AIHA®

Laboratory Quality Assurance Manual

Fifth Edition

Provides the major elements of a reliable laboratory quality assurance program.

Edited by Andrew L. Teague, CIH and Mary E. Eide

AIHA®
Protecting Worker Health

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Laboratory
Quality Manual

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Edited by Andrew L. Teague, CIH, and Mary E. Eide

Editorial Board: Eric L. Botnick, Allen Iske, Mary E. Eide, Scott M. VanEtten, Ram S. Suga, and Andrew L. Teague

Prepared by the American Industrial Hygiene Association
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Preface

To assess chemical and physical agents that affect the environment or employees' health and sense of well-being, industrial hygiene, occupational health and safety, and environmental professionals rely on accurate and precise measurements. Laboratories of all kinds must be sure they produce results of known Quality System (QS), and the key to ensuring quality results lies in a sound QS program. An effective quality program provides maximum benefits when it covers all aspects of monitoring, from preparation for sampling to report generation and record keeping.

This manual describes the major elements of a reliable QS program. The manual was compiled to aid new laboratories in establishing quality programs and to help established laboratories enhance existing programs. It is not to be used as a "turn-key" document, but as a reference manual, the parts of which can be adapted to the individual laboratory. By incorporating the basic principles outlined in this manual, laboratories can develop comprehensive programs that will assure quality results. These results can be used to show the adequacy of worker health protection programs or compliance with environmental regulations.

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Chapter 1
Introduction

by Andrew A. Teague, CIH

1.1 Introduction

In this manual, key elements in the development and evaluation of a Laboratory Quality System (QS) or laboratory quality assurance (QA) program are presented. The updates found in this edition make the manual consistent with respect to "ISO/IEC 17025:2005 – General Requirements for the Competence of Testing and Calibration Laboratories"[1] and the AIHA Laboratory Quality Program policies.[2]

1.2 Purpose

The purpose of any QS program is to ensure the integrity and validity of reported data. Additionally, court defensibility should be considered the end goal of a QS program that deals with regulatory and compliance samples. The program must encompass all facets of the QS process, from sampling strategy to the final review of reports and aspects in between, including sample preparation and analysis. The chain of custody of the samples and the accompanying quality control (QC) procedures must be documented, and that documentation must be traceable. All aspects of sample handling, data handling, calculation, and QC review and reporting must be formally documented and auditable. An error in these areas makes the reported data highly suspect.

NOTE: Descriptions of the various American Industrial Hygiene Association (AIHA®) Laboratory Accreditation Programs are available from AIHA®. Visit www.aiha.org or call (703) 849-8888 for more information. The references in the bibliography at the end of this chapter provide a more comprehensive treatment of QS programs.

1.3 Scope

The QS policies of a laboratory must be defined clearly in a formal written plan identified as the Quality Manual (QM). The QM establishes the top-level framework for how the laboratory operates, and how it conforms to the requirements established by its certifying or accrediting authorities. QM content should not be as specific as Standard Operating Procedures (SOPs) or work practices, but rather should set the policies by which these more specific instructions are developed. In this fashion all the laboratory's procedures and practices are linked back to the QM. Thus the QM establishes how the laboratory will meet requirements imposed upon the laboratory, and internal activities are evaluated for compliance with QM policies.

The QM should be endorsed by the highest management level of the organization, reviewed at least annually, and updated as needed. Nonetheless, QM content that requires frequent change may best be included in a lower-level document such as an SOP.

The written plan must be available to all personnel. It is essential that the plan is understood by all laboratory personnel and that the policies are followed as written. The References and Further Reading sections at the end of this chapter can provide more specific guidance on establishing QA policies.

1.4 Definitions

1.4.1 Quality System

Quality system is an overall process to provide confidence that the QC program is being applied effectively. The process includes internal audits designed to evaluate all known policies and procedures that affect the quality of the analytical results.

1.4.2 Quality Control

Quality control consists of operational procedures used to ensure that the analytical data are of known acceptable precision and accuracy.

1.5 Elements of a Quality System Program

The following aspects must be incorporated into a QS program:

1.5.1 Description of QS objectives and policies
1.5.2 Quality Manual (QM) maintenance and updated procedures
1.5.3 Organizational chart and personnel responsibilities
1.5.4 Personnel qualifications, training, and training records
1.5.5 Sampling materials and procedures
1.5.6 Chain of custody for samples
1.5.7 Sample processing procedures
1.5.8 Specifications for reagents and standards
1.5.9 Preparation and storage of reagents, standards, and samples
1.5.10 Vendor approval criteria and list(s) of approved vendors
1.5.11 Analytical methodology
1.5.12 Method validation
1.5.13 Data reduction, validation, and reporting including statistics
1.5.14 Metrology (the science of measurements)
1.5.15 Documentation, record keeping, and record retention
1.5.16 Equipment calibration and maintenance procedures
1.5.17 Internal QS procedures
1.5.18 External QS procedures
1.5.19 QS audit procedure, including quarterly reports
1.5.20 Corrective action plan
1.5.21 Preventative Action Plan
1.5.22 Sample retention and disposal
1.5.23 Uncertainty Measurement and Traceability Policies
1.5.24 References

1.6 List of Abbreviations

AA	Atomic Absorption Spectrometer/Spectrometry
AAC	AIHA Asbestos Analysis Committee
AAR	Asbestos Analysts Registry
AAT	AIHA Asbestos Analytical Testing Program
ABIH	American Board of Industrial Hygiene
ACGIH®	American Conference of Governmental Industrial Hygienists
AIHA®	American Industrial Hygiene Association
ASV	Anodic Stripping Voltammetry
CALA	Canadian Association for Laboratory Accreditation Inc.
CCV	Continuing Calibration Verification
CRM	Certified Reference Material
ECD	Electron Capture Detector for Gas Chromatography

ELAP	New York DOH Environmental Laboratory Accreditation Program
ELLAP	AIHA Environmental Lead Laboratory Accreditation Program
ELPAT	Environmental Lead Proficiency Analytical Testing Program
Env.	Environmental
FAA	Flame Atomic Absorption Spectrometer/Spectrometry
FID	Flame Ionization Detector for Gas Chromatography
FPD	Flame Photometric Detector for Gas Chromatography
GC	Gas Chromatograph/Chromatography
GFAA	Graphite Furnace Atomic Absorption Spectrometer/Spectrometry
HEPA	High-Efficiency Particulate Air (filter)
HSE/NPL	Health and Safety Executive/National Physical Laboratory
HPLC	High Performance (Pressure) Liquid Chromatograph/Chromatography
IAQ	Indoor Air Quality
IC	Ion Chromatograph/Chromatography
ICP	Inductively Coupled Plasma Atomic Emission Spectrometer/Spectrometry
ICP/MS	Inductively Coupled Plasma/Mass Spectrometer
ICV	Initial Calibration Verification
ID	Identification (or Identification Number)
IDL	Instrument Detection Limit
IH	Industrial Hygiene or Industrial Hygienist
IHLAP	Industrial Hygiene Laboratory Accreditation Program
IHPAT	Industrial Hygiene Proficiency Analytical Testing Program
ILAC	International Laboratory Accreditation Cooperation
IR	Infrared Spectrophotometer/Spectrophotometry
LCL	Lower Control Limit
LCS	Laboratory Control Sample
LOD	Limit of Detection
LOQ	Limit of Quantitation, which is similar to RL
LWL	Lower Warning Limit
MDL	Method Detection Limit
MS	Mass Spectrometer/Spectrometry
MSD	Mass Selective Detector for Gas Chromatography
N582	NIOSH 582 (or equivalent) Asbestos Analysis Training Course
NACLA	National Council for Laboratory Accreditation
ND	Non-Detectable or Not Detected
NELAC	U.S. EPA National Environmental Laboratory Accreditation Committee
NELAP	U.S. EPA National Environmental Laboratory Accreditation Program
NIOSH	National Institute for Occupational Safety and Health
NLLAP	U.S. EPA National Lead Laboratory Accreditation Program
NPD	Nitrogen Phosphorus Detector for Gas Chromatography
NVLAP	National Voluntary Laboratory Accreditation Program
OSHA	Occupational Safety and Health Administration
PAT	Proficiency Analytical Testing
PCM	Phase Contrast Microscope/Microscopy
PLM	Polarized Light Microscope/Microscopy
PQL	Practical Quantitation Limit
PSV	Potentiometric Stripping Voltammetry
QA	Quality Assurance
QC	Quality Control
QM	Quality Manual
QS	Quality System
QSC	Quality System Coordinator
rad	radioactive
RI	Refractive Index
RL	Reporting Limit, which is always equal to or greater than the MDL
RPD	Relative Percent Difference
SEM	Scanning Electron Microscope/Microscopy

SOP	Standard Operating Procedure
SRM	Standard Reference Material
SW-846	U.S. EPA Solid Waste Document 846–Compendium of Solid Waste Methods
TCLP	Toxicity Characteristic Leaching Procedure (SW-846 1311)
TCD	Thermal Conductivity Detector for Gas Chromatography
TEM	Transmission Electron Microscope/Microscopy
UCL	Upper Control Limit
UPS	Uninterruptable Power Supply
UV/Vis	Ultraviolet/Visible Spectrophotometer/Spectrophotometry
UWL	Upper Warning Limit
XRD	X-ray Diffractometer/Diffractometry
XRF	X-ray Fluorescence Spectrometer/Spectrometry

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1.8 Further Reading

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Chapter 2
Personnel and Training

by Pamela Kostle, CIH

2.1 Introduction

Qualified is defined by Webster as "having the necessary skill, experience, or knowledge to do a particular job or activity."[1] Qualified and properly trained personnel are fundamental to satisfactory laboratory performance. The EPA Manual for Laboratory Certification specifies sufficient personnel at appropriate classification that have the education, training and experience to conduct their responsibilities.[2] A laboratory must establish adequate qualifications for personnel as well as initial and ongoing training programs to develop and maintain abilities to carry out the methods and procedures used by the laboratory. Also, personnel should be knowledgeable of the sampling and analysis process in order to facilitate general communication with field personnel and clients. Effective coordination is essential to provide laboratory clients with satisfactory analytical services.

2.2 Personnel Qualifications

The minimum qualifications for laboratory personnel should be those currently set forth in AIHA Laboratory Accreditation Programs, LLC, or by other relevant accreditation/ qualification programs. AIHA Laboratory Accreditation Programs, LLC, a signatory of the ILAC-MRA has identified minimum criteria for key personnel of Technical Manager, Quality Manager and Analyst.[3] The criteria includes education, experience, technical background and supervision as general requirements and program specific criteria. Some laboratories may want to establish other qualifications, such as additional experience in specific areas or on specific topics, or require that some personnel have advanced scientific degrees, certification in certain areas of expertise such as asbestos counting, and/ or additional chemistry education. The Bureau of Labor statistics lists the following qualities/skills for an Environmental Scientist or specialist: analytical skills, communication skills, interpersonal skills, problem-solving skills, and self-discipline.[4] Qualifications and position descriptions document the minimum responsibilities for the person fulfilling the position. Suggestions on preparing these descriptions are described in United Nations Industrial Development Organizations Complying with ISO 17025.[5]

2.3 Personnel Training

2.3.1 General Considerations

A Quality System (QS) program must include and provide for the ongoing training of laboratory personnel. The training program must be adaptable, written and include demonstrations of proficiency. A record of qualifications and training must be kept for each person.

It is the responsibility of management to establish and implement a training program to ensure that methods and procedures are carried out properly. The program should include, at minimum, sampling techniques, analytical methods, specificity, accuracy and precision monitoring, limits of detection, potential interferences, uncertainty measurement and regulatory/ authoritative limits.

2.3.2 Specific Training

It is the responsibility of laboratory management (however titled) to ensure that all laboratory personnel are trained properly in laboratory techniques, methods of analysis, and QS

procedures. The laboratory should have a written training program or checklist to ensure that all laboratory personnel receive adequate training in laboratory policies, procedures, methods, instrumentation, calculations, reporting, quality control, and safety, with special emphasis on the safe handling of toxic and carcinogenic materials. Training of new personnel should be conducted under the guidance of qualified, experienced analysts. This training should be supplemented by attendance at some of the many seminars and courses that are available, such as those presented by instrument manufacturers. Also, there are courses in fundamentals of industrial hygiene, industrial hygiene chemistry, laboratory accreditation, quality control, asbestos analysis, and lead sampling and analysis, etc., presented by various college and university educational resource centers in addition to the National Institute for Occupational Safety and Health (NIOSH),[6] the U.S. Environmental Protection Agency (EPA),[7] and AIHA.[8] There should be opportunities for personnel to keep current in their fields of interest by attendance at appropriate technical meetings such as the annual American Industrial Hygiene Conference & Exposition (AIHCe).

An analyst must be required to demonstrate proficiency in the performance of new methods or techniques as the last step in the training process prior to conducting analysis of actual field samples. This can be accomplished by the satisfactory analysis of known samples such as certified reference materials (CRM), extra sets of industrial hygiene proficiency analytical testing (IHPAT) program samples from AIHA PAT, extra sets of environmental lead proficiency analytical testing (ELPAT) samples from AIHA PAT, and blind samples prepared by someone else in the laboratory. The use of standard reference materials (SRM) for such purposes is discouraged as it depletes the supply of the materials and other sources of samples are readily available. The laboratory's routine QS program should serve as a continuing check on the analyst's performance.

Records must be kept to document the qualifications and training of all laboratory personnel. The minimum information in these records should include formal education, prior experience, training received on the job, etc. These records should include a description of the course and outline, technique or procedure, and the dates and the person or organization providing the training. The training record shall include the qualifications of the course instructor. A designated management representative should sign the record, indicating satisfactory completion of each phase of training and authorization to operate specific equipment and perform specific analytical methods. Laboratory management shall ensure that the accreditation/recognition programs record keeping requirements specific to the laboratory are met.

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Chapter 3
Uncertainty of Measurement, Error and Sources of Error

by Evan Floyd, PhD

3.1 Introduction

An essential task in a Quality System (QS) program is the detection of error and its elimination or control. This chapter discusses error in sampling and analysis. Error and its various types are defined and ways to detect and control error are presented briefly. Also, specific sources of error are given for consideration during sampling and analysis.

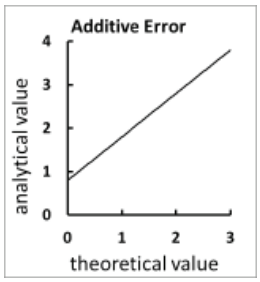
As the first step in the detection of error, the analyst must be familiar with the definitions of the types of error that might be encountered and the relationship of error to accuracy and precision. An understanding of these definitions increases awareness of the potential for error and allows sources and types of error to be detected and classified. Once the sources and types of error are identified, the elimination or control of error should follow.

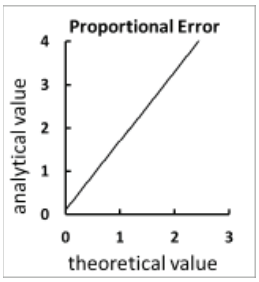
3.2 Definitions

Error cannot be defined without defining accuracy. Therefore, accuracy is defined as the degree of agreement between the measured value and the true value. Error is defined as the degree of disagreement of a measured value with an accepted reference value (i.e., the "true" value). Error has both systematic and random components.[1]

Systematic error is synonymous with determinate error, assignable error, or "bias" and is a consistent deviation in the results of measurements from an accepted reference value. The cause of bias may or may not be known but is considered to be assignable. Systematic errors can be classified as "additive" or "proportional." The error is additive if it is a constant value regardless of the amount or concentration of the analyte (see Figure 3.1.a). A plot of observed values as a function of theoretical values would be linear. It would have a slope of unity and have a non-zero intercept equivalent to the additive systematic error value.

A systematic error is said to be proportional if the magnitude of the error is proportional to the concentration of the analyte. If there is proportional systematic error, then a plot of observed values as a function of theoretical values would demonstrate a slope different from unity (see Figure 3.1.b). Systematic error may be a more complex function of concentration and could result in a curvilinear slope for such a plot (see Figure 3.1c).

a.) 

b.) 

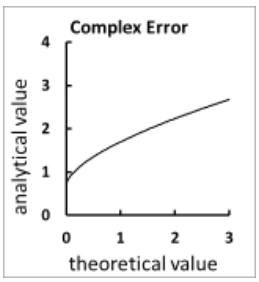
c.) 

Figure 3.1. — Plots that Demonstrates Types of Systematic Error a.) Additive b.) Proportional c.) Complex error.

Although specific sources of determinate (systematic) error are listed for various aspects of sampling and analysis in a later portion of this chapter, the following are illustrative examples[2]:

- Poor recovery of the analyte from the sample media
- Incorrect preparation of standards
- Improper instrument calibration
- Contaminated reagents
- Human visual acuity deficiencies
- Human carelessness or personal bias
- Improper selection of sampling or analytical method
- Improper shipment and storage of samples
- Interferences from other elements or compounds
- Saturation or breakthrough of the sample media

Random error is synonymous with indeterminate error or non-assignable error(3) and often is defined in terms of the precision of measurements. Random error is characterized by irregular variation in repeated observations or measurements. Because the variations are random in nature, they are unpredictable individually. The cause of this type of error generally cannot be assigned. Random error statistics usually are represented by a normal distribution – a distribution having a frequency vs. size curve that is bell-shaped and symmetrical about the arithmetic mean size. However, measurements of small entities with possibly large errors in the upward but not downward direction (zero-limited, non-negative measures) give a skewed distribution curve that is often represented by a lognormal distribution (i.e., the logarithms of the measurements are normally distributed). Random error can be estimated statistically from the precision of replicate measurements.

Potential Sources of random error are typically identified as sources of variation inherent to the specific steps of an established sampling and analytical method. Examples include variations in physical measurements of volume and mass, variations in instrument response, electrical line voltage transients, and sample heterogeneity.

For a quantity or concentration that is derived from the measurement of several variables, an estimate of the overall uncertainty in the derived quantity caused by random error can be made by combining the contribution of each variable by the law of the propagation of errors. Implicit in the law is the assumption that each contributing variable is independent. For linear relationships, the overall variance is the weighted average of the individual variances with degrees of freedom used as weighing factors. For products and ratios, the squares of the relative errors (i.e., the squares of the coefficients of variation) are additive. The overall variance is the square root of the sum of the squares of the coefficients of variation for each measured variable. A more thorough presentation of this mathematical approach is given by Mandel and Nanni.[1]

An example of the use of the law of the propagation of errors is provided by NIOSH in the validation of personal air sampling and analytical methods.[3] The concentration of an air contaminant is the ratio of a measured contaminant mass to a measured air volume. Consequently, NIOSH defines the overall coefficient of variation as the square root of the sum of the squares of the coefficients of variation for sampling (CVs), analysis and desorption efficiency (CVA+DE), and flow (CVF).

3.3 Detection and Elimination or Control of Errors

The QS measures, discussed in other chapters of this manual, are useful in the detection and elimination or control of errors. Quality control charts provide a means for identifying and separating systematic and random errors. The random variations produce an irregular pattern on the chart. Trends, shifts, or other extremes can be attributed to systematic and, therefore assignable error. Other techniques used to detect systematic error include the analysis of spiked samples and the use of an independent analytical method for comparison.

Systematic errors can be eliminated or minimized by a variety of techniques. Corrective actions may be classified according to whether they are applicable before, during, or after using the method. The analysis of blind, field-, and laboratory-spiked samples, the use of internal standards, the use of continuing calibration standards, and the elimination or reduction of interferences, are activities that will help identify and reduce systematic errors.

Random errors can never be eliminated, but they can be reduced by strictly adhering to established methods. The distribution and range of random error can be determined by the analysis of replicate samples and also by collaborative studies among laboratories proficient in a specific method.

3.4 Sources and Types of Error

Specific examples of sources of error are presented in this section for both sampling and analysis. These include sampling, sample shipment and storage, sample preparation, standard and spike preparation, sample analysis, data interpretation, and report generation. Table 3.1 lists some of the more common errors encountered. However, the list should not be considered to be all-inclusive.

Table 3.1 — Sources of Potential Error

Type	*Element Sources*
Variation in pump flow rate	Improper pump calibration: - Inadequate frequency - Incorrect flow rate calculation - Calibrator not corrected for temperature and/or pressure
Sampling	Improper media selection Improper media preparation Selection of improper flow rate Improper sample volume (high or low) Improper sample mass (high or low) Improper area sampled (high or low) Use of contaminated sampling equipment Failure to submit field blanks with samples Insufficient interference information Improper sample handling Inadequate or improper sample labeling
Shipping/Storage	Improper sample preservation or protection in shipment Temperature limitations exceeded in storage or shipment (high or low) Containers which have not been checked for interferences Bulk samples shipped with samples to be analyzed Cross contamination during shipment or storage Excessive storage interval Excessive shipment interval Failure to ship spikes/ blanks with the samples Damage to sample in shipping and storage
Standards Preparation/ Spike Preparation	Unknown purity of solvents and/ or reagents Improper calibration of volumetric equipment Improper volumetric handling techniques Improper cleaning and storage of glassware Incorrect calculation of mass levels applied Use of a non-traceable or contaminated standard Uncalibrated balance Use of non-certified volumetric glassware

Table 3.1 — Sources of Potential Error (continued)

Type	*Element Sources*
Sample Preparation	Non-quantitative sample transfer Improper dilution technique Variability in size and processing techniques for subsampling of bulks Cross contamination in handling sample Contaminated glassware Inadequate QC samples Unknown solvent purity
Analytical Personnel	Experience, training, and techniques of analysts Subjective interpretations in some analyses (e.g. microscopic analysis of fungal organisms or asbestos)
Analytical Instruments	Improper instrument parameter used Non-regulated power sources Nonlinear working range Improper instrument calibration Instrument drift Matrix effects [**NOTE:** See vendor's troubleshooting guide for typical instrumental problems or contact vendor's application specialist for unusual mechanical problems.]
Methodology	Incorrect recoveries of QC samples Excessively high blanks Deviations from documented procedures Improper calculations of mass concentration Inadequate extraction efficiency data over the range of analysis
Data Interpretation	Improper evaluation of reported results Discounting large breakthrough results on solid sorbent samples Erroneous calculation Failure to consider additive effects Ignoring unacceptable QC results
Report Generation	Illegible handwriting Transposition of data and/or results (Decimal point errors are the most common) Data entry errors Typographical errors Erasures or white-out on final reports Inadequate auditing procedures Lack of pertinent analytical comments concerning problems encountered during analysis Failure to follow chain-of-custody procedures Improper data and report storage

3.5 Significant Figures and Rounding

Significant figures indicate the precision of the value. The last reported significant figure can be expected to vary by ±1 due to random error. For example, if the precision of an analysis is 5%, one should not report more than three significant figures and two would be more appropriate.

Rounding of values should be done with two important rules in mind. First and most important, do not round until the last math operation is complete or rounding error could be introduced. Second, be consistent in how numbers are rounded. Consult NIST for guidance on rounding numbers.[4]

3.6 Estimation of Uncertainty

AIHA Laboratory Accreditation Programs, LLC has information on Uncertainty of Measurement and Traceability of Measurement on their website. There are policies, guidance documents, and workbook examples posted on the AIHA-LAP, LLC website under "Policy Modules".[5] These documents incorporate ISO/IEC 17025 "General requirements for the competence of testing and calibration laboratories".[6] The requirement to have procedures describing how uncertainty will be calculated and reported can be addressed with a single procedure, within each analytical method, or in any way a laboratory chooses as long as all requirements, including identifying contributors to uncertainty for each method or type of method are met.

3.6.1 Requirements for the Estimation of Uncertainty

Laboratories accredited under the AIHA-LAP, LLC Accreditation Program shall fulfil the following requirements with respect to the estimation of uncertainty of measurement for tests associated with their scope of accreditation:

3.6.1.1 Laboratories shall be able to demonstrate their ability to estimate measurement uncertainty for all accredited quantitative test methods. In those cases where a rigorous estimation is not possible, the laboratory must make a reasonable attempt to estimate the uncertainty of test results. All approaches that provide a reasonable and valid estimation of uncertainty are equally acceptable.

3.6.1.2 Laboratories shall make independent estimations of uncertainty for tests performed on samples with significantly different matrices. For example, estimations made for filter samples cannot be applied to bulk samples.

3.6.1.3 Estimations of measurement uncertainty are not needed where the reported test results are qualitative. Laboratories are, however, expected to have an understanding of the contributors to variability of test results. Examples of such tests are those that report only organism identifications or presence/absence.

3.6.1.4 Laboratories shall have a written procedure describing the process used to estimate measurement uncertainty, including at a minimum:

3.6.1.4.1 Definition of the measurand — the quantity intended to be measured. The specification of a measurand requires knowledge of the kind of quantity, description of the state of the phenomenon, body, or substance carrying the quantity, including any relevant component, and the chemical entities involved. In chemistry, "analyte", or the name of a substance or compound, are terms sometimes used for 'measurand'. This usage is erroneous because "analyte" refers to type but not quantity.

3.6.1.4.2 Identification of the contributors to uncertainly. These can include sampling or sub-sampling, transportation and sample handing including storage, preparation of samples, environmental measurement conditions, personnel carrying out tests, variations in the test procedure, measurement instruments, calibration standards or reference material, methods of generating test results, and corrections for systematic errors.

3.6.1.4.3 Details of the approaches used for estimating measurement uncertainty, such as Type A and/or Type B. Type A is the evaluation of a component of measurement uncertainty by the statistical analysis of measured quantity values obtained under defined measurement conditions. Type B is the evaluation of a component of measurement uncertainty determined by means other than a Type A evaluation.

Type A approach can include uncertainty specified within a standard or validated method, laboratory control samples or matrix spikes, duplicate data, and Proficiency Testing (PT) sample data. When using the Type A approach, laboratories shall utilize one or more of the following options. These options are generally considered from 1) most suitable, to 4) least suitable:

1) Uncertainty specified within a standard method. In those cases where a well-recognized test method (such as a peer-reviewed AOAC, NIOSH, OSHA, ASTM, etc. method), specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, laboratories need not do anything more than follow the reporting instructions as long as they can demonstrate they follow the reference method without modification and can meet the specified reliability.
2) Laboratory Control Samples (LCS) and Matrix Spikes. In cases where matrix specific LCS (CRM or media spikes) and/or matrix spike data are available, include uncertainty estimated from the standard deviation of long term data collected from routine sample runs for existing test methods or from the standard deviation of the LCS or matrix spike data for method validation/verification studies for new test methods.
3) Duplicate Data. In cases where sub-sampling occurs and there are data over the reporting limit, include uncertainty estimated from long term duplicate data collected from routine sample runs for existing test methods or method validation/verification studies for new test methods.
4) Proficiency Testing (PT) Sample Data. In cases where the previous options are not available and where PT samples are analyzed with sufficient data above the reporting limit, pooled PT sample data can be used to estimate uncertainty.

3.6.1.4.4 Identification of the contributors of variability for qualitative test methods.

3.6.1.4.5 All calculations used to estimate measurement uncertainty and bias.

3.6.1.4.6 The reporting procedure.

3.6.1.5 Laboratories are required to re-estimate measurement uncertainty when changes to their operations are made that may affect sources of uncertainty.

3.6.1.6 Laboratories shall report the expanded measurement uncertainty, along with the reported analyte concentration, in the same units as analyte concentration, when it is relevant to the validity or application of the test results, or a customer's instructions so requires, or the uncertainty affects compliance to a specification limit.

3.6.1.7 When reporting measurement uncertainty, the test report shall include the coverage factor and confidence level used in the estimations (typically k = approximately 2 at the 95% confidence level).

3.6.1.8 When the test method has a known and uncorrected systematic bias, it shall be reported separately from the test result and uncertainty estimation, as a probable bias value.

3.6.2 Summary of Guidance Documents Steps to determine uncertainty:

3.6.2.1 Review and identify the contributors.

3.6.2.2 Determine if contributors are accounted for with existing QC data.

3.6.2.3 Compile the applicable QC data and any other contributors and perform calculation of combined uncertainty.

3.6.2.4 Calculate combined uncertainty (SDc).
It may be beneficial to use RSD instead of SD as it ameliorates the magnitude dependence concentration based SD. Sources that have an SD of less than 1/3 of the largest SD can be eliminated.

$$SD_{combined} = \sqrt{(SD_1^2 + SD_2^2 + \cdots + SD_n^2)}$$

3.6.2.5 Calculate the expanded uncertainty.
Apply the appropriate coverage factor 'k'. Calculate the expanded uncertainty by multiplying the combined standard uncertainty by the appropriate coverage factor (k) to give an expanded uncertainty with the desired confidence level. The factor k is the confidence interval Student distribution t-factor for n-1 degrees of freedom. For a confidence level of 95%, k is approximately 2 for a data set of 30 points or more, for normally distributed data sets. Expanded measurement uncertainty = k x SDc.

