements are not used shall be documented. In spiking each sample matrix, only one spiked sample for each analyte and major matrix constituent is required to be run on each sample matrix if that spike passes the acceptance criteria. If it does not meet the acceptance criteria, a sufficient number of additional spikes shall be analyzed in order to identify and resolve the source of the interference.
5.4. Routine analysis of spiked samples may be required by other authorities. For the purposes of this standard, spiked samples are used to determine the effect of sample matrix on measuring analyte concentration. Therefore, analysis of spiked samples is only warranted for initial method validation or when matrix changes are encountered. The chemistry staff must have procedures in place to ensure a changing matrix does not have an adverse effect on measuring any analyte.

6.0 Determining Blank Interferences

6.1. Blank interferences can affect analytical method quantification. Therefore, each analytical method important to plant operations is to be evaluated for blank interferences. Evaluation includes as appropriate:

   6.1.1. Sample blank
   6.1.2. Reagent blank

6.2. Identified interferences are documented, resolved or adjusted for as appropriate.

7.0 Comparison to Existing Methods

7.1. An acceptable method for validating a new method is to compare the results of both methods to a common sample or standard. If the two methods provide results that are not statistically different from each other then the new method may be deemed analytically equivalent. If Valid Requirements are not used, the verification results and the basis for the acceptance criteria (e.g., QC limits, 80-120%, etc.) are to be documented.

Note: Statistical tests such as the Student’s “t” test and the Fisher “F” test may be used. (6) This comparison does not provide assurances that the new method’s LOD, UQL, uncertainty, matrix affects, blank interferences, etc. are similar to the original method. These determinations/verifications must also be completed as appropriate.

8.0 Method Re-validation

8.1. Method re-validation is required when any part of the analytical method undergoes a change, such as major maintenance, reagent modification or matrix change.
Note: This document provides a list of relevant references. The references below are in no particular order of importance. The user of these standards is to be cognizant of regulations and commitments that may lend favor to one reference over another.

REFERENCES

Philosophical References

A3 - Standard for LOD/LOQ/UQL

LOD/LOQ/UQL

1.0 General

1.1. Many references exist that provide adequate discussion on low-level detectability. Most references evaluate the standard deviation (SD) of a blank or low-level standard and mathematically derive a detection limit by multiplying the SD by a factor. The determination of these values are not exact calculations, rather they are the best estimates derived using conditions that simulate those of the sample. For the purpose of this Standard, the Power Plant Chemistry Advisory Group is providing relevant references for determining low-level detection limits (commonly referred to as Limit of Detection, LOD) for the betterment of the programs in place at participating laboratories. See references 2, 3, 4, 5, 6, and 7 below.

1.2. Although the preferred method of determining the LOD is statistical, if there is no regulatory or business need to utilize this method, or if the instrument does not allow for such a determination, then the instrument manufacturer’s LOD or operating range is used.

2.0 Reporting Data at or Near Detection

2.1. The purpose for determining a detection limit is to provide the user of the analytical data assurance that the quantity reported is in fact greater than background/blank, i.e., greater than zero. By definition analytical uncertainty at a concentration just greater than zero can be as much as 60% (e.g., Lloyd Currie’s Lc the “Decision point”). As analyte concentration increases, the relative uncertainty associated with analytical results decreases. Currie shows analytical uncertainty of approximately 30% at Ld the “Detection point” (also known as LOD) and 10% uncertainty at Lq the “Quantification Point” (also known as limit of quantification (LOQ)). This discussion shows that one purpose for establishing low limit detection points is to associate a level of uncertainty with analytical data. Data near Ld has greater uncertainty than data near Lq.

2.2. All analytical data have value. It is incumbent on the user of the data to be cognizant of the uncertainty associated with any data they may use for decision-making purposes. If data uncertainty is large due to measuring near the detection limit, then the user must take this into account when making operational decisions.

2.3. For the purpose of this standard, power plant chemistry low-level data will be reported down to LOD. Data less than LOD will be reported as the value of LOD.

3.0 Using the Lowest Non-zero Calibration Standard as LOD

3.1. Another practice is to not calculate LOD but rather develop a calibration range that encompasses the concentrations of interest and then use the lowest non-zero calibration standard as the LOD.
3.2. Using the lowest non-zero calibration standard as LOD is appropriate under the following conditions:

3.2.1. The lowest non-zero calibration standard is not less than the instrument supplier’s published detection limits.
3.2.2. There is no regulatory, business or other need to report concentrations below this concentration.

4.0 Frequency for Determining LOD

4.1. LOD is determined during initial method development or method validation.

4.2. Once established, LOD is evaluated periodically based on instrument/method stability. If the method is known to be stable for long periods of time then the LOD evaluation period may be long (e.g., several years between evaluations). The basis for the frequency of evaluations is documented for each important analyte.

4.3. Periodic evaluation of LOD by:

4.3.1. A statistical evaluation may be performed by comparing the SD of a low-level standard, analyzed seven (7) times over several days, to the SD used in the initial LOD determination.
4.3.2. The Fisher “F” test\(^{(8.a)}\) is an acceptable test for this analysis. Since the SD of a low-level standard may have a high uncertainty, a two-tailed 99\% confidence coefficient is recommended for the F test. If the evaluation shows no statistically significant change in SD (i.e., passes the F test) then the original LOD is retained. If the evaluation shows a statistically significant change in SD (i.e., fails the F test) then a new LOD is calculated.

5.0 Upper Quantification Limit (UQL)

5.1. The upper quantification limit for each method is equal to the concentration of the highest calibration standard, unless otherwise documented (e.g., in the case of on-line instrumentation if the Valid Requirement allows the use of the upper end of the manufacturer’s range).
Note: This document provides a list of relevant references. The references below are in no particular order of importance. The user of these standards is to be cognizant of regulations and commitments that may lend favor to one reference over another.

REFERENCES

Philosophical References

2. Protocol for the sampling and analysis of industrial /municipal wastewater (1999), MISA, Ministry of the Environment (Ontario)

Calculational References

3. ASTM D 4210-89: Standard Practice for Intra laboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data
5. Lloyd A. Currie, (1968) Limits of Qualitative Detection and Quantitative Determination
7. Ellis Horwood PTR Prentice Hall, Statistics for Analytical Chemistry, Miller and Miller
A4 - Standard for Determining QC Check Concentration and Frequency

QC CHECK CONCENTRATION

1.0 General

1.1. At least three (3) separate assessments are used to evaluate the effectiveness of a laboratory Quality Management System.

1.1.1. Laboratory capability is assessed through the participation in interlaboratory comparison sample analysis. (See Standard for Intra/Interlaboratory QA/QC)\(^{(1)}\)

1.1.2. Analyst capability is assessed through the participation in an intralaboratory QA/QC program. (See Standard for Intra/Interlaboratory QA/QC)\(^{(1)}\)

1.1.3. Instrument capability is assessed for drift/failure by analyzing a known standard at a specified frequency. (See Standard for Control Charting)\(^{(1)}\)

1.2. A separate assessment of instrument detection limits is performed (see A3 - Standard for LOD/LOQ/UQL)\(^{(1)}\)

1.3. This standard is written to provide additional information relevant to 1.1.3 above "Instrument capability is assessed for drift/failure by analyzing a known standard at a specified frequency" in as much as it provides a standard for determining the concentration at which a QC check standard is prepared for analysis.

2.0 Background

2.1. Historically in power plant laboratories, QC check standards were analyzed at a concentration approximately equal to the expected concentration of the system being monitored. This practice seemed reasonable when system concentrations, and therefore QC check standard concentration, were well above the limit of detection (LOD) for the analyte of interest. However, as system concentrations and therefore QC check standard concentrations have lowered, it has become more difficult to reliably prepare and analyze a QC check standard at this lower concentration. In some cases, the capability of the analyst to prepare a QC check standard near an LOD is tested rather than assessing instrument drift/failure. Therefore, in cases where the expected concentration is near the LOD, this historical practice has become unreasonable.

3.0 Objective of a QC Check Standard

3.1. The objective for analyzing a QC check standard, at a specific frequency and concentration, is to assess instrument drift/failure at the time of analysis. For this Standard, it is important to state that the purpose of analyzing a QC check standard is not to verify instrument LOD. Instrument LOD determination and verification are discussed separately (See A3 - Standard for LOD/LOQ/UQL).

3.2. Given the objective for analyzing a QC check standard is to assess instrument drift/failure, then the concentration of the QC check standard must:

3.2.1. Be at a concentration high enough to assess instrument drift/failure, while not being confounded by the error associated with low-level analysis

3.2.2. Be low enough to reflect instrument drift/failure in the normal working range of the instrument (i.e., the mid-point of the calibration)
4.0 Frequency of QC Check Standard

4.1. For instruments calibrated with each use, analyze a QC check standard at the beginning and end of the sample run. If a large number of samples are to be run, then analyze a QC check standard at the beginning, end, and every 10th sample of the run.

4.2. For those instruments that are not calibrated with each use, analyze QC check standards at a frequency that provides the necessary degree of reliability and confidence needed. Consideration shall be given to the level of risk the laboratory is willing to assume in regard to evaluation of data that was produced between QC check standards. The longer the frequency between QC check standards, the more data are at risk of being unacceptable, provided a confirmed failure of a QC check standard occurs.

4.3. In any case, sample data produced between acceptable and unacceptable QC check standards is evaluated to determine the effect of the failure on these data.

5.0 QC Check Standard Concentration

5.1. Two methods are shown for how to determine the concentration of the QC check standard. One method for linear calibrations and one for non-linear calibrations are shown. The concentration of the QC check standard must be such that its measurement precision allows for the detection of any significant slope changes.

5.1.1. For linear calibrations, a QC check standard concentration of between 10 x LOD and the mid-point of the calibration is used as a starting point. If 10 x LOD is beyond the mid-point of the calibration and the precision meets the VRs, then use the mid-point. If 10 x LOD is less than the mid-point, and the precision meets the VRs, then use any concentration greater than 10 x LOD but less than the mid-point of the calibration curve.

5.1.2. For non-linear calibrations, QC check standard concentrations should be close to the expected sample concentration. If multiple concentration ranges are expected, multiple QC check standards are analyzed. How close the QC check standard concentration should be to the expected sample concentration depends on how non-linear the calibration curve is. For most non-linear curves, ± 20% is adequate.

6.0 QC Check Standard Response Time

6.1. For those situations when the response time to reach a predetermined QC check standard concentration is part of the VR, the response time required to reach a specified recovery (e.g. 90% of the full value) shall be measured and controlled to within predetermined specifications.

7.0 Periodic Verification of Calibration at Concentrations Near LOD

7.1. Periodically each laboratory verifies that calibrations are not pivoting on their axis by analyzing a low-level standard. This standard shall be prepared at a concentration where dilution error or background contamination can be minimized. Standard concentration for this verification is recommended to be between LOD and LOQ.

7.2. Acceptance criteria for this periodic verification may be as large as ± 60% (95% confidence coefficient) at concentrations near LOD or ± 20% (95% confidence coefficient) at concentrations near LOQ.
Note: This document provides a list of relevant references. The references below are in no particular order of importance. The user of these standards is to be cognizant of regulations and commitments that may lend favor to one reference over another.

REFERENCES

Philosophical References


Calculational References


A5 - Standard for Control Charting

DEVELOPING CONTROL CHARTS

1.0 General

1.1. Control charting is an effective means of preventing or identifying non-conformances in the laboratory’s Quality Management System (QMS). When properly created and implemented, control charts provide ongoing assurance that the selected analytical method is capable of producing data that is in a state of statistical process control and meets the laboratory’s valid requirements. Control chart limits can be imposed either statistically or non-statistically. Statistically imposed limits are preferred except in situations as described in section, “Setting non-statistically based limits”, below.

