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## 1. Introduction

It is critical to all laboratory operations that quality control protocols be established. It is the quality control protocols that ensure the results generated by any laboratory are valid and defensible. In the absence of good quality control protocols, results generated by any laboratory are questionable and possibly unreliable. Through the proper use of quality control techniques such as: Blanks, Duplicates, Analytical Standards, Reference Materials, Proficiency Testing Programs, and the establishing of Control Charts, a laboratory can control the quality of its data and ensure that the users of the results have the most reliable data possible.

The appropriate and necessary quality control techniques used vary by method. There are three categories of analysis that can be identified to help determine which of the quality control techniques are needed to ensure the validity of the final results: 1) methods of analysis that utilize analytical instruments that require calibration, 2) methods of analysis that utilize analytical instruments that do not require calibration, and 3) wet chemistry methods of analysis that do not utilize analytical instruments. While there are variants to these categories all tests can be generally placed into one of these three. These categories are outlined below along with the specific quality control techniques that are applicable to each.

Proficiency testing programs are applicable to all three categories and are especially helpful in monitoring the effectiveness of the quality control techniques applied in a laboratory. Proficiency testing allows the laboratory to test their methods and analysts using blind samples and then compare their results to that of peer laboratories. The value of regular participation in proficiency testing programs cannot be overstated – it is essential to managing a successful laboratory.

## 2. Definitions

### 2.1 Quality Control Samples

Special samples that are analyzed along with the regular samples for the purpose of validating the analysis and proving that the results generated on the regular samples are accurate to a defined degree.

#### 2.1.1 Blank

A Quality Control Sample that is known to have a zero or non-detectable level of the analyte of interest.

#### 2.1.2 Sample Duplicate

A sample that has been previously analyzed and is analyzed a second time.

#### 2.1.3 Analytical Standard

A Quality Control Sample with a documented known value of the analyte of interest. The purpose of Analytical Standards is to either calibrate or validate an analytical instrument or validate an analytical method. Analytical Standards must be traceable to high purity reagents and have a Certificate of Guarantee stating the analyte concentrations and respective uncertainties.

##### 2.1.3.1 Calibration Standard

An Analytical Standard that is used for calibration of an instrument.

##### 2.1.3.2 Validation Standard

An Analytical Standard that is used for validation of either a calibrated instrument or an analytical test method.

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**2.1.3.2.1 Reference Material**

A Validation Standard that is used for validation of an analytical test method.

**2.1.4 Bracketing**

Bracketing is a technique whereby an Analytical Standard is run at the beginning, every ten samples, and at the end of an Analysis Queue. If the results of the Analytical Standards analyzed before the test samples and at the end of the samples are both within the pre-established Control Limits then the results for all samples between the two are considered to be valid.

**2.2 Control Charts**

Graphical charts showing the results of Quality Control Samples with defined Control Limits.

**2.3 Control Limits**

The lower and upper limits of acceptance set for Quality Control Samples.

**2.4 Analysis Queue**

An Analysis Queue is defined as a batch of samples and related Quality Control Samples that are analyzed in sequence without interruption. If the Analysis Queue is interrupted by the analyst or by any other event (e.g. instrument failure, power failure, computer control failure, etc.), the Analysis Queue is considered stopped and a new Analysis Queue must be started. The only sample results that may be reported when an Analysis Queue is interrupted are those samples that are between Validation Standards that are within the Control Limits.

## 3. Quality Control for Analytical Instruments that Require Calibration

**Examples:** Gas Chromatography (GC), High Performance Liquid Chromatography (HPLC), Ion Chromatography (IC), pH meter, etc.

The following Analysis Queue must be followed for instrumentation that requires calibration.

**3.1 Calibration Standards**

A minimum of three Calibration Standards that cover the anticipated range of analyte concentrations must be utilized and the resulting calibration curve must achieve a correlation coefficient of 0.9900 or better for all analytes or the calibration process must be repeated (for pH Meter calibration a slope of >95% must be achieved).

**3.2 Blank**

The Blank must show non-detectable results for all analytes of interest or the calibration process must be repeated. Analysis may not continue until a Blank that shows non-detectable results for all analytes of interest is achieved.

**3.3 Validation Standard**

The Validation Standard must be from a different production lot than the Calibration Standards. The results for all analytes must fall within the predetermined Control Limits before proceeding with sample analysis. If any analytes in the Validation Standard fall outside of the Control Limits, any acquired data may not be reported and the instrument needs to be recalibrated and analysis restarted.

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**3.4 Samples (i.e. Unknowns)**

Unknown samples requiring analysis. A maximum of 10 samples may be run between Validation Standards.

**3.5 Sample Duplicate**

One Sample Duplicate must be included per Analysis Queue.

**3.6 Bracketing Validation Standard**

The Validation Standard must be from a different production lot than the Calibration Standards. The results for all analytes must fall within the predetermined Control Limits before any sample results are reported. It is required that the Bracketing Validation Standard be analyzed at a minimum of every 10 samples and at the end of each Analysis Queue. The only sample results that may be reported are those that fall between two Validation Standards that meet the Control Limits for all analytes of interest. If any analytes in the Bracketing Validation Standard fall outside of the Control Limits, any acquired data may not be reported and the instrument needs to be recalibrated and analysis restarted.

**Control Chart Requirement**

Results of all Validation Standards that are used to accept data must be entered into a database that can be used to generate Control Charts demonstrating compliance of all Validation Standards to be within the Control Limits.