3.6.2.6 Reporting test results with the expanded measurement uncertainty, for example total benzene concentration of 88 ug/sample + 11 ug/sample at the 95% confidence level (k=2).

3.6.2.7 Where bias is present, report it along with the uncertainty as a probable bias in a manner such as the following example: total lead concentration of 78 ug/sample ± 12 ug/filter at the 95% confidence level (k=2). This method has an average recovery of 94%, or at this level, a probable bias of -5 ug/filter.

3.6.2.8 Alternate forms of reporting uncertainty and bias are acceptable as long as required information is clearly presented.

3.6.2.9 During assessment and surveillance of a laboratory, the assessor will evaluate the capability of the laboratory to estimate the measurement uncertainty for test methods included in the laboratory's scope of accreditation. The assessor will verify that the methods of estimation applied are valid, all significant contributors to uncertainty have been considered, and all the criteria of the AIHA-LAP, LLC policy are met.

3.6.2.10 Refer to the AIHA-LAP, LLC *Guidance on the Estimation of Uncertainty of Measurement* for suggestions and examples for implementing the policies and helpful references.[5] Example Excel spreadsheets are also on the website.

3.7 References

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3.8 Further Reading

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Chapter 4
Traceability of Measurement

by Periyasamy Subramanain, PhD, CIH

4.1 Introduction

AIHA® Laboratory Accreditation Programs, LLC has information on Uncertainty of Measurement and Traceability of Measurement on their website.[1] These Policies and Guidance Documents follow ISO/IEC 17025 "General requirements for the competence of testing and calibration laboratories"[2], ILAC-P10 Policy on Traceability of Measurement Results[3], ILAC-G24 Guidelines for the determination of calibration intervals of measuring instruments[4], and CALA A61 CALA Traceability Policy.[5]

Traceability is one of the critical pieces of information necessary to produce a valid test result. The traceability chain of analytical standards and related equipment requires that each step of the analysis must have the information on calibration, uncertainty, and traceability recorded. See AIHA Laboratory Accreditation Policy Module H Traceability of Measurement for definitions of the terms used in the accreditation policies involving traceability and uncertainty found on their website.[1] Traceability is characterized (in ILAC documents and the VIM) by: a) an unbroken chain of comparisons going back to stated references acceptable to the parties, usually a national or international standard; b) the uncertainty of measurement for each step in the traceability chain must be calculated or estimated according to agreed methods and must be stated so that an overall uncertainty for the whole chain may be calculated or estimated; c) each step in the traceability chain must be performed according to documented and generally acknowledged procedures and the results must be recorded; d) the laboratories or bodies performing one or more steps in the traceability chain must supply evidence for their technical competence (e.g. by demonstrating that they are accredited for that activity); e) the chain of comparisons must, where possible, end at primary standards for the realization of the SI units; and f) calibrations must be repeated at appropriate intervals; the length of these intervals will depend on a number of variables (e.g. uncertainty required, frequency of use, way of use, stability of the equipment).

4.2 Policies

Traceability is characterized by the following AIHA® LAP, LLC policies or requirements:

4.2.1 Laboratories are required to demonstrate, when possible, that their analytical results are traceable to the SI (International System of Units) through an unbroken chain of calibrations within the measuring system. This requirement can be met for weights (masses), balances, thermometers, volumetric ware (e.g., mechanical pipettes) and stage micrometers.

4.2.2 Laboratories accredited by AIHA-LAP, LLC shall demonstrate, when possible, that calibrations of critical equipment and hence the measurement results generated by that equipment, relevant to their scope of accreditation, are traceable to the SI through an unbroken chain of calibrations.

4.2.3 External calibration services shall, wherever possible, be obtained from providers accredited to ISO/IEC 17025 by an ILAC recognized signatory. Calibration certificates shall be endorsed by a recognized accreditation body symbol. Certificates shall indicate traceability to the SI or reference standard and include the measurement result with the associated uncertainty of measurement.

4.2.4 Where traceability to the SI is not technically possible or reasonable, the laboratory shall use certified reference materials provided by a competent supplier, or use specified methods and/or consensus standards that are clearly described and agreed to by all parties concerned. A competent supplier is a National Metrology Institute (NMI) or an accredited reference material producer (RMP) that conforms with ISO Guide 34 in combination with ISO/IEC 17025, or ILAC Guidelines for the Competence of Reference Material Producers, ILAC G12. Conformance is demonstrated through accreditation by an ILAC recognized signatory.

4.2.5 Reference materials shall have a certificate of analysis that documents traceability to a primary standard or certified reference material and associated uncertainty, when possible. When applicable, the certificate must document the specific ISO certified reference material for traceability. In the US, it is usually a NIST SRM® or NMI certified reference material.

4.2.6 Calibrations performed in-house shall be documented in a manner that demonstrates traceability via an unbroken chain of calibrations regarding the reference standard/material used, allowing for an overall uncertainty to be estimated for the in-house calibration.

4.2.7 Calibrations shall be repeated at appropriate intervals, the length of which can be dependent on the uncertainty required, the frequency of use and verification, the manner of use, stability of the equipment, and risk of failure considerations.

4.2.8 Periodic verifications shall be performed to demonstrate the continued validity of the calibration at specified intervals between calibrations. The frequency of verifications can be dependent on the uncertainty required, the frequency of use, the manner of use, stability of the equipment, and risk of failure considerations. Examples of periodic verifications may include (but not exclusive to): a) checking balance with calibrated masses; b) verifying thermometers with a bath checked by a reference thermometer; and c) measuring a known mass of water with a mechanical pipettes/dispensers/dilutors at a known temperature. The calibration of analytical instruments is verified by the routine use of Continuing Calibration Verification (CCV) standards as prescribed by the analytical method, accreditation requirements, or laboratory policies. Examples of the uncertainties associated with the calibration of thermometers and pipettes are listed in Tables 4.2 and 4.3.

4.2.9 The laboratory shall have procedures describing their external and internal calibration and verification activities and frequencies, and the actions to follow if the equipment is found to be out of acceptable specification.

4.2.10 Laboratory staff performing in-house calibrations and verifications shall have received documented training.

4.3 Measurement

The frequency by which the calibration of equipment is performed will depend on many variables. Table 4.1 provides some minimum requirements for specific equipment, which is not a list of recommended frequencies and is not an inclusive list of all equipment. It includes a list of reference standards and support equipment commonly found in laboratories performing industrial hygiene related analyses that require calibration. ILAC-P10 Policy on Traceability of Measurement Results[3] the following list of considerations a laboratory may wish to consider along with the analyses it performs, its customer's needs and various aspects of its operation :

- uncertainty of measurement required or declared by the laboratory
- risk of a measuring instrument exceeding the limits of the maximum permissible error when in use;
- cost of necessary correction measures when it is found that the instrument was not appropriate over a long period of time;
- type of instrument;

- tendency to wear and drift;
- manufacturer's recommendation;
- extent and severity of use;
- environmental conditions (climatic conditions, vibration, ionizing radiation, etc.);
- trend data obtained from previous calibration records;
- recorded history of maintenance and servicing;
- frequency of cross-checking against other reference standards or measuring devices;
- frequency and quality of intermediate checks in the meantime;
- transportation arrangements and risk; and
- degree to which the servicing personnel are trained

Table 4.1 — Minimum Calibration/Verification Frequency Requirements for Common Reference Standards and Support Equipment

Reference Standard / Equipment	*Calibration Frequency*	*Verification Frequency*
Reference Thermometer	Initial and if damaged	Not applicable
Working Thermometer	Not applicable	Initially, then annually
Reference Masses	Initial and every 5 years	Not applicable
Working Masses	NA	Initially, then annually
Stage Micrometer	Initial and if damaged	Not applicable
Balance	Initial and following service/ repair or when verification fails	Each day of use using external masses
Mechanical Pipettes	Initial and when verification fails	Annually
Volumetric Containers for critical functions (non-Class A)	Not applicable	Each lot prior to use

NOTE 1: For some laboratories, this list may not be complete. It is the responsibility of each laboratory to identify all reference standards and support equipment whose calibration has a significant impact on analytical uncertainty.
NOTE 2: It is the laboratory's responsibility to establish a calibration and verification schedule suitable to the use of equipment
NOTE 3: Laboratories should be mindful of ISO/IEC 17025, clause 5.6.1 when developing the schedule, "All equipment used for testing and/or calibrations, including equipment for subsidiary measurements (e.g. for environmental conditions having a significant effect on accuracy or validity of the results of the rest, calibration or sampling shall be calibrated before being put into service. The laboratory shall have an established program and procedure for the calibration of its equipment."[2]
NOTE 4: Laboratories should be prepared to show supporting data and rationale for the schedule chosen.

Examples of the uncertainty associated with the calibration of thermometers and pipettes are listed in Tables 4.2 and 4.3. These uncertainties should be taken into consideration when developing a calibration schedule, so that all are addressed in that schedule.

Table 4.2 — Uncertainty Contribution Table for Thermometers

Contribution (nomenclature)		*Distribution*	*Estimated Value*
u_r	Standard uncertainty of the nominal values of the reference thermometer.	Normal	Expanded uncertainty on the calibration certificate of the reference thermometer divided by 2 (coverage factor – k)

Table 4.2 — Uncertainty Contribution Table for Thermometers (continued)

Contribution (nomenclature)		*Distribution*	*Estimated Value*
s_p	Standard deviation of the set of calibration readings	Normal	Standard deviation of the set of calibration measurements.
u_1	Standard uncertainty of the readability and resolution of the working thermometer	Uniform (Square)	Smallest gradation of the working thermometer divided by √3. Use ONLY if Sp = 0

Table 4.3 — Uncertainty Contribution Table for Pipettes

Contribution (nomenclature)		*Distribution*	*Estimated Value*
u_r	Standard uncertainty of the nominal values of the reference balance.	Normal	Expanded uncertainty on the calibration certificate of the reference balance divided by 2 (coverage factor – k)
S_p	Standard deviation of the set of calibration readings	Normal	Standard deviation of the set of calibration measurements.
S_T	Standard deviation of corrections caused by temperature (ΔT) when the temperature differs from standard temperature (200C). The thermal coefficient of expansion of water is 0.00021 per 1° Celsius at 20° Celsius.	Uniform (Square)	Relative Standard Deviation = (ΔT x 0.0002) / (√3) in milliliters per milliliter
u_1	Standard uncertainty of the readability and resolution of the working volumetric instrument	Uniform (Square)	Smallest gradation of the working volumetric instrument divided by √3. Use ONLY if Sp = 0

4.4 Calibration Certificates

Calibration certificates accompanying reference standards, or provided in support of other calibration services are traceable to the equipment via serial numbers. The certificates of reference standards and the calibration certificates of equipment should be kept for the life of the equipment or the length of time of legal requirements if this is longer. These records should be maintained following the guidelines of ISO/IEC 17025 section 4.13, Control of Records. The following will appear on an ISO/IEC 17025 compliant calibration certificate for reference standards and support equipment: a) a statement, supported by a recognized accreditation body symbol, that the calibration laboratory is accredited to ISO/IEC 17025; b) the serial number of the measuring equipment/reference standard being used to calibrate your reference standard or equipment and a statement that the measuring equipment is traceable to SI units through an NMI; and c) the measurement range for which your equipment or reference standard was calibrated and the specific uncertainty measurements for that range.

There are calibration laboratories accredited to ISO/IEC 17025 which can be used to calibrate reference standards and equipment. A copy of their accreditation and scope of their accreditation should be maintained with the calibration records.

In-house calibrations by laboratory personnel should be well documented with lot numbers and serial numbers, along with reference information. These calibrations should follow AIHA-LAP, LLC policies and ISO/IEC 17025 policies. Calibrations of measuring systems (i.e. organic,

inorganic, microbiological) must follow the documented instrument calibration procedure defined by the reference method, regulatory standard, other consensus testing method, or laboratory standard operating procedures and utilize reference materials that satisfy traceability requirements in accordance with section 5.0 of the AIHA-LAP, LLC Traceability of Measurement Policy. This requires laboratories to maintain records of all certificates of analyses and calibrations. These records need to have serial numbers and lot numbers on them.

The traceability to the SI for some chemical and biological measurements is not possible. For chemical measurements the ideal is the traceability to the mole and for biological measurements no SI has been defined. For microbiological measuring systems, the use of reference cultures (materials) from an accredited or recognized microbiological reference material producer is the best practice for traceability of the measuring system. For chemical measuring systems, the use of reference material from accredited reference material producers or NMIs, when possible, is the best practice for traceability of the measuring system. With over 10,000 possible chemical and microbiological measurands, reference standards from accredited producers are not always available. Refer to Section 5.6 of the AIHA-LAP LLC Guidance on Traceability of Measurement Document for information regarding selection of a supplier of reference materials.

4.5 Calibration Laboratories

Selecting a laboratory for calibration or an accredited reference material provider involves requesting the accreditation certificates in the fields in question from an ILAC Signatory. This includes recognition through regional cooperation such as APLAC and/or IAAC. The following websites provide some sites to find accredited calibration laboratories and reference material producers:

- SCC/CLAS – http://inms-ienm.nrc-cnrc.gc.ca
- NVLAP - http://ts.nist.gov/Standards/scopes/programs.htm
- A2LA – http://www.a2la.org/dirsearchnew/newsearch.cfm
- IAS – http://www.iasonline.org/Calibration_Laboratories/CL.html
- L-A-B – http://www.l-a-b.com/content/search-l-a-b-accredited-laboratories
- ACLASS - http://www.aclasscorp.com/Directory/tabid/113/Default.aspx
- Perry Johnson Laboratory Accreditation, Inc. -www.pjlabs.com

Primary reference standards and secondary reference standards should be obtained from ISO certified suppliers. In the US, they may also be obtained from NIST or from an NMI. The NIST website describing available standard reference materials is http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PROGRAM_INFO.cfm. The following websites present information on available primary reference standards for chemicals: www.virm.net and www.bipm.org. The Virtual Institute for Reference Materials (VIRM) states “The central mission of this ‘Virtual Institute’ is to be a Knowledge Network and a facility to encourage the interaction between all stakeholders in the field of Reference Materials (Certified Reference Materials, Quality Control Materials) for analysis.” The Bureau International des Poids et Mesures (BIPM) website presents the Key Comparisons Database (KCDB) from national metrology institutes. The BIPM website identifies primary reference standards in a variety of media.

4.6 References

1. **AIHA Laboratory Accreditation Programs, LLC:** *AIHA Laboratory Accreditation Policies and Documents*. [Online] Available at: http://www.aihaaccreditedlabs.org/2014PolicyModules/Pages/2014AccreditationPolicies.aspx (accessed June 2014).
2. **International Organization for Standardization:** General requirements for the competence of testing and calibration laboratories; ISO/IEC 17025: 2005, ISO 9000 Certification Standards. [Online] Available at http://www.iso.org/iso/iso_catalogue.htm (accessed December 2013).

3. **International Laboratory Accreditation Cooperation (ILAC):** *ILAC Policy on Traceability of Measurement Results* [Online] Available at: https://www.ilac.org/486.html (accessed December 2013).
4. **International Laboratory Accreditation Cooperation (ILAC):** *Guidelines for the determination of calibration intervals of measuring instruments* [Online] Available at: https://www.ilac.org/guidanceseries.html (accessed December 2013).
5. **Association for Laboratory Accreditation Inc.:** *CALA Traceability Policy CALA A61* Canadian, [Online] Available at: http://www.cala.ca/A61-CALA_Trac.pdf (accessed December 2013).

4.7 Accreditation Websites

The following documents provide the basis for and assist with application of the principles stated in the AIHA-LAP, LLC Policies.

1. AIHA-LAP, LLC Policy Appendix G on the Estimation of Uncertainty of Measurement http://www.aihaaccreditedlabs.org/policymodules/Documents/ApGPolicyEstimationUnce rtaintyMeasurement.pdf (accessed December 2013).
2. AIHA-LAP, LLC Guidance on the Estimation of Uncertainty of Measurement http://www.aihaaccreditedlabs.org/policymodules/Documents/GuidanceEstimationUncertaintyMeasurement.pdf (accessed December 2013).
3. AIHA-LAP. LLC Policy Appendix H Traceability of Measurement http://www.aihaaccreditedlabs.org/policymodules/Documents/ApHPolicyTraceabilityMeasurement.pdf (accessed December 2013).
4. AIHA-LAP, LLC Guidance on Traceability Measurement http://www.aihaaccreditedlabs.org/2013PolicyModules/Documents/8%202%202%20LAP%20Guidance%200n%20Traceability%200f%20Measurement_R2%20FINAL.pdf (accessed June 2014)
5. CALA Traceability Policy, CALA A61 http://www.cala.ca/A61-CALA_Trac.pdf.
6. CALA Example Internal Calibration Workbook http://www.aihaaccreditedlabs.org/policymodules/Documents/CALAInteCalibrationWorkbook.xls (accessed December 2013).
7. General requirements for the competence of testing and calibration laboratories, ISO/IEC 17025:2005 http://www.iso.org/iso/iso_catalogue.htm (accessed December 2013).
8. ISO/IEC 17025:2005 Guidelines for the determination of calibration intervals of measuring equipment, ILAC G 24:2007 http://www.ilac.org/documents/ILAC_G24_2007.pdf
9. Guidelines for the Selection and Use of Reference Materials, ILAC G9:2005 http://www.ilac.org/documents/ILAC_G9_2005_guidelines_for_the_selection_and_use_ of_reference_material.pdf (accessed December 2013).
10. ILAC Policy on the Traceability of Measurement Results, ILAC P10:2002 http://www.ilac.org/documents/ILAC_P10_2002_ILAC_Policy_on_Traceability_of_Meas urement_Result.pdf (accessed December 2013).
11. International vocabulary of metrology— Basic and general concepts and associated terms (VIM), VIM JCGM 200:2012 http://www.bipm.org/en/publications/guides/vim.html (accessed December 2013).
12. Metrological Traceability Of Measurement Results In Chemistry: Concepts And Implementation (IUPAC Recommendations 2009), International Union of Pure and Applied Chemistry (IUPAC), Paul De Bièvre1, René Dybkaer, Aleš Fajgelj And D. Brynn Hibbert, http://www.iupac.org/web/ins/2001-010-3-500 (accessed December 2013).
13. NISTIR 6919 – Recommended Guide for Determining and Reporting Uncertainties for Balances and Scale http://ts.nist.gov/WeightsAndMeasures/upload/NISTIR6919.pdf (accessed December 2013).
14. NIST Special Publication 1088- Maintenance, Validation, and Recalibration of Liquid-in-Glass Thermometers http://www.nist.gov/cgi-bin/view_pub.cgi?pub_id=900914 (accessed December 2013).

Chapter 5
Sampling Procedures

by Charles (Gus) Manning, PhD, CIH

5.1 Introduction

To ensure the scientific reliability of the data generated, quality assurance (QA) elements must be included in the field sampling procedure from the conception of the sampling strategy to the delivery of samples to the laboratory. Sample result validity is aided by adhering to established calibration, sampling, handling, identification, and chain-of-custody procedures. These QA elements, outlined in the following sections, provide the framework on which the validity of analytical results acquired from field samples. Accordingly, sampling procedures should be evaluated against the criteria outlined herein and should be carefully documented. See Figure 5.1 for a system flow chart. Sampling is a critical link in hazard evaluation.

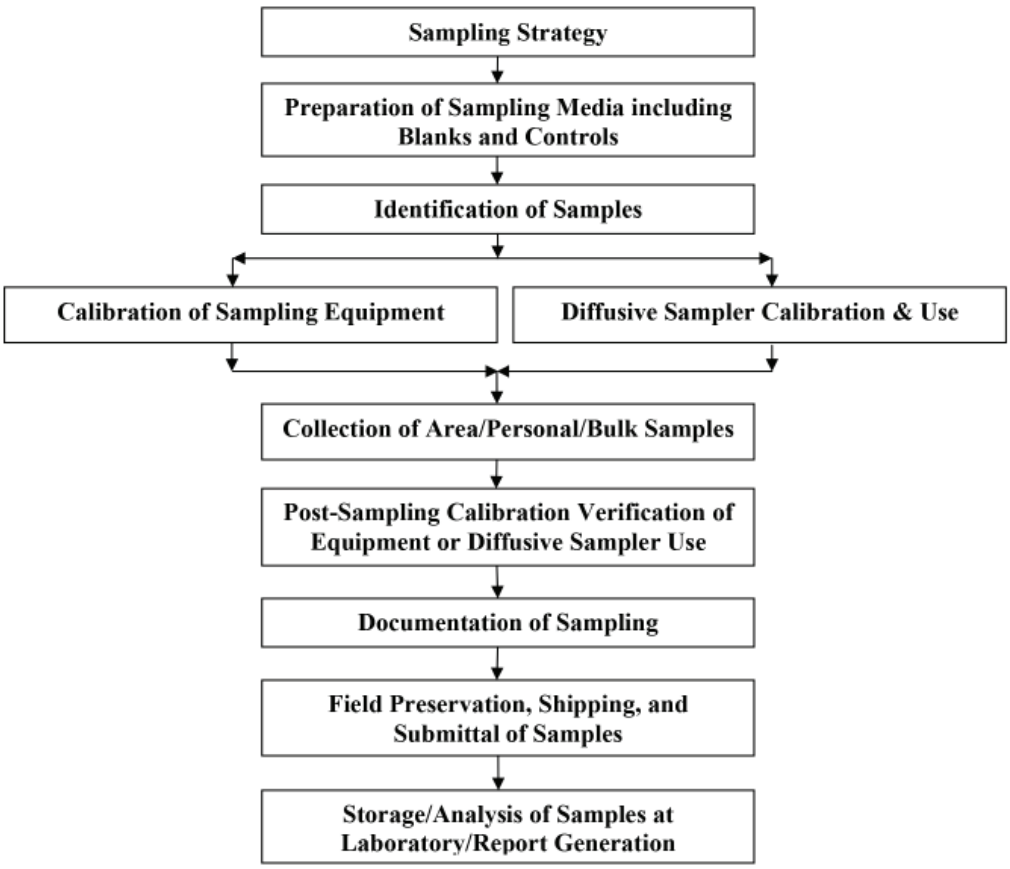


Figure 5.1 — System Flow Chart

5.2 Sampling Strategy

Sampling locations, timing, and duration should closely represent the typical locations, timing, and duration of expected exposures. Thus, persons representing each group expected to experience similar exposures should be sampled during the entire time during which exposures are possible. Further, sampling should be repeated with a frequency necessary to ensure accurate representation of daily exposure variations. For further guidance on this subject, consult references 1 and 2 of this chapter.

5.3 Preparation of Sampling Media Including Blanks and Controls

Samplers should be prepared as directed in an evaluated analytical method or should be acquired from a vendor who provides an evaluated method. Prior to their use, samplers should be packaged and stored in a manner that protects samplers from damage and from extrinsic contamination (i.e. contaminants unrelated to the environment to be sampled). To identify and correct for extrinsic contamination of samplers, the following additional steps may be taken.

5.3.1 Media Blanks and Reagent Blank Evaluation

It is essential that all new lots of sorbent tubes, passive monitors, filters, wipes, bottles, or other sampling media be tested by the laboratory for unacceptable contamination prior to use in the field. This result would be called a method blank or a media blank. Reagents used in the analysis must be checked for analyte interferences. The appropriate mixture of reagent chemicals is analyzed without the analyte in the mixture. This result would be called the reagent blank. Both of these blanks are taken through the preparation and analytical sequences, just as a sample would be. An appropriate acceptance criteria level for contaminants would be less than or equal to the reporting limit (RL) of the analysis.

5.3.2 Quality Control (QC) Samples or Lab Control Samples

Validation of sample collection and analysis methods by means of blanks and spiked samples (unexposed samples inoculated with a known quantity of contaminant) should be performed whenever possible to aid in verification of sample integrity. The results can show potential contamination or recovery problems and might indicate a need to modify the sampling plan or re-validate the sampling method. Each QC Sample should have definitive acceptance criteria. Acceptance criteria for QC samples must be documented for each analysis under the lab's scope of accreditation. Included in this documentation would be spiking levels (where appropriate), frequency, acceptance criteria, corrective actions, and criteria for rejection of samples.

5.3.3 Field Blanks

The most commonly used field control is the field blank or trip blank. This may be a collection device or solution supplied by the laboratory or prepared by the individual taking the samples. Field blanks must be handled in exactly the same manner as field samples, except that the blank is not exposed to the environment (e.g., sample ends of charcoal tubes are broken off in the field and polyethylene caps are placed immediately onto the tube ends, or a wipe is removed from its container and immediately stored away in a hard-walled sample container). If field blank values are higher than lab media blanks described in section 5.3.1, the sample set results must be carefully evaluated for bias.

5.3.4 Spiking of Sampling Media in the Field

Spikes, or inoculated control samples, may be applied in the field in a similar fashion as the lab control samples described in section 5.3.1, and are taken through the preparation and analytical sequences, just as a sample would be. The level of analyte in the spiked sample should approximate the level expected in field samples or the regulatory action limit, preferably both.

Laboratory recovery data from spiked collection media will assist in the overall evaluation of sampling and analytical methods.

5.3.4.1 Laboratory Spikes
The laboratory spike is a laboratory-generated QC sample on or in the same matrix as the sample(s) undergoing analysis. These laboratory spikes, also sometimes known as laboratory control samples (LCS), are analyzed as regular samples, and their results become part of the ongoing QA program. Recovery and precision data generated through this type of spike should be plotted on control charts to provide information on analytical precision, analyst bias, sample contamination or reactivity, instrument performance, sample handling, and calibration.

5.3.4.2 Field Spikes (inoculated or liquid spikes)
Sampling media spiked in the field by liquid inoculation may be generated by field personnel and may be shipped to the laboratory for analysis with the sample set. These field spikes also provide information about the stability of the sample and retention of the analyte of interest and its recovery. However, unless they are exposed to the actual conditions of field sampling, they will provide no information about how the method responds to the environmental sampling conditions. Field spikes may be redundant if field personnel can gain access to the laboratory data describing its use of blanks and spiked control samples.

5.3.4.3 Field-Exposed Spikes and Vapor Spikes
Sampling media spiked by liquid inoculation in the laboratory and then exposed in the field may give an indication of the effect of environmental conditions on the sample. This may be part of an overall method evaluation which should also include exposure to known vapor concentrations under environmental conditions to be expected in the field. In many cases, the lab evaluation of controlled vapor spikes under simulated field conditions has been found to be a superior method of method evaluation.

5.3.4.4 Field Duplicates
Field Duplicates offer a means of assessing the reproducibility of lab analysis and/or of the sampling environment. To utilize field duplicates, care must be taken to ensure that the samples are collected from the exact same environment. Although it is frequently assumed that samples taken side-by-side will be identical, air sampling environments are commonly inhomogeneous, and this assumption may be unwarranted.

5.4 Identification of Samples

The sample history or chain of custody usually begins at initiation of sample collection. A unique identifier, blind to the analysts, must be assigned to each field sample, bulk sample, collection device or solution, and to each blank and spike. Care must be taken to avoid the use of duplicate identifiers (a common problem). A field sampling data sheet corresponding to each sample or a group of similar samples is prepared. Several samples from the same area could be grouped on a sample sheet designed for multiple entries. Several important items can be included when defining the unique sample number. For example, a code table can be developed to define each sampling device or collecting solution. The table can include codes for the area sampled, the date of collection, and the sample sequence.

5.5 Calibration and Use of Sampling Equipment

5.5.1 General Accuracy Criteria

Whether used in the laboratory or in the field, instruments used in air sampling should be traceable to appropriate standards, such as those available from an ISO certified supplier, or

in the U.S., from NIST, calibration of field equipment should be made with at least secondary standards traceable to primary standards.

Field instruments are commonly calibrated prior to and after each use or on a scheduled, periodic basis. For a QA program to have credibility, accuracy must be traceable at all performance levels and such accuracy must be documented.

Calibrations of sampling pumps, rotameters, wet and dry test meters, equipment for measuring pressure, vacuum and temperature, and portable detection instruments should be traceable to NIST standards. Each calibration device must have a written calibration program containing the following items:

5.5.1.1 Identification

5.5.1.2 Descriptions, including manufacturer, model number, and serial number

5.5.1.3 Location of use or storage

5.5.1.4 Calibration instruction

5.5.1.5 Calibration/calibration verification interval

5.5.1.6 Date of last calibration/calibration verification

5.5.1.7 Name of the person who performed the calibration/calibration verification

5.5.1.8 Date next calibration/calibration verification is due

5.5.1.9 Calibration/calibration verification data (curves, etc.)

While periodic calibration of many instruments may be subcontracted to vendors, frequent calibration of air sampling pumps is necessary to ensure that the pump sampling rate calibrations (hence, calculated sample volumes) are valid at the time and place of sampling.