2.0 Collecting Data

2.1. Statistically imposed limits are based on the statistical analysis of a minimum of 20 QC check standard data. In order to determine these limits, QC check standard data must be collected under appropriate conditions:

2.1.1. The laboratory environment should be typical (e.g., temperature, humidity variations, etc.).
2.1.2. The equipment used must be the equipment that will be used to generate sample data.
2.1.3. The reagents and standards used must be of the same grade that will be used to generate sample data.
2.1.4. The staff used to generate the control chart data, must be similar in ability to the staff that will be used to generate the sample data.
2.1.5. Data collection is at a frequency and concentration that reflects the normal QC check standard data frequency and concentration.
2.1.6. Control chart limits during data collection shall be set not to exceed the valid requirements.

3.0 Evaluating Data Normality

3.1. After data collection, an evaluation is performed to determine if the data collected is normally distributed and can be used to establish statistically based control chart limits. If the data collected is not normally distributed, non-statistical limits may be imposed as described below.

4.0 Evaluating Data Precision

4.1. After data analysis for normality, perform an evaluation to determine if the data collected meet the precision required as stated in the valid requirements. If the data meet the precision requirements specified in the valid requirements, then that instrument may be used for analysis. If the data does not meet the valid requirements, then an evaluation of the valid requirements and instrument capabilities is made. The evaluation determines if the valid requirements should be changed or if the method should be changed.
5.0 Centering Control Chart

5.1. When the data are shown to be normally distributed and meet the precision of the valid requirements then a determination is made as to where to center the control chart:

5.1.1. If the data pass the Student’s “t” test, it is centered at the target (i.e., the theoretical QC check standard analyte concentration).

5.1.2. If the data fail the Student’s “t” test, but do not exhibit a persuasive bias (i.e. the mean is <1 standard deviation (SD) from the target value), the control chart is centered at the mean.

5.1.3. If the data fail the Student’s “t” test and does exhibit a persuasive bias (i.e., the mean is > 1 SD from the target value), the control chart is centered at the target and the cause of the bias is investigated and eliminated where possible. Non-statistical limits are set not to exceed the valid requirements for the analyte in question. Additional QC check standard data are collected and evaluated for the potential future use of statistics.

6.0 Setting Statistically Based Limits

6.1. The limits applied to the control chart are termed ‘Warning Limits’ (set at ±2 SDs) and ‘Control Limits’ (set at ±3 SDs). Approximately 5% of data generated is predicted to exceed the warning limits. Exceeding the control limits is statistically improbable (~3 in a 1000) and is an indication that instrument performance has changed and may no longer be in a state of statistical process control. An investigation of the analytical system is warranted.

7.0 Setting Non-statistically Based Limits

7.1. Non-statistical (or manual) limits may supersede statistical limits for a number of reasons, namely:

7.1.1. Reasonable manual limits (i.e., that at a maximum meet the valid requirements for the test) may be enforced during the period of time when data are being collected for statistical limits.

7.1.2. Manual limits may be imposed that reflect published, vendor or regulatory limits (in this case these are the valid requirements).

7.1.3. In those situations where the data collected are not normally distributed (i.e., fail the normality test) manual limits that at a maximum meet the valid requirements, are used.

8.0 Other Types of Control Charts

8.1. Spike, duplicate, etc., are prepared using the same philosophy, but with chart factors customized to their type.
9.0 On-Going Use of Control Charts

9.1. All QC check standard data shall be recorded on the same quality control chart. For statistically based control charts, data are evaluated for on-going control chart checks and for long-term trends using statistical evaluations. For non-statistically based control charts, data are evaluated not to exceed the valid requirements.\(^{(2,3,4,5)}\)

10.0 On-going Checks\(^{(3,4,5)}\)

10.1. For statistically based control charts, the result of each QC check standard is evaluated to determine if any of the following conditions exist:

10.1.1. If the result of the QC check standard is within three (3) SDs from the centerline of the control chart, then the instrument is in service.

10.1.2. If the result of the QC check standard is beyond three (3) SDs from the centerline of the control chart then a backup QC check standard is immediately analyzed. If the result of the backup QC check standard is within three (3) SDs from the centerline of the control chart, the instrument is kept in service. If the result of the backup QC check standard is beyond three (3) SDs from the centerline of the control chart, the instrument is placed out of service. The instrument may not be placed back in service without an evaluation or corrective actions being taken. To place an instrument back in service one (1) QC check standard must be within three (3) SDs from the centerline of the control chart.

10.1.3. If two (2) consecutive QC check standards are beyond two (2) SDs from the centerline of the control chart, then an evaluation is performed.

10.1.4. If the average of seven (7) consecutive QC check standards is beyond one (1) SD from the centerline of the control chart, then an evaluation is performed.

10.2. For non-statistically based control charts, the result of each QC check standard is evaluated to be within the limits prescribed on the control chart. Instrument availability is determined using a similar procedure as described above for statistically based control charts. Returning an instrument to service should also follow the procedure used for statistically based control charts discussed above.\(^{(1)}\)

11.0 Long-term Trends\(^{(1,8e)}\)

11.1. For statistically based control charts, long-term trends shall be evaluated for shifting precision, bias and normality. The evaluation is performed quarterly using a minimum of 20 QC check standard data. If less than 20 data are available, the evaluation is postponed until enough data are available.

11.2. All three (3) evaluations must pass. If any of the evaluations fail, the analytical process is reviewed and compared to the valid requirements. The aim of the long-term trend evaluation is to identify when a significant trend has occurred that warrants developing a new control chart. New control charts are developed after careful consideration of all factors relevant to determining if a long-term trend has developed. One evaluation failing one time would not necessarily constitute a new control chart being developed. Instrument performance may return to original behavior after an appropriate period. Developing a new control chart and new limits should be rare.

11.3. For non-statistically based control charts, long-term trends shall include an equivalent evaluation as the procedure used for statistically based control charts above.
Note: This document provides a list of relevant references. The references below are in no particular order of importance. The user of these standards is to be cognizant of regulations and commitments that may lend favor to one reference over another.

REFERENCES

Philosophical References


Calculational References

   a. Grubbs, test for outliers
   a. Dixon’s Q, test for outliers
   b. Kolmogorov-Smirnov (K/S), test for normality
   c. RSD, testing for precision
   a. Outlier determination
   b. Meeting precision requirements for Valid Requirements
   c. Centering control charts based on Student’s t: test
   d. Setting non-statistically based control chart limits
   e. Shifting precision (F test), shifting bias (t test), normality (K/S test)
A6 - Standard for Inter- and Intra-laboratory Programs

INTER-LABORATORY AND INTRA-LABORATORY PROGRAMS

1.0 General

1.1. Inter-laboratory and Intra-laboratory are distinct but complementary programs that allow a laboratory to demonstrate analytical capabilities for both the overall laboratory and for each individual analyst.

Note: Current practices do not allow for effective methodologies to be put in place for the use of Inter/Intra-laboratory programs for the assurance of data produced by on-line instrumentation, so on-line instrumentation is excluded from this section.

1.2. The inter-laboratory program is intended to demonstrate a laboratory’s overall capabilities in meeting its valid requirements pertaining to a given analysis. The intra-laboratory program is intended to provide assurance that each individual analyst is capable of meeting the laboratory’s valid requirements for a given analysis.

1.3. The objective of the inter-laboratory program is to check one laboratory’s analytical capability relative to a group of peer laboratories. The focus of the inter-laboratory program is external. It is primarily used to identify laboratory-wide bias. An inter-laboratory program typically involves submitting blind sample laboratory results to a program administrator for evaluation. The program administrator compares laboratory results and typically calculates statistically based agreement criteria. Each laboratory is compared to the grand average of the peer laboratory results. Agreement is indicated if the results are within ± 3 SD. The results of this comparison provide valuable information to the participants. The results show how one laboratory’s analytical capability compares to others. Non-agreement indicates systematic error for the failing laboratory. The value of this program is that one laboratory demonstrates agreement with all their peer laboratories, with their potentially different QMS.

1.4. The objective of the intra-laboratory program is to check individual analyst proficiency. The focus of intra-laboratory program is internal. It can be used to identify individual analyst bias. This program involves each analyst submitting blind sample results to a program administrator for evaluation. The program administrator compares all individual results and typically calculates statistically based agreement criteria. Each analyst is compared to the grand average of the laboratory results. Agreement is indicated if the results are within ± 3 standard deviations (SD), expressed as a percent based on control chart QC check standard limits of the average of the individual blind sample results. The results of this comparison provide valuable information to the participants. They show how one analyst's analytical capability compares to others. Non-agreement indicates systematic error for the failing analyst.

1.5. Agreement criteria other than statistically based criteria may be necessary for small groups of data. Typically minimums of seven (7), preferably 20-30, data are used to calculate statistical limits. If fewer than seven (7) data are to be evaluated, the program administrator shall document the basis for non-statistically based limits.
1.6. Complementary to the inter-laboratory and intra-laboratory is the analysis of a routine QC check standard (addressed separately in the Standard for Control Charting). These three (3) checks provide assurance that the laboratory is producing results that:

- Are similar to its peers (inter-laboratory program)
- Demonstrate individual proficiency (intra-laboratory program)
- Demonstrate the instrument capability at a specific moment in time (routine QC check standards)

2.0 Inter-laboratory Program

2.1. The parameters to be analyzed include those that are deemed important to the continued safe and efficient operation of the plant (e.g., fuel warranty items, tech specs, etc.). Each laboratory shall participate in an inter-laboratory program annually.

2.2. The inter-laboratory program must consider the following:

2.2.1. The material to be analyzed must be stable and match, if necessary, the power plant’s sample matrix.

2.2.2. The concentration is unknown at time of analysis.

2.2.3. The sample is analyzed as if it were a routine lab sample. Only one datum is to be reported. Averaged results are not to be reported.

2.2.4. Acceptance criteria shall be statistically derived (± 3 SD of the inter-laboratory program unless otherwise specified by the inter-laboratory program administrator, or regulatory agency. Statistically derived acceptance criteria include techniques for identifying and rejecting outliers as well as showing the data used are normally distributed.

2.3. Specific results for each laboratory as well as results for all laboratories participating must be made available to all laboratories in the inter-laboratory program. If the laboratory’s result falls outside the acceptance criteria, the laboratory must take appropriate actions. The cause of the discrepancy is to be determined and corrective actions are to be taken and monitored for effectiveness.

2.4. If laboratories perform radioactive analyses, they shall participate in inter-laboratory programs, at least annually, per category of radioanalytical analysis.

3.0 Intra-laboratory Program

3.1. As with the inter-laboratory program, the parameters to be analyzed include those that are deemed important to the continued safe and efficient operation of the plant. The intra-laboratory program requires each analyst to analyze each important analyte annually in order to maintain qualifications.

3.2. The intra-laboratory program must consider the following:

3.2.1. The material to be analyzed must be stable and match, if necessary, the power plant’s sample matrix.

3.2.2. The concentration is unknown at time of analysis.

3.2.3. The sample is analyzed as if it were a routine lab sample. Only one datum is to be reported. Averaged results are not to be reported.
3.2.4. The acceptance criterion is based on control chart, check standard limits (i.e., ± 3 SD) for each parameter analyzed. Control chart limits are used as a percentage to establish the acceptance criteria for the intra-laboratory program.

