Arizona Department of Weights and Measures

Quality Assurance/Quality Control Plan
Development Policy

for

Compliance with the Arizona Cleaner Burning Gasoline Rule,
Title 20, Chapter 2, Article 7

Revision 0

Effective Date: December 19, 1997

Table of Contents

This document was prepared as an account of work by *Quality by Design* for the Arizona Department of Environmental Quality, in cooperation with the Arizona Department of Weights and Measures.

1.0 Introduction

1.1 This document describes the program requirements of the Arizona Department of Weights and Measures for the development of a laboratory testing Quality Assurance/Quality Control (QA/QC) plan required under the Arizona Cleaner Burning Gasoline (CBG) Program, codified in the Arizona Administrative Code (AAC) Title 20, Chapter 2, Article 7. The requirements outlined in this document are intended to ensure that data generated in accordance with the Arizona CBG regulatory requirements are technically defensible, legally admissible, and consistent with national and international standards for laboratory testing. Laboratories which provide analytical results under the Arizona CBG program must prepare and implement a QA/QC Plan compliant with the requirements of this document, and must provide this QA/QC Plan to the Arizona Department of Weights and Measures for review and approval prior to performing testing required as part of the Arizona CBG program. The rule provides exemptions from the laboratory QA/QC Plan submittal requirements for facilities that conduct independent testing program or utilize computer-controlled in-line blending equipment and are operating under an exemption from EPA.

1.2 This document provides policies for the preparation of the laboratory QA/QC plans. Although contract-specific or project-specific requirements may alter, add to, or replace individual elements of the baseline QA program, the QA/QC Plan should describe the laboratory's minimum Quality Assurance policies and procedures for the testing of Arizona CBG or AZRBOB, as well as the process for identifying, documenting, and implementing project-specific requirements, so that confidence in the laboratory's independence of judgment and integrity is maintained at all times. Nothing in this document relieves any program participant from the responsibility of complying with contract requirements or with applicable federal, state, or local regulations. The Arizona Department of Weights and Measures (ADWM) should be notified of substantive technical conflicts between this document and other applicable requirements.

1.3 Program components that are considered essential are specified throughout this document by the use of the terms "shall" or "must." Information that is provided as guidance that constitutes an acceptable means of accomplishing a desired objective is designated by the terms "should" (recommended) or "may" (permissible). Examples are used extensively throughout this document. These examples are not requirements but are illustrations of the intent of the policy or samples of how laboratories may deal with the issue being discussed.

2.0 Definitions Used in This Policy Document

2.1 Accuracy: The ability of a test to report the true or expected value of the quantity of concern.

2.2 ASTM: American Society for Testing and Materials.

2.3 AZRBOB: A petroleum-derived liquid as defined in AAC R20-2-701.

2.4 CBG: Cleaner Burning Gasoline, as defined in AAC R20-2-701.

2.5 Cross contamination: The process by which an analyte in one sample or container is transmitted into another sample, thereby contaminating the second sample.

2.6 Data Reduction: The process of making calculations and manipulating analytical data for use in a laboratory report.

2.7 External proficiency testing: The testing of a known value check sample from a source outside of the laboratory, and for which the acceptability of the test results are determined by a process independent of the laboratory's control.

2.8 Known value check sample: A sample that is of a comparable matrix as the samples to be tested, for which the true or expected value of the quantity of concern is known.

2.9 MDL: Method Detection Limit

2.10 Precision: The closeness of agreement between two or more analyses of the same sample. Precision may be defined as the "repeatability" criteria as listed in each ASTM method.

2.11 Replicate testing: Performing the same analysis on two or more aliquots or subsamples from a single sample and thereby obtaining two or more analytical results.

2.12 RF: Response factor

2.13 RSD: Relative Standard Deviation

2.14 Senior Laboratory Management: An employee of an organization which has a laboratory who has the authority to commit the organization to the policies of the QA/QC Plan and who has the responsibility for implementing the QA/QC Plan.

2.15 SOP: Standard Operating Procedure

3.0 General Requirements for the Laboratory Quality Assurance/Quality Control Plan

3.1 A laboratory's quality assurance (QA) and quality control (QC) documentation defines the quality management system of the laboratory and establishes policies for accomplishing, maintaining, and improving that system. For the purposes of this document, a laboratory's top level quality assurance document which defines QA policies will be referred to as a QA/QC Plan, although in practice, it may be referred to as a QA Manual, QA Program Plan, or similar title.

3.2 The QA/QC Plan defines and documents policies and objectives for good laboratory practices and the quality of client services. Although contract specific or project-specific requirements may alter, add to, or replace individual elements of the baseline QA Program, the QA/QC Plan must describe the laboratory's minimum QA policies and procedures by providing a complete description of the scope, approach, and implementation of the laboratory's overall QA Program for the testing of Arizona CBG or AZRBOB.