Results of all Sample Duplicates that are analyzed in conjunction with reported sample results must be entered into a database that can be used to generate Control Charts demonstrating compliance of the difference between sample Duplicate sets to be within the Control Limits.

## 4. Quality Control for Analytical Instruments that do not Require Calibration

**Examples:** Karl Fischer, Density Meter, Acidity Titration (coulometric), etc.

The following Analysis Queue must be followed for instrumentation that does not require calibration.

**4.1 Validation Standard**

The results for all analytes must fall within the predetermined Control Limits before proceeding with sample analysis. If any analytes in the Validation Standard fall outside of the Control Limits, any acquired data may not be reported and the analysis must be restarted.

**4.2 Samples (i.e. Unknowns)**

Unknown samples requiring analysis. A maximum of 10 samples may be run between Validation Standards.

**4.3 Sample Duplicate**

One Sample Duplicate must be included per Analysis Queue.

**4.4 Bracketing Validation Standard**

The results for all analytes must fall within the predetermined Control Limits before any sample results are reported. It is required that the Bracketing Validation Standard be analyzed at a minimum of every 10 samples and at the end of each Analysis Queue. The only sample results that may be reported are those that fall between two Validation Standards that meet the Control Limits for all analytes of interest. If any analytes in the Bracketing Validation Standard

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fall outside of the Control Limits, any acquired data may not be reported and the analysis must be restarted.

**Control Chart Requirement**

Results of all Validation Standards that are used to accept data must be entered into a database that can be used to generate Control Charts demonstrating compliance of all Validation Standards to be within the Control Limits.

Results of all Sample Duplicates that are analyzed in conjunction with reported sample results must be entered into a database that can be used to generate Control Charts demonstrating compliance of the difference between sample Duplicate sets to be within the Control Limits.

## 5. Quality Control for Wet Chemistry Methods – i.e. Methods that do not use Analytical Instruments

**Examples:** Total Solids, Dextrose Equivalent, Brix, Acidity Titration (colorimetric), etc.

The following Analysis Queue must be followed for methods that do not use analytical instruments. For methods that do not use analytical instruments for analysis the Analysis Queue should be viewed as a group/batch of samples and standards analyzed either together or within a reasonable amount of time of one another. Under no circumstances can an Analysis Queue extend beyond the same working day to be considered a batch.

### 5.1 Validation Standard/Reference Material

If any analytes in the Validation Standard/Reference Material fall outside of the Control Limits, any acquired data may not be reported and the analysis must be restarted – i.e. the batch of samples reanalyzed.

### 5.2 Samples (i.e. Unknowns)

Unknown samples requiring analysis.

### 5.3 Sample Duplicate

One Sample Duplicate must be included per Analysis Queue.

**Control Chart Requirement**

Results of all Validation Standards/Reference Materials that are used to accept data must be entered into a database that can be used to generate Control Charts demonstrating compliance of all Validation Standards/Reference Materials to be within the Control Limits.

Results of all Sample Duplicates that are analyzed in conjunction with reported sample results must be entered into a database that can be used to generate Control Charts demonstrating compliance of the difference between sample Duplicate sets to be within the Control Limits.

# Laboratory Quality Control Protocol

| Analysis queue requirements | Instruments requiring calibration-GC, HPLC, IC, pH meter, etc. | Instruments that do NOT require calibration- KF, Density, Titration, some Brix, etc. | Wet Chemistry Methods with no instrumentation-Solids, DE, some Brix, some titrations, starch, etc. |
|---|---|---|---|
| **Calibration Standards** | Minimum of 3 calibration standards covering range of concentrations<br>$R^2 \geq 0.9900$<br>pH meter slope >95% | NA | NA |
| **Blank** | ND for all analytes | NA | NA |
| **Validation Standard**<br>**or**<br>**Reference Material**<br>Note: must be from a different production lot than the calibration standards | 1) After calibration standards before samples<br>2) Every 10 samples<br>3) End of queue | 1) Beginning of queue<br>2) Every 10 samples<br>3) End of queue | Reference Material can be utilized in place of a validation standard |
| **Samples** | Unknowns; max of 10 samples between validation standards | Unknowns; max of 10 samples between validation standards | Unknowns |
| **Sample Duplicate** | One unknown sample analyzed in duplicate | One unknown sample analyzed in duplicate | One unknown sample analyzed in duplicate |
| **Bracketing Validation Standard**<br>Note: must be from a different production lot than the calibration standards | End of queue | End of queue | NA |

**Validation and Reference Material acceptable criteria:**

- The results for all analytes must fall within the predetermined Control Limits before proceeding with the sample analysis. If any analytes fall outside of the Control Limits, any data acquired may not be reported and the queue must be restarted.

**Bracketing Validation Standard acceptable criteria:**

- The results for all analytes must fall within the predetermined Control Limits before any sample results are reported.
- Required to be analyzed at a minimum of every 10 samples and at the end of each queue.
- The only sample results that may be reported are those that fall between two Validation Standards that meet the Control Limits for all analytes of interest. If any analytes fall outside of the Control Limits, any acquired data may not be reported and analysis queue restarted. If applicable; the instrument needs to be recalibrated as well.

**Control chart requirements:**

- Results of all Validation Standards that are used to accept data must be entered into a database that can be used to generate Control Charts demonstrating compliance of all Validation Standards to be within the determined Control Limits.
- Results of all Sample Duplicates that are analyzed in conjunction with reported sample results must be entered into a database that can be used to generate Control Charts demonstrating compliance of the difference between Sample Duplicate sets to be within the Control Limits.

*See the enclosed for the full Laboratory Quality Control (LQC) Protocol*