Example: If the control chart were centered at 1.00 ppm, with a ± 3 SD of ± 0.10 ppm, (i.e., ± 10%), then an intra-laboratory program average of 0.8 ppm would have acceptance criteria of ± 0.08 ppm, (i.e., ± 10%).

3.3. If the individual analyst's result falls outside the acceptance criteria, the analyst must be retested as soon as possible. If the analyst passes the retest, then no further action is required. If the analyst fails the retest, then the analyst is disqualified from further like analysis. The cause of the failure is to be determined and the analyst is to be retrained. To requalify, the analyst must demonstrate competence by satisfactorily completing another intra-laboratory sample.
Note: This document provides a list of relevant references. The references below are in no particular order of importance. The user of these standards is to be cognizant of regulations and commitments that may lend favor to one reference over another.

REFERENCES

Philosophical References

SIGNIFICANT DIGITS

1.0 General

1.1. Data, for both bench and on-line instrumentation shall be reported with an adequate number of significant digits in order for the data to meet its valid requirements. Data that have inadequate significant digits may lead to inappropriate actions being taken when a process being monitored is suspected to be in compliance. Data with more digits than appropriate may convey a false sense of analytical capability.

2.0 Significant Digits Calculations

2.1. Calculational methods for determining the appropriate number of significant digits are specified in the references below.\(^{[1,2]}\)
Note: This document provides a list of relevant references. The references below are in no particular order of importance. The user of these standards is to be cognizant of regulations and commitments that may lend favor to one reference over another.

REFERENCES

Calculational References

1. ASTM E29 - 08 Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
2. Protocol for the sampling and analysis of industrial /municipal wastewater (1999), MISA, Ministry of the Environment (Ontario)
A8 - Standard for Stating Analytical Uncertainty

ANALYTICAL UNCERTAINTY

1.0 General

1.1. Power Plant Laboratories are not required to report uncertainty with all analytical data. This standard provides for both bench and on-line instrumentation, the approach adopted by the Power Plant Chemistry Advisory Group for calculating uncertainty for wet chemistry analysis should an uncertainty statement be needed.

1.2. All analytical data generated have an uncertainty. Uncertainty as defined by the Power Plant Chemistry Advisory Group is the repeatability/reproducibility of a QC check standard data set at a specified confidence coefficient. In other words, if the data were generated at any moment in time what would the probable range of outcomes be at a specified confidence coefficient (e.g., 99% or 95%). Analytical data can be expressed with a ± quantity at the specified confidence coefficient with this understanding.

2.0 Uncertainty Calculations

2.1. The range of probable outcomes is determined by applying a factor times the SD of a normal distribution of data. Approximately 95% of the data fall within ± 2 SD and 99.7% of the data fall within ± 3 SD from the average of the data. More precisely the factor is the Student's “t” value for 95% or 99% confidence coefficient, for n-1 degrees of freedom, using a two tailed factor.
Note: This document provides a list of relevant references. The references below are in no particular order of importance. The user of these standards is to be cognizant of regulations and commitments that may lend favor to one reference over another.

REFERENCES

Philosophical References


Calculation References

ON-LINE INSTRUMENTATION

1.0 General

1.1. As on-line instrumentation in the power plant setting has evolved, it has moved from often being perceived as a trending methodology to one that is used for critical decision-making. Unit operation, as well as environmental and safety decisions are now routinely based on on-line instrumentation outputs. This transformation has necessitated QA/QC protocols be developed in order to ensure the accuracy and reliability of the data being produced. Appropriate methods of demonstrating the ability of on-line instrumentation to meet valid requirements are:

- The line method
- The reference method
- The standard injection method

1.2. The line method of verification of on-line analyzers, as discussed in ASTM D 3864, involves the use of a ‘referee’ instrument. The referee instrument, which could be a second on-line analyzer or a laboratory analyzer is calibrated (whenever possible these calibrations must be traceable back to a national or international standard) and QC checked. The on-line instrument result and the referee instrument result are then compared (preferably statistically) and a decision is then made as to the quality of the on-line reading.

1.3. The reference method of verification of on-line analyzers, as discussed in ASTM 3864, also involves a ‘referee’ instrument, however rather than the process fluid being passed through both instruments, a reference standard is used. The on-line result and the referee instrument result are then compared (preferably statistically) and a decision is then made as to the quality of the on-line reading.

1.4. The preferred method of on-line instrument verification is standard injection. The standard injection method involves the injection of a known concentration of the analyte into the analyzer (whenever possible the analyte concentration must be traceable back to a national or international standard). The result generated is then compared (preferably statistically) to the true concentration and a decision is then made as to the quality of the on-line reading. If a nationally or internationally traceable standard is used, further verification via comparison with laboratory instrumentation is not required.

1.5. No matter what method of verification is utilized, proper sample conditioning must be in place (sample temperature, flow, pressure, etc.).

1.6. It is understood that many facilities have on-line instrumentation in place that may not have been chosen with valid requirements in mind, however the process of demonstrating the on-line instrumentation’s ability of meeting suitable valid requirements must be confirmed.

2.0 On-line Instrumentation QA/QC Requirements

2.1. Table 1 below indicates by instrument type the calibration techniques, frequency, check technique, and check frequency required to ensure the validity of the instrument output.
### Table 1 - On-line Instrumentation QA/QC Requirements

<table>
<thead>
<tr>
<th>Instrument Type</th>
<th>Calibration Technique</th>
<th>Calibration Frequency</th>
<th>Calibration Check Technique</th>
<th>Calibration Check Frequency</th>
</tr>
</thead>
</table>
| Dissolved Oxygen                       | • Water saturated air calibration AND  
• Zero check using sodium sulphite or nitrogen                                            | • When placed in service OR  
• When Calibration Check fails                                                            | • Line sample method (use statistics and control chart differences) OR  
• Known addition (i.e. Faraday cell) OR  
• Calibration gas (use statistics and control chart values)                              | • Immediately after calibration AND  
• weekly                                                                                   |
| Colorimetric (Silica, Phosphate, Chlorine, Ammonia, Hardness, Hydrazine)                  | • Injection of a known standard AND  
• Injection of a blank                                                                       | • When placed in service OR  
• When Calibration Check fails OR  
• When standards or reagents change OR  
• After an instrument outage, using at least one standard                                   | • Standard injection with a concentration >10 x LOD and <midpoint OR  
• Line sample method (use statistics and control chart values)                             | • Immediately after calibration AND  
• Before changing reagents AND  
• weekly                                                                                   |
| Sodium (Ion Selective Electrode)        | • Injection of a known standard AND  
• Double Known Addition                                                                      | • When placed in service OR  
• After a probe change OR  
• When Calibration Check fails                                                             | • Line sample method (use statistics and control chart differences) OR  
• Known addition (use statistics and control chart value) OR  
• Standard Injection (Span Check)                                                            | • Immediately after calibration AND  
• Before and after changing reagents AND  
• Weekly                                                                                   |
| Ion Chromatography                      | • Injection of a known series of standards AND  
• Injection of a blank                                                                       | • When placed in service OR  
• When Calibration Check fails                                                             | • Line sample method (use statistics and control chart differences) OR  
• Standard Injection (use statistics and control chart values)                             | • Immediately after calibration AND  
• After maintenance AND  
• daily                                                                                     |
| Conductivity                           | • Factory calibrated                                                                      | • Factory calibrated                                                                    | • Line sample method (use statistics and control chart differences) OR  
• Standard Injection (e.g. KCl) (use statistics and control chart values)                  | • Monthly (meter used for line comparison to be validated annually)                        |
| pH                                     | • Injection of two standards at least two pH units apart                                   | • When placed in service OR  
• When Calibration Check fails                                                             | • Line sample method (use statistics and control chart differences) OR  
• Grab comparison OR  
• Calculated pH vs. Observed pH (provided the concentrations of the species contributing to the pH are known) | • Weekly                                                                                   |
<table>
<thead>
<tr>
<th>Instrument Type</th>
<th>Calibration Technique</th>
<th>Calibration Frequency</th>
<th>Calibration Check Technique</th>
<th>Calibration Check Frequency</th>
</tr>
</thead>
</table>
| Turbidity               | • Injection of a known standard  
                      • Vendor provided standard                                                      | • When placed in service OR  
                      • After maintenance (e.g. lamp replacement) OR  
                      • When Calibration Check fails                                                 | • Line sample method (use statistics and control chart differences) OR  
                      • Optical comparison using a known standard (other than the standard used for calibration) | • Weekly                      |
| Hydrazine (amperometric)| • Potentiometric – zero and one point trim calibration (either using standard or comparing to a bench analyzer)  
                      • Ion selective electrode (ISE) – 2 standards a decade apart in concentration | • When placed in service OR  
                      • After maintenance (e.g. filter or reagent change) OR  
                      • When Calibration Check fails                                                  | • Line sample method (use statistics and control chart differences) OR  
                      • Standard Injection (use statistics and control chart values) OR  
                      • Known addition                                                                  | • Weekly                      |
| Total Organic Carbon (TOC)| • Factory calibrated OR  
                      • In situ one point calibration                                                  | • When placed in service AND annually (either return to manufacturer or in situ) OR  
                      • After maintenance (e.g. lamp change) OR  
                      • When Calibration Check fails                                                   | • Line sample method (use statistics and control chart values)                      | • Monthly                    |
| Dissolved Hydrogen      | • One point calibration with a known standard (e.g. a certified gas or VCT bomb)  
                      • Grab sample comparison                                                        | • When placed in service OR  
                      • After maintenance (e.g. membrane replacement) OR  
                      • When Calibration Check fails                                                   | • Line sample method (use statistics and control chart differences) OR  
                      • Standard Injection (use statistics and control chart values) OR  
                      • Calculated hydrogen vs. Observed hydrogen (provided the concentration of hydrogen is known) | • Monthly                    |
| Chlorine (ion selective electrode or amperometric)| • One point calibration with a known standard AND  
                      • Grab sample comparison                                                        | • When placed in service OR  
                      • After maintenance (e.g. probe replacement) OR  
                      • When Calibration Check fails                                                   | • Line sample method (use statistics and control chart differences) OR  
                      • Standard Injection (use statistics and control chart values) OR  
                      • Grab comparison                                                                  | • Weekly                      |
| Oxidation Reduction Potential (ORP)| • One point calibration with a known standard                                      | • When placed in service OR  
                      • When Calibration Check fails                                                   | • Standard Injection (use statistics and control chart values)                      | • Monthly                    |
REFERENCES

Philosophical References

A10 – Standard for Analytical Radiation Detection Instrumentation

RADIOCHEMISTRY

1.0 General

1.1. In order to minimize the effect of detector/cave contamination on reported sample activities, background determinations over the channels of interest shall be trended on a statistically based control chart, with increases above an established limit (i.e. +3 standard deviations (SD), and not to exceed valid requirements) investigated for its impact on detection limits. If a peak search of a background count identifies the presence of by-product material, action is required (such as decontamination of the detector/cave). When the presence of by-product material cannot be eliminated, the impact of the by-product material shall be taken into account for sample results generated.

1.2. Appropriate corrections for radioactive decay shall be performed. Procedures shall be implemented to minimize the potential for cross contamination.

1.3. The ongoing verification of analytical performance shall be demonstrated by the use of an instrument response check source(s). If an instrument response check source becomes damaged, a new instrument control chart can only be established through the use of a new source, after verification that the instrument calibration has not changed. Otherwise, the instrument shall be recalibrated.

1.4. The laboratory shall have in place an appropriate method for determining and reporting low-level activities. A routine program for monitoring of background is particularly important. Minimum Detectable Concentration (MDC or however named) or "less than MDC" should never be recorded in databases in lieu of a concentration "less than MDC" since this practice creates an uncorrectable bias.