3.3 The minimum elements of the laboratory QA/QC Plan shall include:

1. Signature Page
2. Table of Contents
3. Program Organization
4. Sample Collection Control
5. Sample Analysis Protocols and Controls
6. Data Reduction, Review, and Reporting
7. Document and Data Control
8. Data Assessment and Corrective Action Process
9. Quality Assurance Reviews and Reports
10. Subcontracting
11. Preventative Maintenance
12. Reports to the Arizona Department of Weights and Measures
13. References
14. Definitions and Abbreviations
15. Attachments

3.4 If a QA/QC Plan does not include all of the information required by the ADWM Cleaner Burning Gasoline Program in this document, the laboratory must provide the required information by some equivalent method and cross-reference to the elements listed in Section 3.3. The equivalent method must be accompanied by signature approvals at the same levels of authority required for release of the QA/QC Plan.

Example: Some required information is not present in a QA/QC Plan that is already in existence, but is in a Standard Operating Procedure (SOP). The SOP may be attached as an appendix or exhibit to the Quality Assurance/Quality Control Plan.

4.0 Contents of the Quality Assurance/Quality Control Plan

4.1 Signature Page

The laboratory QA/QC Plan must include a signature page. This page must include this certification statement:

"I hereby certify and attest that I am the {position} with {company} , that I have the authority to act on behalf of and bind the company with regards to this QA/QC Plan, that the foregoing QA/QC Plan is true and accurate to the best of my knowledge, that the QA/QC plan is a laboratory policy document, that the management of the company is committed to the implementation of the plan, will provide the resources necessary for the plan's implementation, and that all necessary laboratory staff have been made aware of the policies contained herein.

The effective date and revision number of this QA/QC Plan is _____. This QA/QC Plan is being submitted pursuant to the Arizona Administrative Code Title 20, Chapter 2, Article 7.

_____
Signature

_____
Printed/Typed Name

_____
Title

_____
Company

_____
Date"

4.2 Table of Contents

The Table of Contents must list each major topic and its location in the document. Elements of the plan may be presented in any order. A list of appendices or attachments which augments the laboratory QA/QC Plan must be included. The laboratory must include a cross-reference which details the correlation between an existing QA/QC Plan and the requirements of this document.

4.3 Program Organization

4.3.1 Description

The laboratory shall be legally identifiable as to name, business, and physical location. It shall be organized and shall operate in such a way that its permanent, temporary and mobile facilities used to analyze Arizona CBG or AZRBOB meet the requirements of this policy document. This section must have:

4.3.1.1 A brief description of the number of employees, and all laboratory facility(ies) and layout involved in analyzing Arizona CBG or AZRBOB samples.

4.3.1.2 An estimate of the number and type of samples analyzed .

4.3.1.3 A list of major laboratory equipment and computerized systems (e.g., chromatography software, LIMS system) used to analyze Arizona CBG or AZRBOB.

Example: "This laboratory is a full service facility employing 38 staff. It performs the analysis of Fuels and Volatile Organics by gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS). No high pressure liquid chromatography is performed. Reid Vapor Pressure is performed by ASTM D-5191 and sulfur by X-ray Spectrometry. In addition, the laboratory performs wastewater analysis, for which metals are performed by Inductively Coupled Plasma (ICP) and Atomic Absorption (AA) spectroscopy and classical wet chemistry methods are performed by both automated and manual techniques. There is a Sample Receiving area and adequate hoods for sample extractions and digestions.

"The facility is approximately 7,000 square feet and conflicting operations have been segregated to the extent practical. Two different physical locations, separated by a driveway, with spot ventilation, external air handling and fume hoods serve to minimize contamination problems. The laboratory operates a single shift, but individual employee hours are staggered so that the facility is staffed from 8:00 AM into the early evening.

"A listing of the test methods that the laboratory routinely performs is in Appendix A, a listing of the laboratory's Standard Operating Procedures is in Appendix B, and a listing of major laboratory

equipment is in Appendix C."

4.3.2 Management

This section should contain a description of the organizational entities involved in data collection activities. The laboratory shall:

4.3.2.1 Have managerial staff with the authority and resources needed to discharge their duties.

4.3.2.2 Specify and document the responsibility, authority and interrelation of all personnel who manage, perform or verify work affecting the quality of calibrations and tests.

4.3.2.3 Have a technical manager (however named), who is familiar with the test methods and procedures, the objective of the calibration or test and the assessment of the results, and who has overall responsibility for the technical operations.

4.3.2.4 Have a quality manager (however named) who has responsibility for the quality system and who has the authority to recommend and implement corrective actions in response to quality issues. The quality manager shall have direct access to the highest level of management at which decisions are made or actions are taken on laboratory policy or procedures, and to the technical manager. It is recognized that in some laboratories, the quality manager may also be the technical manager or deputy technical manager.

4.3.2.5 Nominate deputies who carry out the responsibilities of the technical manager and the quality manager in case of absence of the technical or quality manager.