5.5.2 Record of Performance

A master log should be maintained listing the repair history, current status, and next scheduled calibration date, if applicable, for each piece of equipment. This log is necessary to ensure that sampling devices are delivering reliable flow rates.

5.6 Diffusive Sampler Calibration and Use

Diffusive samplers, also known as passive monitors or personal monitoring badges, are lightweight samplers worn in the breathing zone of personnel. Unlike active samplers, diffusive samplers are characterized by fixed sampling rates. That is, for a given model of sampler, each contaminant sampled has a distinct sampling rate (on that sampler) that is determined by its diffusion coefficient (a constant of nature). Sampling rates for a diffusive sampler are normally determined by its manufacturer and published for users. Such published sampling rates amount to the manufacturer's calibration of a diffusive sampler. Accordingly, diffusive sampler users should acquire the sampling rate for each contaminant sampled from the manufacturer by reference to the model number of the sampler. Users should also review documentation to verify that the sampler manufacturer has performed appropriate testing to evaluate the sampler under its expected conditions of use.

5.6.1 Field Evaluation of Diffusive Samplers

A monitoring method should be evaluated in the field only after a successful laboratory validation program has been completed.[3,4]

A diffusive sampler may be compared with the active sampler reference method (e.g., OSHA) by placing the active and diffusive samplers side-by-side under the same conditions and at the same sampling locations. While there is no requirement to perform side-by-side comparisons, they can be helpful in satisfying users that two samplers perform similarly. Statistical side-by-side method comparisons may often be inconclusive due to the variations that occur under field conditions, especially the concentration variation between a diffusive sampler and an active sampler placed alongside, but which is actually drawing air from adjacent locations. Accordingly, OSHA and other agencies who evaluate samplers tend to rely more on lab validations that simulate field conditions under rigorously controlled conditions.

5.6.2 Availability of Information on Sampler Evaluation

Sampler manufacturers should provide users with sampler documentation including instructions for use, evaluations performed[3,4], and recommended conditions for use (commonly available at a web-site). Users should review manufacturer's documentation to ensure that the conditions of expected use (especially expected contaminant concentrations, humidity, and sampling duration) are within the range covered by the manufacturer's evaluation and recommendations. Evaluations published by third parties (commercial or government organizations, e.g., OSHA) can also provide technical information to support diffusive sampler use.

5.6.3 Limitations of Use

Diffusive sampler users should be aware of certain limitations on their use as follows.

5.6.3.1 Minimum Air Velocity — A certain amount of air movement (usually 30 ft/min) is necessary at the sampler face to avoid starvation effects and provide accurate sampling.

5.6.3.2 Aerosols — Aerosols cannot be sampled accurately by a diffusive sampler as larger airborne particles do not follow known laws of diffusion.

5.6.3.3. Low Sampling Rates — Since diffusive samplers typically employ lower sampling rates than active sampling pumps, limits of detection may be higher than expected. Where applicable, lower detection limits may be achieved through longer sampling times.

5.6.3.4 Capacity Limitations — Samplers (active or diffusive) utilizing charcoal adsorbent are subject to sample retention and recovery reduction when contaminant levels exceed sorbent capacity. Users can usually stay within sample capacity limits by estimating sample loadings from expected sampling time and concentration.

5.6.3.5 Environmental Conditions — All samplers (active or diffusive) are subject to the manufacturer's recommended conditions of use which must be limited to environmental conditions for sampling and holding and time durations within which sampler performance has actually been evaluated. While sampler use outside of these conditions may be successful, such use can only be supported by subsequent research or evaluation.

5.7 Documentation of Sampling

The sampling data sheet must provide a complete, traceable record that will withstand legal or compliance scrutiny. This sheet should represent the compilation of all sampling information and be easily linked to the analytical result(s) from the laboratory. All sampling personnel should have signed this document. Data entry into the bound, pre-numbered pages of a note-

book with each page signed and dated offers the most defensible record. Computer records should have back-up storage to properly maintain the records.

All pertinent facts about each collected sample should be recorded on the field sampling data sheet. An example is at the end of this chapter as Attachment A. Sample information requirements will vary somewhat among organizations and types of samples collected but probably will include many of the following items:

5.7.1 Date, start/finish time, temperature, pressure, and relative humidity
5.7.2 Wind direction and speed
5.7.3 Plant, department, shift, process description
5.7.4 Sampling location (to include surface color and substrate)
5.7.5 Name, job classification, and/or alpha-numeric identifier of the person sampled (Social security numbers should be avoided if possible)
5.7.6 Task description
5.7.7 Personal protective equipment (PPE) worn
5.7.8 Worker's comments concerning exposure potential
5.7.9 Observations of the workplace and unusual conditions during sampling
5.7.10 Sample media (manufacturer, catalog number, lot or batch number, etc.)
5.7.11 Sample type, collection rate, and duration of sampling
5.7.12 Sample pump type, model, serial number and calibration information
5.7.13 Substance monitored
5.7.14 Sample identifier (unique to each sample)
5.7.15 Sample preservation used (if any)
5.7.16 Analysis required and possible interferences
5.7.17 Sampler's name, signature and date
5.7.18 Sampler's observations of sampling
Any other pertinent data needed to identify the sample or to aid the laboratory analyst should be included. Examples of typical industrial hygiene and lead-based paint sampling data sheets are included at the end of this chapter.

5.8 Field Preservation, Shipping, and Precautions

5.8.1 Preservation to Prevent Sample Contamination

Without exception, all bulk samples should be shipped separately from samples collected to determine employee or area exposure levels. Care must be taken to ensure that representative material is selected for the bulk sample.

The laboratory should define the amount of bulk sample required regardless of the matrix sampled. Since the laboratory is faced with the disposal of hazardous materials, excessive amounts must be avoided. The sample container used must be compatible with the sample. The shipping container should be packed properly to absorb leakage and/ or breakage of the sample container and should be labeled completely so that the carrier can take appropriate precautions.

5.8.2 Field Preservation

Samples must be submitted to the laboratory for analysis as soon as possible after collection and within the recommended holding time. The laboratory should provide information regarding the proper preservation requirements for all samples. References such as the OSHA Sampling and Analytical Methods[4], NIOSH Manual of Analytical Methods[5] and the various EPA[6] waste sampling protocols should be consulted for sample preservation requirements. Special requirements should be documented in the method.

5.8.3 Shipping Procedures

If it is necessary to ship samples by commercial carrier, follow the appropriate shipping regulations, in the US it is adherence to DOT packaging and labeling regulations. These regulations state what can be shipped and how it must be packaged for shipment. Individual rapid-delivery services (e.g., FedEx and UPS) may have additional requirements.

The following packaging precautions should be taken:
[For Example: Do not package asbestos monitoring cassettes with polystyrene packing materials: static electricity effects might remove fibers from the filter.]

5.8.3.1 Cassettes, sorbent tubes, passive monitors, etc., must be properly sealed.

5.8.3.2 Sufficient packing material must be used to prevent breakage.

5.8.3.3 The shipping container must be secure and must be labeled properly.

5.8.3.4 Custody seals, if used, must be intact at the time of shipping.

5.8.4 Special Precautions

Samples should be packed and shipped in such a fashion with timing such that the samples remain within limits of environmental conditions and holding times specified in the analytical method. Direct contact with water/ice should be avoided as this can alter the chemicals collected on the media, alter the media, and make it difficult for the analyst to extract the analytes from the media.

Samples should be shipped by traceable express mail within a guaranteed delivery time.

A complete copy of sampling documents should accompany samples to the laboratory.

Excessive temperature can cause loss of sample during shipping. For example, charcoal tubes or Diffusion Monitors sitting in a car on a hot sunny day might be exposed to temperatures above 49°C (120°F), causing desorption and/ or migration of some adsorbed organics.

The method of shipment should be traceable. Sample arrival should be confirmed by contacting the laboratory. Paying for overnight delivery will not guarantee that samples will be delivered by the following day. However, it does give added traceability in case of sample shipment misplacement.

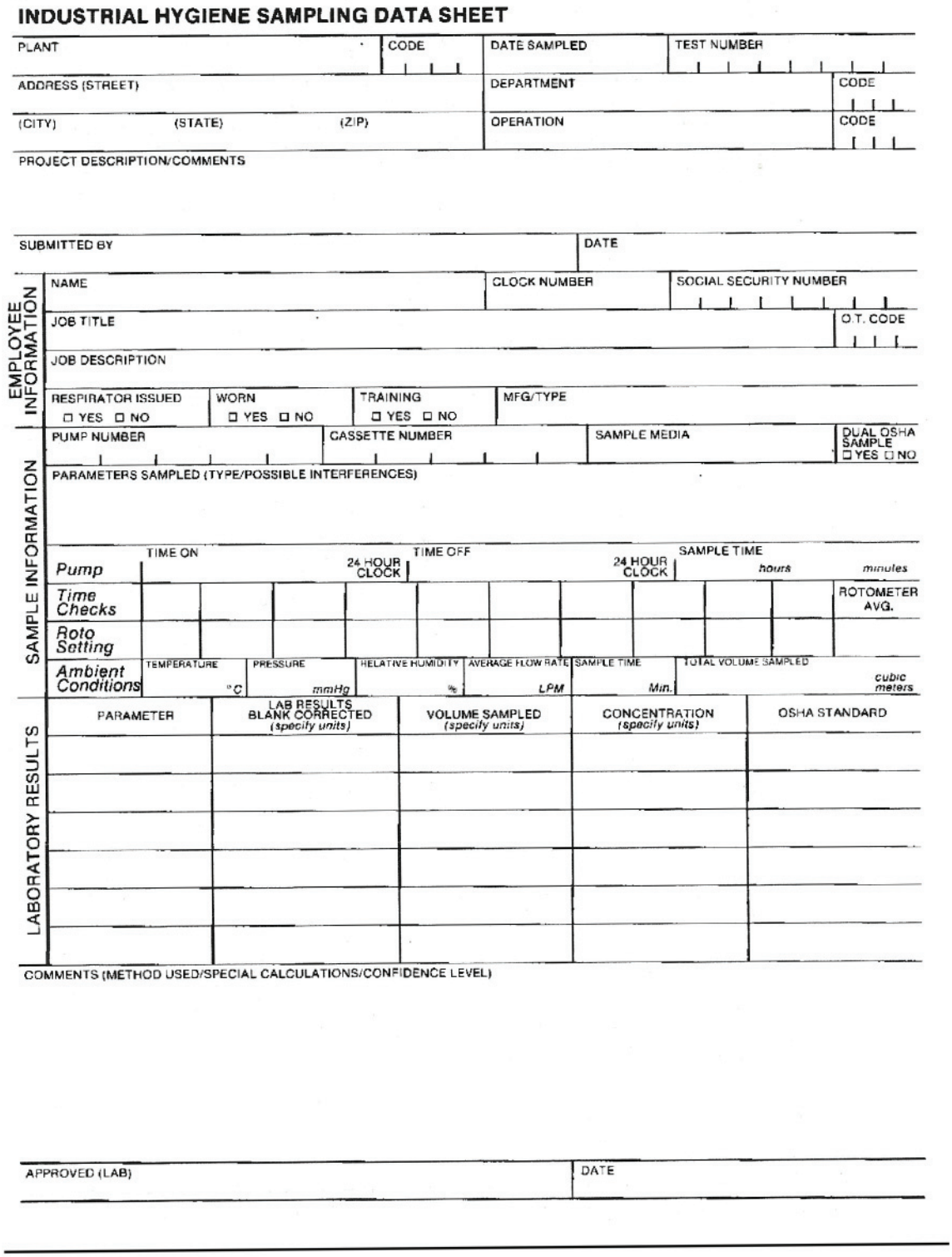
A copy of the sample information must accompany the packaged sample to ensure proper laboratory identification and handling.

5.9 References

1. **Leidel, N.A., K. Busch, and J. Lynch:** *Occupational Exposure Sampling Strategy Manual* (DHEW/NIOSH Publication No. 77-173). 1977. [Online] Available at: http://www.cdc.gov/niosh/docs/77-173/ (accessed January 2014).
2. **American industrial Hygiene Association (AIHA):** Exposure Assessment Committee, *A Strategy for Assessing and Managing Occupational Exposures*, AIHA, Fairfax, VA 2006, [Online] Available at https://www.aiha.org/get-involved/VolunteerGroups/Pages/Exposure-Assessment-Strategies-Committee.aspx (accessed January 2014).
3. **American National Standard Institute:** ANSI/ISEA 104-1998 (R2009) American National Standard for Air Sampling Devices — Diffusive Type for Gases and Vapors in Working Environments, New York, [Online] Available at http://webstore.ansi.org/default.aspx (accessed January 2014).

4. **Hendricks, W.:** *Development of a Protocol for Laboratory Testing of Diffusive Samplers*. 1996, United States Department of Labor, Occupational Safety & Health Administration. [Online] Available at https://www.osha.gov/dts/sltc/methods/studies/3movm/3movm.html (accessed January 2014).
5. **National Institute for Occupational Safety and Health:** *NIOSH Manual of Analytical Methods.* [Online] Available at http://www.cdc.gov/niosh/nmam/ (accessed January 2014).
6. **U.S. Occupational Safety and Health Administration (OSHA):** *OSHA Sampling and Analytical Methods*. [Online] Available at https://www.osha.gov/dts/sltc/methods/ (accessed January 2014).

Attachment A: An example of an Industrial Hygiene Sampling Data Sheet.



INDUSTRIAL HYGIENE SAMPLING DATA SHEET

PLANT	CODE	DATE SAMPLED	TEST NUMBER
ADDRESS (STREET)		DEPARTMENT	CODE
(CITY) (STATE) (ZIP)		OPERATION	CODE
PROJECT DESCRIPTION/COMMENTS			

SUBMITTED BY		DATE	
EMPLOYEE INFORMATION			
NAME		CLOCK NUMBER	SOCIAL SECURITY NUMBER
JOB TITLE			O.T. CODE
JOB DESCRIPTION			
RESPIRATOR ISSUED ☐ YES ☐ NO	WORN ☐ YES ☐ NO	TRAINING ☐ YES ☐ NO	MFG/TYPE
SAMPLE INFORMATION			
PUMP NUMBER	CASSETTE NUMBER	SAMPLE MEDIA	DUAL OSHA SAMPLE ☐ YES ☐ NO
PARAMETERS SAMPLED (TYPE/POSSIBLE INTERFERENCES)			

	TIME ON			24 HOUR CLOCK	TIME OFF			24 HOUR CLOCK	SAMPLE TIME hours		minutes
Pump											
Time Checks											ROTOMETER AVG.
Roto Setting											
Ambient Conditions	TEMPERATURE °C		PRESSURE mmHg		RELATIVE HUMIDITY %	AVERAGE FLOW RATE LPM	SAMPLE TIME Min.		TOTAL VOLUME SAMPLED cubic meters		

LABORATORY RESULTS

PARAMETER	LAB RESULTS BLANK CORRECTED (specify units)	VOLUME SAMPLED (specify units)	CONCENTRATION (specify units)	OSHA STANDARD

COMMENTS (METHOD USED/SPECIAL CALCULATIONS/CONFIDENCE LEVEL)

APPROVED (LAB)	DATE

Chapter 6
Sample Receipt and Handling

by David Sandusky, CIH

6.1 Introduction

Ensuring the integrity of compliance samples is imperative if the results are ever challenged by a regulatory authority. Any sample that may be introduced as evidence in a courtroom must have the same integrity. Written procedures, proof of training (in the form of written training records), proper use of sample seals and chain-of-custody forms provides documentation that legally defensible handling procedures were followed.

This chapter describes appropriate procedures to ensure sample integrity through chain-of-custody records. The custody of samples normally passes from field personnel to the laboratory sample custodian to the analyst, with intermittent involvement by carrier agents and laboratory supervisory personnel. Although this chapter is concerned primarily with laboratory procedures, it must be remembered that the chain-of-custody process must start in the field. Also, it is best to assume that all analytical data will be used as evidence in a court of law. The guidance provided in this chapter is useful in meeting the requirements of AIHA-LAP, LLC policy. (1) (As of 2014 Section 2A.5.8.1 states: "The laboratory shall have a written description of the chain-of-custody and sample receiving procedures followed in the laboratory. Procedures shall include criteria for rejection of samples.")

6.2 Chain of Custody

Improper sample and data handling and inadequate chain-of-custody procedures can affect the acceptability of even the most accurate and precise analytical results. Therefore, it is essential that samples be collected and handled properly. It is important for a chain of custody to be maintained to document that the samples have been received in acceptable condition, preserved, stored and processed properly from the time of collection to the time the analytical results are received by the customer.

Sample receipt and log books should be hardbound with sequentially numbered pages, although the use of other formats may be acceptable if detailed entry, handling, and other procedures and practices are in place. An adequate computer-based laboratory sample logging system may be effective to this end. Entries should be clear and concise with appropriate cross-referencing information to enable an auditor to determine the status of an in-process sample or to trace the final data file and report. Custodial signatures, and dates and times should be readily available and retrievable.

The use of standardized forms and sample seals is a way to document chain of custody and sample integrity. (Examples of these forms and a sample seal are included at the end of this chapter.) All pertinent information must be obtained before starting the chain of custody. This information should include sampling conditions, when the samples were collected, the type of sample, the sampling media, the sampling method, and any other information that might be required by the laboratory to handle and analyze the samples properly. Each sample container should be sealed to prevent tampering. Each sample should be identified uniquely and be traceable to its corresponding data sheet. The samples and copies of their paperwork must be packed properly and shipped to the laboratory according to the appropriate shipping regulations, in the US it is DOT regulations. Bulk samples should be shipped separately from air samples to prevent contamination. Upon arrival at the laboratory, each sample should be assigned a unique labora-

tory sample identification number and should be logged into a register. A log of all personnel handling the samples should be maintained. Each sample should be checked on receipt for visible damage or leakage, an intact sample seal, and identification corresponding to assignments on the data sheet. Any unusual observation should be recorded on the data sheets and in the register. Samples must be stored properly in a secured area until analysis.

Upon receipt by the analyst, the condition of the samples and their seals should be documented on the data sheets and also in the laboratory log book before the seal is broken. The analyst should not break the sample seal until it is absolutely necessary. If the sample cannot be safeguarded by the analyst, it may be necessary to place it in a secured area to ensure that tampering cannot occur. The analyst should verify and document that the proper media was used for the requested analysis.

6.3 Information Reporting

6.3.1 Sampler Reporting

Samples should arrive at the laboratory with information provided by the sampling data sheet, transport records, and chain-of-custody records. This should include:

6.3.1.1 Name and contact information of the person who collected the samples – the sampler;

6.3.1.2 Time and date the sampling was performed;

6.3.1.3 Location of sampling;

6.3.1.4 Description of sampling method used;

6.3.1.5 Preservation method used, if applicable;

6.3.1.6 Unique sample identifier for each sample submitted;

6.3.1.7 Sample air volume and/or flow rate and time;

6.3.1.8 Possible interferences;

6.3.1.9 Number of samples submitted, including field blanks;

6.3.1.10 Date samples were shipped to laboratory;

6.3.1.11 Date analysis results are required;

6.3.1.12 Method of transportation of sample to laboratory;

6.3.1.13 Chain-of-custody record; and

6.3.1.14 Name, address and contact information of person(s) to receive the analysis report.

6.3.2 Laboratory Reporting

The following information must be verified and documented (hardcopy and/or electronically):

6.3.2.1 Date of sample arrival at the laboratory

6.3.2.2 Condition and number of samples and their seals

6.3.2.3 Name of the laboratory staff member who accepted the samples

6.3.2.4 Appropriateness of sample packaging

6.3.2.5 Manner of sample preservation

6.3.2.6 Manner of sample storage

6.3.2.7 Date of sampling and maximum holding time, if appropriate

6.3.2.8 Correspondence of field sample identification numbers to request form

6.3.2.9 Appropriateness of media selection for analyte(s) requested

6.3.2.10 Appropriateness of air flow rates and volumes

6.3.2.11 Dates and signatures for persons who obtained and relinquished custody of sample

The following information should be documented after analysis is complete:

6.3.2.12 Date(s) sample(s) was/were prepared and analyzed

6.3.2.13 Analytical procedure(s) used

6.3.2.14 Equipment / Instrumentation used

6.3.2.15 Detection and/or quantitation limits

6.3.2.16 Results of analysis

6.3.2.17 Documentation of all appropriate QC samples and their results

6.3.2.18 Signature(s) of preparer(s) and analyst(s)

6.3.2.19 Signature(s) of reviewer(s) and date(s) reviewed

6.3.3 Sample Handling Considerations

Samples should be logged into the laboratory record system as soon as possible. Samples should be stored in a central area so that they can be located easily. Access to this area should be limited and samples should be locked up until assigned to an analyst. Certain samples might require storage in a locked refrigerator or other secured area. Care must be taken to ensure that sorbent samples are not stored in the same package, cabinet, refrigerator, or freezer with bulk samples of volatile liquids. Some samples might be light-sensitive or have limited storage life. These factors determine how samples should be stored and prioritized for analysis. Laboratory records should indicate whether the samples were disposed of or returned to the client after the analysis was completed.

6.3.4 Use of Sample Seals

Although sample seals (also known as custody seals) may not be considered necessary during routine monitoring, they can be valuable if analytical data are used as evidence in court proceedings. OSHA uses an official sample seal on all samples collected by its staff, as shown in Figure 6.1.

Figure 6.1 — OSHA Official Sample Seal

The use of sample seals provides additional evidence of sample integrity. An effectively designed sample seal should use an adhesive that will not allow the seal's removal without breaking it. Some adhesives contain solvents that might interfere with the analysis. The seal should provide enough space for adequate sample identification, including the sampler's signature. Sample seals normally will prevent caps on samples from vibrating loose during transit to the laboratory if applied in the following manner:

6.3.4.1 Sorbent tubes: Wrap end to end, over the end caps.

6.3.4.2 Filter cassettes: Wrap across the cassette covering the inlet and outlet plugs.

6.3.4.3 Vials containing bubbler solutions: Wrap top to bottom, across the top of the cap.

6.3.4.4 Hard-walled tubes with, for example, wipe samples: Wrap across the top of the cap.

6.3.5 Contract Laboratory

Subcontracted laboratories may be used when a laboratory does not have the required capability or capacity to analyze certain samples properly and safely. Clients should be informed when testing will be performed by a subcontract laboratory. The handling and chain-of-custody procedures at the contract laboratory must be investigated to ensure that they are adequate. If they are not, the required procedures must be specified and should be an extension of the customer's handling and chain-of-custody procedures. The Subcontract laboratory must hold an appropriate accreditation by a reputable Accrediting Body, such as ISO or AIHA, for the required analysis. With AIHA-LAP, LLC Accredited Laboratories, their accredited Fields of Testing (FoT) and specific methods can be checked at the AIHA-LAP, LLC website[1] by selecting "Accredited Laboratories" followed by selecting the appropriate Laboratory Accreditation Program.

A file must be maintained that contains the following information (at a minimum):

6.3.5.1 Certification or Accreditation certificates (check the dates)

6.3.5.2 Scope of accreditation certificate

6.3.5.3 Results of current and relevant proficiency testing programs

6.3.5.4 Current copies of each analytical method performed which should include sample collection information. The current method may also be accessed as needed from the internet.

6.3.6 Documentation Forms

Some examples of forms used for documentation are included at the end of this chapter (see Attachment B, C, and D). They include the following:

6.3.6.1 OSHA Air Sampling Worksheet

6.3.6.2 OSHA Air Sampling Report

6.3.6.3 OSHA Chain-of-Custody Record

6.4 References

1. **AIHA Laboratory Accreditation Programs, LLC:** *AIHA Laboratory Accreditation Policies*. [Online] Available at: http://www.aihaaccreditedlabs.org (accessed June 2014).

Attachment B: OSHA Air Sampling Worksheet submitted with samples to lab for analysis.

Air Sampling Worksheet

U.S. Department of Labor
Occupational Safety and Health Administration

1. Reporting ID	**2.** Inspection Number	**3.** Sampling Number ▶		
4. Establishment Name		**5.** Sampling Date	**6.** Shipping Date	
7. Person Performing Sampling (Signature)		**8.** Print Last Name	**9.** CSHO ID	
10. Employee (Name, Address, Telephone Number)		**14.** Exposure Information	a. Number	b. Duration
		c. Frequency		
		15. Weather Conditions	**16.** Photo(s) Y	
11. Job Title	**12.** Occupation Code			
13. PPE (Type and effectiveness)		**17.** Pump Checks and Adjustments		

18. Job Description, Operation, Work Location(s), Ventilation, and Controls

Cont'd

19. Pump Number:

Sampling Data

20. Lab Sample Number						
21. Sample Submission Number						
22. Sample Type						
23. Sample Media						
24. Filter/Tube Number						
25. Time On/Off						
26. Total Time *(in minutes)*						
27. Flow Rate ☐ *l/min* ☐ *cc/min*						
28. Volume *(in liters)*						
29. Net Sample Weight *(in mg)*						
30. Analyze Samples for:	**31.** Indicate Which Samples To Include in TWA, Ceiling, etc. Calculations					

32. Interferences and IH Comments to Lab	**33.** Supporting Samples a. Blanks: b. Bulks:	**34.** Chain of Custody	Initials	Date
		a. Seals Intact?	Y N	
		b. Rec'd in Lab		
		c. Rec'd by Anal.		
		d. Anal. Completed		
		e. Calc. Checked		
		f. Supr. OK'd		

Case File Page / of

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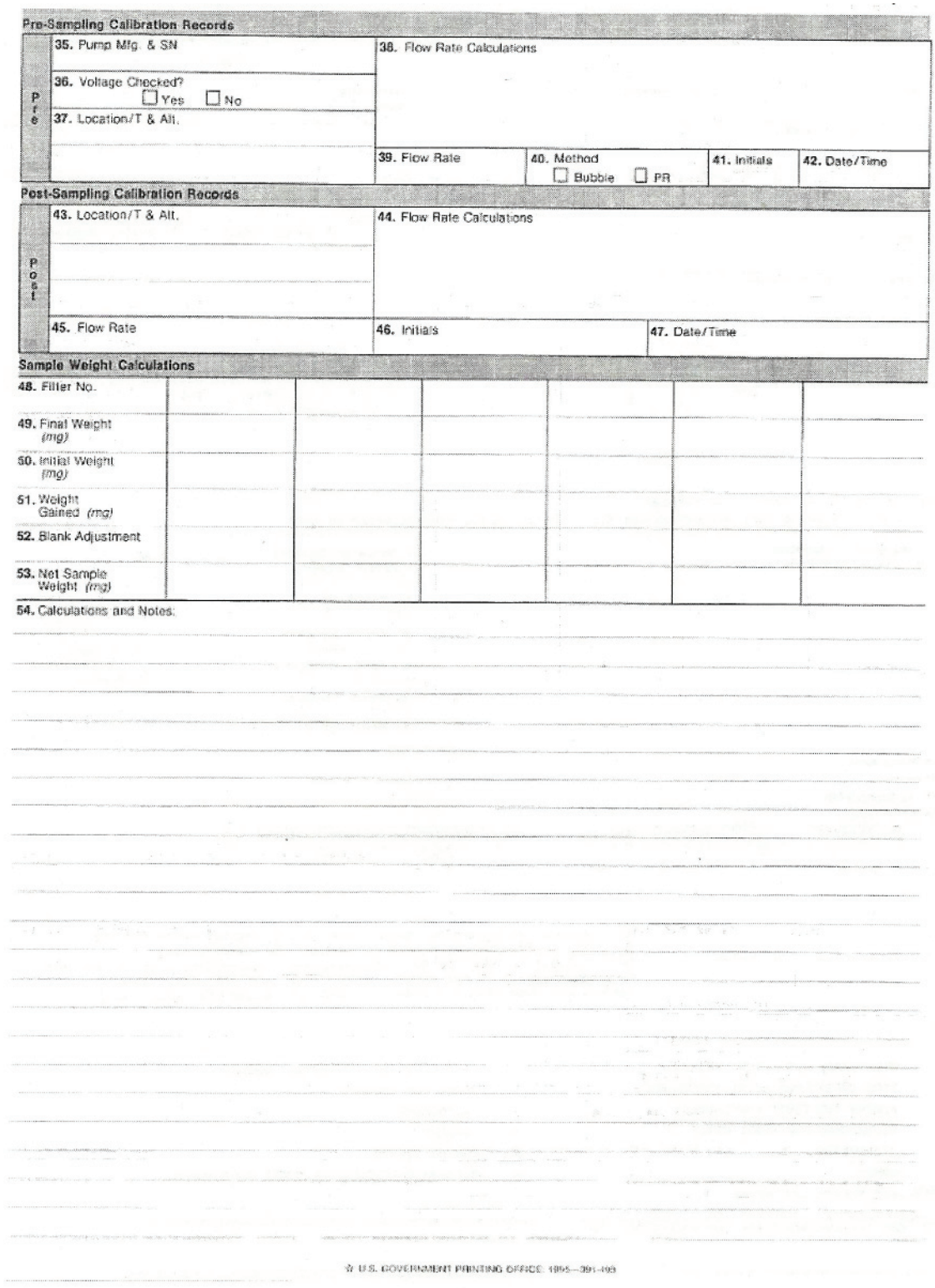
Attachment B: OSHA Air Sampling Worksheet submitted with samples to lab for analysis. (continued)



Pre-Sampling Calibration Records

Pre

35. Pump Mfg. & SN

36. Voltage Checked? ☐ Yes ☐ No

37. Location/T & Alt.

38. Flow Rate Calculations

39. Flow Rate

40. Method ☐ Bubble ☐ PR

41. Initials

42. Date/Time

Post-Sampling Calibration Records

Post

43. Location/T & Alt.

44. Flow Rate Calculations

45. Flow Rate

46. Initials

47. Date/Time

Sample Weight Calculations

48. Filter No.						
49. Final Weight *(mg)*						
50. Initial Weight *(mg)*						
51. Weight Gained *(mg)*						
52. Blank Adjustment						
53. Net Sample Weight *(mg)*						

54. Calculations and Notes:

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Attachment C: OSHA Sample Report Sheet

Air Sampling Report U.S. Department of Labor Occupational Safety and Health Administration.