1.5. An on-site process to determine total uncertainty for each measurement process should be developed.\(^{(4)}\)

2.0 Gamma-ray Spectroscopy Calibration Technique

2.1. The system shall be calibrated with traceable sources for each required source-to-distance configuration (also referred to as detector geometry).

2.2. The source used to calibrate the detection system shall contain gamma-rays that cover the energy range of interest and be counted for a sufficient duration in order to obtain a resolution precision of less than 0.2 keV.\(^{(1)}\)

2.3. Pulse pileup and coincidence summing of gamma rays shall be accounted for by the calibration method or analytical procedures.

2.4. The calibration spectrum shall acquire a minimum of 10,000 counts in each gamma-ray peak of interest.\(^{(5)}\)
3.0 Gamma-ray Spectroscopy Calibration Frequency

3.1. The system shall be calibrated (efficiency, energy and peak resolution) when placed in service and as necessary (following unresolved calibration check failures, critical component change-outs, etc.)\(^{(2,3)}\).

4.0 Gamma-ray Spectroscopy Calibration Check Technique

4.1. Ongoing analytical system performance shall be checked by use of a sealed source containing gamma-ray energies that cover the range of interest. Statistics shall be used where appropriate (e.g. recording source activity or background checks) and professional judgment shall be used to impose appropriate tolerance limits when statistically derived limits are not deemed appropriate (e.g. recording peak channel numbers).

4.2. Energy, efficiency and resolution calibrations shall be checked periodically by counting source emitting gamma rays whose energies are precisely known and cover the region of interest (typically about 100 keV and greater than 1300 keV). One counting geometry shall be used each time for these checks. Deviations at both high and low energies shall be measured and recorded periodically and any unexpected changes investigated.\(^{(1)}\)

4.3. The source used does not necessarily need to be a primary-grade calibration source, but a sealed source that is well characterized and stable. The purpose of this QC source is to validate that the detector performance is reproducible.\(^{(4)}\)

4.4. The count time used shall obtain a relative count uncertainty of at least <1% (10,000 net counts minimum).\(^{(6)}\)

5.0 Gamma-ray Spectroscopy Calibration Check Frequency

5.1. The system’s performance shall be checked immediately after calibration and:

- Daily when in use
- After calibration or maintenance that could affect instrument performance
- After system upsets, (e.g. loss of power or temperatures greater than or equal to 80°F\(^{(1)}\) (27°C))

5.2. Quality control checks for energy calibration, efficiency calibration and resolution, may be combined.\(^{(4)}\)

6.0 Liquid Scintillator

6.1. The laboratory shall ensure that procedures are in place to minimize the sample counting effects caused by such interferences as quenching, sample color and turbidity, chemiluminescence and static electricity. Before counting it is advisable to equilibrate the counting vials in the liquid scintillation counter for light and temperature adaptation.\(^{(7)}\)

6.2. The laboratory shall ensure the matrix of the calibration test-source is appropriate for the sample types to be analyzed. Sample preparation may be required prior to analysis to ensure sample matrix interferences are minimized. The ratio of sample volume to scintillation-cocktail volume must match the calibration test source.\(^{(4)}\)
6.3. The laboratory shall ensure the method background is properly characterized. In order to minimize the effect of contamination on reported sample activities, background determinations over the channels of interest shall be trended on a statistically based control chart, with increases above an established limit (i.e. +3 SD, and not to exceed valid requirements) investigated for its impact on detection limits.

7.0 Liquid Scintillator Calibration Technique

7.1. Calibration shall be performed with appropriately quenched cocktails (at least five points) incorporating suitable radionuclides in order to ensure the required sample energy regions of interest are established. (4)

7.2. Suitable traceable sources not exceeding 5,000 counts per second (6) are used to ensure the decay products of interest achieve a minimum of 10,000 counts (relative counting error of 1%).

8.0 Liquid Scintillator Calibration Frequency

8.1. The system shall be calibrated when placed in service and as necessary (following unresolved calibration check failures, critical component change-outs, etc.). (2, 3)

9.0 Liquid Scintillator Calibration Check Technique

9.1. Ongoing system performance shall be verified by use of a sealed source containing unquenched reference standards that cover the energy range of interest. Statistics shall be used where appropriate to ensure ongoing system performance (e.g. recording source activity or background checks). (4)

10.0 Liquid Scintillator Calibration Check Frequency

10.1. The system’s performance, including a background determination, shall be checked immediately after calibration and:

- Daily when in use
- After calibration or maintenance that could affect instrument performance
- After system upsets (e.g. loss of power or temperatures greater than or equal to 80°F (27 °C))

10.2. Calibrations shall be checked by counting a source with precisely known emitted energies.

11.0 Proportional Counters

11.1. Instrument parameters such as operating voltage, detector counting efficiencies and crosstalk factors shall be verified prior to initial use to ensure manufacturer’s recommended settings are valid. (4)

11.2. The laboratory shall ensure that factors such as sample geometry, back-scatter and self-absorption are incorporated in the determination of sample activity. (6)

11.3. Sources used for calibration shall be replaced at a frequency that ensures their continued effectiveness, taking into account source half-lives and leakage. (2) When gross sample activities are reported, the calibration nuclide shall be noted. (6) Sources used for calibration shall be similar in energy and type of radiation to the nuclide(s) of interest being measured.
11.4. The laboratory shall ensure the method background is properly characterized. In order to minimize the effect of contamination on reported sample activities, background determinations shall be trended on a statistically based control chart, with increases above an established limit (i.e. +3 SD, and not to exceed valid requirements) investigated for its impact on detection limits.

12.0 Proportional Counters Calibration Technique

12.1. Calibration shall be performed with suitable radionuclides in order to ensure the required sample energy regions of interest are established.

12.2. Suitable traceable sources not exceeding 5,000 counts per second\(^{6}\) are used to ensure the decay products of interest achieve a minimum of 10,000 counts (relative counting error of 1%).

13.0 Proportional Counters Calibration Frequency

13.1. The system shall be calibrated when placed in service and as necessary (following unresolved calibration check failures, critical component change-outs, etc.).\(^{2,3}\)

14.0 Proportional counters calibration check technique.

14.1. Ongoing system performance shall be verified by counting a sealed source of sufficient activity (not exceeding 5,000 counts per second\(^{6}\)) and a system background. Decay products of interest shall achieve a minimum of 10,000 counts (relative counting error of 1%). Statistics shall be used to where appropriate to ensure ongoing system performance (e.g. recording source activity, etc.).

15.0 Proportional Counters Calibration Check Frequency

15.1. The system’s performance, including a background determination, shall be checked immediately after calibration and:

- Daily when in use
- After calibration or maintenance that could affect instrument performance
- After system upsets, e.g. loss of power or temperatures greater than or equal to 80ºF (27ºC)

15.2. Calibrations shall be checked by counting a source with precisely known emitted energies.
REFERENCES

Philosophical References

3. Clevesori, Lenore & Greenberg, Arnold & Eaton, Andrew (Eds.)1999, Standard Methods for the Examination of Water and Wastewater 20th addition, American Public Health Association
4. Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP) (July 2004), EPA 402-B-04-001C, Sections 18.3.4 and 19.0
5. ASTM E 181-98 (Reapproved 2003), Standard Test Methods for Detector Calibration and Analysis of Radionuclides.
6. ASTM D 7282-06, Standard Practice for Set-up, Calibration and Quality Control of Instruments Used for Radioactivity Measurements.
A11 – Standard for Bench Top Instrumentation

BENCH TOP INSTRUMENTATION

1.0 General

1.1. The Power Plant Laboratory's analytical instrumentation is required for a variety of uses, such as:

- Ensuring safety
- Ensuring Regulatory requirements are met
- Ensuring operational limits are not exceeded
- Verification of On-line Instrumentation operation (see Appendix 9, Reference Method)

1.2. In order to ensure that Valid Requirements are being met, an effective Quality Control program must be in place. An adequately designed QC program ensures a cost effective level of effort is in place to demonstrate this.

2.0 Bench Top Instrumentation QA/QC Requirements

2.1. Table 1 below indicates by instrument type the calibration techniques, frequency, check technique, and check frequency required to ensure the validity of the instrument output.

<table>
<thead>
<tr>
<th>Instrument Type</th>
<th>Calibration Technique</th>
<th>Calibration Frequency</th>
<th>Calibration Check Technique</th>
<th>Calibration Check Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inductively coupled plasma spectrophotometer</td>
<td>Injection of a known series of standards AND Injection of a blank</td>
<td>When placed in service OR When Calibration Check fails OR After instrument maintenance that affects instrument performance</td>
<td>In accordance with Appendix 4 above OR After instrument maintenance that may impact instrument performance</td>
<td></td>
</tr>
<tr>
<td>Balance</td>
<td>Certified weights</td>
<td>Annual OR When Calibration Check fails</td>
<td>Weights measured in region of interest Tolerances based limits are in accordance with certification or other uncertainty statements</td>
<td>Daily when in use</td>
</tr>
<tr>
<td>Gas Chromatograph</td>
<td>Injection of a known series of standards</td>
<td>When placed in service OR When calibration Check fails</td>
<td>Standard injection in the region of interest utilizing statistics for acceptance criteria</td>
<td>In accordance with Appendix 4 above OR After instrument maintenance that may impact instrument performance</td>
</tr>
<tr>
<td>Ion Chromatograph</td>
<td>Injection of a known series of standards AND Injection of a blank</td>
<td>When placed in service OR When calibration Check fails</td>
<td>In accordance with Appendix 4 above OR After instrument maintenance that may impact instrument performance</td>
<td>In accordance with Appendix 4 above OR After instrument maintenance that may impact instrument performance</td>
</tr>
<tr>
<td>Instrument Type</td>
<td>Calibration Technique</td>
<td>Calibration Frequency</td>
<td>Calibration Check Technique</td>
<td>Calibration Check Frequency</td>
</tr>
<tr>
<td>-----------------------</td>
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<td>-----------------------------</td>
<td>----------------------------------------------------------</td>
</tr>
<tr>
<td>Conductivity Cell</td>
<td>• Factory calibrated OR • With a known standard OR • With a calibrated 'referee' instrument</td>
<td>• When placed in service OR • When calibration Check fails</td>
<td>• Statistical analysis of measurements made of known potassium chloride solutions</td>
<td>• Monthly OR • After instrument maintenance that may impact instrument performance</td>
</tr>
<tr>
<td>pH</td>
<td>• Use of 2 standards at least 2 pH units apart</td>
<td>• When placed in service OR • When calibration Check fails OR • Daily when in use</td>
<td>• Check with a third independent standard</td>
<td>• After calibration</td>
</tr>
<tr>
<td>UV/Vis Spectrophotometer</td>
<td>• Injection of a known series of standards AND • Analysis of a blank</td>
<td>• When placed in service OR • When calibration Check fails</td>
<td>• In accordance with Appendix 4 above</td>
<td>• In accordance with Appendix 4 above OR • After instrument maintenance that may impact instrument performance</td>
</tr>
<tr>
<td>AA</td>
<td>• Injection of a known series of standards AND • Injection of a blank</td>
<td>• When placed in service OR • When Calibration Check fails OR • After instrument maintenance that affects instrument performance</td>
<td>• In accordance with Appendix 4</td>
<td>• In accordance with Appendix 4 above OR • After Calibration</td>
</tr>
<tr>
<td>XRF</td>
<td>• Analysis of a known series of standards</td>
<td>• When placed in service OR • When Calibration Check fails OR • After instrument maintenance that affects instrument performance</td>
<td>• In accordance with Appendix 4</td>
<td>• In accordance with Appendix 4 above OR • After Calibration</td>
</tr>
<tr>
<td>TOC</td>
<td>• Injection of a known series of standards AND • Injection of a blank</td>
<td>• When placed in service OR • When Calibration Check fails OR • After instrument maintenance that affects instrument performance</td>
<td>• In accordance with Appendix 4</td>
<td>• In accordance with Appendix 4 above OR • After Calibration</td>
</tr>
<tr>
<td>Potentiometric Titration</td>
<td>• normalize titrant AND • analysis of a blank</td>
<td>• When placed in service OR • When Calibration Check fails OR • After instrument maintenance that affects instrument performance OR • new titrant</td>
<td>• In accordance with Appendix 4</td>
<td>• In accordance with Appendix 4 above OR • After Calibration</td>
</tr>
<tr>
<td>Instrument Type</td>
<td>Calibration Technique</td>
<td>Calibration Frequency</td>
<td>Calibration Check Technique</td>
<td>Calibration Check Frequency</td>
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<tr>
<td>-------------------------------------</td>
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<td>---------------------------------------------------------------------</td>
<td>--------------------------------------------------------</td>
</tr>
<tr>
<td>FTIR</td>
<td>• analysis of a known series of standards within appropriate ranges • appropriate blank for range</td>
<td>• When placed in service OR • When calibration Check fails • After instrument maintenance that may impact instrument performance</td>
<td>• In accordance with Appendix 4</td>
<td>• In accordance with Appendix 4 • After calibration</td>
</tr>
<tr>
<td>Selective Ion Electrode</td>
<td>• Use of minimum of 3 standards covering a range of at least 2 decades</td>
<td>• When placed in service OR • When calibration Check fails • After instrument maintenance that may impact instrument performance</td>
<td>• In accordance with Appendix 4</td>
<td>• In accordance with Appendix 4 • After calibration</td>
</tr>
<tr>
<td>Turbidity</td>
<td>• Use of minimum of 3 standards over region of interest</td>
<td>• When placed in service OR • When calibration Check fails • After instrument maintenance that may impact instrument performance</td>
<td>• In accordance with Appendix 4</td>
<td>• In accordance with Appendix 4 • After calibration</td>
</tr>
<tr>
<td>Fixed Pipettes (for volumetric use only)</td>
<td>• NA</td>
<td>• NA</td>
<td>• Gravimetric check corrected for temperature and pressure • ASTM E 542 • Or manufacturer recommendation</td>
<td>• When placed in service • 1/qtr</td>
</tr>
<tr>
<td>Variable pipettes (for volumetric use only)</td>
<td>• NA</td>
<td>• NA</td>
<td>• Gravimetric check corrected for temperature and pressure over the range of interest • ASTM E 1154 • Or manufacturer recommendation</td>
<td>• When placed in service • 1/qtr</td>
</tr>
</tbody>
</table>
REFERENCES