4.3.3 Staff Qualifications and Personnel Training Records

The QA/QC Plan shall describe the policy through which the laboratory can demonstrate each analyst's proficiency and ability to perform the test properly. The laboratory must develop and document procedures for training each analyst in all methods that each analyst is conducting.

4.3.3.1 The training method and time period shall be documented.

4.3.3.2 In-house training records shall be maintained and be available for audit for each analyst.

4.3.3.3 At a minimum, the records shall include the analyst's name, the method(s) for and date(s) on which the analyst has completed training, a brief description of the training and the final demonstration of proficiency, the trainer who is certifying completion of each training session, the date(s) when recertification training is needed and the date(s) recertification was completed (if appropriate).

4.3.3.4 Only analysts who have completed training may perform analytical methods independently. An analyst in training must be directly supervised by an analyst who has completed training.

4.3.3.5 The laboratory may include continuing education in the in-house training records, such as additional education courses, professional seminars attended, and company sponsored training courses.

Example: The laboratory may establish minimum job description qualifications for each type of position in the laboratory or may require a set number of supervised on-the-job training, and may couple these requirements with a policy that each analyst demonstrate proficiency by successfully analyzing an unknown sample.

4.3.4 Laboratory Organizational Chart.

The laboratory shall provide a description of the laboratory organization or an organization chart which indicates supervisor's responsibilities, oversight responsibilities, and independence of the quality manager. If desired, the laboratory may provide short resumes or job descriptions of key laboratory staff. At a minimum, the information must include:

4.3.4.1 Laboratory name.

4.3.4.2 Laboratory address(es).

4.3.4.3 Laboratory positions or the position titles.

4.3.4.4 Direct and indirect lines of authority and supervision within the laboratory.

4.3.4.5 Effective date of the organizational chart or description.

4.4 Sample Collection Control

4.4.1 Sample Representativeness

The laboratory or any party responsible for sampling shall describe procedures for the collection of samples which will ensure that the samples are representative of the material being sampled. These procedures must be referenced to a standard method such as ASTM D-4057 or the API MPMS (Chapter 8.1, Manual for Sampling of the Petroleum and Petroleum Products). These procedures which are included or referenced in the QA/QC Plan must include but are not limited to:

4.4.1.1 Policies or procedures for sample container cleaning and decontamination.

4.4.1.2 SOPs and/or policies for documenting sampling procedures.

4.4.1.3 A test criteria which documents that the samples are truly representative of the material being sampled.

Example: The QA/QC Plan should address how the laboratory can be assured that a sample from a tank is representative of the entire contents of the tank, e.g., through the use of purging of sample lines, mixing, random or stratified subsampling, timed subsampling, and/or placement of the sample intake line. The taking of multiple samples from a tank, such as composite, bottom, top, and middle samples, each of which are checked for specific gravity, is an acceptable example of stratified subsampling, providing that an acceptance criteria window is established.

4.4.2 Criteria for Rejection of Samples

The laboratory shall describe the inspection process or procedures for identifying and rejecting unusable or improperly handled samples.

Example: Several ASTM methods require that the sample container be filled to about three quarters full . The QA/QC Plan would identify at what point the sample container is inspected, the person responsible for inspection, and the criteria or the process by which a sample is rejected if the sample container is damaged or improperly filled to less than half.

4.4.3 Sample Tracking

The laboratory shall describe the policies and procedures for sample tracking within and outside of the laboratory. The system used to track individual samples from sample receipt through data reporting, archival, and disposal must be described.

4.4.4 Shipping, Storage, and Archival of Samples

The QA/QC Plan must provide a description of the policies or procedures that will ensure that samples are shipped, stored, and archived in a manner to prevent cross-contamination and maintain sample integrity. The QA/QC Plan must describe the length of archival time and the procedure for ensuring that samples are not prematurely disposed. The minimal archival time period is 30 days. If requested by the Arizona Department of Weights and Measures, individual samples must be stored for up to 180 days.

4.5 Sample Analysis Protocols and Controls

4.5.1 Analytical Methods

The QA/QC Plan must include a listing of the analytical methods used by the laboratory. The analytical methods must be those approved for use under the Arizona CBG program per AAC R20-2-759. The listing must:

4.5.1.1 Include full references as described in Section 4.14.

4.5.1.2 Identify any major deviations from or modifications to the approved published methods.

Example: The USEPA has issued draft guidance which suggests that minor deviations, such as using a different size beaker or a different chromatography column, do not require documentation beyond routine revisions of the laboratory's analytical SOP. Major deviations, such as replacing

a

gas chromatograph's flame ionization detector with a flame photometric detector, either are not permitted or are to be documented to meet or exceed the Quality Control criteria of the reference method.