Page 1

1. Reporting ID	2. Inspection Number	3. Sampling Number

4. Establishment Name

5. CSHO ID	6. Sampling Date	7. Shipping Date	8.Date Result Received

9. Job Title	10.Occupational Code	11. Number Exposed

12. Frequency of Exposure

Exposure Summary

14. Substance Code	15. Rqstd	16. Smpl Type	17.Exp Type	18.Exp Level	19. Units	20. PEL	21. Adj	22. Severity	23. Citation information: No Cit	FTA	Over Exp	Eng	PPE	Trng	Med	OTI

TWA calculated on actual time sampled
The I. H. is free to make changes on the Form 91B and submit them directly to IMIS

26.Analyst's Comments
(Analytical Method)

27.Chain of Custody	Init.	Date
a. Seals Intact		
b. Rec'd In Lab		
c. Rec'd by Anal.		
d. Anal. Completed		
e. Calc. Checked		
f. Supr. OK'd		

28 Submission number

29 Lab Sample No. (Minutes/Type)

30. Analyte	31. Analysis Results/	32. Sample included in calculations of

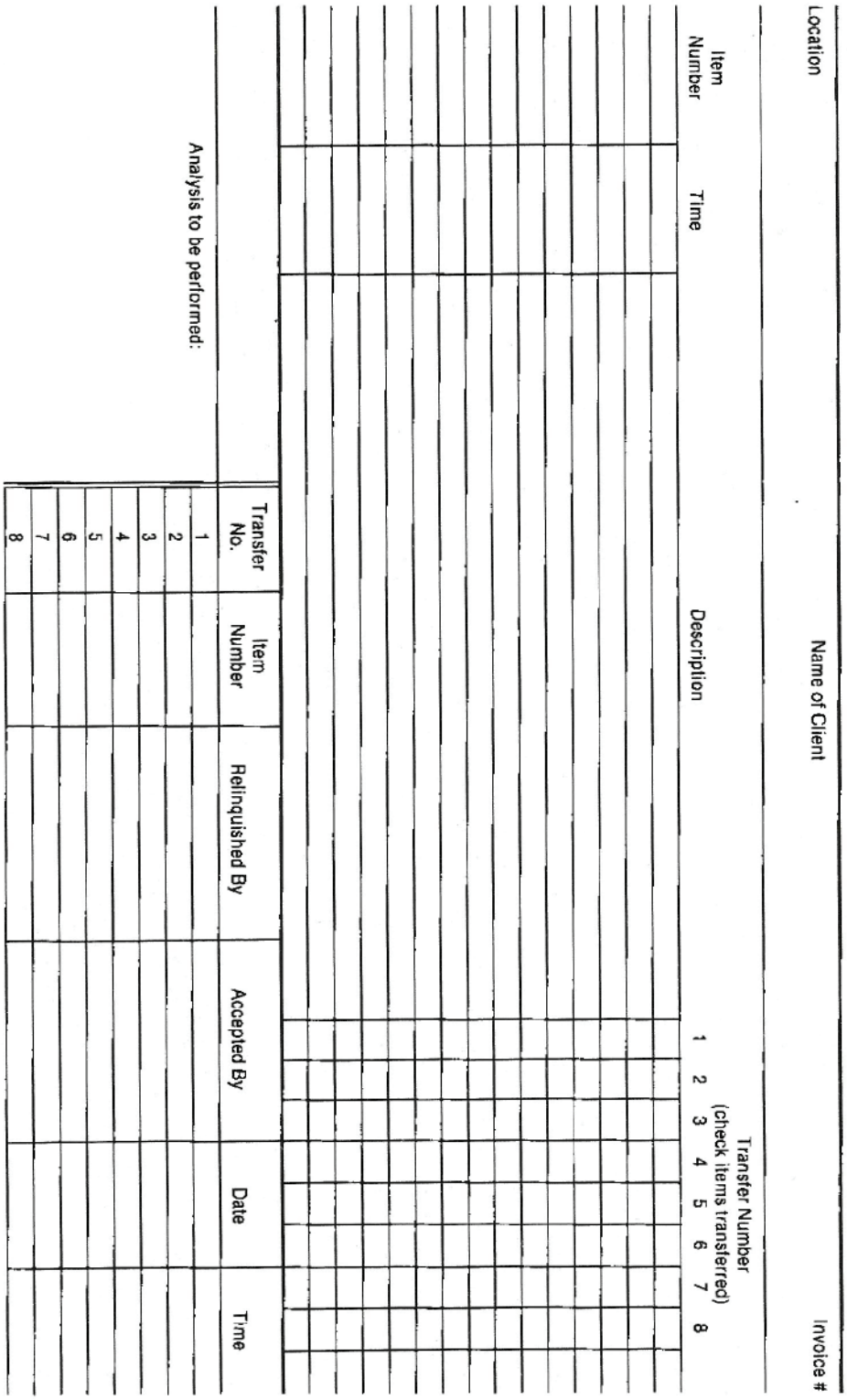
33. Analyte Code SAE Value

The Sampling and Analytical Error (SAE) is the current value for the specific chemical(s) and should be used for the calculations. Blank values are reported for reference only. Appropriate blank corrections have been applied to the samples by the Salt Lake Technical Center. Blank results are less than the reporting limit(s) unless otherwise noted.

L	MILLIGRAMS PER LITER (URINE)	D	MICROGRAMS PER DECILITER (BLOOD)
C	PICO CURIES PER LITER (RADON GAS)	P	PARTS PER MILLION
F	FIBERS PER CUBIC CENTIMETER	X	MICROGRAMS
M	MILLIGRAMS PER CUBIC METER	%	PERCENT
Y	MILLIGRAMS	E	FIBERS PER MM2
N	NONE	G	MILLION PARTICLES PER CUBIC FOOT (MPPCF)
BM/S	Bar Meters per Second		

ND The results are below the detection limits.

Attachment D: An example of a chain of custody sheet submitted with sample for analysis.



CHAIN-OF-CUSTODY RECORD

Location | Name of Client | Invoice #

Item Number | Time | Description | Transfer Number (check items transferred) 1 2 3 4 5 6 7 8

Analysis to be performed:

Transfer No.	Item Number	Relinquished By	Accepted By	Date	Time
1					
2					
3					
4					
5					
6					
7					
8					

Chapter 7
Intra-laboratory and Inter-laboratory Quality Testing

by Keith Nicholson, CIH

7.1 Introduction

Vital parts of the laboratory quality management system are the quality assurance (QA) and quality control (QC) programs. The QC program consists of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. The aim is to provide quality that is satisfactory, adequate, dependable and economical. The QA program is an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure a product or service meets defined standards of quality within a stated level of confidence.[1] The parts of a comprehensive QA program are discussed throughout this manual. To assure that these programs are functioning as desired, they are subjected to system and procedural audits by both internal and external auditors at specified frequencies with proper documentation. This chapter follows the policies outlined in ISO/IEC 17025 and AIHA-LAP, LLC Policies.[1,2]

7.2 Intra-laboratory Aspects

Intra-laboratory aspects include those which occur within the laboratory and involve the following:

- Use of standard operating procedures (SOPs)
- Calibration of instrumentation and equipment to ensure accuracy
- Duplication of analyses to ensure precision
- Analysis of known analytical standards
- Analysis of spiked samples to check for matrix interferences and to monitor accuracy
- Analysis of blanks to check for contamination
- Acceptance criteria for QC Samples
- Corrective actions for out of compliance QC samples

7.2.1 Standard Operating Procedures

Using SOPs helps to control the variability in the laboratory. These procedures describe the steps in a process and how each step should be accomplished. This provides greater assurance that different individuals will perform the process in the same manner and that the process will be performed the same way each time it is used.[1] The analytical method, consensus standards, and governmental methods websites can be useful in the development of SOPs.

SOPs can be used as training documents, simplifying the training of laboratory personnel, and serve as references if a question arises about the process. They provide a historical record of how a process was done at any given time. Whenever changes occur in a laboratory process, the associated SOPs are revised to reflect the changes. Copies of older SOPs should be maintained in order to preserve the historical record, but these copies must be stored in such a manner that they will not be used in place of a current SOP.[1]

Generally, a laboratory will use published methods to perform analyses. Often, the method may not specify the exact process to be used, or there are differences between the written

method and the way it is implemented in the laboratory. Deviations or clarifications to the method must be documented in laboratory records. It is prudent to document the laboratory's implementation of a method in an SOP.[1]

By standardizing procedures, individual errors that need to be controlled can be identified and minimized. These errors include: carelessness, lack of knowledge, calculation errors, use of contaminated or improper reagents, poor manipulative techniques, use of dirty glassware, poorly prepared standards, and improperly calibrated instruments.

7.2.2 Control of Reference Standards, Reference Materials, and Reagents

To provide a high level of accuracy, the chemicals and references used by the laboratory must be procured from vendors that have been approved by the laboratory and must be controlled from initial receipt through storage and use. Procedures must be in place for the storage and use of the materials in order to minimize degradation and contamination.[1]

A reference standard is an object that has a measured physical property (e.g., mass, length) determined to a stated uncertainty.[1] In an analytical chemistry laboratory, these include balance weights, thermometers, micrometers, and some timers. Their values must be traceable to NIST or other internationally recognized standards or equivalent. These standards should be handled with the utmost care in order to prevent damage that may alter the value. They should be recalibrated periodically. Reference standards should be initially and periodically calibrated by a calibration laboratory that is accredited to ISO/IEC 17025. Standards used on a routine basis should have their accuracy verified on a regular basis. Other references are not generally traceable directly to an ISO recognized supplier, or in the US, a NIST standard; rather they are traceable through other properties. For example, volume traceability of pipettes is often traced by weighing a dispensed volume at a known temperature and pressure and calculating the actual volume dispensed. The mass, temperature, and pressure are individually traceable to an ISO recognized supplier, or in the US, a NIST making the volume calculated traceable. It is important to document all reference standards and any recertifications which occur.[1,2]

A reference material is a material of sufficiently homogenous composition that has a physical (e.g., viscosity, particle size) or chemical (e.g., pH, constituent concentration) measured property determined to a stated uncertainty.[1] They are often diluted to known concentrations and then used to prepare instrument calibration curves. Documentation should show traceability to an ISO recognized supplier, or in the US, a NIST Standard Reference Materials® (SRMs), other national standards, or, if not available, to materials of the highest purity available. Certificates of analysis document the purity of the material, the concentration of the material, associated uncertainty, and traceability. Whenever possible these materials should be obtained from a supplier that is a reference material provider that is accredited to ISO Guide 34.[3]

A reagent is a chemical used in an analysis, but is not the specific analyte of concern. These chemicals should be labeled at the time of receipt with the date of receipt, the name or initials of the person who received it, and expiration or reevaluation date. Appropriate testing and proper care of all critical reagents should be carried out. All reagents must meet established method and QC parameters before being released for laboratory usage and records maintained of the receipt, including any quality certifications.[1]

Expiration dates provided by manufacturers of reference materials and reagents should be honored. In the absence of a manufacturer expiration date, the laboratory assigns an expiration date based on the stability of the material. Expired chemicals should be discarded or reevaluated. Expired chemicals are reevaluated in accordance with a documented procedure with specific pass/fail requirements and records of the reevaluation kept. Expired chemicals maintained for any reason are clearly identified as expired and removed from routine storage areas.[1]

Facilities must be available to store the reagents and reference materials safely and according to manufacturer or laboratory-established criteria—whichever is more stringent. Use of properly ventilated storage areas for organic solvents is very important. Chemical inventories must be maintained. A file of safety data sheets (SDS) for all hazardous chemicals used in procedures is required by law.[4]

Records of preparation and analysis of all calibration standards must be retained. Solution preparation conditions, reagent sources (e.g., lot numbers), and all assays should be traceable and documented. Reagents generated on site (such as purified water) must meet criteria appropriate for their intended use. Records of calibration standards must provide explicit traceability back to the parent reference materials.[1,2]

7.2.3 Control of Equipment, Instrumentation and Supplies

In order to produce reliable results, laboratory equipment and instrumentation must be maintained in accordance with manufacturer instructions and accepted laboratory procedures. Records need to be maintained for all instrument maintenance and for all calibrations or calibration verifications.[1]

Equipment that is used to measure volumes, masses, etc. must meet appropriate specifications when procured. If adjustment or change in the measuring system is possible, the accuracy of the equipment must be determined and documented on a regular basis. Common items that require periodic calibrations or verifications include balances, mechanical pipettes, ovens, and heating and cooling equipment. When possible, the calibrations or verifications should be done so that the results are traceable to an ISO recognized supplier, or in the US, a NIST SRMs® or other national standards.[1,2]

Instruments need to be calibrated using standards or reference materials of known property or concentration. These should be analyzed periodically to confirm the accuracy of a procedure. The calibration must be based on materials that are traceable to an ISO recognized supplier, or in the US, a NIST SRMs®, other national standards, or, if not available, to materials of the highest purity available.[1,2]

In general errors can be controlled by standardizing the reagents, using calibrated volumetric glassware and weights, recognizing and correcting personal bias (e.g., color estimation), eliminating chemical interferences, and correcting for physical influences, such as the effect of temperature and light in the visible and ultraviolet spectral region.

Various quality checks help to establish that the instrument is properly calibrated and that the sample preparation procedures did not introduce any errors. Quality checks are solutions of known concentration and/or analytical standards which are not specifically standards. These quality checks should have predetermined acceptance criteria and may include the following:

7.2.3.1 Initial Calibration Verification (ICV). A standard solution (or set of solutions) used to verify calibration standard levels. The ICV shall be prepared independently from the calibration standards (from a stock solution having a different manufacturer or different manufacturer's lot identification or as an independent preparation from a neat material).[1]

7.2.3.2 Continuing Calibration Verification (CCV). A standard solution (or set of solutions) analyzed periodically to verify freedom of excessive instrumental drift. [1]

7.2.3.3 Reporting Limit Verification (RLV). A standard that is prepared at or below the laboratory's reporting limit for the analysis to demonstrate that the instrumental setup for the analysis is capable of differentiation of the analyte at the reporting limit from that of the analytical baseline.[1]

7.2.3.4 Check standards can be used to monitor instrument performance over time. These are effective when the same instrument setup is used for the analysis. By recording and monitoring the results of the check standard analysis, subtle changes in the instrument performance can be observed and corrected.

7.2.3.5 Duplicate analyses can be divided into two types:

7.2.3.5.1 Replicates, where the sample is analyzed multiple times on the instrument in order to evaluate the precision of the instrument.

7.2.3.5.2 Duplicate samples, where a second sample is prepared and analyzed in order to evaluate the precision of the full analytical process. For industrial hygiene samples duplicate field samples are generally not possible, therefore duplicate QC samples are prepared and analyzed.

7.2.3.6 Blanks serve several purposes in the analysis.

7.2.3.6.1 Calibration Blanks are calibration standards with no analytes present, prepared in the same matrix as the calibration standards.

7.2.3.6.2 Calibration Verification Blanks (ICB and CCB) demonstrate that the instrument is able to return to baseline after the analyte is detected. They also provide a means to monitor instrument baseline drift.

7.2.3.6.3 Reagent Blanks are samples consisting of reagent(s), without the target analyte(s) or sampling media, introduced into the analytical procedure and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. The reagent blank is used to measure contamination contributed from the sample preparation, equipment, and reagents.

7.2.3.6.4 Method Blanks are unexposed sampling media or reagent(s), not taken to the field or shipped, but carried through the complete sample preparation and analytical procedure. The blank is used to assess possible background contamination from the analytical process.[1] These are reagent blanks that also contain the sampling media.

7.2.3.6.5 Other blanks can be introduced at any stage of the sample preparation process to demonstrate that analytes or interferences are not introduced by the sample preparation procedures, or to quantitate the apparent amount that is introduced.

7.2.3.7 There are numerous other checks that can be applied, depending on the instrumentation. These include, for example, various mass spectrometer tuning parameters and spectral interference checks for inductively coupled plasma instruments.

Supplies that affect the quality of the analysis must be procured from vendors that meet requirements established by the laboratory. The quality of the supplies must be verified and records maintained. Any certifications from the manufacturer are maintained as part of the record. For example, disposable volumetric ware must either be certified by the manufacturer to meet a specification, or the laboratory must verify that it meets pre-established criteria, generally by a statistical sampling. In either approach, documentation must be maintained showing that it meets requirements (see Chapter 4 Traceability of Measurement).[1]

7.2.4 Quality Control Samples

Quality control samples consist of media that have a known theoretical value, and mimic, as closely as practical, the samples submitted to the laboratory for analysis. The purpose of

these samples is to demonstrate that the analytical process produces an accurate result. They should be analyzed on a regular basis, and, preferably, with each batch of samples processed by the laboratory.[1,2]

Depending on laboratory policy, the theoretical values of the QC samples may or may not be known to the analyst. It is possible to occasionally introduce QC samples into the laboratory's sample stream as regular samples so that their presence is unknown, or blind, to the analyst.

The results from the analysis of QC samples provide the data used to establish control limits for the analysis, to evaluate analytical error, and to aide in the data validation process, as described in Chapter 10 of this manual.

QC samples are one aspect of the overall quality control program. The analytical methods, instrument calibration performance as compared to specifications and past history, the results of QC sample analyses, and the observations of a trained analyst all contribute to the quality of the result reported to the customer. When the result of a QC sample is outside of control limits, all aspects of the analysis should be considered in order to determine the source of the problem. The QC result failure starts the investigation, but it is the purpose of the investigation to determine the quality of the result being reported to the customer and to qualify the result, if necessary.

7.3 Inter-laboratory Aspects

In addition to internal quality controls, external controls are needed to ensure the analytical integrity of the laboratory and to provide greater uniformity throughout the industry. There are several methods by which this may be accomplished.

7.3.1 Proficiency Testing Programs

A laboratory may participate in a proficiency-testing program on a voluntary basis, with no interest in accreditation. Other laboratories may seek accreditation from AIHA Laboratory Accreditation Programs, LLC (AIHA-LAP)[5] and/or other accreditation entities, or may be required to become accredited under state or federal or contractual requirements.[1,2] These programs usually require the laboratory to demonstrate an acceptable level of performance through the analysis of known samples as part of a proficiency-testing program, such as the AIHA Proficiency Analytical Testing, LLC's (AIHA-PAT) Industrial Hygiene Proficiency Analytical Testing (IHPAT) or Environmental Lead Proficiency Analytical Testing (ELPAT) programs. Results indicate how well the laboratory is performing in comparison to other (reference) laboratories performing similar analyses. The laboratory must demonstrate a continued satisfactory level of performance to maintain accreditation. Many states require satisfactory participation in proficiency programs such as the EPA WP and WS series to be certified or accredited to perform analysis on drinking water and wastewater. These proficiency samples are also available to laboratories for use in their external QC programs without involvement with a state program.

7.3.2 Exchange of Samples with Other Laboratories

An excellent external QA/QC program includes periodically exchanging samples with one or more laboratories, and comparing the results. This is useful when a laboratory analyzes samples for which there is no proficiency-testing program. This method can also be used to supplement a proficiency-testing program. Care should be taken to ensure that no sample deterioration occurs during or prior to the exchange.[1,6]

Typically samples are prepared by a single laboratory and distributed to the other participants. The laboratory then receives the results from the participants and performs a statistical evaluation of the data. This responsibility can be rotated through the participants. The protocols for the exchange should be written and agreed upon by all of the participants.

7.3.3 Reference Laboratories

A third method for external quality control consists of sending duplicate samples to a commercial laboratory for analysis, and comparing their results with the results from the laboratory that originally analyzed the samples. A statistically significant number of samples for a given analyte must be submitted for this technique to be valid. If submitting duplicates collected in the field, evaluations must recognize variability in the collection, even if the samples are collected side-by-side.[1,6]

The reference laboratory should be one that consistently demonstrates a capability to analyze the desired types of samples with an acceptable degree of accuracy and precision. AIHA-LAP accredited laboratories are required to demonstrate an acceptable level of performance through the analysis of known samples, as part of the AIHA-PAT IHPAT and/or ELPAT programs. Results indicate how well the laboratory is performing in comparison with other laboratories performing similar analyses. The laboratory must demonstrate a continued satisfactory level of performance to maintain accreditation.

7.4 Audits

The purpose of an audit is to ascertain whether the system or process is being conducted according to established procedures and requirements. The audit can also serve as a means to evaluate the system or process and recommend improvements.[1,2]

Audits should be conducted by qualified personnel using a predefined checklist. As much as possible, the auditor should be independent of the operation undergoing audit. Internal audits should be conducted at least annually. External audits will be conducted in accordance with the external organization's policies.

Audit deficiencies should be entered into the laboratory's corrective action system to assure that the corrections are made and follow-up determination of the cause is scheduled, as appropriate. Any suggestions or "opportunities for improvement" should be carefully evaluated for potential preventive action.

It should be remembered that an audit is only a "snapshot" of the process. An audit finding might be isolated to that particular incident, or, it may be indicative of a problem that is more extensive. Additional audits may be warranted. The corrective actions taken in response to an audit finding should carefully consider the impact of the finding on the quality system.

7.4.1 Internal Audit

This is generally an audit performed by personnel from within the laboratory organization; however, the auditor could be a consultant to the laboratory.(1,2) These audits can be divided into two types:

7.4.1.1 Procedural audits, where an analyst is observed performing a procedure. The purpose of this type of audit is to determine if procedures are being performed in accordance with a standard operating procedure or method.

7.4.1.2 System audits, where the laboratory system is evaluated to make sure that it meets the requirements of a higher or external (e.g., customer, regulatory, or accrediting) organization.

7.4.2 External Audit

These audits are performed by an external organization to determine if the laboratory is operating in accordance with regulatory, contractual, or accreditation requirements.[1,2,6]

7.5 References

1. **AIHA Laboratory Accreditation Programs, LLC:** *AIHA Laboratory Accreditation Policies.* [Online] Available at: http://www.aihaaccreditedlabs.org (accessed June 2014).
2. **International Organization for Standardization:** General requirements for the competence of testing and calibration laboratories; ISO/IEC 17025: 2005. [Online] Available from http://www.iso.org/iso/home/store/catalogue_tc/catalogue_detail.htm?csnumber=39883.
3. **International Organization for Standardization:** ISO Guide 34 [Online] Available at http://webstore.ansi.org/RecordDetail.aspx?sku=ISO%20Guide%2034:2009&source=google&adgroup=iso9&gclid=CjkKEQjwwbCcBRCxvJn9-N6dorwBEiQAVriOigy2Hi-dwzLEFeEDfJtd0XT5C-ZX0ahN8q2WaFQ3FkEbw_wcB (accessed June 2014).
4. **Chapter 13:** *The Occupational Environment: Its Evaluation, Control, and Management*, 3rd edition, Anna, D. (ed.). American Industrial Hygiene Association. 2011.
5. **AIHA Laboratory Accreditation Programs, LLC:** AIHA-LAP Proficiency Analytical Testing Programs [Online] Available at: http://www.aihapat.org/Pages/default.aspx (accessed June 2014).
6. **American Society for Testing and Materials (ASTM):** *ASTM E691 Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method*. [Online] Available at http://webstore.ansi.org (accessed June 2014).

Chapter 8
Analytical Methods

by Dan Pastuf

8.1 Introduction

In all Quality System Programs, proper sample collection and analysis depends on several factors that should be taken into consideration during the planning, selection, validation, and routine use of the analysis method. This chapter provides guidance in the selection, validation, and documentation of analytical methods.

A complete analytical method consists of both sampling and analysis. The sampling aspect relates to the collection or separation of the component(s) of interest from the environment onto or in clean media, followed by transportation and storage. Analysis consists of sample preparation, measurement, and data reporting, with estimation of method uncertainty (precision and accuracy). Proper documentation of analytical methods, including the planning and selection process, validation, traceability, and revision history, is a pivotal part of laboratory quality assurance. AIHA Laboratory Accreditation Programs, LLC, has information on methodology, documentation, traceability, and Uncertainty of Measurement on their website.[1] These Policies follow ISO/IEC 17025 "General requirements for the competence of testing and calibration laboratories"[2], ILAC-P10 Policy on Traceability of Measurement Results[3], ILAC-G24 Guidelines for the determination of calibration intervals of measuring instruments[4], and CALA A61 CALA Traceability Policy.[5] Definitions of the terms used in this chapter can be found at these references and in the previous chapters in this book. Additional guidance on method development procedures can be found on the OSHA and NIOSH websites.[6,7]

8.2 Method Planning and Selection

8.2.1 Initial Planning

It is essential for the laboratory analyst to interact with field personnel and assist in identifying the proper collection method for the analyte(s) based on the data quality objectives (DQOs) of the analysis. The method must meet realistic expectations on sensitivity, specificity, accuracy, reliability, precision, interferences, matrix effects, limitations, cost, and timeliness. The factors to be included in the initial stages of methods development are varied and complex; some are often overlooked. A thorough assessment of the requirements placed on the data and method is essential to the production of a cost-effective method that produces the desired results. Some of the major considerations are discussed in this chapter.

8.2.2 Degrees of Confidence

The desired degree of confidence to be placed on the analytical method must be estimated for two categories: qualitative and quantitative.

8.2.2.1 Qualitative Degrees of Confidence

The degree of acceptable error in analyte identification should be considered. The following paragraphs give examples of qualitative degrees of confidence. When it is imperative that the identification has a high degree of certainty, the analyses should be confirmed using independent methods. For example, an analytical method by inductively coupled plasma atomic emission spectroscopy (ICP-AES) for the measurement of a metal might be verified with atomic absorption spectrometry (AAS). If the

requirements are less stringent, comparison of analyte analytical characteristics with literature values might prove acceptable (e.g., a gas chromatography [GC] retention index could be measured on one column and compared with those found on two other columns for identification of the compound of interest as compared with the more expensive identification on a mass spectrometer).

Another means of varying the degree of confidence is through the selection of the detection technique. If the analytical method can provide multiple characteristics per analyte, the degree of confidence will be greater than with a method that provides only one. For example, a high performance liquid chromatographic (HPLC) technique that uses the ratio of absorption measurements at two wavelengths has a greater degree of confidence than an HPLC method which gives the retention characteristic at only one wavelength. A more definitive identification could be achieved by a more expensive technique such as diode array or LC-mass spec.

The degree and type of sample preparation will have a significant role in identification, since selective extraction can limit the degree to which interferences will be present. For example, an extraction technique with a certain solvent may reduce the interferences to a closely related group of compounds of similar physical or chemical makeup. For example, choice of desorption solvent for a charcoal tube can affect the interference concentration if it does not desorb well with the solvent chosen for the analyte of interest.