Philosophical References

A12 – Standard for a Quality Management System Assessment

QUALITY MANAGEMENT SYSTEM (QMS) ANALYSIS

1.0  General

1.1. A QMS Assessment provides an assessment of a laboratory’s ability to meet the Power Plant Chemistry Advisory Group’s (PPCAG) Standard requirements.

1.2. A request for a QMS Assessment is submitted to the PPCAG’s Executive Committee for consideration at least three months prior to the preferred QMS Assessment date. This allows for the selection of prospective QMS Assessment Team members and for the members to obtain travel authorizations. At a minimum, the QMS Assessment Team should consist of:
   - One (1) qualified QMS Assessment Team Director
   - Two (2) members

1.3. Formalization of the QMS Assessment Team’s membership shall be complete two months prior to the proposed QMS Assessment completion date.

1.4. The laboratory shall supply to the QMS Assessment Team specific target areas for the QMS Assessment (e.g., the QMS, procedures, instrumentation method development, training records, etc.) for the targeted analytical protocols.

1.5. The QMS Assessment Team shall comply with the following:
   - Development of a site specific agenda in accordance with specific targets requested by the laboratory
   - Meet prior to start of QMS Assessment
   - Conduct the entrance meeting on the first day of the QMS Assessment
   - Tour the facilities
   - Conduct the QMS Assessment
   - Conduct an exit meeting with laboratory management to present the Draft QMS Assessment Report
   - Release the Final QMS Assessment Report to the PPCAG and laboratory.

1.6. Upon completion of the QMS Assessment Report, the laboratory shall document corrective actions taken to address any findings and document situations deemed unnecessary to correct due to the business needs of the organization.

1.7. Request by the laboratory for a QMS Assessment

1.7.1. The QMS Assessment is voluntary; therefore, the requesting laboratory is responsible for contacting the PPCAG Executive Committee to request a QMS Assessment.
2.0 Preliminary Activities

2.1. The Laboratory is responsible for providing necessary on-site support as follows:

- Security access, badging, and/or escorts as necessary
- A secure method of transmittal of personal information, directly to the security authority, if required prior to arrival onsite
- Meeting room for entrance and exit meetings
- Meeting room for QMS Assessment Team members
- Tour of the facilities (laboratories, on-line instrumentation, chemical storage, etc.)
- Access to laboratory personnel for interviews, at approved times in accordance with agenda
- Access to on-line and bench-top instrumentation
- A primary and alternate laboratory contact to ensure effective communication between the QMS Assessment Team and the laboratory (additional data, coordination of observations and interviews, etc.)
- Access to quality control documentation, i.e., procedures, training records, instrumentation method development and quality control records
- Appropriate personal protective equipment (PPE) for QMS Assessment Team personnel

2.2. QMS Assessment Team composition

2.2.1. The QMS Assessment Team consists of at least two individuals trained in the disciplines to be reviewed. The PPCAG Executive Committee selects the QMS Assessment Team off-site members from a pool of volunteers. These team on-site members should have as a minimum:

- An in-depth knowledge of the PPCAG Standard
- Orientation by the Team Director
- Knowledge of the areas to be assessed and experience in laboratory quality management practices
- Good interpersonal and communication skills
- Approval from their company to participate in the QMS Assessment
- Approval from the PPCAG Executive Committee

2.2.2. The QMS Assessment Team Director shall have as a minimum:

- Resume' indicating relevant laboratory and previous assessment experience
- Completed formal training approved by the Executive Committee
- Completed a formal QMS Assessment Team Director training program approved by the PPCAG Executive Committee
- Good organizational and scheduling skills
- Participated in at least one QMS Assessment acting as a Team Director under the tutelage of a qualified Team Director or served as a QMS Assessment Team Director on a formal industry audit to ISO/IEC 17025, or equivalent, standards
- A recommendation from another team director
- Approval of the PPCAG Executive Committee
- Completed a formal QMS Assessment Team Director under the tutelage of a qualified Team Director training program
2.3. Target areas for analysis

2.3.1. The QMS Assessment Team, as a minimum, reviews the laboratory’s QMS and specific analyses, such as pH, cation conductivity, ICP-MS, AA, LSC, gamma spectrometry, online sodium, etc. as requested by the laboratory.

2.3.2. Applicable analytical procedures shall be provided to the QMS Assessment Team Director by the laboratory one month prior to the scheduled QMS Assessment.

2.4. Agenda

2.4.1. The QMS Assessment Team Director shall develop an agenda in consultation with the laboratory contact and QMS Assessment Team members. The agenda specifies times for the following:

- Entrance meeting
- Facilities tour
- Interview schedules
- Observation opportunities
- End of day briefings
- Exit meeting

2.4.2. The laboratory contact ensures that the times noted on the agenda are reasonably attainable, e.g. the required time for the QMS Assessment Team members to acquire the necessary site security access, length of analytical reviews and availability of analysts, etc.

3.0 Site visit activities

3.1. QMS Assessment Team meeting prior to commencement of QMS Assessment

3.1.1. The QMS Assessment Team consists of members with diverse backgrounds; therefore, the members are to meet prior to going on site to discuss the purpose of the visit, the Standard, the checklists, and the final report.

3.2. Entrance meeting

3.2.1. A meeting with the QMS Assessment Team and laboratory contacts shall be scheduled prior to conducting interviews and observations. The purpose of the meeting is to confirm the agenda (minor changes made as necessary) and to discuss the terminal objective of the QMS Assessment (i.e. for the laboratory to demonstrate conformance to this Standard).
3.3. Tour of facilities

3.3.1. The purpose of the tour is to orient the QMS Assessment Team in regards to sampling, analysis, and records locations. The tour should include, as required, the following areas:

- On-line instrumentation
- All laboratories
- Management office locations
- Training facilities
- Sample panels
- Quality control records

3.4. Completion of the QMS Assessment

3.4.1. QMS Assessment Team members perform the QMS Assessment using the QMS Assessment forms (Appendix 11 attachments 11.1, 11.2, and 11.3). The information gathered in this manner is the basis of the draft QMS Assessment presented at the conclusion of the QMS Assessment. The QMS Assessment Team conducts daily briefs with the laboratory to allow for any misunderstandings, etc. to be resolved.

3.5. Exit meeting

3.5.1. The purpose of the exit meeting is for the QMS Assessment Team to present the draft QMS Assessment report documenting any observed findings between the requirements of this Standard and the conduct of the laboratory.

3.6. Release of final QMS Assessment report

3.6.1. The QMS Assessment Team Director shall complete the final QMS Assessment report and submit the report within one month to the PPCAG Executive Committee and to the requesting laboratory.

4.0 Alternatives to a formal QMS Assessment

4.1. Other methods of conducting a QMS Assessment include, but are not limited to, self-assessments, peer assessments, and internal audits in accordance with site-specific or company audit requirements.
REFERENCES

1. Power Plant Chemistry Advisory Group Standard, Rev 4
2. Canadian Association for Laboratory Accreditation, PO2-Program Description, Section 2 The Accreditation Program for Testing Laboratories, 2009
4. EPA QA/G-8 (2002) Guidance on Environmental data Verification and Data validation,
5. INPO 06-007, (2006) Guidelines for Conduct of Chemistry at Nuclear Plants,

FORMS

12.1 QMS Analysis team Director Preparation Checklist
12.2 QMS Analysis QMS Review
12.3 QMS Analysis Method Review
POWER PLANT CHEMISTRY ADVISORY GROUP

QMS Assessment  Team Director Preparation Checklist

Attachment 12.1

LABORATORY NAME

______________________________

Date of QMS Assessment

QMS Assessment scheduled by Executive Committee and requesting Laboratory  Date Complete _____________

<table>
<thead>
<tr>
<th>Primary Contact</th>
<th>E-mail</th>
<th>Phone</th>
<th>Position</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Secondary Contact</td>
<td>E-mail</td>
<td>Phone</td>
<td>Position</td>
<td>Comments</td>
</tr>
</tbody>
</table>

Team members identified  Date Complete _____________

<table>
<thead>
<tr>
<th>Name</th>
<th>Position</th>
<th>Company</th>
<th>Contact information e-mail</th>
<th>Contact information phone</th>
<th>Qualified in accordance with standard</th>
</tr>
</thead>
<tbody>
<tr>
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Areas of focus identified and team members assigned

<table>
<thead>
<tr>
<th>Focus Area</th>
<th>Team Member(s)</th>
</tr>
</thead>
<tbody>
<tr>
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</tbody>
</table>

Date Complete _____________

Agenda Compiled and submitted to team members and laboratory for concurrence

Date Complete _____________

Procedures and data requested from Laboratory

Date Complete _____________

Procedures and data received from Laboratory

Date Complete _____________

Verified team members have made travel arrangements

Date Complete _____________

Verify with Laboratory that the following facilities/equipment available:

- Team meeting room
- Communications
- Office equipment to include, printers, projector, paper, pens, sticky notes, etc.
- Personal Protective Equipment
- Escorts
QMS Assessment

LABORATORY NAME

___________________________________

LABORATORY REPRESENTATIVE(s)

___________________________________

___________________________________

___________________________________

Team Member(s)

___________________________________

___________________________________

DATE

___________________________________
<table>
<thead>
<tr>
<th>Item</th>
<th>Section</th>
<th>Standard’s Requirement</th>
<th>Documentation Review</th>
<th>Implementation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Q01</td>
<td>3.0</td>
<td>Verify that the laboratory is committed to meeting the requirements of:</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Regulatory authorities</td>
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<tr>
<td></td>
<td></td>
<td>- This Standard</td>
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<tr>
<td></td>
<td></td>
<td>- Public safety</td>
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<td></td>
<td></td>
<td>- Plant safety</td>
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<td></td>
<td></td>
<td>- Plant requirements</td>
<td></td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>3.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>02</td>
<td>3.0</td>
<td>Verify that the necessary authority and resources are granted to:</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Implement the QMS</td>
<td></td>
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</tr>
<tr>
<td></td>
<td></td>
<td>- Maintain the QMS</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Ensure continual improvement</td>
<td></td>
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<td></td>
<td></td>
<td>- Initiate actions to minimize/prevent departures from the QMS</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1 – Complies with standard; 2 – Does not comply with standard; 3 – not applicable

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<table>
<thead>
<tr>
<th>Item</th>
<th>Section</th>
<th>Standard’s Requirement</th>
<th>Documentation Review</th>
<th>Implementation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>03</td>
<td>2.4</td>
<td>Verify that the requirements of this Standard are applicable to all work carried out (i.e. central and satellite facilities, as well as on-line instrumentation).</td>
<td>□</td>
<td>□</td>
</tr>
<tr>
<td>04</td>
<td>3.0</td>
<td>Verify that the laboratory’s internal structure as well as its place in the parent organization is clearly defined (e.g. through organization charts, etc.).</td>
<td>□</td>
<td>□</td>
</tr>
<tr>
<td>05</td>
<td>3.0</td>
<td>Verify that the responsibilities and authorities of personnel performing, reporting or verifying work affecting quality are clearly defined (e.g. during back shifts).</td>
<td>□</td>
<td>□</td>
</tr>
<tr>
<td>06</td>
<td>3.0</td>
<td>Verify that the laboratory has in place a technical management, with clearly defined roles and authorities, to ensure adequate resources are available to meet the predetermined Valid Requirements.</td>
<td>□</td>
<td>□</td>
</tr>
</tbody>
</table>

1 – Complies with standard; 2 – Does not comply with standard; 3 – not applicable
<table>
<thead>
<tr>
<th>Item</th>
<th>Section</th>
<th>Standard’s Requirement</th>
<th>Documentation Review</th>
<th>Implementation</th>
</tr>
</thead>
<tbody>
<tr>
<td>07</td>
<td>3.0</td>
<td>Verify that the laboratory has in place a quality management, with clearly defined roles and authorities, to ensure the QMS is implemented and followed and has access to that level of management at which critical decisions are made.</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>08</td>
<td>3.0</td>
<td>Verify that the laboratory has in place a clearly defined substitution process for key staff.</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>09</td>
<td>3.0</td>
<td>Verify that the laboratory has in place a process to ensure the authority to suspend activities, deemed to be outside the QMS specifications, is clearly defined and understood by the staff.</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>10</td>
<td>3.0</td>
<td>Verify for those situations where the requirements of this Standard are not supported by the business needs of the organization, and so not implemented, that details are documented.</td>
<td>1</td>
<td>2</td>
</tr>
</tbody>
</table>

1 – Complies with standard; 2 – Does not comply with standard; 3 – not applicable
<table>
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</thead>
<tbody>
<tr>
<td>Q02</td>
<td>01 4.0</td>
<td>Verify that the QMS requirements are documented and that the laboratory has in place a process to ensure staff are aware of, understand and implement the program and are aware of the importance of their activities in attaining its overall objectives.</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td>02 4.0</td>
<td>Verify that a Quality Manual (however named) is in place that includes or references:</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Top management’s commitment to developing/implementing a QMS</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Top management’s commitment to meeting regulatory requirements</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Top management’s authorization of a quality policy and objectives</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• The structure of documentation used in the QMS</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
</tbody>
</table>

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<tbody>
<tr>
<td>03</td>
<td>4.0</td>
<td>Verify that a quality policy statement includes:</td>
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<tr>
<td></td>
<td></td>
<td>· Management’s commitment to professional practices</td>
<td></td>
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</tr>
<tr>
<td></td>
<td></td>
<td>· A statement of the laboratory’s standard of service</td>
<td></td>
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<td></td>
<td></td>
<td>· A requirement that staff implement the quality documentation</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>· Management’s commitment to comply with this Standard</td>
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<td></td>
</tr>
</tbody>
</table>

Q03 DOCUMENT CONTROL

<table>
<thead>
<tr>
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</tr>
</thead>
<tbody>
<tr>
<td>01</td>
<td>7.0</td>
<td>Verify that procedures are in place to control all QMS documentation and are:</td>
<td></td>
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</tr>
<tr>
<td></td>
<td></td>
<td>· Approved by authorized personnel</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>· Reviewed periodically</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>· Available where required</td>
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<td></td>
<td></td>
<td>· Removed when deemed obsolete</td>
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</tbody>
</table>

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</tr>
</thead>
<tbody>
<tr>
<td>02</td>
<td>7.0</td>
<td>Verify that a readily available master list (or equivalent) is available for all internal and external documents that affect the QMS (including the currently used versions).</td>
<td>[ ] [ ] [ ]</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td>03</td>
<td>7.0</td>
<td>Verify that all quality documentation is uniquely identified, including:</td>
<td>[ ] [ ] [ ]</td>
<td>[ ] [ ] [ ]</td>
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<tr>
<td></td>
<td></td>
<td>- Date of issue</td>
<td>[ ] [ ] [ ]</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Revision number</td>
<td>[ ] [ ] [ ]</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Issuing authority</td>
<td>[ ] [ ] [ ]</td>
<td>[ ] [ ] [ ]</td>
</tr>
<tr>
<td>04</td>
<td>7.0</td>
<td>Verify that changes to documentation are reviewed and approved by the same level of authority that performed the original review and approval and that they had access to the pertinent information.</td>
<td>[ ] [ ] [ ]</td>
<td>[ ] [ ] [ ]</td>
</tr>
</tbody>
</table>

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### Standard’s Requirement Documentation Review

<table>
<thead>
<tr>
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<th>Standard’s Requirement</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>Implementation</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>05</td>
<td>7.0</td>
<td>Verify that procedures are in place to ensure:</td>
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<tr>
<td></td>
<td></td>
<td>The requirements for hand-written changes are defined</td>
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<td></td>
<td></td>
<td>Computerized amendments are adequately controlled</td>
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<td></td>
<td></td>
<td>Other work instructions (e.g. ‘job aides’) are authorized and valid</td>
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<tr>
<td>Q04</td>
<td>VALID REQUIREMENTS (VR)</td>
<td>Verify that policies and procedures are in place to determine the Valid Requirements (i.e. required accuracy and precision) of each technique, e.g.:</td>
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<tr>
<td>01</td>
<td>6.0, A1</td>
<td>Test method validation</td>
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<td></td>
<td></td>
<td>Equipment</td>
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<td>Training</td>
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<td></td>
<td>Measurement traceability</td>
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<tr>
<td></td>
<td></td>
<td>Environmental conditions</td>
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<tr>
<td></td>
<td></td>
<td>Sampling and sample handling</td>
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</tr>
</tbody>
</table>

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### Item 02: A1
Verify that, for each method, the VR is clearly documented, by stating it as:

- A maximum deviation from the target (bias), or
- A maximum deviation at a specified concentration (precision), or
- A stated maximum error (combining bias and precision)

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</thead>
<tbody>
<tr>
<td>02</td>
<td>A1</td>
<td>Verify that, for each method, the VR is clearly documented, by stating it as:</td>
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</tbody>
</table>

#### Q05: SUBCONTRACTING OF TESTS

Verify that the subcontracted laboratory is competent (i.e., conforms to the requirements of this Standard or ISO 17025).

<table>
<thead>
<tr>
<th>Item</th>
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</tr>
</thead>
<tbody>
<tr>
<td>01</td>
<td>17.0</td>
<td>Verify that the subcontracted laboratory is competent (i.e., conforms to the requirements of this Standard or ISO 17025).</td>
</tr>
</tbody>
</table>

#### Q06: PURCHASING SERVICES/SUPPLIES

Verify that the purchasing process allows for the laboratory to specify technical content of the material or service.

<table>
<thead>
<tr>
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</tr>
</thead>
<tbody>
<tr>
<td>01</td>
<td>17.0</td>
<td>Verify that the purchasing process allows for the laboratory to specify technical content of the material or service.</td>
</tr>
</tbody>
</table>

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<tbody>
<tr>
<td>02</td>
<td>17.0</td>
<td>Verify that all supplies affecting quality are stored appropriately and not used until verified compliant with specifications.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>03</td>
<td>17.0</td>
<td>Verify that all supplies affecting quality are obtained from approved suppliers (a list to be maintained).</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Q07</td>
<td>NONCONFORMANCES</td>
<td>Verify that procedures, related to reported results that did not conform to the QMS requirements, are in place to ensure:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>5.0</td>
<td>• Responsibilities are defined</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Authorities are defined</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>• Significance is determined</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>• Proper notifications are made</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>• Corrective actions are followed</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Responsibility for authorizing resumption of work is defined</td>
<td></td>
<td></td>
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</tbody>
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<tbody>
<tr>
<td>Q08</td>
<td></td>
<td><strong>PREVENTATIVE ACTIONS</strong></td>
<td>1  2  3</td>
<td>1  2  3</td>
</tr>
<tr>
<td>01</td>
<td>5.0</td>
<td>Verify that a process is in place to improve the QMS (e.g. through proactive programs, user feedback and complaints, etc.).</td>
<td></td>
<td></td>
</tr>
<tr>
<td>02</td>
<td>5.0</td>
<td>Verify that procedures are in place for the documentation of preventive actions.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>03</td>
<td>5.0</td>
<td>Verify that preventive actions put in place are monitored to evaluate their effectiveness.</td>
<td></td>
<td></td>
</tr>
</tbody>
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<tbody>
<tr>
<td>Q09</td>
<td>5.0</td>
<td>Verify that a corrective action program is in place that ensures:</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td>01</td>
<td></td>
<td>- Confirmation of the non-conformance</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Cessation of use of the method until resolution of the problem</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Investigation as to whether like processes are affected</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Performing remedial action to correct the problem</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Determination as to whether retesting should be performed</td>
<td></td>
<td></td>
</tr>
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<tr>
<td></td>
<td></td>
<td>02 5.0 Verify that a corrective action program is in place that defines the appropriate authority required for the development and implementation of the actions and includes:</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Cause analysis</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Identification of possible corrective actions</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Selection of the most appropriate corrective action</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Development and implementation of necessary changes</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td>- Monitoring for the effect of any changes made</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- The laboratory authority authorized to permit the resumption of work</td>
<td></td>
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<tr>
<td>Q10</td>
<td>16.0</td>
<td>Q10 CONTROL OF RECORDS</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>01 Verify that a process is in place to ensure continued legibility and that retention times are adequate to meet both quality assurance and regulatory requirements.</td>
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<tr>
<td>02</td>
<td>16.0</td>
<td>Verify that a process is in place to ensure the prevention of alteration of stored records and the backup of electronic files.</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td>03</td>
<td>16.0</td>
<td>Verify that a process is in place to ensure all data needed to repeat the test under similar conditions is available, e.g.:</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<tr>
<td></td>
<td></td>
<td>- Personnel involved</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Instrumentation parameters</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Procedure used</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Certified calibration certificates</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Raw data is available</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td>04</td>
<td>16.0</td>
<td>Verify that a process is in place to ensure that:</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Data is recorded in a permanent medium</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Changes made do not obscure the original data</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<tr>
<td></td>
<td></td>
<td>- Changes are traceable</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
</tbody>
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<tr>
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<td></td>
<td>1 2 3</td>
<td>1 2 3</td>
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<tr>
<td>05</td>
<td>16.0</td>
<td>Verify that a process is in place to ensure standardized forms used to record raw data are authorized.</td>
<td></td>
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<tr>
<td>Q11</td>
<td>INTERNAL AUDITS</td>
<td></td>
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<tr>
<td>01</td>
<td>18.0</td>
<td>Verify that a process is in place to ensure internal audits are periodically conducted and:</td>
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<td></td>
<td>• Follow a predetermined schedule</td>
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<td>• Conducted by trained personnel</td>
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<td></td>
<td>• Corrective actions are acted on</td>
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<td></td>
<td>• Records are maintained</td>
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<td>• Follow-up audits are performed if required</td>
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<td></td>
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<td>• Significant findings are reported to the necessary authorities</td>
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</tbody>
</table>