4.5.1.3 Include a policy which sets criteria for the approval of deviations and modifications in the event that the laboratory develops modifications to the method. This policy must include a procedure for communicating the approval and implementation of changes and deviations to the analytical staff as well as the process for identifying, documenting, and implementing project specific requirements. Major deviations from the analytical methods are prohibited unless this policy is included in an ADWM approved QA/QC Plan or an approved amendment thereto.

Example: The QA/QC Plan might state, "Changes to existing methods may not be performed in the laboratory until they are demonstrated to be equivalent in terms of precision and accuracy to the existing method through the use of multiple sample equivalence studies. Prior to use, a SOP must be developed and issued to each analyst in conjunction with a training/discussion period. Analysts must then demonstrate their proficiency in the method as described in Section XX.YY."

4.5.2 Glassware and Equipment

The laboratory must describe the process and policies that ensure the use of clean and uncontaminated glassware and equipment in the analytical process.

4.5.3 Reagents, Standards, and Chemicals

The QA/QC Plan must include policies and procedures which describe how the laboratory avoids deterioration or damage to reagents and standards during receipt from the vendor, storage, handling, and preparation. This must include:

4.5.3.1 A policy establishing shelf life for all chemicals.

4.5.3.2 A policy establishing minimum quality grades for those chemicals and reagents which do not have quality grades defined by the referenced analytical method.

Example: The QA/QC Plan might note that all reagents are a minimum of ACS Reagent grade and that standards be made from neat or 99% purity stock materials.

4.5.3.3 A policy establishing documented traceability for standards, calibration materials, and quality control checks to national standards. If traceability is not applicable, the QA/QC Plan must set a policy or procedure which provides evidence of correlation of test results to other testing facilities.

Example: The QA/QC Plan should establish a policy that the vendor certifications for all calibration standards be maintained on file and that a standard preparation logbook traces the working standards. In the case where there are no national standards, comparability may be established through the use of external proficiency samples or participation in a quarterly or semi-annual round robin with other laboratories.

4.5.3.4 A policy to ensure the security of the chemicals.

4.5.3.5 A policy for the appropriate storage of chemicals which may degrade or decompose, and the segregation of incompatible chemicals.

Example: The QA/QC Plan may state that gas chromatography calibration standards must be stored apart from both samples and stock reagents.

4.5.4 Calibration Procedures and Data Generation

The QA/QC Plan must provide a description of the general procedures for calibration and data generation which, at a minimum, is compliant with the approved method. This should include:

4.5.4.1 The use of Standard Operating Procedures, including a policy or procedure for periodic review and approval.

4.5.4.2 The mode and frequency of initial and continuing calibrations. For laboratories which certify Arizona CBG or AZRBOB, the minimum requirements and acceptance criteria for initial and

continuing calibration listed in Tables 1 and 2 shall apply. Laboratories which only perform screening tests which are not used for the certification of Arizona CBG or AZRBOB must include procedures which are compliant with all of Table 2 and the final row of Table 1, titled "Screening Tests".

4.5.4.3 The preparation of and use of reagents and standards.

4.5.4.4 The frequency of analysis and the acceptance criteria for all quality control and blank materials. For laboratories which certify Arizona CBG or AZRBOB, the minimum requirements for quality control checks listed in Tables 1 and 2 shall apply. Laboratories which only perform screening tests which are not used for the certification of Arizona CBG or AZRBOB must include procedures which are compliant with all of Table 2 and the final row of Table 1, titled "Screening Tests". If the acceptance criteria are statistically developed from historical or inter-laboratory data, the procedure described in the QA/QC Plan must include the process for identifying and discarding statistical outliers.

4.5.4.5 The use of any third party reference materials or of additional calibration or quality control checks which the laboratory has implemented, with their associated acceptance criteria.

Example: As denoted by the use of the word "or" in Table 1, the laboratory has several choices in the use of calibration checks and the demonstration of accuracy and precision. Possible alternatives which a laboratory might choose to implement for the analysis of gas chromatography might be:

1. Analysis of a Secondary Source check sample only once after the initial calibration, followed by the analysis of a mid-level calibration standard as a CCV at the beginning of an analytical run, every tenth sample, and after the final sample could demonstrate calibration acceptability. Accuracy and precision could be demonstrated by the analysis of a sample duplicate and a spike every 20th sample or in every analytical batch, whichever is more frequent.
2. Analysis of a Secondary Source check sample as a CCV at the beginning, every tenth sample, and after the final sample could demonstrate the initial calibration acceptability, continuing calibration verification, and accuracy. Sample analysis in duplicate would demonstrate precision.
3. Analysis of a mid-level calibration standard as a CCV at the beginning, every tenth sample, and after the final sample could demonstrate calibration verification. Analysis of the Secondary Source once per analytical batch could demonstrate accuracy and precision if acceptance criteria were established for accuracy (e.g., the percent recovery of each

individual sample) and for precision (e.g., the relative percent difference between repeated analyses of the same sample).