8.2.2.2 Quantitative Degrees of Confidence

8.2.2.2.1 Sensitivity

For an analytical method, sensitivity refers to the ability of the method to detect small amounts of, or small changes in the amount of, the analyte of interest. For example, an analytical method able to detect or differentiate microgram quantities of an analyte would be more sensitive than a method whose ability to detect or differentiate milligram quantities. Sensitivity is not merely a function of how low the method detection limit is (such as 1 microgram versus 1 milligram in a sample), but also of how well the method detects small changes. The ability to distinguish between 1.0 and 1.1 micrograms of an analyte would denote greater sensitivity than the ability to distinguish between 1 and 2 micrograms. Greater sensitivity can provide greater confidence in evaluating data for such purposes as exposure assessment against a limit value. However, greater sensitivity often comes with greater cost, requiring evaluation of cost versus benefit.[6,7]

8.2.2.2.2 Specificity (Selectivity)

For an analytical method, specificity (or selectivity) refers to the ability of the method to respond uniquely to the analyte of interest; that is, its ability to accurately measure an analyte, both qualitatively and quantitatively, even in the presence of other, potentially similar, components. Important factors in determining method selectivity include freedom from interference by other components and good precision and accuracy.

One example involves the colorimetric determination and X-ray diffraction (XRD) determination of crystalline silica. The colorimetric determination is sensitive for a class of compounds—in this case all crystalline silica—while the XRD method is both sensitive and specific for crystalline silica in several forms, as quartz,, cristobalite, and tridymite. XRD analysis would be the preferred method because of its sensitivity and specificity.

Accuracy and precision are of prime importance in method selectivity. Standards used in determining unknown sample concentrations must be stable. Calibration standards that are not stable can still be used by applying derivatization if a stable derivative can be formed. For example, the standards could be injected onto the collection media that contains a derivatizing agent. For example, 2,6-toluenediisocyanate reacts rapidly with itself to form polymers, but can easily be collected as the monomer on a media containing a derivatizing agent specific for isocyanates, and the monomer is derivatized into a unique compound which has a good storage time. Results must be reproducible, accurate, and preferably exhibit linearity in the calibration curve over a defined working range. Certain atomic absorption (AA) data systems will automatically run in first-, second-, or third-order fit calibration modes. The operator must be aware of this and note any calibration problems, particularly if the system is running a third-order fit.[1]

A common means of determining the specificity of a given method is through comparison with an independent method—usually one that has a history of accuracy in determining true analyte content under field challenge conditions. The independent method may be one that uses a superior (or, at least, different) mode of detection. For example, electron microscopy may be considered an independent method in substantiating the presence of asbestos fibers on a filter that was analyzed originally by phase contrast microscopy.

Caution must be exercised when comparing results between two methods, since the independent method might not be as accurate as the trial method. As a rule, it is wise to verify both methods under field conditions, so that the results of the trial method can be compared with both theoretical and independent method results.

8.2.2.2.3 Analyte Concentration Range

A useful concentration or mass range for calibration and analytical method purposes is one that represents levels 0.2 to 2.0 times the target level of concern. Examples include the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Value (TLV®) or other appropriate exposure limit, the concentration range usually observed in the field, or the current regulatory limit for the target analyte.

Currently, the AIHA IHPAT[8] program has established mass ranges of metals to represent 0.5 to 2.0 times the TLV®, based on a 200 L air sample. The mass ranges of organic solvent vapors represent 0.1 to 2.0 times the TLV®, based on a 100 L air sample. Mass ranges may need to be adjusted depending on the sample volume. In the case of silica and asbestos, targets are designed to fall within the working range for the analytical method (currently <3000 fibers/mm^2 of filter surface for asbestos and 0.025–0.150 mg for silica [quartz]). Excluding asbestos, the relationship between the mass of analytes and the analytical results should be linear over the working range of the method.

Working ranges may vary with matrix. For example, the ranges for lead in paint, dust, and soil for ELPAT samples are 0.05% (w/w) -5% (w/w), 50–5000 ug/wipe, and 50–2000 ppm, respectively.

When the analyte concentrations are greater than the expected working range, the analyst must dilute the sample to bring it into the desired mass range. If the analyte mass is below the established working range, the

analyst must extend the lower mass limit by using additional analytical standards, if possible, or report the result as "less than" the lowest analytical standard used. Concentration preparations may be useful in achieving lower limits, but they also concentrate any matrix interferences present and may adversely affect accuracy and/or precision.

8.2.3 Method Selection

One of the more difficult problems of industrial hygiene and environmental monitoring may be the selection of an appropriate method. The analytical technique must be appropriate for the needs of the client, and the sampling technique must effectively collect the contaminant in a manner compatible with the analytical technique. In some instances, the client may specify or propose the method to be used. The lab must advise the client when a proposed method is not suitable or has been superseded.

The lab should employ recognized, published methods where possible, ensuring that the most current method is used. AIHA maintains a Field of Testing list that identifies published reference methods and governmental methods.[9] Alternatively, use of methods published in national or international standards is encouraged by ISO 17025.[2] Another source of methods is through AIHA's Industrial Hygiene Methods Exchange Network (IHMEN). The IHMEN has been developed by the AIHA Sampling and Laboratory Analysis Committee (SLAC) in order to share information on IH sampling and analysis methods that are not part of the public literature. A database of these unpublished methods can be found on the SLAC website.[10] Laboratory-developed methods may be used if they meet the client's needs, the lab has validated them, and they are demonstrated to be proficient. In any event, the client is to be notified of the method being utilized.

To choose the sampling and analytical method properly, one must know the physical state of the analyte and whether it might change state during the time between sampling and analysis. For example, will the solid that was sampled sublime or melt during transport? If so, then could it be shipped with a cold pack or overnight to prevent loss? Additional questions could be explored before the choice of sampling and analytical method is chosen.

The analytical capabilities of the laboratory and the technical expertise of the analyst must be taken into account. Does the laboratory have the equipment and instrumentation required to perform the requested analysis? Has the lab validated the method (See Section 8.3) and is it currently proficient in that area in its accreditation? (See Section 7.3)

The cost of analysis is often a significant factor, particularly if the monitoring has not been anticipated or budgeted for. Although often overused, the term "cost-effective" is appropriate. For example, if the cost of a particular method can be cut in half, then twice the number of samples can be analyzed for the same amount of money.

These considerations are difficult to balance, but must be considered when choosing a method. Ultimately, the chosen method must be fit-for-purpose to meet the stated DQOs.

8.3 Method Validation

Validation of analytical methods is essential to confirm their fitness for their intended use(s). The use of standard methods is strongly encouraged because there typically has been some degree of validation already performed. Even for standard methods, validation may be required if some aspect of how the method will be used, such as the sample matrix or the analyte concentration range, is outside of the scope of the standard method, or if there are anticipated issues (such as with interferences or method detection limits) not considered by the standard method. For modified standard methods, and for non-standard methods, validation is a specific requirement of ISO 17025.[6]

8.3.1 Validation Guidelines and Techniques

Method validation should be guided by a documented plan that defines testing conditions, types of samples, and success criteria (such as quality and performance objectives). These criteria typically include precision, accuracy, range, detection limits, sensitivity, specificity, repeatability, and robustness against interferences. Validation performance may include techniques such as calibration with reference standards; comparison of results with independent analysis methods and/or with other laboratories; and evaluation of the various factors (such as sample preparation, dilution factors, sample transport) to the method results or to method uncertainty.

8.3.2 Examples

Even accepted and documented methods must be validated for specific samples at times. Examples of method validation performed by the laboratory are listed below:

8.3.2.1 Generating and analyzing samples from test atmospheres to verify the accuracy of the method at various contaminant levels;

8.3.2.2 Determining desorption efficiencies over the concentration range of interest for analytes collected on a solid sorbent;

8.3.2.3 Verifying breakthrough volumes for analytes collected on solid sorbents or in impinger solutions under various conditions, such as at different temperatures and humidities;

8.3.2.4 Determining the storage recoveries for up to two weeks;

8.3.2.5 Generating and analyzing parallel samples to verify the precision of the overall method;

8.3.2.6 Checking the effects of expected sample interferences on the method; and

8.3.2.7 Using an alternate accepted procedure to verify the accuracy of the trial method.

If an accepted method must be modified to meet specific analytical requirements, the revision must be planned carefully. It must be approved by laboratory management (however titled), and must be documented in the QA program.[1] Implementing procedures should clearly state that the method is a modification (example: "This method is a modification of NIOSH Method 7903").

8.4 Method Document Control

Guidelines for the establishment of a policy for document control needs to be in the Quality Manual. This includes all method documents and their modifications, and all of the standard operating procedures (SOPs) used by the laboratory. The methods used by the laboratory for analysis of each parameter, including any modifications, and SOPs must be specified and approved by the laboratory director and/or supervisor. They must also have controlled distribution, so that each analyst has access to a copy of the currently approved method.

The active (i.e., in-use) analytical methods manual must be reviewed according to a documented schedule (annually to triennially would be appropriate). Revisions to active analytical methods require the same level of review and approval as the original method.
Outdated methods should be removed from the active methods manual and placed in an inactive methods file or archive.

Analytical methods should be numbered or otherwise coded to reflect method revisions. For example, Method 422.3 could represent the third revision of Method 422.

The analytical method should be listed with the laboratory data on the analytical report.[1]

8.5 Method Format

Every analytical method should contain certain elements to ensure the desired degree of precision and accuracy. The method documentation should include all templates used for method development and write-up and each template should have its own document control number. Also, every method must be clearly written, with sufficient detail, so that it can be readily followed by the user. Items that should appear in the procedure are discussed below. A consistent order for presenting these items should be utilized for all method procedures.

8.5.1 Method Title and Identification

Since the titl e may be used to catalog or reference the method, it should be brief, yet should contain enough information to allow classification by title alone. If it is not feasible to convey the desired amount of information in the title, an abstract may be added. Other information associated with the title should include a unique method identifier for unambiguous method identification, revision dates, authors, and approvals.

8.5.2 Principles

The underlying principles of analysis should be discussed briefly, even for analytical techniques that are widely known. These underlying principles should be discussed in greater detail for new techniques or applications of existing procedures. The principles should be described in sufficient detail for the analyst to estimate the specificity of the method for the analyte(s) of interest, as compared with other contaminants that might be present in the environment or matrix sampled.

8.5.3 Scope

A monitoring method should provide the user with the concentration or mass range over which the method is appropriate, the expected sample matrix, sampling media capacity, recommended flow rate, and recommended minimum and maximum sample volumes, etc., over which the method is valid.

8.5.4 Interferences and Special Precautions

A section of the method should point out all aspects of the protocol that might pose significant problems if not dealt with properly, such as chemical or physical interferences. Techniques that are particularly susceptible to errors should be discussed thoroughly in the context of the experience of the author(s) in eliminating or minimizing the causes of those errors (e.g., critical steps). Any special environmental conditions required to perform the method should be clearly identified.[1]

Health and safety precautions should be stressed. In some instances, they warrant special notation when particularly hazardous situations might arise during the application of the analytical method. Remember, all materials are toxic at some concentration.

8.5.5 Equipment, Reagents, and Consumables

The analysis method should include a listing of the analytical equipment (including instrumentation and labware), reagents, consumables, and reference materials required to perform the method. Sufficient detail should be provided to aid the user in assembling the required items, for example include model number, serial number and manufacturer of the instrument, supplier(s) and lot numbers of the reagents, etc.

8.5.6 Sampling

If not covered in separate sampling procedures, the analysis method should include details of how samples are collected, with special attention given to sample collection media (e.g., filters, impinger solutions, surface wipes, preservatives etc.) being utilized.

8.5.7 Sample Preparation

The method must include specific directions for sample preparation. These may involve several physical and/or chemical operations, each of which may add varying degrees of bias and/or lack of reproducibility to the analytical result because of contamination or analyte losses. For example, a contaminant may affect the qualitative identification of an analyte, just as it may affect quantitation. To trace the potential causes of bias and variance, sample preparation must be documented thoroughly.

Any special requirements for handling samples should be highlighted. In many cases, the sample itself may be hazardous. There may also be special requirements to maintain sample integrity.

The analytical method should specify how to correct the results for blank contamination. Representative field blanks should accompany all samples submitted to the laboratory. Abnormally high blanks should be investigated for acceptability on a case-by-case basis, and should be described in the report of results. Some analytical methods state that there is a high blank for that media, or circumstances which cause high blanks. The industrial hygienist should be instructed to avoid these circumstances which cause high blanks. Occasionally, a bad batch of media may cause high blanks. The manufacturer of the media should be contacted if this is suspected.

Analyte recovery should be addressed in the method. Recovery of the analyte from the sample matrix or sampling media is termed "extraction efficiency," or "desorption efficiency" if the analyte is adsorbed or absorbed onto the collection media. When recovery is significantly different from 100%, or when the laboratory QA/QC criteria indicate, a suitable correction factor may be applied if the bias is deemed correctible. Spiking procedures consistent with published requirements and manufacturer's recommendations should be included in the method, when appropriate.[1,6,7]

8.5.8 Analysis

8.5.8.1 Reagent Preparation

The method should provide detailed requirements for reagent preparation, or refer to other procedures that contain these requirements. Precautions for safe handling of carcinogens, teratogens, embryotoxins, etc., as well as flammable, toxic, or irritating chemicals must be highlighted.

8.5.8.2 Standards Preparation

Preparation of analytical standards used to calibrate analytical instrumentation should be described, in addition to limitations such as shelf life. Certification of highly purified reference materials (e.g., CRMs and SRMs) should be documented, along with all lot numbers of all the reagents used as required by ISO 17025[6] and AIHA policy.[1]

8.5.8.3 Instrument Parameters

Specific or typical instrument operating parameters must be identified so that the analyst can accurately and easily configure the instrument.

8.5.8.4 Calibration

Calibration is the process by which the response of the analytical device is measured at several analyte concentrations of synthetic standards. This should be documented in the method. The assumption is made that the chemical of interest will behave the same whether in a standard or a sample collected in the workplace. Accuracy of calibration is essential for both qualitative and quantitative analysis. Standards must be analyzed in the same time frame and under conditions identical to those used for the analysis of the sample (i.e., same acid concentration, pH, derivative reagent concentration, etc.). No analysis of samples shall be performed prior to calibration or calibration verification of the instrument.

A key purpose of calibrating an instrument or measurement system is to demonstrate traceability of the measurements to applicable primary standards. The analytical method should state, or reference, information which establishes this traceability. Typically, calibration of a working instrument would involve working standards or secondary reference materials which, through an unbroken series of comparisons, is linked to a primary standard (such as a certified reference material).[1,2]

The method of calibration should cover each type or group of similar standards. The calibration curve should consist of a minimum of a blank and three standards (unless otherwise determined) that bracket the expected sample concentration range. Reporting numerical data results beyond the range of the calibration curve, either greater than the highest standard or less than the lowest analytical standard, shall not be done since instrument response outside of that range may vary.

8.5.9 Acceptance Criteria

The analysis method should include criteria for accepting (or rejecting) the results of the analysis. For example, for the AIHA Laboratory Accreditation, LLC, Policies[1] requires documentation of acceptance criteria for determination of reporting limits, performance at the reporting limit, calibration curves and standards, calibration verification standards, and laboratory control samples. Additional guidance may be obtained from ASTM, EPA, NIOSH, OSHA, and other governmental websites.[6,7,11,12]

8.5.10 Data Recording

The analysis method should include information on what data are to be recorded and how results are calculated and reported. Any reviews of data prior to reporting should also be specified. These can be in hard copy or electronic record.

8.5.11 Method Uncertainty

The analysis method should provide an estimate, or steps for developing an estimate, of the uncertainty associated with the method. This may, for example, take the form of a statement of precision and bias, or it may be an analysis of uncertainty components, as described in the ISO Guide to Expression of Uncertainty in Measurement.[13]

8.6 Analytical Terms and Limits

8.6.1 Method Detection Limit (MDL)

AIHA's Environmental Lead Laboratory Accreditation Program (ELLAP) Specific Additional Requirements are described in Policy Module 2C. A requirement of these policies is the initial and annual (minimum) determination of MDLs for each method employed, according to 40 CFR Part 136, Appendix B. The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is

determined from analysis of a sample in a given matrix containing the analyte.[14]

For example, the MDL for lead in paint is the smallest measurable (non-zero) concentration of lead taken from a paint sample by a certain validated digestion/extraction and analysis method. Notice that there would be a different MDL for settled dust by wipe sampling, another for airborne dust collected using a filter cassette, and yet another for lead in soil, even if similar digestion/extraction and/or instrument analysis procedures are used for each. Each sample media matrix has a unique MDL given in units specific to the media, even if the analyte (Pb) is the same for each. (Note: A different wipe could/would have a different MDL, due to the matrix change.)Determination of the MDL involves preparation and analysis of spiked sampling media digestates/extracts, at low lead concentration. (Note: Liquid-standard spiking of clean matrix material is allowed for the determination of an MDL.) To determine an MDL based on the method found in 40 CFR Part 136, digest/extract and analyze a minimum of seven spikes with concentration no more than five times the resulting MDL (make a guess at the MDL), and determine the standard deviation of the results. The MDL is the standard deviation multiplied by a factor from the Tables of Students' "t" Values at the 99% confidence limit. For a set of seven replicates the factor is 3.143 and the calculation is demonstrated by Equation 1 (where MDL is method detection limit and SD is standard deviation).

$$MDL = 3.143\ SD \qquad \text{(Equation 1)}$$

Another method that may be used to determine an MDL, but does not require an estimate of the (actual) MDL can be found in several references and texts, e.g., Fundamentals of Analytical Chemistry by Skoog, Holler and Crouch (6th ed.).[15] This process involves analysis of the digestates/extracts from at least seven examples of the blank matrix. The standard deviation of the results is calculated and entered into a relationship (Equation 2), which considers degrees of freedom of the process.

$$MDL = t\ S\ [(Nj + N_b / Nj\ N_b)]^{05} \qquad \text{(Equation 2)}$$

where:

MDL = the method detection limit;
t = a factor from the Students' "t" Values, = 3.143;
S = the standard deviation of the concentration of lead found in the blank media digestates/extracts;
Nj = the number of times an unknown sample is to be analyzed,

where usually

Nj = 1;
N_b = the number of blank media digestates/extracts analyzed, where N_b s 7.

For example:

When Nj = 1 and N_b = 7, Equation 2 simplifies to Equation 3.

$$MDL = 3.360\ S \qquad \text{(Equation 3)}$$

Note that this approach gives a more conservative MDL than that from Equation 1.
AIHA Accreditation/EPA Recognition for analysis of environmental lead requires that the MDL for each matrix be determined at least annually.[12,14]

8.6.2 Instrument Detection Limit (IDL)

This is the lowest concentration at which the instrumentation can distinguish the analyte content from background generated by the matrix. It is usually determined by the manufacturer

for use in advertising and promotion. In atomic adsorption (AA) or inductively coupled plasma (ICP) analyses, the IDL can be determined from blank, acidified, deionized water as the matrix, with the same calculation methods used to determine an MDL.[16]

8.6.3 Lowest Standard Determined (LSD)
Lowest Standard Reported (LSR)
Lowest Standard Used (LSU)

These, along with several others not listed, are used to refer to the lowest non-zero concentration standard used in the instrument calibration process. The lowest- and the highest-concentration calibration standard define the endpoints of the calibration line. A laboratory may not report sample data beyond these values (i.e., extrapolation of a calibration line should not be allowed). AIHA Accreditation/EPA Recognition requires that no data be reported as either below the LSD or above the highest standard determined.[12,14]

8.6.4 Limit of Detection (LOD)

The use of this term is more a function of the context of its use than of a specific definition. In some conversations, the LOD is the IDL. In other conversations, it is the MDL. Care must be taken to remain aware of the topic under discussion when this term is used. For example, in conversations involving lead analysis, the LOD being discussed is most likely an MDL, since there are several different lead sample matrices to be considered. The units used for the LOD may aid in understanding what/which MDL is being discussed.[1,2,11,12,14]

8.6.5 Instrument Quantification Limit (IQL) and Method Quantification Limit (MQL)

Definitions of these terms are similar to the associated detection limits discussed above. The difference between the two terms is the presence of the sampling matrix in determination of the MQL. Actual quantification limits are expensive and time consuming to determine, and when determined are based on the statistics of measurement at a chosen confidence level (e.g., 95% confident that the result of a single sample analysis result will fall within ± 5% of the actual value). Most laboratories avoid defining the MQL, and utilize a Reporting Limit (RL; see discussion to follow) for data reporting.

8.6.6 Limit of Quantification (LOQ)

Use of this term is similar to use of LOD, in that it can change meaning as the discussion changes. Sometimes it is used as a substitute for IQL, and other times as a substitute for MQL. Common usage is as a substitute for MQL. As with IQL and MQL, most laboratories avoid defining them by utilizing a Reporting Limit (RL) for report of sample data. This needs to be determined for each method, for example for an ICP method this is typically 100 times the baseline noise. Parameters for this determination need to be documented in the method.

8.6.7 Reporting Limit (RL)

The RL is the lowest concentration of analyte in a sample that can be reported with a defined, reproducible level of certainty.[3] This value is used to replace the various quantification limits, and describes what a lab has chosen as the lowest analyte value they can confidently report for the matrix of interest. Laboratories can use any reasoning in the choice of the RL value, so long as the value chosen is used consistently and is not smaller than the lowest calibration standard. All sample analysis data then reported is equal to or greater than the RL, and thereby equal to or greater than the lowest standard. All reported data are therefore “on scale” or “within range” of the calibration line used in the analysis. Use of the RL in this way is acceptable and common practice. Note that for environmental lead, the RL must be at least twice the MDL.

8.6.8 Standard Reference Material (SRM)

These are primary standards, and are the top-of-the-heap as far as standards are concerned. They are the "gold standards" of the analysis industry. Primary standards should be obtained from ISO certified suppliers. In the US, SRMs are available from the National Institute of Standards and Technology (NIST), are of known and certified (the Certificate of Analysis is enclosed) concentration, are expensive, and are irreplaceable.[17] An SRM comes in a single, finite-quantity batch, which once gone is gone forever. Some SRMs have already been depleted, and are gone. AIHA, as an organization that accredits laboratories under the National Lead Laboratory Accreditation Program (NLLAP), and the EPA (the agency which "recognizes" laboratories as able to perform environmental lead analyses) "encourage" laboratories not to use SRMs as LCSs. SRMs are to be used to verify other (i.e., secondary) standards. Conservation of these assets is considered a good thing.

8.6.9 Laboratory Control Sample (LCS)
Laboratory Control Standard (LCS)

An LCS is a reference material, based on the same matrix as the samples being analyzed (e.g., use a paint dust-on-wipe LCS when dust wipes are being analyzed). It is a secondary standard established concentration independent of the instrument calibration and traceable to a primary standard. A primary standard should not be used as an LCS. The digestion/extraction and analysis of a LCS is performed to demonstrate the performance of the lab as compared to performance of other laboratories performing similar analysis. Comparison of analysis data to the LCS quoted value is a measure of the inter-laboratory variation. AIHA Accreditation/EPA Recognition requires statistical reduction of LCS analysis data.

8.6.10 Certified Reference Material (CRM)

In general, these are secondary standards that are of like-matrix to the samples being analyzed and have a certificate from the manufacturer/supplier that states the matrix and analyte content, the method used to analyze the material, and the NIST SRM, or ISO certified reference material, it was analyzed against. In common language, in the US the manufacturer/supplier has "certified" that this standard is "traceable" to a NIST SRM by some process.[17] There is much discussion about the traceability process, and many and varied concerns as to the viability of such processes. From these discussions, the liability for the quality of a CRM falls to the manufacturer/supplier, but the responsibility for verification of a secondary standard falls to the laboratory. Any and every secondary standard should be verified prior to use, either by comparative analysis to another secondary standard in current use, or by analysis comparison to an SRM (the best approach).

8.6.11 Precision

Precision refers to how well the individual measurements, in a group of measurements of the same parameter or property, agree with one another. Typical expressions of precision include standard deviation (absolute or relative), variance, or range.[3]

8.6.12 Bias

Bias refers to a consistent deviation, either positive or negative, from a known true value.

8.6.13 Accuracy

Accuracy refers to how well an observed value agrees with a known or accepted value, and includes a combination of precision and bias.[3]

8.7 Summary Discussion of Detection and Reporting Limits

Consider the following relationships and generalities.[17]

IDL < MDL

The instrument detection limit (IDL) is always smaller and usually very much smaller than the method detection limit (MDL) because there is a matrix present for the latter.[6]

MDL < MQL

The MDL is always smaller than the method quantification limit (MQL). Often, the MQL is significantly (2 to 10 times) larger than the MDL.

IDL < MDL < MQL<LOQ < LSD < RL

The IDL is always smaller than the instrument quantification limit (IQL).

The IQL is always smaller and usually very much smaller than the method quantification limit (MQL).

The lowest standard determined (LSD) could be equal to or larger than the MQL, but never smaller. More often than not, the LSD is significantly larger than the MQL.

The current AIHA LAP policy is the reporting limit (RL) should be at least twice the MDL.

Many laboratories use the LSD as the RL.

8.8 Quality Control Sample Acceptance Criteria

QC Samples are needed to verify the analytical data from a validated method that is being used for production samples. The analyst must have a set of acceptance criteria that can be used to make up the QC samples, analyze them, and determine if the results are acceptable. The ISO Guide to the Expression of Uncertainty in Measurement (GUM) can be useful in determining acceptance criteria.[13] The following table is an example of how these criteria can be translated into a method of practice.

Table 8.1 — Example QC Table.

Sample Type	Frequency	Spike Level	Acceptance Criteria	Corrective Actions

8.9 References

1. **AIHA Laboratory Accreditation Programs, LLC:** *AIHA Laboratory Accreditation Policies*. [Online] Available at: http://www.aihaaccreditedlabs.org/2013PolicyModules/Pages/2013AccreditationPolicies.aspx (accessed December 2013).
2. **International Organization for Standardization:** General requirements for the competence of testing and calibration laboratories; ISO/IEC 17025: 2005, ISO 9000 Certification Standards. [Online] Available at http://www.iso.org/iso/iso_catalogue.htm (accessed December 2013).

3. **International Laboratory Accreditation Cooperation (ILAC):** *ILAC Policy on Traceability of Measurement Results*. [Online] Available at: https://www.ilac.org/486.html (accessed December 2013).
4. **International Laboratory Accreditation Cooperation (ILAC):** *Guidelines for the Determination of Calibration Intervals of Measuring Instruments*. [Online] Available at: https://www.ilac.org/guidanceseries.html (accessed December 2013).
5. **Canadian Association for Laboratory Accreditation Inc.:** *CALA Traceability Policy CALA A61* [Online] Available at: http://www.cala.ca/A61-ALA_Trac.pdf (accessed December 2013).
6. **U.S. Occupational Safety and Health Administration (OSHA):** *Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Methods*. [Online] Available at: https://www.osha.gov/dts/sltc/methods/chromguide/chromguide.pdf (accessed June 2014).
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Chapter 9
Equipment Calibration and Maintenance

by Mary E. Eide

9.1 Introduction

Accurate calibration and careful attention to the maintenance of all equipment are essential components of a laboratory QS program. The Quality Manual should have policies for documentation of all calibrations and maintenance of each piece of equipment in the laboratory. The equipment log should include a chronological record of preventative and emergency maintenance performed on any equipment. The logs include a record of calls, service technician summaries, records of calibration by the manufacturer, routine user maintenance, and other information as required by these policies. Equipment logs are kept for at least 3 years. The air sampling equipment may be calibrated with a primary calibration instrument or by a secondary calibration instrument. The air sampling equipment calibrated from a secondary calibration instrument should have traceability to a primary calibration instrument. The National Technology Transfer and Advancement Act of 1995 established a common testing strategy for commercial and federal agencies.[1] The U.S. Government published the National Standards Strategy for the United States in 2000, which made American National Standards Institute (ANSI) as the lead agency for establishing the testing methods.[2] Most ANSI and American Society for Testing Materials (ASTM) methods for calibrating instruments specify traceability to a National Institute of Standards and Technology (NIST) primary calibration instrument. Outside the US, the ISO standards specify traceability to an ISO certified supplier. For many instruments, the primary or secondary calibration equipment is traceable to NIST following procedures by American National Standards Institute (ANSI), American Society for Testing Material (ASTM), and Instrument Society of America (ISA). Global bodies for standardization and standards development are the International Electrotechnical Commission (IEC) and the International Organization for Standardization (ISO). The IEC provides technical specifications and publishes international standards for electrical, electronic, and related technologies. ISO provides procedural guidance and fills the need for standards outside of the electrical and electronic disciplines, and includes members from all over the world, including the United States. European standards development bodies also exist including the Comité Européen de Normalisation (CEN) and the Comité Européen de Normalisation Electrotechnique (CENELEC).