Q12  MANAGEMENT REVIEWS

| 01   | 19.0    | Verify that a process is in place to ensure that top management carries out an annual review of the QMS with laboratory management, including quality management, to ensure its continuing suitability. |                      |                |

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<tbody>
<tr>
<td>02</td>
<td>19.0</td>
<td>Verify that a process is in place to ensure the following areas are reviewed:</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Policies and procedures</td>
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<tr>
<td></td>
<td></td>
<td>- Internal audit findings</td>
<td></td>
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<td></td>
<td></td>
<td>- Corrective and preventive actions</td>
<td></td>
<td></td>
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<td></td>
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<td>- External audit findings</td>
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<td>- Changes in volume/type of work</td>
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<td>- Laboratory user feedback</td>
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<td>- Complaint resolution</td>
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<td>- Improvement initiative results</td>
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<td></td>
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<td></td>
<td></td>
<td>- Staff training</td>
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</table>

| 03   | 19.0    | Verify that a process is in place to ensure that actions are carried out within an appropriate and agreed to timetable and that records of actions and resolutions are maintained. |                     |                |

1 – Complies with standard; 2 – Does not comply with standard; 3 – not applicable
<table>
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<th>Implementation</th>
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<tbody>
<tr>
<td></td>
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<td>1</td>
<td>2</td>
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<tr>
<td>Q13</td>
<td>PERSONNEL</td>
<td></td>
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</tr>
<tr>
<td>01</td>
<td>11.0</td>
<td>Verify that technical personnel:</td>
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<td></td>
<td></td>
<td>- Are authorized to perform tasks independently</td>
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<td></td>
<td></td>
<td>- Demonstrate their capabilities through job performance measures</td>
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<td></td>
<td></td>
<td>- Are adequately supervised</td>
<td></td>
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<tr>
<td>02</td>
<td>11.0</td>
<td>Verify that a Systematic Approach to Training (SAT) program is in place that:</td>
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<td></td>
<td></td>
<td>- Identifies training needs</td>
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<td></td>
<td></td>
<td>- Provides the required training</td>
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<td>- Provides evaluation of training effectiveness</td>
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<td></td>
<td>- Provides retraining as required</td>
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<td></td>
<td></td>
<td>- Is adequately documented</td>
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</table>

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<tbody>
<tr>
<td>03</td>
<td>11.0</td>
<td>Verify that current job descriptions are available for all laboratory staff.</td>
<td></td>
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<tr>
<td>04</td>
<td>11.0</td>
<td>Verify that management has authorized and maintains dated records for specific personnel to:</td>
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<tr>
<td></td>
<td></td>
<td>- Perform sampling</td>
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<td></td>
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<td>- Perform testing</td>
<td></td>
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<td></td>
<td></td>
<td>- Authorize results</td>
<td></td>
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<td></td>
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<td>- Operate equipment</td>
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<td></td>
<td></td>
<td>- Evaluating QC data</td>
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<td>- Conducting Internal Audits</td>
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<td></td>
<td></td>
<td>- Authorizing procedures</td>
<td></td>
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<tr>
<td>05</td>
<td>11.0</td>
<td>Verify that the laboratory has an intra-laboratory program in place to evaluate the staff’s proficiency in performing analytical determinations against predetermined conditions.</td>
<td></td>
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</tbody>
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<tr>
<td></td>
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<td>1 2 3</td>
<td>1 2 3</td>
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<tr>
<td>Q14</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>01</td>
<td>12.0</td>
<td>Verify that accommodation and environmental conditions are adequate to meet the Valid Requirements and:</td>
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<td></td>
<td></td>
<td>• Are documented and recorded</td>
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<td></td>
<td></td>
<td>• Include adequate housekeeping</td>
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<td></td>
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<td>• Lead to termination of testing when conditions are unacceptable</td>
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<td></td>
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<td>• Include effective separation between areas of incompatibility</td>
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<td></td>
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<td>• Include routine feedback to management on safety issues</td>
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<tr>
<td>Q15</td>
<td></td>
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<tr>
<td>01</td>
<td>8.0, A2</td>
<td>Verify that the laboratory uses appropriate methods to ensure VR are met, based on:</td>
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<td></td>
<td></td>
<td>• International standards</td>
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<td></td>
<td>• National standards</td>
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<td>• Regional standards</td>
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<td></td>
<td></td>
<td>OR, if necessary</td>
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<td></td>
<td></td>
<td>• Published methods or</td>
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<td></td>
<td></td>
<td>• As specified by the manufacturer</td>
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</tbody>
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<tbody>
<tr>
<td>02</td>
<td>8.0</td>
<td>Verify that the laboratory uses an appropriate method of validation, such as:</td>
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<td></td>
<td></td>
<td>- Analysis of Standard Reference Materials</td>
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<td></td>
<td></td>
<td>- Analysis of Reference Materials, the validity of which is documented</td>
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<td></td>
<td></td>
<td>- Use of an alternative method</td>
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<td></td>
<td></td>
<td>- Use of recovery studies</td>
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<tr>
<td>03</td>
<td>8.0, A2</td>
<td>Verify that the laboratory’s validation of a method confirms, as appropriate:</td>
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<td></td>
<td></td>
<td>- The number of standards needed</td>
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<td>- The calibration curve</td>
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<td>- The LOD and UQL</td>
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<td></td>
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<td>- Matrix interference</td>
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<td></td>
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<td>- Blank interference</td>
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<tbody>
<tr>
<td>04</td>
<td>9.0, A2</td>
<td>Verify that the laboratory has a process in place to ensure the method is re-validated if the method undergoes change (major maintenance, reagent modification, matrix change, etc.).</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>05</td>
<td>8.0</td>
<td>Verify that the laboratory participates in proficiency testing programs at a regular frequency to evaluate the laboratory’s performance against predefined conditions.</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>06</td>
<td>8.0, A2</td>
<td>Verify that method validation includes:</td>
<td>1</td>
<td>2</td>
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<tr>
<td></td>
<td></td>
<td>- Records of validation</td>
<td></td>
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<td></td>
<td></td>
<td>- Procedure used</td>
<td></td>
<td></td>
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<tr>
<td>07</td>
<td>8.0, A2</td>
<td>Verify that the laboratory ensures the range and accuracy are relevant the intended use.</td>
<td>1</td>
<td>2</td>
</tr>
</tbody>
</table>

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<tr>
<td>08</td>
<td>8.0</td>
<td>Verify that the laboratory, upon request, is able to provide an estimate of measurement uncertainty.</td>
<td></td>
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<tr>
<td>Q16</td>
<td>EQUIPMENT</td>
<td></td>
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<tr>
<td>01</td>
<td>9.0</td>
<td>Verify that all equipment used for producing test results is:</td>
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<tr>
<td></td>
<td></td>
<td>• Available and functioning properly</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<td></td>
<td></td>
<td>• Capable of meeting VR</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<tr>
<td></td>
<td></td>
<td>• Checked and calibrated as specified prior to use</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<td></td>
<td></td>
<td>• Operated by authorized personnel</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<td></td>
<td></td>
<td>• Maintained and operated as per current instructions</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<td></td>
<td></td>
<td>• Uniquely identified, where applicable</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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</tbody>
</table>

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<tbody>
<tr>
<td>02</td>
<td>9.0</td>
<td>Verify that equipment records are maintained and include:</td>
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<td></td>
<td></td>
<td>• Make, model and serial numbers</td>
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<td></td>
<td></td>
<td>• Information re the instrument’s ability to meet specifications</td>
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<td>• Manufacture’s instructions</td>
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<td></td>
<td>• Calibration history and due date for next calibration</td>
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<td>• Damage and repairs made</td>
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<tr>
<td>03</td>
<td>9.0</td>
<td>Verify that equipment procedures are in place for:</td>
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<td></td>
<td></td>
<td>• Use</td>
<td></td>
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<td></td>
<td></td>
<td>• Planned maintenance</td>
<td></td>
<td></td>
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<tr>
<td>04</td>
<td>5.0, 9.0</td>
<td>Verify that equipment deemed defective is clearly identified as such and addressed under a non-conformance procedure.</td>
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<tr>
<td>05</td>
<td>9.0</td>
<td>Verify that the equipment calibration status, including the expiry date is available (not applicable to equipment calibrated on an as-used basis).</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td>06</td>
<td>9.0</td>
<td>Verify that equipment that goes outside the direct control of the laboratory is validated to ensure it meets its VR prior to return to service.</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td>Q17</td>
<td>10.0</td>
<td>MEASUREMENT TRACEABILITY</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
</tr>
<tr>
<td>01</td>
<td>10.0</td>
<td>Verify that suppliers of calibration services provide:</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<td></td>
<td></td>
<td>• Evidence of their competence, traceability and capability</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<td></td>
<td></td>
<td>• Certificates that contain the measurement results</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<td></td>
<td></td>
<td>• Certificates that include measurement uncertainties</td>
<td>☐ ☐ ☐</td>
<td>☐ ☐ ☐</td>
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<td>02</td>
<td>10.0</td>
<td>Verify that the laboratory utilizes:</td>
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<tr>
<td></td>
<td></td>
<td>- Certified Reference Materials (CRM) OR</td>
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<td>- Reference materials that are traceable to a CRM</td>
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<td>- Instructions for alternatives when the above are not available</td>
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<td></td>
<td></td>
<td>- Procedures for the transport and storage of reference materials</td>
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<tr>
<td>Q18</td>
<td>SAMPLING</td>
<td></td>
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<tr>
<td>01</td>
<td>13.0</td>
<td>Verify that the laboratory has sample procedures in place and readily available for those samples that are in its area of responsibility.</td>
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<td>02</td>
<td>13.0</td>
<td>Verify that the sampling procedures include or reference:</td>
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<tr>
<td></td>
<td></td>
<td>• Sample location</td>
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<tr>
<td></td>
<td></td>
<td>• Sample container type</td>
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<td></td>
<td></td>
<td>• Sample container rinsing requirements</td>
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<td></td>
<td>• Safety requirements</td>
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<td>• Valving sequences (as appropriate)</td>
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<td></td>
<td>• Auto sampler identification (as appropriate)</td>
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<td></td>
<td></td>
<td>• Environmental conditions (if relevant)</td>
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### Standard for a QMS Assessment