4.6 Data Reduction and Review

The QA/QC Plan must provide a description of the general procedures for data reduction and in-house review which occur prior to reporting the test results to the Arizona Department of Weights and Measures.

4.6.1 The QA/QC Plan must include a description of the review process, identify the responsibilities of each person associated with the data, and identify who has authority to release the data at each step of its processing.

4.6.2 In-house review must include an appraisal of blanks and QC checks, precision and accuracy, control limits, and detection/reporting limits. The frequency of review must include a peer or supervisory review of a minimum of 10% of all analytical data generated by staff that have completed the training described in Section 4.3.3, a 100% review of data generated by staff in training, and a 100% review of all data which is manually entered into computers.

4.6.3 Computer software must be documented in an owner's manual or, if developed internally, in the code. Software formulas and data transfer must be validated. Policies and procedures must be established for maintenance, security of data, and proper functioning and calculations. A procedure should be established to prevent unauthorized access and changing of computer records.

Example: Commercial chromatography software quality is well established and the programming code is difficult to modify. The QA/QC Plan may require that only spot checking upon installation. But software that is developed in-house is easily corrupted or may be subject to changes by staff. This software, such as spreadsheets used for calculating sample concentration or mass balance, may need to be validated annually by a manual check of the calculations.

Table 1: General Requirements for Initial and Continuing Calibration

Instrument/ Method	Calibration Standards Used	Calibration Frequency	Computation Process and Acceptance Criteria *	Continuing Calibration and Acceptance Criteria	Other Criteria (Method Specific)	Accuracy	Precision
Gas Chro-matography, Gas Chro-matography/ Mass Spec-troscopy, Gas Chro-matography/ Fourier Transform Infrared Spectro-scopy	Per method or 3 points minimum	After major changes or as troubleshooting procedure for a QC check that is out-of-control.	Per method or Linear Regression with a Correlation Coefficient ≥ 0.995 or Response Factor with %RSD ≤ 10%	Run CCV: 1. Daily or before each day of use, and 2. After every 20 samples, and 3. At closing to bracket the final samples. Criteria = True Value ± 10% or statistically derived 95% or 99% confidence interval.	Run Secondary Source* once after initial calibration and before analyzing samples. Analyze surrogates as required. Acceptance criteria is defined by the source of the standard (e.g., NIST, API) or as described in Section 4.5.3.3	Analyze Secondary Source* daily or with every analytical batch, whichever is more frequent; or 1 Matrix Spike for every 20 samples or analytical batch, whichever is more frequent. Acceptance criteria for percent recovery is defined by statistically derived confidence interval.	Analyze Secondary Source* daily or with every analytical batch and compare to previous analysis; or Prepare and analyze sample duplicates at the frequency defined in the method. If the frequency is not defined, prepare and analyze 1 sample in duplicate for every 20 samples or analytical batch, whichever is more frequent. Acceptance criteria for reproducibility is defined by a statistically derived 95% or 99% confidence interval or by the "repeat-ability" criteria in the referenced ASTM method.

Table 1: General Requirements for Initial and Continuing Calibration							
Instrument/ Method	Calibration Standards Used	Calibration Frequency	Computation Process and Acceptance Criteria *	Continuing Calibration and Acceptance Criteria	Other Criteria (Method Specific)	Accuracy	Precision
Distillations (ASTM D86)	None	N/A	Per method	Run Toluene check standard every 6 months	Run Secondary Source* monthly Acceptance criteria is defined by the source of the standard (e.g., NIST, API) or as described in Section 4.5.3.3	N/A	Same as above
Reid Vapor Pressure (Setavap & ASTM D5191)	McLeod Gauge match to pressure transducer	Every 6 months or, after major changes, or as troubleshooting for a QC check that is out-of-control.	None	Run known value check sample daily.	None	N/A	Same as above

Table 1: General Requirements for Initial and Continuing Calibration							
Instrument/ Method	Calibration Standards Used	Calibration Frequency	Computation Process and Acceptance Criteria *	Continuing Calibration and Acceptance Criteria	Other Criteria (Method Specific)	Accuracy	Precision
Sulfur (ASTM D2622)	Per Method (5 point)	After major changes or as troubleshooting for a QC check that is out-of-control.	Linear Regression. Criteria is 1% Coefficient of Variation for counts per Note 4 in method.	Run CCV: 1. Daily or before each day of use, and 2. After every 20 samples, and 3. At closing to bracket the final samples. Criteria = True Value ± 10% or statistically derived 95% or 99% confidence interval.	Run Secondary Source* once after initial calibration and before analyzing samples. Acceptance criteria is defined by the source of the standard (e.g., NIST, API) or as described in Section 4.5.3.3	Analyze Secondary Source* daily or with every analytical batch, whichever is more frequent Acceptance criteria for percent recovery is defined by statistically derived confidence interval.	Same as above