Traceability requirements for AIHA accreditation require the maintenance of accurate records of all calibrations and calibration verifications of all instruments and equipment used in an accredited laboratory. These records must be traceable to the serial number of the instrument or equipment. The records must be retained for at least the lifetime of the equipment or longer as required by the laboratory accreditations, and if there are legal requirements for a longer retention period. An example of records which must be retained past the lifetime of an instrument is any sample which may be potentially involved in litigation. AIHA Laboratory Accreditation Programs, LLC has information on Traceability on their website.[3] The AIHA Environmental Lead Laboratory Accreditation Program (ELLAP) requires retention of records for 5 years after the use.[4]

9.2 Sampling and Analytical Instrumentation

The accuracy of laboratory measurements can be no greater than the accuracy of the equipment with which the samples are taken and analyzed. It is critical for all such equipment to be

calibrated accurately and maintained properly. Some examples of the equipment that should be considered are listed below, but should not be limited to those listed as new equipment is produced each year by manufactures and must be included:

9.2.1 Sampling pumps

9.2.2 Soap bubble (frictionless piston) flowmeters

9.2.3 Digital flowmeters

9.2.4 Wet test meters

9.2.5 Dry test meters

9.2.6 Rotometers

9.2.7 Thermometers

9.2.8 Vacuum and pressure gauges

9.2.9 Microscopes

9.2.10 Balances and weights

9.2.11 Volumetric glassware

9.2.12 Digital burettes and pipettes

9.2.13 Analytical field instruments

9.2.14 Gas chromatographs (GC)

9.2.15 Infrared spectrophotometers (IR)

9.2.16 Ultraviolet/visible spectrophotometers(UV/Vis)

9.2.17 Atomic absorption spectrophotometers (AA)

9.2.18 Inductively coupled plasma atomic emission spectrophotometers (ICP-AES)

9.2.19 X-ray fluorescence spectrophotometers (XRF)

9.2.20 High pressure liquid chromatographs (HPLC)

9.2.21 Ultra-high pressure liquid chromatograph (UPLC)

9.2.22 Ion chromatograph (IC)

9.2.23 Gas Chromatography mass spectrometry (GC-MS)

9.2.24 Inductively coupled plasma mass spectrometry (ICP-MS)

9.2.25 Capillary Electrophoresis (CE)

The calibration of air sampling pumps is performed before and after each episode of sampling with the media in line, as the media provides resistance affecting the flow rate. Do not use the same individual media for the calibration and for sampling as the individual media may become contaminated during calibration from chemicals in the calibration room. ASTM D5337-04

Standard Practice for Flow Rate for Calibration of Personal Sampling Pumps provides a guide to the calibration of air sampling pumps.[5] Aerosol air sampling pumps should be calibrated following ASTM Standard D6061-01 (2007) Standard Practice for Evaluating the Performance of Respirable Aerosol Samplers.[6] OSHA sampling methods require the air sampling pumps perform within 5%, as a performance criteria of 5% is used in their calculation of the Sampling and Analytical Error (SAE).[7] U.S. Environmental Protection Agency (EPA) has specific requirements for air quality monitoring found in 40 CFR Parts 50, 53, and 58. Part 50 is the National Primary and Secondary Ambient Air Quality Standards, Part 53 is the Ambient Air Monitoring Reference and Equivalent Methods, and Part 58 is the Ambient Air Quality Surveillance.[8,9] Records should be kept of the pre/post calibrations of each sampling episode. When post-calibration results differ from pre-calibration results by more than 5% the validity of the samples are in question and samples should be retaken. When post/pre-calibration results differ by less than 5% the flow rate used for total volume calculations will be determined by professional judgment. For most instances use the average of the pre and post calibrations.[10] By using the lower flow rate number the calculation errs on the side of increased worker protection. For enforcement purposes the larger flow rate must be used as it errs on the side of the employer.[6]

European Standard EN 1232 has performance criteria for battery powered air sampling pumps with nominal volumetric flows of 5 mL/min to 5 L/min along with laboratory testing methods for pump performance tested under specific laboratory conditions.(11) EN 12919:1999 has performance criteria for pumps with nominal flow rates over 5 L/min.[12]

To adequately establish an audit trail for traceability, ISO and NIST recommends that a proper calibration result include (1) the assigned value, (2) a stated uncertainty, (3) identification of the standard used in the calibration, and (4) the specifications of any environmental conditions of the calibration when correction factors should be applied if the standard or equipment were to be used under different environmental conditions.[13] Records of the calibration of the secondary standards and its primary standard should be maintained together to provide the proper traceability of the equipment calibration. Any manufacturer's certificates of traceability to ISO or NIST certification should be added to the audit trail of a particular piece of equipment. AIHA ELLAP requires retention of these records for 5 years after use[4], and legal requirements may require retention for a longer period of time if the samples are involved in litigation.

The primary and secondary calibration standards which can be calibrated following ASTM D1071-83(2008) include: cubic-foot bottle, immersion type of moving-tank type; portable cubic-foot standard (Stillman-type); fractional cubic-foot bottle; burettes, flasks, and other volumetric measuring devices; calibrated gasometers (gas meter provers); gas meters (displacement type) liquid-sealed relating drums; gas meters (displacement type) diaphragm- or bellows-type meters, equipped with observation index; gas meters (displacement type) rotary displacement meters; gas meters (rate of flow) porous plug and capillary flowmeters; gas meter (rate of flow) float (variable area, constant head); orifice flow nozzle; and venturi-type flow meters.[14]

The International Organization for Standardization (ISO) series 9000 standard requires that all measurements that affect quality shall be calibrated at prescribed intervals with certified equipment having a known valid relationship to nationally recognized standards. Certification to the ISO 9000 Quality System Standards is primarily in reference to the global business environment but also has an impact on calibration laboratories.[15] The ability to document equipment traceability will no doubt take on greater and greater significance as organizations develop international traceability standards.

9.3 Instrument Maintenance

Each instrument should have its own separate maintenance log. Key information documented in this log should include the information listed, as applicable to instrument design, construction, etc. Below is a list of information which should be included in the maintenance log:

9.3.1 Instrument name

9.3.2 Manufacturer name, address, and contact telephone number

9.3.3 Model number

9.3.4 Serial number

9.3.5 Date of purchase

9.3.6 Location or site where stored, kept, or secured

9.3.7 Method of calibration/calibration verification

9.3.8 Calibration/calibration verification schedule

9.3.9 Date of last calibration/calibration verification and the person performing it

9.3.10 Dates of previous calibrations/calibration verifications and the persons performing them

9.3.11 Dates of all maintenance procedures, an account of what was performed, and by whom the maintenance was performed

9.3.12 Records of dates of outside service, accounts of what was performed, and by whom the service was performed

9.3.13 Telephone number(s) for and names of service personnel

Whenever possible, analytical standards used should be ISO or NIST traceable or a comparable authority. When this is not possible or feasible, the most reliable available standard should be used to minimize overall method error. It may be necessary for the analyst to make the calibration standards using reagent grade chemicals or chemicals with certificates of analysis. The interval between instrument calibrations is best guided by the manufacturer's instructions, but this may be modified by user experience and frequency of instrument use, the type of analysis being performed and instructions in the method of analysis being used, or the need to conform to any requirements set forth by accrediting organizations or regulatory agencies. The calibration of analytical instruments is verified throughout each analysis by the routine use of Continuing Calibration Verification (CCV) standards as prescribed by the analytical method, accreditation requirements, or laboratory policies.

To the extent it is practical, a good supply of spare parts should be kept on hand to promote quick repairs and reduce instrument downtime. Recalibration after maintenance or repair might be necessary, depending on the maintenance and repairs performed.

9.4 Glassware and Volumetric Apparatus

Cleanliness of the glassware used is critical. Since industrial hygiene and environmental chemistry generally involve analysis of microgram (µg) quantities or less, it is extremely important that glassware and other containers used in the analysis be scrupulously clean. Beakers and other glassware should be washed, or at least rinsed out, as soon as possible after use. Soaking glassware heavily contaminated with metals in a 10% nitric solution overnight before washing can aid the cleaning process. Salt deposits should never be allowed to dry onto the walls of containers. In routine practice, labware should be washed thoroughly with a high quality, non-contaminating, warm detergent solution, rinsed thoroughly with tap water, then rinsed with distilled or deionized water, and heat or air dried.

To ensure the removal of traces of heavy metals such as lead, cadmium, or mercury, glassware should be rinsed in nitric acid, followed by several rinses of distilled or deionized water prior to analysis for these elements. Trace organic contaminants should be removed by pre-rinsing glassware with the solvent used in the extraction or desorption portion of the analysis. If this is not possible, glassware should be soaked in a surfactant solution, which will dissolve the contaminants, overnight before washing. The washing procedure should be documented as part of the laboratory's QA/QC program.

Analytical accuracy depends highly on the accuracy of the volumetric flasks, pipettes, micropipettes, etc., with which samples, spikes, and standards are prepared. Only Class A quality volumetric glassware should be used when the highest degree of accuracy is needed.

Pipette, micropipette, and syringe calibration should be verified by mass. The pipette is filled to the mark with water, which is then allowed to drain into a tared container on an analytical balance capable of weighing to the nearest 0.1 mg, for pipettes holding 100 mL or less. The measured weight of the water is divided by the density of the water at the temperature of use to calculate the actual volume of the pipette using the following equation. These calibrations should be performed initially upon receipt and on a periodical schedule. All records of these calibrations should be readily accessible and retained the required period of time.[3,4]

$$Volume\ (mL) = \frac{w}{d}$$

where:

w = mass of water in grams
d = density (grams/mL @ T°C
T = temperature of the water

9.5 Electrical Power Source

For laboratory equipment to function properly, a good source of electrical power should be available. Some instruments might even need a constant voltage transformer. A stable power source is critical for instruments containing integrated circuits.

Intermittent transient spikes in line voltage could cause serious problems, including circuit failure. To avoid these problems, isolation transformer/voltage regulators should be used to isolate the electronics from power line variations. Transient protection may also be needed. Some newer instruments already incorporate these features in their instruments. The ultimate protection comes from a high quality Uninterruptible Power Supply (UPS) that has internal isolation circuitry. The manufacturer should be consulted for the specific power requirements for their instrumentation.

Certification of equipment may be done by NIOSH, Mine Safety and Health Administration (MSHA), or by a contract testing laboratory such as Underwriters Laboratories (UL), or by a third party testing such as Safety Equipment Institute (SEI). These organizations may certify the equipment or oversee part or all of the assembly process. Outside of the U.S. third party certification is usually performed to see if the equipment conforms to the performance standards of that particular country, and may include an audit of the manufacturer's quality management system to ensure each piece of equipment conforms to that country's performance standards. In Europe the third party certifies the instrument and places the certification mark of Conformite' Européen (CE) on the instrument. Equipment can also be internationally certified by ISO.

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Chapter 10
Data Validation and Interpretation

by Jeffery A. Cooper

10.1 Introduction

An evaluation of the reliability of analytical results is described in this chapter. The process involves various statistical techniques that put the data in context (i.e., do the results appear reasonable?). Based on the evaluation, results are accepted, or corrective actions are taken.

Data validation and interpretation refer to the process of evaluating the results of sample analysis. Data validation is an attempt to prevent the release of incorrect data. The process serves as a final screen before the analytical results are used in making decisions. It is important that data validation be performed as soon as possible after the data collection to facilitate timely corrective actions.[1–3]

10.2 Data Review Procedure

The request for analysis submitted to the laboratory with the samples and all data generated by the laboratory must be reviewed after analysis and before releasing a final report to the customer. The report released to the customer should identify both the analyst(s) and reviewer(s), and be signed by someone with the authority to release data. The entire review process should be documented. The following factors should be reviewed[1–3]:

10.2.1 Proper collection of the sample took place. Verify that the proper sampling media and the recommended sampling method were used.

10.2.2 Ensure that the sample was analyzed correctly. Verify that the method was appropriate for the analyte, was capable of quantifying results at the level of the reported analytical result, and that the results were not affected by interferences.

10.2.3 Review the summary of the sample information. All the pertinent information on a sample should be recorded in one place, including sampling description and analysis parameters. Examples of sampling information should include start and finish sampling time, sampling device used, air sampling device flow rate, date, and location, if known to the laboratory. Examples of analysis information include method, date, aliquot size, dilutions, desorption solvent, extraction/desorption time, and instrument parameters.

10.2.4 Review the calculations for accuracy. Calculate the results from the original data to ensure that the proper calculation was performed. The data should be displayed in an orderly fashion with enough information to explain the calculation.

10.2.5 Check for accurate recording of data and its transferal from the primary data form to the final report. Transcription of data must be done carefully and accurately.

10.2.6 All chromatograms or recorder printouts should be included with the data. Check to see that all QC work had been done and is within acceptance limits.

10.2.7 All data pages should be signed and dated by the analyst and the chemist or supervisor responsible for the analyses, and by the reviewer and/or approval authority. This double checking should catch errors before reports are issued.

10.3 Quality Control Charts

QC charts are used to reveal variation in analytical performance from an established historical record for the same procedure. Several methods for the use of QC charts, including selection criteria, are included in References 1 through 4 of this chapter. A comprehensive approach has been developed for a broad range of sample procedures for large workloads[5], and a helpful guide to the use of control charts has been published by the Ford Motor Company.[6]

Accuracy and precision control charts are separate documents that work together to monitor the reliability of the analytical method. Control charts are a quick way to detect changes or trends in accuracy and precision. These tools can be used to identify assignable causes such as systematic bias or random errors. These techniques allow for control of the uncertainty involved in interchanging operators and instruments. They may help to reduce the need for repeat measurements and enhance the reliability of the data generated by the laboratory. Without an indication of reliability, it is difficult to support analytical results and the judgments based on them.

Control charts can be used to differentiate between systematic (determinate) and random (indeterminate) error. Control charts indicate the variation in the analytical results. They can be based on a number of parameters (e.g., the range or standard deviation of replicate field sample analyses may be used). Figure 10.1 is an example of a range/mean vs. analysis number plot. It shows the variation in the range/mean ratio for duplicate analyses. (There will be no results less than zero, of course, since the range cannot be negative. The plot was generated by a computer program.)

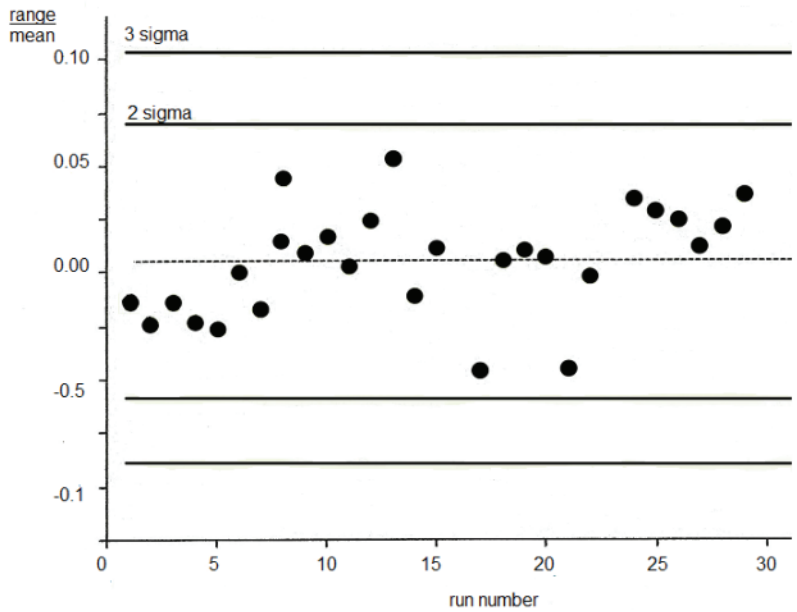

Figure 10.1 — Plot of duplicates-range variation of a formaldehyde in 1% sodium bisulfite (sigma = standard deviation)

For "real world" samples, however, where there are wide variations in the concentrations of the samples, the range or standard deviation can show wide variations. The range divided by the mean (R/X) or the coefficient of variation (CV) can be used to eliminate this problem. Plotting these measures of variation allows precision control over a greater range if field sample results are used to establish control limits. A check of homogeneity of imprecision can be made by measuring the RSD.

Usually, X bar (average) charts or X bar and R (range) charts are maintained for routine work.[2–6] Once control charts have been established, laboratory-specific acceptance criteria must be determined, and defined to assist the analyst in making a "go/ no-go" decision. These criteria must be established by laboratory management and must be documented for each analysis performed. Such documentation should include actions to be taken if a suspicious trend develops or an outlier is obtained. QC samples should be analyzed prior to the analysis of samples to ensure that the reagents and instruments are performing as expected. QC samples should also be randomly placed within a batch of samples to ensure continuing control. Control criteria must be satisfied before samples are analyzed.[2,3,4,5,6]

A synthetic "lab standard" may be prepared, matching the analyte matrix as closely as possible. The lab standard is analyzed 30 times for temporary control limits and 60 times for permanent control limits for the control charts. As a general rule, the warning limits are set at two standard deviations (± 2 s.d.) from the mean of the standard results, and the control limits are set at three standard deviations (± 3 s.d.) from the mean of the standard results. If the plotted value is outside the warning limit but inside the control limits, the analysis may be continued, but the possible sources of error must be evaluated. If the value is outside the control limits, the analysis must be discontinued and corrective actions taken and documented. Then the control standard sample must be rerun to verify return to control.[8]

Variations in precision may be caused by a number of factors. Some of the more common factors include different analysts, equipment, reagents, daily variations, and sample heterogeneities. Possible problem trends can be predicted by several consecutive points on the chart showing consistent movement away from the mean value line. As a rule of thumb, seven points on the same side of the mean line indicate that likely there is a bias ($P < 0.01$). These variations may be the result of reagent degradation, sample handling, a systematic analytical variation, or equipment malfunction.

The Youden two-sample plot (see Figure 10.2) is another way to evaluate precision.[12] The two-sample plot is a plot of one sample result against another. It has the advantage that both random and systematic errors can be observed. Ideally, the point plotted should lie along a diagonal line (slope = 1) intersecting zero. The distance of the point from the diagonal line is an indication of the random variation. The distance of the point up or down the line from a known target value is an indication of the systematic bias of the results.

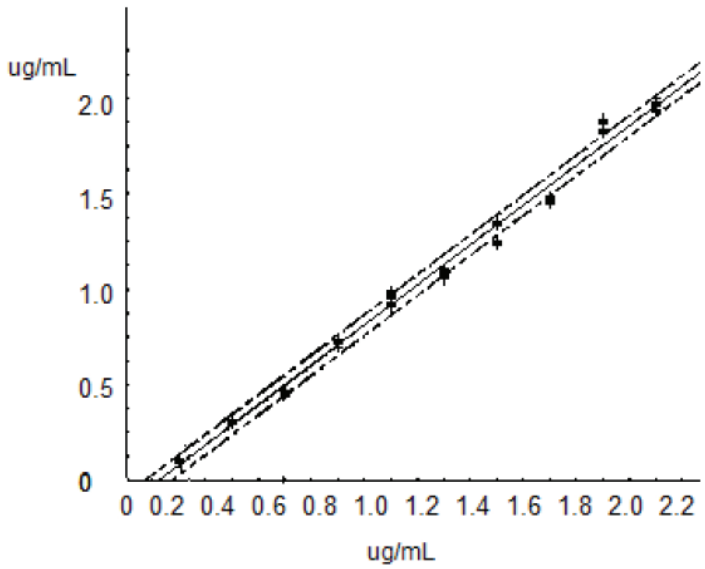

Figure 10.2 — Youden two-sample plot of formaldehyde in 1% sodium bisulfate, Duplicate A vs. Duplicate B including regression with 99% confidence limit lines drawn.

The cu-sum (cumulative sum) chart is another method used to demonstrate analytical precision. It is much more sensitive to variation than the other charts because it uses the square of the difference between duplicate values. It also requires that if the result lies outside the control limits, the analyst must stop and start a new chart to get the point within the control limits. This forces the analyst to stop and evaluate the problem.[4]

10.4 Occasional Samples

Maintaining the quality of analysis of occasional samples, those analyzed between once and four times a year, does not lend itself easily to statistical analysis; however, procedures have been developed for the evaluation of quality. A combination of spiked and replicate samples is necessary for establishing the quality of results.[7]

Once reproducibility has been established, it is important to be able to demonstrate the accuracy of that result. This can be done by using a control chart of the percent recovery of a sample of known concentration with either a known reference material or a spiked field sample. The spiked field sample analysis demonstrates accuracy within the matrix of the sample. The analysis of the standard reference material gives an absolute indication of the method accuracy. Trends in the accuracy of results on the control chart may be caused by degradation of the standards or reagent or by a systematic bias due to the instrument or personnel.

10.5 Statistical Analysis for Quality Control of Precision

Most Laboratory QC Managers are familiar with the techniques for generating traditional control charts for reference and spike samples by taking the mean and standard deviation of the data, and plotting warning and control limits at +2σ and +3σ respectively (*see* Figure 10.3).[1,4,8,10]

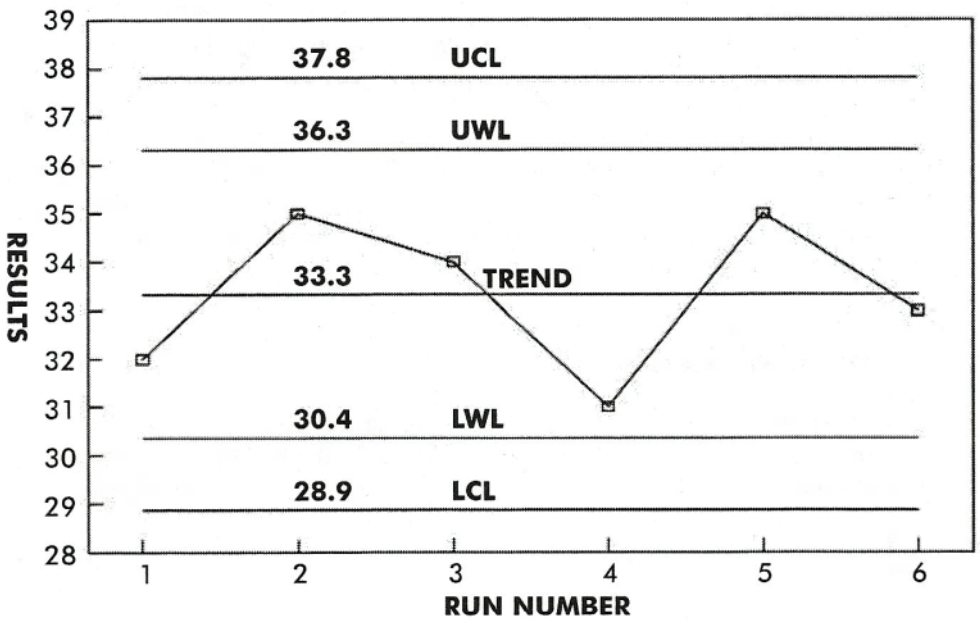

Figure 10.3 — Reference Control Chart

Establishing and plotting control limits for duplicate data, however, is another matter entirely. Here, we see what is essentially the same approach; calculating the mean and standard deviation of the difference, or relative percent difference (RPD), between the two results, then setting up control limits as one would for reference and spike samples. Although this approach is

widely used, it is wrong as there is not multiple analyses on differing days so no trending can be calculated.

There is no significance to using the mean of the RPD as a mean because there is no trend line for duplicate data. If there is one, it would be zero. Instead, the RPD should be treated in the same manner as the difference between the values and the mean for reference and spike data (i.e., the data should be taken and calculated as a standard deviation.) We will take the example data in Table 10.1, and calculate it several ways to illustrate what we mean.

Table 10.1 — Example Data

Run #	*Result 1*	*Result 2*	*RPD*	*ABS RPD*
1	41	37	10	10
2	37	42	-13	13
3	47	51	-8	8
4	28	24	15	15
5	26	30	-14	14
6	29	27	7	7

Table 10.1 (the ABS RPD column) and Figure 10.4 show the data calculated and plotted using the erroneous approach. Although the appearance looks comforting, it is meaningless. There are no lower control limits for duplicate data, and the mean is not the desired target.

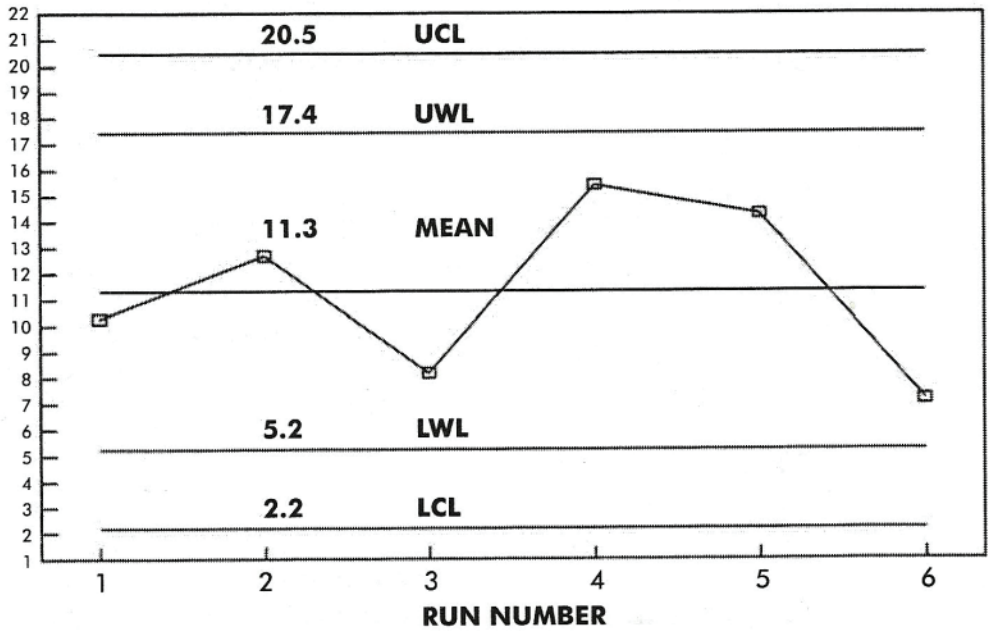

Figure 10.4 — Duplicate Control Chart using the wrong method

Probably the simplest approach is the one recommended by Paul Britton, the Statistical Data Manager for EPA's proficiency program and Results Advisor for ASTM Committee D19 on water. His approach is the same as the erroneous one, except that the sign of the RPD is kept. Using this approach, the resulting distribution is Gaussian, and we can look for trends in the first vs. second result. The mean converges at zero, as we would expect. Table 10.1 and Figure 10.5 show this approach.[4,10]

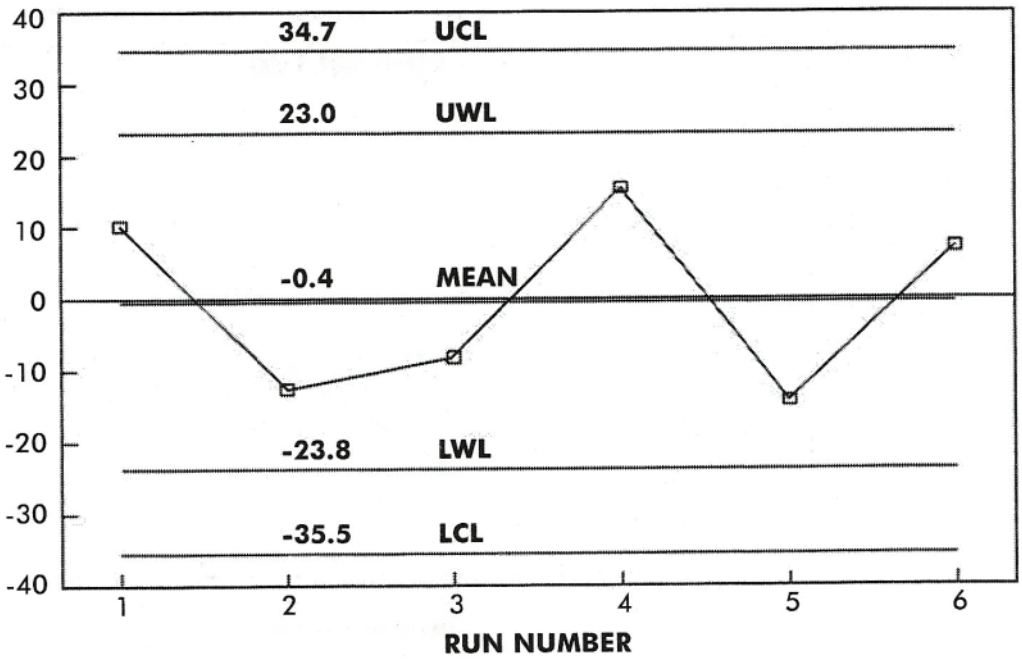

Figure 10.5 — Duplicate Control Chart using Britton's Method

There are other approaches. Taylor uses the root-mean-square approach, where he takes the root-mean-square sum of the RPD values and uses that value as a standard deviation. He then takes 2x and 3x that value as control limits - the trend line is zero. This produces a 1-way control chart, which is totally suitable for duplicate data. Figure 10.6 shows such a graph using the data in Table 10.1.[8]

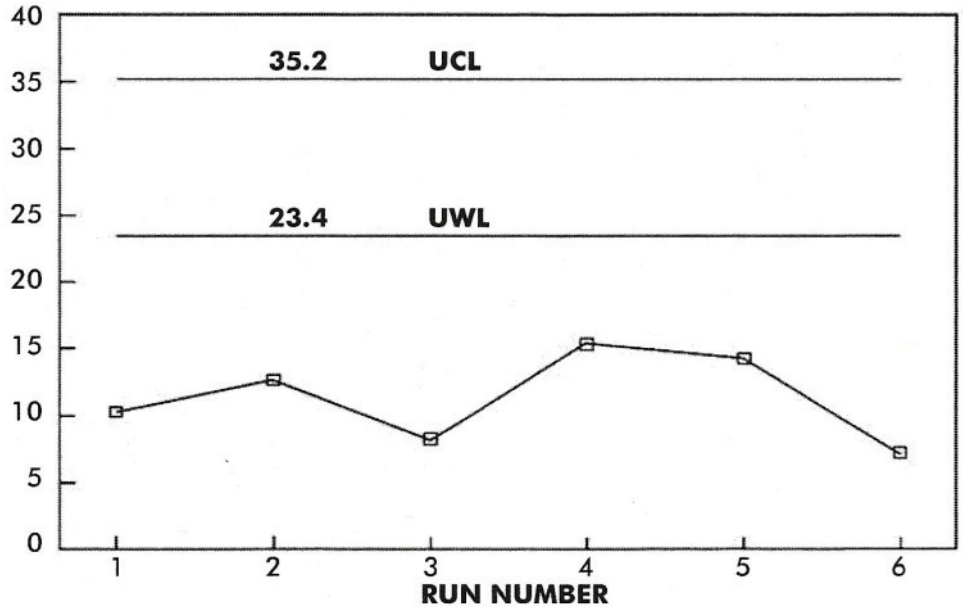

Figure 10.6 — Duplicate Control Chart by Taylor's Method.