#### Item Section Standard's Requirement Documentation Review Implementation

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<tr>
<td>Q19</td>
<td>13.0</td>
<td><strong>SAMPLE HANDLING</strong></td>
<td>1  2  3</td>
<td>1  2  3</td>
</tr>
<tr>
<td>01</td>
<td></td>
<td>Verify that, whether or not laboratory staff obtain the sample, a process is in place to ensure samples are:</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>• Stored and handled in a manner to protect their integrity</td>
<td>1  2  3</td>
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<tr>
<td></td>
<td></td>
<td>• Uniquely identified</td>
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<tr>
<td></td>
<td></td>
<td>• Examined on receipt to ensure they are fit for purpose</td>
<td>1  2  3</td>
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<tr>
<td></td>
<td></td>
<td>• Maintained in such a way so as to avoid deterioration</td>
<td>1  2  3</td>
<td></td>
</tr>
<tr>
<td>Q20</td>
<td>14.0</td>
<td><strong>CONTROL OF DATA</strong></td>
<td>1  2  3</td>
<td>1  2  3</td>
</tr>
<tr>
<td>01</td>
<td></td>
<td>Verify that independent verification and rationality checks are used to ensure the correctness of data when produced through calculational techniques.</td>
<td>1  2  3</td>
<td></td>
</tr>
<tr>
<td>02</td>
<td>14.0</td>
<td>Verify that a process is in place to ensure factors embedded in software (spreadsheets and the like) are verified when they are updated.</td>
<td>1  2  3</td>
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<tr>
<td>03</td>
<td>14.0</td>
<td>Verify that laboratory developed software is sufficiently validated.</td>
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<tr>
<td>04</td>
<td>A5</td>
<td>Verify that a process is in place to ensure that data collected for the preparation of a control chart is appropriate, e.g.:</td>
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<td></td>
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<td>- Environmental conditions are typical</td>
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<tr>
<td></td>
<td></td>
<td>- Equipment is that which is normally used for sample analysis</td>
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<td></td>
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<td>- Reagents are the same grade as is normally used for sample analysis</td>
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<td></td>
<td>- Staff is similar in ability as is used for normal sample analysis</td>
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<td>- Data is collected at the normal frequency and concentration</td>
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<tr>
<td></td>
<td></td>
<td>- Temporary limits set during data collection do not exceed the VR</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1 – Complies with standard; 2 – Does not comply with standard; 3 – not applicable
<table>
<thead>
<tr>
<th>Item</th>
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<th>Documentation Review</th>
<th>Implementation</th>
</tr>
</thead>
<tbody>
<tr>
<td>05</td>
<td>A5</td>
<td>Verify that control charts are evaluated on an on-going basis to detect trends and to ensure VR are not exceeded.</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td>06</td>
<td>A5</td>
<td>Verify that data used in the preparation of control charts is statistically analysed, i.e.:</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Evaluation of data normality</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Evaluation of data precision</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Evaluation of control chart centre</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Setting of statistical limits</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Setting of manual limits, if warranted</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
</tbody>
</table>

1 – Complies with standard; 2 – Does not comply with standard; 3 – not applicable
<table>
<thead>
<tr>
<th>Q21</th>
<th>REPORTS</th>
<th>Documentation Review</th>
<th>Implementation</th>
</tr>
</thead>
<tbody>
<tr>
<td>01</td>
<td>15.0</td>
<td>Verify that, although simplified reports are normally used, relevant data such as listed below are readily available:</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Sample time</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Analysis time</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Analytical units</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Analyst</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Sample identifier</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Method used</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Equipment used</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• QA/QC results</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Traceability</td>
<td></td>
</tr>
<tr>
<td>02</td>
<td>15.0</td>
<td>Verify that for those instances a deviation from the normal protocol was made, a notation to alert the end user is recorded.</td>
<td></td>
</tr>
</tbody>
</table>

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### POWER PLANT CHEMISTRY ADVISORY GROUP
**QMS Assessment  Method Review**  
Attachment 12.3

#### LABORATORY NAME


---

#### Team Member(S)


---

#### DATE


---

#### METHOD BEING ASSESSED


---

<table>
<thead>
<tr>
<th>ANALYST(S)</th>
<th>ANALYTICAL INSTRUMENTATION</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

#### INTERLABORATORY SAMPLE COMPLETED IN THE PAST 2 YEARS? – [ ] Yes          [ ] No

If Yes – results of interlaboratory sample(s)  [ ] Pass  [ ] Fail

If fail – was corrective action plan implemented?  [ ] Yes  [ ] No
<table>
<thead>
<tr>
<th>Item</th>
<th>Section</th>
<th>Standard’s Requirement</th>
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<th>Implementation</th>
</tr>
</thead>
<tbody>
<tr>
<td>M01</td>
<td>6.0, A1</td>
<td>VALID REQUIREMENTS</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td>01</td>
<td>6.0, A1</td>
<td>Verify that the Valid Requirements have been determined so as to ensure, e.g.:</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Appropriate methods</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Appropriate equipment</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Appropriate training</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Measurement traceability</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Appropriate environmental conditions</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Appropriate sampling and sample handling</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td>M02</td>
<td>7.0</td>
<td>DOCUMENTATION CONTROL</td>
<td></td>
<td>1 2 3</td>
</tr>
<tr>
<td>01</td>
<td>7.0</td>
<td>Verify that the current authorized method, supporting work instructions and sampling instructions are readily available to the analyst</td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
</tbody>
</table>

1 – Complies with standard; 2 – does not comply with standard; 3 – not applicable
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td>M03</td>
<td></td>
<td><strong>METHOD VALIDATION</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>8.0, A2, A7, A8</td>
<td>Verify that the method has been validated for the following (as applicable)</td>
<td>□ □ □</td>
<td>□ □ □</td>
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<td></td>
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<td></td>
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<tr>
<td></td>
<td></td>
<td>• LOD/LOQ/MDL/UQL</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Precision and bias</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Concentration range</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Number of standards required</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Measurement uncertainty</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Effect of any known interference</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Appropriate significant digits</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
<tr>
<td>M04</td>
<td></td>
<td><strong>PROCEDURE</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>7.0</td>
<td>Verify that all steps in the method enable the production of competent results.</td>
<td>□ □ □</td>
<td>□ □ □</td>
</tr>
</tbody>
</table>

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<tr>
<td></td>
<td></td>
<td></td>
<td>1 2 3</td>
<td>1 2 3</td>
</tr>
<tr>
<td>M05</td>
<td>SAMPLING</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
| 01   | 12.0    | Verify that appropriate instructions are provided and implemented, for those samples that are obtained by laboratory staff (does not apply to on-line instrumentation) e.g.,  
  - Field filtration  
  - Chemical preservation  
  - Sample containers  
  - Storage conditions  
  - Holding time                                                                                                                   |                      |                 |
| 02   | 12.0    | Verify that appropriate sampling instructions are provided and implemented, for those samples that are obtained by laboratory staff (does not apply to on-line instrumentation) e.g.,  
  - Sample location (room, valve, etc.)  
  - Flow rate, flush time  
  - Operating state of unit and system  
  - Approved alternate sample point  
  - Personal protective equipment                                                                                                  |                      |                 |

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<tbody>
<tr>
<td>M06</td>
<td>A9, 2.4</td>
<td>Verify that the on-line instrument's ability to meet the Valid Requirements has been</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>demonstrated, e.g.:</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Line Method</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Reference Method</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Standard Injection Method</td>
<td></td>
<td></td>
</tr>
<tr>
<td>02</td>
<td>A9</td>
<td>Verify that proper sample conditioning is in place (as applicable), e.g.:</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Pre-filtration</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Temperature regulation/correction</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Flow regulation</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Pressure regulation</td>
<td></td>
<td></td>
</tr>
</tbody>
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<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>1  2  3</td>
<td>1  2  3</td>
</tr>
<tr>
<td>M07</td>
<td>CALIBRATION</td>
<td>Verify that method calibration requirements (as appropriate) are included or referenced in the test method.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
| 01   | 8.0, 10.0, A2 | - Reagent blank  
- Standard/sample matrix matched  
- Adequate number of standards  
- Traceable standard used (to national or international if possible) |                      |                |
| M08  | METHOD QUALITY CONTROL | Verify that quality control requirements are included or referenced in the method (as appropriate). |                      |                |
| 01   | 10.0, A5  | - Duplicates  
- Reference standard  
- Method blank  
- Control charting  
- Control standard (independent from calibration standards) |                      |                |

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<tr>
<td></td>
<td></td>
<td></td>
<td>1  2  3</td>
<td>1  2  3</td>
</tr>
<tr>
<td><strong>M09</strong></td>
<td>METHOD CONTENT</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>7.0, 9.0</td>
<td>Verify that all supporting work instructions are available to the analyst; e.g.</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Glassware cleaning instructions</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Supporting test methods</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>- Equipment instruction manuals</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Reference texts</td>
<td></td>
<td></td>
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</table>

<table>
<thead>
<tr>
<th>Item</th>
<th>Clause</th>
<th>Standard’s Requirement</th>
<th>1  2  3</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>M10</strong></td>
<td>CONDUCT OF TESTING</td>
<td></td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>6.0, 7.0, 8.0</td>
<td>Verify the test, including all supporting work instructions are performed as documented.</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Item</th>
<th>Clause</th>
<th>Standard’s Requirement</th>
<th>1  2  3</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>M11</strong></td>
<td>EQUIPMENT</td>
<td></td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>9.0</td>
<td>Verify that all necessary instrumentation is available and functioning properly</td>
<td></td>
</tr>
</tbody>
</table>

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<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>02</td>
<td>9.0</td>
<td>Verify that all out of service equipment is clearly marked and checked before return to service to ensure it meets its Valid Requirements.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>03</td>
<td>9.0</td>
<td>Verify that all equipment that requires periodic calibration clearly indicates the next required calibration date (not required for equipment calibrated 'as-used').</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>04</td>
<td>9.0</td>
<td>Verify that a planned and implemented instrumentation maintenance program is in place.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>05</td>
<td>9.0</td>
<td>Verify that all temporary equipment (e.g. during repairs to permanent equipment, etc.) meets the level of performance of the permanent equipment (i.e. meets method’s Valid Requirements).</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M12</td>
<td></td>
<td>SUPPLIES</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>01</td>
<td>10.0, 17.0</td>
<td>Verify that all supplies are available and meet any predefined requirements.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>02</td>
<td>10.0, 17.0</td>
<td>Verify that all supplies are stored appropriately (temperature, incompatibility, etc.).</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
<tr>
<td>03</td>
<td>17.0</td>
<td>Verify all reagents are properly labeled and indicate expiry date.</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
<tr>
<td>04</td>
<td>9.0</td>
<td>Verify that all labware is properly cleaned.</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
<tr>
<td>M13</td>
<td></td>
<td><strong>RECORD KEEPING</strong></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>01</td>
<td>15.0, 16.0</td>
<td>Verify records relating to the method are maintained, e.g.</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Analyst's notes</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Record of non-conformances</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Reagent preparation log</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Equipment preparation log</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Gravimetric, volumetric and temperature traceability</td>
<td>☐</td>
<td>☐</td>
<td>☐</td>
</tr>
</tbody>
</table>

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