Table 1: General Requirements for Initial and Continuing Calibration							
Instrument/ Method	Calibration Standards Used	Calibration Frequency	Computation Process and Acceptance Criteria *	Continuing Calibration and Acceptance Criteria	Other Criteria (Method Specific)	Accuracy	Precision
Sulfur (ASTM D5453 or D4045)	Per Method	After major changes or as troubleshooting for a QC check that is out-of-control.	Per method or Linear Regression with Correlation Coefficient ≥ 0.995	Run CCV: 1. Daily or before each day of use, and 2. After every 20 samples, and 3. At closing to bracket the final samples. Criteria = True Value ± 10% or statistically derived 95% or 99% confidence interval.	Run Secondary Source once after initial calibration and before analyzing samples. Acceptance criteria is defined by the source of the standard (e.g., NIST, API) or as described in Section 4.5.3.3	Analyze Secondary Source* daily or with every analytical batch, whichever is more frequent Acceptance criteria for percent recovery is defined by statistically derived confidence interval.	Same as above
Fluorescent Indicator Adsorption (ASTM D1319)	None	N/A	N/A	None	None	N/A	Same as above

Table 1: General Requirements for Initial and Continuing Calibration

Instrument/ Method	Calibration Standards Used	Calibration Frequency	Computation Process and Acceptance Criteria *	Continuing Calibration and Acceptance Criteria	Other Criteria (Method Specific)	Accuracy	Precision
Screening Tests (e.g., Near Infrared, Fourier Transform Infrared Spectro-scopy)	Per manufacturer's instructions	Per manufacturer's instructions	Per manufacturer's instructions	Per manufacturer's instructions	Positive hits/ compliance exceedences must be confirmed at a laboratory which certifies Arizona CBG or AZRBOB.	Per manufacturer's instructions	Per manufacturer's instructions

* Second source check sample must be a gasoline or the same matrix as the samples.

Table 2: Summary of Periodic Calibration and Quality Control Requirements.

Instrument	Calibration Check Frequency	Calibration Check Process	Acceptance Limits
Analytical Balances	First use in a 24 hour period Monthly Annually	Check with traceable Class S weight Check with full range of weights External service and calibration	True value ± 0.01% True value ± 0.01% ----
Top Loading Scales	First use in a 24 hour period Monthly Annually	Check with traceable Class S weight Check with full range of weights External service and calibration	True value ± 0.1 % True value ± 0.1 % ----
Thermometers	Annually	Calibrate in constant temperature bath against NIST traceable thermometer	True value ± 0.5 °C
Pipettors	Monthly	Gravimetric check	High volume (>100 μL): ≤ 1.0% relative error and RSD Low volume (<100 μL): ≤ 2.0 % relative error and RSD

4.7 Document and Data Control

Policies and procedures for establishing and maintaining control of the QA/QC Plan, administrative and analytical Standard Operating Procedures, and any other laboratory documents that the laboratory may choose must be described. This description shall include:

4.7.1 Procedures for the distribution of current versions of both controlled and uncontrolled documents.

4.7.2 Procedures for cataloguing and archiving obsolete versions.

4.7.3 Procedures for preparation, modification, review, and approval of documents by authorized persons prior to use.

4.7.4 The responsibility and a schedule for the review and revision of quality assurance program documentation must be defined.

4.7.5 Approval, distribution, and maintenance of the QA/QC Plan as a controlled document. Each page must include a page number, revision number, and date of release.

4.7.6 A policy for archival and retrieval of raw data, supporting documentation, and electronic media generated during sample analysis.

4.7.7 A description of document storage policies, including a description of any off-site storage facilities.

4.7.8 The laboratory's policy for, and length of, long-term retention of records. At a minimum, retention of records must comply with the Arizona CBG rules.

4.8 Data Assessment and Corrective Action Process

The QA/QC Plan must establish, implement, and document procedures for assessing data quality and for a corrective action system for process improvement.

4.8.1 This system must be able to identify responsibilities for the various laboratory position classifications (i.e., chemist, supervisor, QA Manager); document and identify defects or out-of-control events found on the bench, in data review, or statistically determined; trace defects to their root cause; correct them when possible; identify follow-up actions to

prevent recurrences; track closure for out-of-control events; and provide for periodic senior laboratory management review of trends.

4.8.2 The QA/QC Plan must define which defects and out-of-control problems are to be documented. The laboratory staff should not be generating documentation for minor problems, yet the needed significant corrective actions are to be identified. In essence, the QA/QC Plan should provide a definition which makes clear that defects which are systematic or which affect test results must be documented and resolved using the corrective action system. However, certain minor problems, such as a problem which is identified before the analysis of samples may, if desired, not be documented and tracked.