A similar approach is used by the New York State Department of Health Environmental Laboratory Approval Program.[9] They calculate the mean of the RPD, but use that as a measure of standard deviation. They take 3.27 times this value as the single control limit, thus also producing a 1-way control chart, but with a single control limit. Figure 10.7 shows such a graph using the data in Table 10.1.[9]

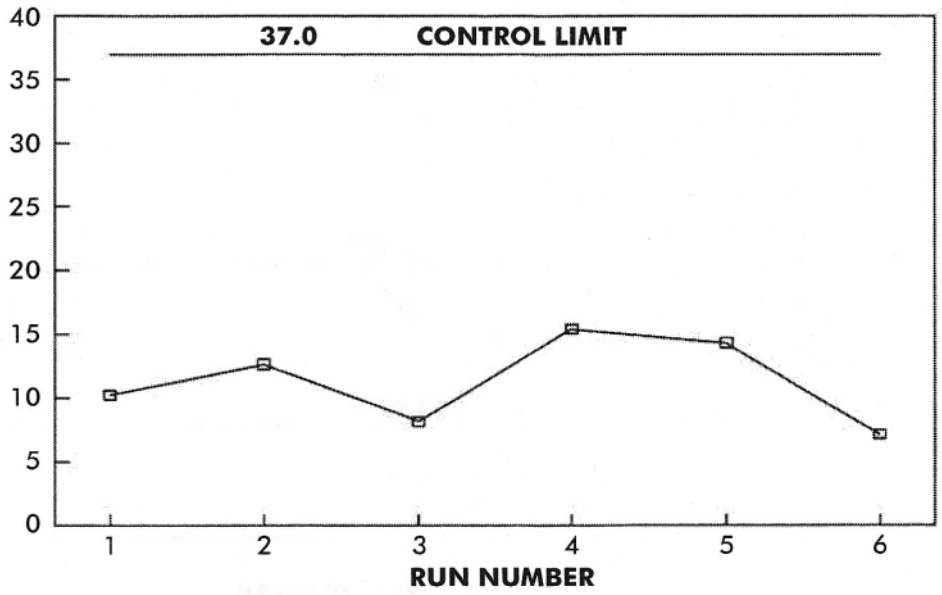

Figure 10.7 — Duplicate Control Chart using NYDOH Method

All of these protocols provide statistically similar and valid results, giving the Laboratory QC Manager a wide variety of options for establishing and plotting control limits for duplicate QC data.

10.6 Rejection of Results

10.6.1 Breakthrough

Breakthrough may result when the quantity of analyte sampled either exceeds the capacity of the sampling medium or is collected on the media at an improper flow rate. It is identified when the amount of analyte found in the backup section divided by the total found in both sections exceeds a specified value.[13] Sample results may be rendered invalid depending on the percentage of breakthrough in the backup section of the sorbent tube or sampling device. In some cases, the backup section is higher than the front section, suggesting that the tube might have been sampled backward. In other cases, one analyte may have displaced another from the front section and into the backup section. The fact that a sample result is questionable does not necessarily mean that the data need to be thrown out, but they should be interpreted with care. The possibility and feasibility of resampling should be considered in this case. If the accuracy of a result is questioned because of breakthrough, the result should be reported as a minimum value with a note that concentrations are likely to be higher. In general, if the percentage of the amount of analyte found in the backup section of the sampler exceeds 10% of the sum of both the front and backup sections of sorbent tubes, that sample should be considered invalid because of significant breakthrough from the front section to the backup section and possible sample loss from the backup section. Consult the manufacturer for guidance on breakthrough criteria for media other than sorbent tubes.

It is important to use sampling devices within their capacity limits so that breakthrough does not occur. When breakthrough does occur, the potential cause should be determined before resampling. Breakthrough can be caused by incorrect sampling flow rate or time, interferences, high humidity, high temperature, an improper sampling device, analyte migration from the front section to the backup section during storage before analysis, etc.

10.6.2 Blanks

Results may be rejected because of questionable blank values. High blank values may be caused by contamination of the sampling medium. This can occur in manufacturing or handling. If possible, additional media blanks should be analyzed to ensure sampling medium lot integrity. The analyst, laboratory supervisor or quality officer, and the field personnel should determine whether reported data should be corrected using blank analysis results. If a relatively high blank is subtracted from the samples, it is important that the average of several blanks be used. All of these situations must be documented.

A problem in dealing with blanks is that the contaminant source is not always constant. One approach would be to decide whether all of the samples are contaminated equally. If there is a consistent source of contamination, then sample results should be at or above the blank level. If there is no consistency, some unexplained phenomenon might have occurred, and the blank should not be subtracted from the analytical result. Keep in mind that subtracting the blank might reduce sample results to lower than what is truly present. It is useful to prepare a control chart of blank values for each method to determine a range of acceptable blank values.

10.6.3 Unacceptable Spike Recovery

Unacceptable spike recovery is based on expected recovery levels. These are determined by observing the historical variation in spike recoveries. QC charts can be used to predict acceptable spike recovery.

Poor spike recovery can result in the rejection of data. Judgment must not be made on only one or two spikes, but on three or more. When poor recoveries are observed, the analytical procedure must be evaluated. Check the standardization, response factors, allocating, etc., then evaluate the spiking technique. Make sure the correct volume and concentration of the spiking solution were used with the proper technique. For example, spiking styrene into air in a Saran® bag is a poor technique. The styrene adsorbs onto the bag walls, giving low airborne concentration levels.

If the cause of the failure cannot be determined, the following corrective action should be performed. If possible, reprepare the samples along with new spike recoveries. Since most industrial hygiene methods require either digestion or desorption of the entire sample a thorough investigation must be performed to determine the root cause of the failure. If the cause of the failure is a representative of the entire preparation batch then it is recommended to recovery correct the batch with the QC bias. If the cause of the failure is isolated just to the spike recoveries then proceed with reporting of the data through normal policies documented for your facility. Additional spikes can be prepared by qualified laboratory personnel to isolate the root cause. If poor recoveries continue, the data for the method are questionable and further evaluation shall be performed to remedy the situation.

10.6.4 Incorrect Sampling Medium

If an improper sampling device is used, the sample should be recollected properly. Resampling will save the expense of performing a method evaluation for one set of samples. If the samples cannot be recollected on the correct sampling medium, an effort should be made to determine if the chemical(s) can be recovered with reasonable accuracy and precision across the entire range of the method. For industrial hygiene analyses, spike recoveries of 75%–125% are generally acceptable.[13,14] For environmental analyses, spike recoveries of 90%–110% are generally

acceptable for EPA protocols.[10] Accreditation/recognition may require other limits. Check with the accreditation/recognition entity for requirements prior to release of analysis results. Please note that this procedure will only verify recovery of the analyte from the media it will not verify whether or not the analyte can be collected on the media during sampling.

10.6.5 Tests for Internal Consistency

When the data are homogeneous, as in the replication of a closely controlled sample, several statistical tests can be applied. These tests check selected values in a data subset that appears atypical when compared with a larger data population. Common anomalies of this type include unusually high or low values (outliers) and large differences in adjacent values. These tests will not detect systematic errors, which alter all values of the data set by either an additive or multiplitive factor (e.g., an error in reading the scale of a meter or recorder). The following tests for internal consistency are listed as examples. For a more extensive treatment of these tests, see References 5, 8, 10, and 11 of this chapter.

10.6.5.1 Data Plots

Data plotting is one of the most effective means of identifying possible data anomalies; however, plotting all data points may require considerable manual effort or computer time. The number of data plots required can be reduced by plotting only those data that have been identified by a statistical test (or tests) to be questionable (e.g., a Dixon ratio Q-test). Nevertheless, data plots often identify unusual data that would not ordinarily be identified by other internal consistency tests.

10.6.5.2 Dixon Ratio Test

The Dixon ratio test is the simplest of the statistical tests recommended for evaluating the internal consistency of data. The test for the largest value requires only the identification of the lowest (X_1) and two highest values (X_{n-1} and X_n) in the data set. The ratio (R) is calculated as:

$$R = (X_n - X_{n-1}) / (X_n - X_1)$$

R is calculated and compared with a tabulated value in the appropriate table.[8,9] Consistency is indicated by a ratio near zero. A possible data anomaly is indicated by a ratio near unity. This test is ideally suited for moderately sized data sets (e.g., a month of daily average values). The critical values of the ratio are derived from the assumption of a normal distribution. Non-normal data distributions, which are observed more frequently with industrial hygiene monitoring data, may require a logarithmic transformation to produce a lognormal distribution.

10.6.5.3 Grubbs Test

This test, like the Dixon ratio test, assumes a normal distribution. It requires computation of the mean (X) and the standard deviation(s) of the data. The test statistic (T) is calculated as follows:

$$T = (X_n - X) / s$$

where Xn is the largest value in the data set. The calculated T is compared with a tabulated value at an appropriate level of risk.[15]

10.6.5.4 Gap Test

This test identifies possible data anomalies by examining the length of the gap (or distance) between the two largest values (X_n and X_{n-1}), the second and third largest

values (X_{n-1} and X_{n-2}), and similarly for other gaps. The two-parameter exponential distribution is fitted to the upper tail of the distribution of the sample data, and the probabilities of the observed gap sizes are determined. If the probability is very small, the larger value is considered to be a possible data anomaly.[15]

10.6.5.5 Johnson "p" test

This test fits a distribution function to the upper tail of the sample data distribution and then compares the consistency of the largest value with that predicted by the fitted distribution (e.g., lognormal or Weibull distribution).[15]

10.7 Corrective Action Plan

A corrective action plan is a formal procedure to evaluate "out-of-control" data. The plan is based on an evaluation of the critical parts of an analytical procedure. It provides for allowable variation in the critical parts of the procedure and the frequency of checks for this variation. QC data normally are used to indicate this variability, but the corrective action plan is not limited to using QC data. Corrective action can be based on other analytical method parameters (e.g., time or temperature of reaction).[15]

The corrective action plan specifies the action to be taken in case of "out-of-control" data. "Go/no-go" limits for the method are established. When these limits are exceeded, the plan generally specifies that the analyst must stop and report findings to the quality system coordinator (QSC), however titled. Critical areas of the method are evaluated, the deficiencies are corrected, and the samples are reanalyzed if necessary. If reanalysis is not possible, resampling is requested.

A key element of the corrective action plan is the documentation of the critical parameters of the method and their limits. When an "out-of-control" situation is encountered, the limits that were exceeded and the corrective action that was taken should be documented.

10.8 System Audits

The purpose of the audit is to verify that all actions adhere to approved QS requirements. A QS audit must be performed periodically (at least yearly) and a written report issued. At least quarterly a report of the quality assurance for the samples analyzed should be given to laboratory management for information and approval of actions required/requested. This quarterly report may contain the internal system audit report, proficiency program performance, nonconformities that occurred in that quarter along with the corrective actions and preventative actions taken to deal with each nonconformity.

The following checklist should be helpful for the audit:

10.8.1 Have corrective actions been instituted for out-of-control situations? What are the results of these actions?

10.8.2 Have analytical and monitoring instrument calibration schedules been followed and documented?

10.8.3 Have analytical and monitoring instrument maintenance schedules been met and documented?

10.8.4 Have the appropriate number of internal QA checks been made and documented?

10.8.5 Have all samples been analyzed by approved procedure(s)?

10.8.6 Have the procedures been validated in the laboratory and in the field?

10.8.7 Have all sample results been evaluated by appropriate personnel?

10.8.8 Have results from all inter-laboratory tests and round-robin (e.g., AIHA IHPAT or ELPAT) samples been checked and documented?

10.8.9 Have corrective actions been instituted for outliers resulting from round-robin tests?

10.8.10 Have all new methods of sampling and analysis been validated for adherence to appropriate criteria and proper documentation?

10.8.11 Have an appropriate number of internal QA samples been sent to contract laboratories, and have the results been documented?

10.8.12 Have the qualifications of contracted laboratories been evaluated?

It is the responsibility of the QSC to compare the results of the audit with the QA standards established for the laboratory and to initiate necessary corrective action.

At least annually, the entire quality system should be reviewed. This includes review of all procedures, personnel qualifications, and all documentation. Examples or the questions which must be answered in the audit are: "Are you doing what you say? Are you saying what you do? Are your policies following the latest changes in AIHA LAP policies?" There should be a match. The report of this activity is also directed to laboratory management for information and approval of actions required/requested.

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Chapter 11
Reporting

by Samuel D. Allen Iske, Jr., PhD, CIH, CSP

11.1 Introduction

Despite scrupulous adherence to the requirements of analytical protocol, if the final report of the analytical results and supporting documentation is incomplete, the data package will be of little value. This chapter discusses the elements of an analytical report and important considerations with regard to data retention and sample archive policy.

Often ignored by many QA documents, the analytical report and the archived data are major pathways back to the control charts, standard samples, and sampling data associated with a particular data set. Although it is difficult to develop a uniform analytical report format since the requirements of customers may differ, this chapter presents critical elements of a report and why they should be considered.

This section also discusses record-keeping practices and considerations for a sample archive. These two areas depend heavily on sample type and regulatory/liability considerations.

Although some samples, such as bulk dust samples can be archived indefinitely, most samples are either totally consumed (e.g. personal monitor media), or will not last very long in storage. However, some OSHA[1] and EPA[2] regulations contain specific sample and record retention policies. AIHA Laboratory Accreditation Policies should also be followed.[3]

11.2 Elements of the Analytical Report

The laboratory analytical report should convey to the customer all of the information needed to evaluate the analytical results and draw conclusions about the exposures or emissions those samples represent.

11.2.1 Laboratory and Customer Identification

Each analytical report should state clearly the name and address of the laboratory and client, and any other relevant information on whoever requested the analytical work. There should be a scheme to uniquely identify each report.

11.2.2 Dates

All reports must be dated. Sample receipt dates and analysis dates should be included. In cases where employee notification of monitoring results is required or there are report deadlines, these dates might have regulatory significance. All other pertinent dates relevant to sample preparation, extraction, storage, or other specified information by client or regulatory agency shall be recorded and archived in each sample data file.

11.2.3 Sample Identification

Each sample should have a unique identifier assigned to it upon receipt in the laboratory. This identifier should be used by analysts during the analysis procedure to ensure that future tracking of the sample can be completed effectively. Sample identifiers often incorporate the date of receipt, not only to facilitate tracking but also to help the laboratory monitor turnaround time. Because the customer has little knowledge of the laboratory identification

(ID) scheme, reporting to the customer should include both the laboratory-assigned sample identification and the customer's sample identification information.

Including other customer information in reporting may be useful, and might include information such as sample location, person sampled, employee identification number (if allowed), sample date, job function, task description, time periods of activities, etc.

11.2.4 Data Presentation

Data should be presented in an unequivocal form with proper units included for each result. Conventional units should always be used (e.g., [µg/100 g] for metals in blood; [ppm(v/v)] for gases and vapors; [µg/ft^2] for lead dust; [mg/m^3] for airborne particulates; [lb/hr] for process emissions; [mg/kg] for lead in soils; and [mg/L], [ppb(w/v)] or [ppm-(w/v)] for liquid environmental samples). The sample matrix should also be described in reporting (i.e., filter type used for sampling airborne dust or fume, sorbent type used for collecting a gas or vapor, a liquid, a soil or solid, etc.).

Terms such as "absent" or "zero" shall never be used. Use of terms such as "none detected" or "not detected (ND)" must be accompanied by the reporting limit (RL) for the method employed, or the Limit of Detection (LOD) accompanied by appropriate qualifiers for measurements made below the Reporting Limit. It is recommended to report the result as "less than" or "<" a specific level in mass and/or concentration units. Results for solid sorbent media should indicate the mass measured on each media section as well as the total mass measured, and may also include the calculated air concentration result for the requested analytes. If additional contaminants are observed during analysis, this information should be reported to the customer. If analysis determines that potential or significant breakthrough has occurred, it should be so noted on the report.

11.2.5 Accuracy and Precision

The Reporting Limit (RL) / Limit of Quantitation (LOQ) must be included in reporting for each parameter reported quantitatively, and the laboratory must also be able to provide the expanded uncertainty estimate for each qualitative result reported (see AIHA-LAP, LLC Policies 2A.5.9[3], 2A.5.10[3], and Appendix G, the Estimation of Uncertainty of Measurement[3]). Including expanded uncertainty and a summary of quality control data (laboratory/ field/ matrix blanks, recovery of spiked samples and control samples, etc.) in reporting is a desirable option. Reporting of analyte breakthrough to the backup section of a sorbent media is essential so that its impact on accuracy may be considered.

11.2.6 Method Reference

A reference notation should be included when utilizing a known reference or regulatory method for sampling and analysis of samples. This reference provides assurance and confidence to the client for evaluation and assessment of their data findings. Specific citation must be made for the sampling and analytical aspects of the method sections. Method identification should be explicit, using designations from NIOSH, OSHA, EPA, consensus standard (ASTM, ISO, etc.), or other in-house developed and validated methods with version number or date of method identified. If no reference method was used, the laboratory must provide a detailed summary of the method utilized for sample preparation and analysis technique Other useful information such as instrumental separation techniques (column specification, column conditions, temperatures, flow rates, etc.), extraction procedures, storage of samples, detection technique, and all other pertinent details should be included. Any and all modifications or deviations from a referenced or specified method should be documented. Future data analysis may determine that a particular technique was subject to a bias; thus, the method referenced may provide information about the reliability of historical data.

11.2.7 Analyst Identification

At a minimum. all reports should be signed by a person with written authority to release analysis results. Best practices have signatures of each analyst, peer-reviewer, quality control reviewer or auditor, and the release authority or designee of the laboratory. The name and title should be printed with the signature.

11.2.8 Calculations

If unique calculations are performed, an example should be included in the report. Some reports to be used for compliance require example calculations. All data on the report should be referenced properly (e.g., notebook number and pages).

11.2.9 Remarks

Any pertinent comments relevant to sample irregularities or problems in analysis must be noted in a "Remarks" section.

11.2.10 Miscellaneous Items

11.2.10.1 If corrections or additions to a test report are made, they must be documented in amended report.

11.2.10.2 Any corrections made to data should be documented with a single strikeout line, the analyst's initials and date, and explanation or reason for the error. All handwritten data should be recorded using indelible ink. Correction fluid must never be used on original laboratory data records.

11.2.10.3 Laboratory records should document all analyses in detail. Include all notebook references with page numbers. Quality control samples included and specific samples analyzed in the data set are identified.

11.2.10.4 The data reduction and review process should include, but not necessarily be limited to, comparison of QC data against established acceptance limits, computation verification, transcription of data, and adherence to the procedures established in the laboratory Standard Operating Procedures (SOPs). The review process should be documented.

11.2.10.5 All aspects of the Laboratory Quality Control policies and procedures must be followed and a summary of the Quality Control results, such as the uncertainty, should be presented in the final report.

11.2.10.6 Significant figures should be statistically correct for the data set or meet requirement by client.[3]

11.2.10.7 Number rounding should follow the rounded off to the desired number of significant figures referenced in the laboratory referenced methods or SOPs or specified by the client.

11.3 Record Retention Program

Good analytical practices require all laboratory records be maintained so as to avoid loss of information and data; this has traditionally meant records be kept electronically or in bound notebooks. Long-term retention of notebooks, logbooks, data sheets, reports, etc., implies the use of numerous filing cabinets or other storage facilities. If space is limited decisions need to be made on the length of holding time, but specific laboratory policy, accreditation, recognition, regulatory requirements must be followed. AIHA-LAP, LLC[3] requirements for minimum

record retention times, specified in the policies for each of its accreditation programs, shall be considered the minimum requirements. A documented tracking system for all data notebooks, files, laboratory data, archives, reports, SOPs, procedures, and other laboratory documents must be kept current and available.

Today, even in smaller laboratories, computerized data base management systems allow for long-term storage of final analytical data, instrument readings, pump flows, and chromatograms. The generation of microfiche, magnetic tape, CD-ROM copies, optical disks, or computer electronic files on servers allows for even very large quantities of hard copy submission forms and sampling logs to be saved in a concise manner.

For some very sophisticated laboratories, tailor-made computer software has been developed to follow samples through the laboratory from login to storage in a predetermined manner. For smaller laboratories with less computer expertise, there are "developed application" programs that allow for data generation, storage, and tracking.

Microfiche, CD-ROM, magnetic tape, optical disks, or computer servers for electronic documents must be stored in an environment similar to that at which the records were generated. The laboratory record retention program should be documented with explicit time intervals for retention of each type of record in storage, and should include required auditing functions to ensure proper retrieval.

11.4 Sample Retention Program

As mentioned above, sample retention is not always feasible and there are few, if any, requirements to keep samples. The rule of thumb for retention is to maintain analytes, blood and urine samples, and desorbing solutions, when possible, for a period long enough to allow the report recipient an opportunity to request a repeat analysis. Samples such as bulk asbestos, soil, solid waste, etc., can be kept indefinitely for as long as space allows, or as contractual obligations require. When a repeat analysis has been performed on a sample that might have changed because of excessive holding time (e.g., organic solvent evaporation), a disclaimer on the analytical report is appropriate. Assure that the retention policy is clearly stated in laboratory SOPs. The laboratory may wish to arrange to return sample hazardous wastes to the customer for disposal.

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Chapter 12
Analysis of Fiber Containing Samples

by Martin Harper, CIH and Donald Halterman, MS.

12.1 Introduction

The information in this chapter will aid laboratories and analysts in meeting the challenges unique to analysis of samples containing fibers. The purpose of the analysis is in some cases to identify fibers, but also to determine concentration in bulk material or air samples.

12.2 Sample Shipment

As discussed in previous chapters, bulk samples should be shipped in separate containers from airborne fiber samples. The bulk samples should be shipped in sturdy, re-sealable, clear containers that have been taped shut to prevent spillage. Air samples received in the same container as bulk samples should be rejected. Care must be taken when opening and closing bulk samples to limit or contain any escaping fibers, to preclude cross-contamination of the bulk samples, and to prevent exposure to personnel in the area.

For air samples, a rigid shipping container should be selected, and cassettes should be packed in a non-contaminating, non-electrostatic medium. The samples must be packed in a manner that prevents them from rattling loose inside the shipping container and must be protected from outside shocks to the container. A mode of transportation least likely to jar the samples in transit should be used.

12.3 Contamination Checks

Periodic monitoring for airborne fibers in the sample preparation and analysis area is recommended to detect the potential for sample cross-contamination and employee exposure. Any detection of asbestos or other interfering fibers makes it necessary to identify the source of contamination and corrective action must be taken before continuing analysis. To prevent cross-contamination, bulk samples should not be manipulated in the same hood as air sample filters.

12.4 Fiber Counting Using Phase Contrast Microscopy (PCM)

12.4.1 Overview

U.S. laboratories performing analysis of airborne asbestos exposure samples must comply with the quality assurance requirements of U.S OSHA's Asbestos Standard Appendix A, CFR 1910.1001.[1] Laboratories outside the United States or its territories may have the option of using other methods such as those from ASTM International, the International Organization for Standardization, or others.[2–6] Appendix A of the OSHA Asbestos Standard specifies analysis per the most current version of NIOSH 7400[7] or OSHA ID-160[8], or an equivalent method (includes TEM method NIOSH 7402 as supplemental to either OSHA ID-160 or NIOSH 7400).[9] The most current versions of these methods at the time of this publication are NIOSH 7400 Issue 2, OSHA ID-160 (July 1997), and NIOSH 7402 Issue 2. Laboratories outside the United States or its territories have the option of using equivalent methods according to AIHA policies.[10]

12.4.2 Microscope Calibration

12.4.2.1 Phase Contrast Microscope Optics

On a daily basis, before routine fiber count analyses, the microscope optics, condenser, phase annulus, phase ring, and phase plate should be properly aligned as per manufacturer's instructions.

12.4.2.2 Health and Safety Executive/National Physical Laboratory (HSE/NPL) Test Slide or Health and Safety Laboratory/ULO Optics (HSL/ULO) Test Slide

The phase-shift detection limit for each analyst/microscope combination should be about 3 degrees measured using the HSE/NPL or HSL/ULO test slide, which consists of seven sets of grooved lines in descending order of visibility from Sets 1 to 7. The requirements for fiber counting based on the HSE/NPL Test Slide are that the microscope optics must resolve the grooved lines in Sets 1 through 3 completely. Sets 4 and 5 should be at least partially visible. Sets 6 and 7 must be invisible. Failure to meet these requirements indicates that the resolution of the microscope is outside of the acceptable range for fiber counting. The requirements for fiber counting based on the HSL/ULO Test Slide are that the microscope optics must resolve the grooved lines according to the accompanying Certificate, provided that the Certificate states that at least one block of lines is expected to be invisible (original Certificates for such slides are printed on red or green backgrounds). Where the Certificate does not state that at least one block of lines should be invisible (original Certificate printed on a yellow background) then it shall not be used. HSL/ULO Test Slides should be checked against the visibility of blocks as stated in the accompanying Certificate. When the visibility of blocks is in accordance with the Certificate the phase shift will be appropriate for fiber counting.[11]

The frequency for performing the resolution check should be established by the lab and stated in its procedures. NIOSH 7400 states that resolution should be checked "periodically." OSHA ID-160 specifies performing this resolution check as part of the alignment routine at the beginning of every counting session, at least daily. Perform the resolution check at a minimum of each time a scope is moved and each time a new microscopist/microscope pairing occurs. If the resolution fails, the microscope optics should be cleaned. If the problem persists, the microscope manufacturer should be consulted and corrective action taken.

12.4.2.3 Graticule

The microscope should be fitted with an eyepiece graticule (Walton-Beckett type G-22; an alternative RIB graticule can be substituted provided it has been shown not to affect the fiber counts[12]) calibrated on at least a yearly, but preferably a monthly basis using an ISO-traceable, NIST-traceable or equivalent stage micrometer for a counting field with a diameter of 100±2 µm. Records of this calibration are to be preserved with sample results. Graticules are custom-made for each microscope. The eyepiece-objective-reticle combination for a microscope must be calibrated initially upon receipt of the graticule, and recalibrated thereafter at a minimum of each time there is change in the optical system (maintenance, repair, or replacement of an optical component of the combination. When a change of interpupillary distance changes the tube length of the microscope, the system must be readjusted to the proper tube length or the altered graticule field area must be used, as long as it is within the 100±2 µm requirement). The laboratory must be aware of whether each microscope automatically compensates for tube length when adjusting interpupillary distance or not.