Examples: After an analytical batch is completed by gas chromatography, the internal standard recovery for 8 of 21 samples is calculated to be out of criteria. The remaining sixteen samples may be reported, but the eight samples may not be reported. Corrective action must be taken and documented which may consist of, but is not limited to, re-analysis, re-extraction, or sample clean-up. In addition, management will periodically review all corrective action documentation to evaluate if internal standard problems occur often and require corrective action. If so, further corrective action may take place in the form of replacement of stock standards, using a different storage location, or training of staff on spiking technique.

In a different situation, if an initial instrument calibration fails to meet the acceptance criteria and a new calibration is generated before any samples are analyzed, the sample results are not affected and the corrective action (recalibration) was successful. The run log should include notes about the initial failure and recalibration but further documentation may not be required.

4.8.3 Laboratory policy and practice for the internal development of control limits must be described. This must include a discussion of the systems for assessment of data quality on a real-time basis, as well as a discussion of systems for long term trend analysis of data. The use of Shewhart Control Charts is required by some ASTM methods, and their use is encouraged throughout the laboratory. Long term trend analysis should include:

4.8.3.1 Establishing and use of warning and control limits.

4.8.3.2 Monitoring for statistical trends.

4.8.3.3 Monitoring for statistical shifts.

Example: When analyzing for MTBE, the laboratory control limits for the second source quality control check sample are 90 to 110 percent recovery, and are tracked on a Shewhart Control Chart. Consecutive values from the previous six analytical runs were 98, 96, 95, 93, 92, and 92 percent. In reviewing the control chart before the sample report is released, the analyst noted the trend and further reviewed the data. Internal standard counts were constant and other surrogates and QC checks were acceptable. Because the laboratory set a policy in the QA/QC Plan that seven consecutive increasing or decreasing results constitute an out-of-control trend, the samples do not need to be re-analyzed. A proactive corrective action is needed, which may be the replacement of the standard stock solution. If the analysis of a seventh sample continues the trend, the associated samples would be out-of-control and would require re-analysis. (NFESC, 1996)

4.8.4 The QA/QC Plan must include a procedure for the resolution of QA/QC issues received from laboratory data users or other parties about the laboratory's activities which entails the insertion of these issues into the corrective action process.

4.8.5 Corrective actions must include the notification of any client whose test results may have been affected.

4.9 Quality Assurance Reviews and Reports

4.9.1 Quality Assurance Audits

The laboratory is expected to regularly and periodically arrange for quality assurance audits, consisting of internal surveillances and external reviews which are carried out by trained and qualified staff. For each type of audit (however named), the frequency, scope, and documentation of internal quality assurance audits conducted in the laboratory must be described.

4.9.1.1 Internal surveillance consists of periodic reviews of the laboratory operations by the Quality Manager or designee for compliance with the policies defined in the QA/QC Plan. The external review consists of a third party review or audit from a party that is independent of the activity being reviewed, and must be performed at a minimum frequency of once per year.

4.9.1.2 The process used to correct deficiencies identified during these audits must be explained, if not already described in the corrective action process.

4.9.1.3 In addition to periodic audits, the laboratory shall ensure the quality of results provided to clients by implementing internal process checks. These checks shall be reviewed by the laboratory's management and may include the following, as appropriate:

4.9.1.3.1 Internal quality control schemes using, whenever possible, statistical techniques.

4.9.1.3.2 Participation in proficiency testing other inter-laboratory comparisons.

4.9.1.3.3 Regular use of certified reference materials and/or in-house quality control samples using secondary reference materials.

4.9.1.3.4 Replicate testing using the same or different methods.

4.9.1.3.5 Re-testing of retained samples.

4.9.1.3.6 Correlation of results for different characteristics of an item.

4.9.2 Quality Assurance Reports to Management

A policy describing the scope, content, and frequency for routine and periodic internal QA reports from the Quality Manager to senior laboratory management must be described. The reports should be prepared annually or more frequently, and should include the status of quality assurance activities and the tracking of the status of corrective actions. Senior laboratory management shall review the QA/QC Program's effectiveness and continuing suitability, and introduce any necessary changes and improvements.

Example: One laboratory's QA report includes these items:

I. Audit Reports
   A. Internal Surveillances
   B. External Audits
   C. Subcontractor Audits

II. Certifications
  A. Pending
  B. Received
III. Performance Evaluation Samples
  A. In-House
  B. Pending
  C. Received (score)
IV. Holding Time Violations
  A. Total Holding Time Violations
  B. Category I - Out of Laboratory Control
  C. Category II - Laboratory Dependent
  D. Category III - Laboratory Reruns
V. Client Specific Quality Assurance Project Plans
  A. Received
  B. Reviewed
VI. Training
  A. In-House
  B. External
VII. Nonconformance Summary
VIII. QC Data/Control Chart Summary
IX. Standard Operating Procedures
  A. Issued
  B. In review
  C. In draft
  D. Needed, assigned
X. Customer Complaint Summary
  XI. Miscellaneous

4.9.3 External Proficiency Testing

The QA/QC Plan for laboratories that certify Arizona CBG or AZRBOB must describe the laboratory's implementation of external proficiency sample testing. This may consist of NIST samples, samples from certifying or regulatory agencies, a commercial proficiency sample service, round robin split samples with at least two other laboratories, or split samples with the Arizona Department of Weights and Measures. If the proficiency sample does not have acceptance criteria defined by the source, the QA/QC Plan must define the acceptance criteria. At a minimum, proficiency samples must be analyzed on a quarterly basis. Proficiency samples which are provided as part of a surveillance or audit may satisfy this requirement.

Example: If the acceptance criteria for the proficiency sample is not defined by the source (e.g., NIST, American Petroleum Institute, commercial vendor) or is part of a round robin study among several laboratories, the acceptance criteria may be established as the true (spiked) value plus or minus a percentage (e.g., 10%), as a statistical (95% or 99%) confidence interval based upon all participating laboratories, or as a statistical definition of an outlier.

4.10 Subcontracting

The QA/QC Plan must include a policy statement which describes the laboratory's assessment of the competency of subcontractors and the criteria for evaluating competency.

4.11 Maintenance

The laboratory policy for scheduling (as applicable), performing, and documenting maintenance for major instruments must be described. This policy must include the corrective maintenance and preventive maintenance which is performed internally and by vendors. At a minimum, preventive maintenance must be performed as described by the owner's manual for each piece of equipment. The responsibilities for performing the maintenance must be identified. The policy must include the acceptance criteria and, if necessary, the use of correction factors for each piece of support equipment, and must demonstrate the return to analytical control following the corrective action, major maintenance, or repairs.

Example: A thermometer's calibration is checked against a NIST traceable thermometer and the acceptance criteria is ±1 °C. The thermometer reads 1.5 °C high. The calibration check is documented in a log. However, the person using the thermometer each day is not likely to check the logbook every day for the correction factor. Therefore, it may be laboratory policy to mark each thermometer with tape which lists the date checked and the 1.5 °C subtraction factor.

4.12 Reports to the Arizona Department of Weights and Measures

4.12.1 The QA/QC Plan must establish a policy for the ad hoc reporting of significant changes in the laboratory, changes in methodologies, or changes in QA procedures as they arise to the Arizona Department of Weights and Measures. The QA/QC Plan must set the criteria and procedure for this reporting. Any major changes or deviations from approved published methods must be reported to ADWM within ten calendar days of the change or deviation. If anything is noted under Section 4.5.1.2, then Section 4.5.1.3 must also be addressed.

Example: The QA/QC Plan describes when a change is reported to the Arizona Department of Weights and Measures and when it is not. A change from an ASTM method to an in-house method should trigger a letter to the Department which informs them of the change. A minor procedural change in a method, such as using a different column in a gas chromatograph should not trigger notification.

4.12.2 An annual update must be sent to the Department of Weights and Measures on or before July 1 of each year, commencing after one year has elapsed since the QA/QC Plan was reviewed. This annual update must:

4.12.2.1 List major changes in the laboratory's operation or confirm that the basic information in the QA/QC Plan, including major equipment and testing methodologies, is substantially unchanged.

4.12.2.2 List changes, if any, in signatories and senior technical staff with review and policy making authority.

4.12.2.3 Provide a summary of external proficiency sample results.

4.12.2.4 Provide a summary of the previous years out-of-control situations and system improvements which were documented in the corrective action system.

4.13 References

The QA/QC Plan must include complete references for all citations and standard methods of analysis, including revision numbers, using standard bibliographical format.

4.14 Definitions and Abbreviations

The QA/QC Plan should include definitions and abbreviations as used in the laboratory.

4.15 Attachments

The QA/QC Plan must include a listing of all attachments and appendices referred to in the body of the text. These may include but are not limited to:

4.15.1 Minimum qualifications and/or staff job descriptions.
4.15.2 Organization charts.
4.15.3 Sample collection and retention time requirements.
4.15.4 Laboratory's major laboratory instrumentation.
4.15.5 Copies or examples of forms and logs in use.
4.15.6 Copies of Standard Operating Procedures needed to comply with the requirements of this QA/QC Plan Policy Document.
4.15.7 Flowcharts and diagrams.
4.15.8 Shewhart Control Charts.

5.0 References Used in the Preparation of this Document

5.1 Guidance On Preparation of Laboratory Quality Assurance Plans, USEPA Document No. 910/9-92-032, Seattle, WA, 1992.

5.2 Navy Installation Restoration Laboratory Quality Assurance Guide, Naval Facilities Engineering Service Center (NFESC), Port Hueneme, CA, February, 1996.

5.3 General Requirements For the Competence of Calibration and Testing Laboratories, ISO Guide 25, International Organization for Standardization, Switzerland, 1990.

5.4 Quality Assurance and Quality Control for Environmental Laboratories Design Guidelines, Electric Power Research Institute, CA, March, 1989.