12.4.3 Filter Sample Preparation and Analysis

During sample preparation, any samples that have wrinkled, torn, or collapsed filters, or loose material in the cassette, should be rejected. Samples without loose material, but which may be overloaded, should be prepared for evaluation by the analyst. All PCM sample preparation and analysis areas, reagents, and tools should be isolated from bulk asbestos sample preparation and analysis areas.

12.4.4 Quality Assurance

12.4.4.1 Reference Slides

A library of permanently mounted reference slides prepared from field samples, assigned by the QA officer, must be maintained for daily use by each analyst. The labels of the reference slides should be changed periodically to prevent analysts from becoming too familiar with the slides.

Each day samples are to be analyzed, the analyst must count a reference slide before counting samples. The result must fall within the documented control limits established for that slide before fiber count analysis may be conducted on field samples.

From blind repeat counts of the reference slides, the laboratory intra- and inter-counter relative standard deviation (Sr) should be determined. At least 20 analyses of each slide are recommended to establish a reliable Sr. Separate Sr values for intra- and inter-counters shall be obtained in at least the following ranges:

12.4.4.1.1 5–20 fibers in 100 graticule fields.
12.4.4.1.2 >20–50 fibers in 100 graticule fields.
12.4.4.1.3 >50 fibers in 100 graticule fields.

12.4.4.2 Recount of Field Samples

Recounts (blind, when possible) should be performed by the same counter on at least 10% of the samples counted. Reject the count pair (count and recount) if the absolute value of the difference between the square roots of the two counts exceeds the test statistic as follows:

$$X = \left(\frac{1}{2}\right)\left(\sqrt{X_1} + \sqrt{X_2}\right)$$

where:

X_1 = original count value, fibers/mm²
X_2 = recount value, fibers/mm²
X = average of the square roots of the two fiber counts (fibers/mm2)

$$\left|\sqrt{X_1} - \sqrt{X_2}\right| > \left|2.77\,(X)\right|\left(\frac{1}{2}\ S_r\right)$$

S_r = Pooled square root scale intra-counter relative standard deviation for the appropriate count range (fibers/mm²) obtained from blind repeat counts (see Section 12.4.4.1).

If the count pair is rejected, the remaining samples in the set must be recounted. Test the recount values against the original count values and discard all rejected paired counts.

12.4.4.3 Standard Relocatable Test Slides

Slides made from preferably proficiency test filters (such as AIHA PAT samples[10]) or if they are not available then certified field samples should also be prepared with a cover slip containing relocatable grids. Euparal mounting medium has been shown to provide long term stability sufficient to allow the use of cover slips with relocatable grids.[13] Each slide is then examined by several other analysts, who must agree on the fibers that should be visible in specified grids. These are then called Standard Relocatable Test Slides, and an analyst must examine specified grids on at least one slide each day that field samples of similar fiber type are to be analyzed before commencing analysis. If the fibers in the field samples are of unknown type or are chrysotile mixed with other fibers, the chrysotile Standard Relocatable Test Slide should be selected for examination. The analyst should achieve a discrepancy score greater than 50 in a chrysotile or Amosite Standard Relocatable Test Slide. The calculation of a discrepancy score is provided in the references.[14,15,16] For each examination, a score is calculated from the number of absolute discrepancies between the reported and verified fibers in each field.

$$\text{Score} = \left[1 - \frac{\textit{no. of discrepancies}}{\textit{no. of verified fibers}}\right] \times 100$$

If the microscope /counter performance is above 50%, the inter-counter variation should be within +/- 20% in counting field samples.

Note: ISO 8672[4] has been revised with a positive international ballot in Committee to incorporate language consistent with that above.

12.4.5 Inter-laboratory Quality Control

It is recommended that the laboratory be enrolled in the AIHA IHPAT program for airborne fiber counts, or the analyst be enrolled in the AIHA Asbestos Analyst Registry (AAR) program, or both. IHPAT sample analyses should be performed by all qualified analysts. However, the results of one preselected analyst should be reported.

A round-robin fiber count exchange program with at least two other independent laboratories using field samples is required by OSHA for the analysis of personal exposure samples. The proficiency testing programs of the AIHA are not acceptable for this requirement. Samples should be exchanged among the laboratories at least semiannually. The results of the round robin should be evaluated statistically and posted in the laboratory for the analysts' viewing. If there is a discrepancy concerning a sample or samples, a recount should be conducted to rectify the problem. If these slides are prepared using cover slips with relocatable grids, analysts can be asked to examine specified grid areas and discrepancies can be more easily evaluated.

12.4.6 Training

Each analyst should receive training equivalent to the NIOSH 582 course "Sampling and Evaluating Airborne Asbestos Dust." This should include training on the application of both "A" and "B" counting rules. Before analyzing any field samples, a new analyst should be monitored for at least a two-week probationary period. During this time, the new analyst must demonstrate his or her ability to meet the established QA guidelines of NIOSH Method No. 7400 and the laboratory or organization. Completion of successful training should be documented through the analysis of Standard Relocatable Test Slides containing chrysotile and Amosite fibers. Documentation of training of the application of "B" counting rules should be through the analysis of a Standard Relocatable Test Slide made from a filter containing synthetic mineral fibers.

12.4.7 Reporting

AIHA policy requires that final reports for PCM testing include: 1) measured fiber density; 2) fibers per cubic centimeter (or total # fibers); and 3) applicable Sr values.

12.5 Airborne Asbestos Fibers by Transmission Electron Microscopy (TEM)

12.5.1 Overview

The following QA procedures are a general treatment of measures that should be used to ensure the adequacy of analytical procedures and equipment calibration for transmission electron microscope (TEM) analysis of airborne asbestos. Many of the items addressed here are derived from the Asbestos Hazard Emergency Response Act (AHERA)[17–20] and National Voluntary Laboratory Accreditation Program (NVLAP)[21,22] criteria.

12.5.2 Calibration

12.5.2.1 Magnification

The TEM should be calibrated monthly with a cross-grating replica placed at the eucentric position and at the magnification(s) used for asbestos fiber counting and identification. If the size of the grid opening is measured with the TEM, the microscope must also be calibrated at that magnification. The magnification should be recalibrated after any maintenance that involves adjustment of the power supplied to the lenses, the high-voltage system, or the mechanical disassembly of the electron optical column, other than filament exchange.

12.5.2.2 Camera Constant

The camera constant of the TEM in electron diffraction (ED) operation mode should be calibrated before ED patterns on unknown samples are indexed. This can be achieved by using a carbon-coated grid, on which a thin film of gold has been sputtered or evaporated. Measurements should be determined with an average camera constant using multiple gold rings. The camera constant is one-half the diameter (D, in millimeters) of the ring multiplied by the interplanar spacing (d, in Ångstroms) of the ring being measured. This calibration should be done weekly.

12.5.2.3 Beam Dose

A chrysotile standard should be examined in the microscope, and a Selected Area Electron Diffraction (SAED) pattern should be obtained for a single fibril > 1 µm in length. The pattern should be checked to ensure it is still visible after 15 seconds for a minimum of 9 out of 10 fibrils. This calibration should be done quarterly.

12.5.2.4 Spot Size

Using a magnification of 15,000X–20,000X, a spot size of approximately 200 nanometers (nm) should be obtained and measured. (The measurement should indicate the spot size is 250 nm). This calibration should be done quarterly.

12.5.2.5 Energy Dispersive X-ray Spectrum (EDS) Peak Position

The EDS system should be calibrated by using two reference elements to verify the energy scale of the instrument. An Al-Cu reference is recommended because its spectrum covers the range of elements found in asbestos minerals. This calibration should be done daily.

12.5.2.6 EDS Resolution

A carbon/manganese-coated grid should be examined to determine the resolution of the Mn K-alpha peak (175 eV or better is required). This calibration should be done semi-annually.

12.5.2.7 EDS K-Factors

NIST Standard Reference Material 2063a[23] and albeit (or acceptable equivalent) should be examined to determine detector sensitivity by monitoring the background-corrected peak intensities for Mg, Si, Ca, Na, Al, and Fe. This calibration should be done semi-annually.

12.5.2.8 Chrysotile Standard

An EDS spectrum should be obtained from a single fiber of a chrysotile standard to demonstrate the presence of both distinctive and significant Mg and Si peaks in the correct ratios. This calibration should be done quarterly.

12.5.2.9 Crocidolite Standard

A crocidolite standard should be examined to confirm the ability of the EDS to detect a statistically significant Na peak. This calibration should be done quarterly.

12.5.2.10 Plasma Asher

The plasma asher should be checked quarterly to set the proper ashing time. The appropriate time is that necessary for approximately 5% of the sample filter to be etched during the etching step of sample preparation (mixed cellulose ester filters only).

12.5.3 Sample Preparation

Items used during sample preparation (such as petri dishes, forceps, screens, scalpels, slides, and glassware) should be meticulously cleaned before use and before contact with subsequent samples.

Filter lots used for sample preparation should be checked for background contamination levels before use. Filter batches must be rejected as contaminated if the average asbestos fiber count exceeds 18 fibers/mm^2, or if a single asbestos fiber count exceeds 53 fibers/mm^2.

Grid batches should be examined for uniformity and size of grid openings (20 openings each of 20 grids per batch of 1000 grids). Manufacturer-calibrated grids are acceptable if the grid opening area is confirmed (size ±10% of nominal) by reviewing at least 25 openings on a total of 10 grids per batch of 1000 grids.

Spectroscopic grade reagents are recommended for sample preparation.

The area in which the filters are prepared should be kept as free of contamination as possible. The use of laminar flow clean benches and fume hoods during prep stages that require volatile chemicals can help achieve this. All prep instruments and tools should be quarantined from other areas of the laboratory, particularly where bulk asbestos samples are analyzed or stored. No interchange of tools, chemicals, filters, cleaning aids, etc., should be permitted.

The plasma asher should be cleaned between each operation. The vacuum evaporator should be cleaned on days when samples are prepared.

A portion of all samples prepared should be re-prepared by a second analyst to ensure uniformity in preparation procedures.

12.5.4 Quality Assurance

12.5.4.1 Blanks

During sample preparation procedures, one laboratory blank should be prepared with each sample set to verify that no contamination exists. It is recommended that a laboratory blank be analyzed with each set, to minimize reanalysis if contamination is found. If the asbestos fiber count on a laboratory blank exceeds 53 fibers/mm2, the entire sample set is suspect and must be re-prepped.

12.5.4.2 Intra-inter-Analyst Reanalysis

At least 2% of all TEM samples should be reanalyzed by the original analyst (intra-analyst) or a second analyst (inter-analyst). The original grid squares should be used for all re-analyses. This information should be used to determine a laboratory coefficient of variation (relative standard deviation) to determine acceptability of future re-analyses.

12.5.4.3 Verified Counting

For a minimum of 1% (NVLAP criteria) of TEM grid openings analyzed, a verified counting analysis should be conducted. The verified counting is done on field samples and documented higher fiber loading samples. 20% of the total verified counting analysis must be done on sample or standards grids that meet the higher level fiber density criterion (6-40 fibers per grid opening) and these grids can be archived for this purpose.

12.5.4.4 Standard Reference Material 1876b

The NIST Standard Reference Material 1876b[24] or an equivalent should be analyzed at least annually by all analysts. The laboratory mean should fall within 80% of the 95% confidence limits as published on the NIST certificate. If the SRM result is out of this range, it should be reanalyzed by the analyst and the quality assurance coordinator to correct any problems encountered. This NIST SRM is currently listed as Discontinued. The NIST website recommends Research Triangle Institute (www.RTI.org) as an alternative source of asbestos reference materials.

12.5.4.5 Standards and Reference Materials

A library of potential interferences and asbestos standards, such as NIST Standard Reference Material 1866a[25], should be maintained. This library should be analyzed periodically to facilitate recognition of asbestos and other fibrous materials, and to verify instrument sensitivity to the materials. This NIST SRM is currently listed as Discontinued. The NIST website recommends Research Triangle Institute (ww.RTI.org) as an alternative source of asbestos reference materials.

12.5.4.6 Verification of Calculations

At least 1% (AHERA criteria) of the samples that are analyzed with an automated data reduction system should be verified by hand calculations. At least 1% (AHERA criteria) of any hand-calculated data should undergo an independent recalculation.

12.5.5 Inter-laboratory Quality Control

The laboratory should be enrolled in a round robin sample exchange program with at least two other laboratories that perform TEM analyses. Field samples should be exchanged between the laboratories at least semiannually. The results should be statistically evaluated. If there is a discrepancy with a sample or samples, there should be a recount to correct the problem. All samples used in the program should be typical of the laboratory's own work load, and the fiber counts should have been verified internally.

The Proficiency Testing Program under NVLAP provides additional external QC checks on sample preparation, analysis, equipment, and interpretation of results.

12.6 Asbestos Bulk Identification Using Polarized Light Microscopy

12.6.1 Overview

Bulk samples for asbestos content are typically analyzed by polarized light microscopy (PLM), such as EPA 600[18], NIOSH method 9002[26], and OSHA Method ID-191.[27] Other methods include analytical electron microscopy and X-ray diffraction.

12.6.2 Calibration

12.6.2.1 Microscope Setup

The alignment of the microscope optics, as per the manufacturer's directions, should be checked before each use to ensure proper illumination. The polarizer and analyzer should be oriented with their privileged directions 90° perpendicular to one another, so that they are at extinction. The polarizer is sometimes referred to as the lower, or substage polarizer, and the analyzer is sometimes referred to as the upper polarizer. The ocular cross hairs must also be aligned with the polarizers in the north-south and east-west directions, in order to properly determine the extinction angles of fibers. The objectives and stage should be centered, to prevent any particles from leaving the field of view during stage rotation and to allow rotation to be referenced to the center of the cross hairs. The condenser should be focused and the field diaphragm should be centered in the field of view, using the ocular cross hairs as a reference.

12.6.2.2 Refractive Index (RI) Liquids

These liquids should be NIST traceable and calibrated monthly using a refractometer or equivalent method. Records should be kept with documentation of purchase, NIST traceability, and monthly calibration of each individual liquid. Any noticeable color change might indicate a change in the liquid's refractive index. Any liquid that has a refractive index greater than ±0.004 of the theoretical value must be replaced. The expired liquid must be disposed of in a manner consistent with waste disposal practices. All RI liquid checks should be documented.

All RI liquids should be stored at a temperature between 54°F and 95°F (12°C–35°C). The temperature of the laboratory should be documented for each day of analysis.

12.6.3 Sample Analysis

12.6.3.1 Stereomicroscopic Examination

A visual examination using a simple stereomicroscope should be performed for all samples. When a sample consists of two or more distinct phases (e.g., the layers of a layered sample), each should be treated as a separate sample, when possible.

Homogeneity, texture, friability, color, and extent of fibrous content should be determined and recorded.

12.6.3.2 PLM Examination

Subsamples of particles should be mounted in RI liquids in a manner that permits the asbestos materials to be visible and distinguishable from everything else. The following optical properties should be evaluated:

12.6.3.2.1 Morphology;

12.6.3.2.2 Color and pleochroism;

12.6.3.2.3 Refractive Indices (±0.005);

12.6.3.2.4 Birefringence;

12.6.3.2.5 Extinction; and

12.6.3.2.6 Sign of elongation.

12.6.4 Quality Assurance

12.6.4.1 Reference Materials

A reference bulk sample should be analyzed at least weekly, preferably each day, prior to analysis. This sample serves to check the microscope optics, the integrity of the RI liquid, and the analyst's ability to perform dispersion staining and polarized light microscopy.

A library of asbestos standards and potential interferences should be maintained and analyzed periodically to facilitate recognition of asbestos and other fibrous materials. If there is a problem related to identification of a particular type of asbestos in a sample, a reference material that contains the type of asbestos suspected in the bulk can be compared to determine whether the characteristics match.

12.6.4.2 Contamination Check

An asbestos-free reference material should be examined before each day of analysis to check for laboratory supplies contamination.

12.6.4.3 Replicate/Duplicate Quality Control Analysis

A blind replicate analysis must be performed on a minimum of 2% of the total samples analyzed. A blind duplicate analysis must be performed on a minimum of 7% of the total samples analyzed. A total of 10% of the samples should be duplicates, replicates, proficiency samples and blanks. If any quantitative results are statistically different or if the qualitative results are different, another analyst should analyze the sample. If this re-analysis verifies a problem, additional quality control measures should be taken.

12.6.5 Inter-laboratory Quality Control

The Proficiency Testing Programs of the NVLAP or AIHA® can assist the laboratory in evaluating its ability to identify and quantify the contents of known bulk samples. Participation in at least one of the programs is recommended.

Also, it is recommended that a laboratory participate in a round-robin exchange program with at least two other laboratories performing PLM analysis of asbestos bulk material. Samples should be exchanged at least semiannually. If there is any discrepancy on result(s) of a sample or samples, a reanalysis should be conducted to rectify the problem. NVLAP requires participation in bulk round robin proficiency samples.[21]

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Chapter 13
Quality Assurance Guidelines for Biological Monitoring

by Richard Mark O'Mara, CIH

13.1 Introduction

This chapter is intended to be used as a resource by industrial hygienists, industrial hygiene chemists, and occupational physicians to provide basic background information for biological monitoring programs and to enhance the quality of data from those programs. Biological monitoring programs are generally more difficult to administer than conventional exposure (air) monitoring programs because of the more personal nature of collecting samples and reporting results, the frequently more complicated sample analysis, and difficulties in data interpretation due to possible exposure outside the workplace. This chapter is not intended to be a laboratory QA manual for biological monitoring. More specific QA considerations for laboratory analysis are addressed in the other chapters of this manual.

All handling of potentially infectious biological fluids must be done in compliance with the OSHA bloodborne pathogens standard.[1]

13.2 Definition

Biological monitoring, or biomonitoring, is a means of assessing the total adsorption of a chemical into a worker by all routes of exposure, including inhalation, skin absorption, and/or ingestion as delineated in the ACGIH TLVs® booklet[2]:

Biological monitoring consists of an assessment of overall exposure to chemicals that are present in the workplace through measurement of the appropriate determinant(s) in biological specimens collected from the worker at a specified time.

The determinant can be the chemical itself or its metabolite(s), or a characteristic reversible biochemical change induced by the chemical. The measurement can be made in exhaled air, urine, blood, or other biological specimens collected from the exposed worker. Based on the determinant, the specimen chosen, and the time of sampling, the measurement indicates either the intensity of a recent exposure, an average daily exposure, or a chronic cumulative exposure.

13.3 Regulations and Exposure Indices

13.3.1 Occupational Safety and Health Administration

Biological monitoring is specified in certain situations by OSHA in substance-specific standards for benzene[3], lead[4], and cadmium.[5] Generally, these standards require biological monitoring if air monitoring data indicate that employee exposure has exceeded a specified threshold. Current regulations should be consulted for details on the requirements for biological monitoring, the frequency and duration of the biological monitoring programs, and corrective action necessary if established concentrations are exceeded.

13.3.2 Biological Exposure Indices

ACGIH® has established biological exposure indices (BEIs®)[6] for 43 organic and inorganic substances and each year propose a list of substances they intend to change or establish. They also list substances and other issues that are under study and solicit information which may assist the ACGIH® BEI Committee in its deliberations. The ACGIH® BEIs® "represent the levels of determinants which are most likely to be observed in specimens collected from a healthy worker who has been exposed to chemicals to the same extent as a worker with inhalation exposure at the TLV®." Like the TLVs®, the BEIs® "do not indicate a sharp distinction between hazardous and nonhazardous exposures."[2]

13.3.3 Other

Biological monitoring guides have also been developed in other countries; the German Research Society, for example, has established Biological Tolerance Values for Working Materials (BATs). For a detailed look at monitoring standards throughout the world, see Cook's Occupational Exposure Limits - Worldwide.[7] It is outside the scope of this document to provide a comprehensive listing of all regulations worldwide, but companies with non-U.S. operations should be aware of and comply with local requirements.

13.4 Program Initiation

13.4.1 Define Scope

As with any program, it is mandatory that the overall scope first be well-defined, both in regard to the activities the program will entail and the results expected of the program. This definition is needed to ensure that the biological monitoring results will provide useful data and will allow appropriate interpretation to accomplish the objective. For biological monitoring data to be meaningful in evaluating and controlling occupational exposure, a detailed understanding is required of a chemical's metabolism and elimination kinetics in the human body. Generally, this information exists for chemicals that have established BEIs® or regulatory biological monitoring requirements.

Biological monitoring can be considered complementary to air monitoring. It is a more complete measure than air monitoring of the true dose received by a worker because all routes of entry and sources of exposure are included. Direct measurement of worker exposure by other routes (i.e. skin and ingestion) can be a challenging project and is beyond the scope of this chapter.

Biological monitoring may be used for a variety of reasons: 1) to comply with regulatory requirements; 2) to confirm air monitoring results; 3) to evaluate the effectiveness of engineering or administrative controls, or personal protective equipment; 4) to estimate dermal or oral exposure; 5) to determine the dose received by workers after responding to emergency situations in which there was no time to prepare air-monitoring equipment; or 6) to identify potential non-occupational exposures. Biological monitoring has proved useful in confirming occasions of dermal or non-occupational exposure in the cases of mercury (fungicides, consumption of fish, etc.) and lead (environmental exposure such as paint).

Although identification of the specific analyte might seem basic to defining the scope of the program, there is an intimate tie between what information you wish to gain from the monitoring, what you will monitor for, and how you will obtain the information. In some cases it is possible to evaluate a combination of acute, intermediate, and chronic exposure. This can be done by appropriately selecting the specific analyte and sample type with an understanding of the biological and metabolic half-life of the material. An excellent discussion of this is found in the "Introduction to the Biological Exposure Indices" in the *ACGIH® Documentation of the Threshold Limit Values and Biological Exposure Indices*.[6]

13.4.2 Populations

Once the decisions have been made on the analyte and time periods of interest, it is possible to determine the populations to be sampled. Normally, these can be broken down into the exposed group and the control group.

13.4.2.1 Exposed Group

Although initially the exposed group might seem to be easily identified, there may be subgroups within the larger population of exposed individuals. In many instances, air monitoring is useful in identifying exposure categories. In an industrial environment, subgroups may be established on the basis of assigned duties, levels of authority, or in some cases, entire shifts. In non-continuous operations — where, for example, there is routinely scheduled downtime over the third shift — maintenance activities carried out on the off-shift might provide a different, perhaps greater, level of exposure than normal production. Other rationales can also be used to establish subgroups (e.g., genetics, smoking, etc.).

Where there are exposure subgroups, variations in the bioassay results among the differently exposed subgroups are expected. When there is significant variation in the monitoring results of a large population, it is advised to obtain detailed information on the work activities of the individuals in the group. This information can help determine whether the variations are due to the presence of subgroups in the population or due simply to differences in metabolism between individuals.

Ideally, every individual in the "exposed" population should be sampled initially. Once a database has been established, based on a minimum of 2 to 3 replicate samplings, the exposed population can be re-evaluated.

13.4.2.2 Control Group

The control group is normally made up of individuals with no industrial exposure to the material(s) of interest. Ideally, controls should be selected to closely mirror the population of the exposed group (i.e. similar age, socio-economic status, etc.). If subgroups have been established in the exposed population, it may be desirable to establish similar subgroups in the control population so that data comparisons can be made between similar exposed and control groups. Samples from the control group are expected to provide normal or background values for the parameter of interest. Although control group samples are somewhat different from blanks in air monitoring, they serve an analogous purpose — to provide a comparison data set to help assess the validity of the data from the exposed group samples.

Selection of the specimen type required for monitoring will greatly impact the availability of control volunteers. The willingness of people to provide samples is normally in direct contrast to the invasiveness of the sample collection procedure. Urine or breath samples are fairly easy to obtain, while for example blood or semen (males only) samples are likely to be more difficult.

Occasionally, unusually high values may be found in the control population, usually as the result of non-occupational exposure to the material being evaluated. For example, elevated lead results were found in samples from a control population as a result of drinking moonshine from a still fabricated using lead solder or background cyanide blood levels in smokers.

13.4.3 Identify a Laboratory

Identifying and choosing a laboratory or laboratories is a critical part of the program and should be completed before samples are collected from the employees. Many laboratories, when approached with a request for analysis, will receive the samples and provide results. However, if the samples are unusual or the analyses requested are not performed frequently or at all by the laboratory, the primary lab may subcontract samples to another laboratory that is more familiar with the analysis, but the submitter should be aware it is happening.

Most commercial laboratories that analyze biological samples are familiar with urine, blood, tissue, etc. However, many are not prepared to deal with breath samples. Because of handling, shipping, and storage problems, breath analysis may be best performed by an in-house or on-site laboratory.

Commercial laboratories and, when possible, in-house analytical laboratories should participate in a variety of quality assurance programs, both internal and external. Although formal programs are not available for all analytes, participation in some aspects of a formal program will provide an indication and a framework for demonstrating that the appropriate quality assurance and control procedures are in place at the laboratory to support the reliability of the program.

The laboratory review should include a discussion of the following issues:

13.4.3.1 Does the laboratory provide sampling and shipping containers with any needed preservatives and instructions?

13.4.3.2 Have chain-of-custody procedures been established for handling of samples?

13.4.3.3 Does the laboratory provide adequate turnaround of samples?

13.4.3.4 Are “rush” analyses available?

13.4.3.5 Can the laboratory provide results by fax, telephone, or electronic data interchange?

13.4.3.6 Will the laboratory provide summary reports as desired on a monthly, quarterly, or annual basis?

13.4.3.7 Will the laboratory provide reports of excursions above your preset limits?

13.4.3.8 Is technical support available for discussion of results?

13.4.3.9 How responsive is the laboratory to efforts to resolve problems?

13.4.3.10 Will the laboratory provide a list of references, analytical methods used, QA/QC procedures, and internal QC data (e.g. control charts)?

13.4.3.11 In what outside proficiency testing programs does the laboratory participate and what are their results?

13.4.3.12 How long will the laboratory maintain records of the analysis of your samples? (Three to five years is typical for “raw” data retention. Currently, there are no existing regulatory requirements.)

13.4.3.13 And, of course, what is the cost, volume discount, multi-site discount, etc.?

The ultimate criterion is laboratory performance. If possible before the final laboratory selection is made, a set of samples having known concentrations should be submitted to the

laboratory disguised as actual biological monitoring samples (see Sections 13.6.2 and 13.6.3 for more information on QA samples). This allows the accuracy and precision of the laboratory's analysis to be evaluated prior to the start of the program. If deficiencies are found, corrective action can be taken (such as working with the lab to improve the results or selecting a different lab).

Once a laboratory has been selected, it should be considered an integral part of the occupational health program. Collaboration with laboratory personnel regarding the scope and objectives can often lead to significant improvements in the overall biological monitoring program.

13.4.4 Confounding Effects of Non-occupational Exposure, Other Possible Interferences, and Specificity of Tests

One of the strengths of biological monitoring that makes it unique from airborne exposure monitoring is that biological monitoring includes all exposures, non-occupational as well as occupational. For those chemicals also present in the non-occupational or recreational environment, this can complicate the interpretation of results.

Heavy metals, for example, can be found in non-occupational settings that might cause elevated concentrations in the blood or urine of workers. Elevated concentrations of lead in blood can result from exposure to lead from residential plumbing or paint, or from ammunition used in recreational use of firearms. Elevated mercury and arsenic levels have been found to correlate with consumption of seafood. Other instances of non-occupational exposure to metals can be found through literature searches of medical databases.

In some cases, the biological monitoring analysis is not specific for a particular chemical but might be for a metabolite or an indicator of the chemical's effect. A recent example is the testing required under the OSHA standard for cadmium[5], which includes a test for ß-2-Microglobulin in urine as well as cadmium in urine and blood. The ß -2-microglobulin is a protein that is used as an indicator of cadmium's adverse effect on the kidneys; however, it is not specific to an effect on the kidneys just by cadmium and might be the result of other unrelated kidney problems. Other similar tests evaluate the impact on an organ where the effect might be from more than one cause.

13.4.5 Employee Communication

Even when not required by OSHA regulations, communication of the biological monitoring program to employees is critical to the program's overall effectiveness. Like other aspects of industrial hygiene, employee cooperation and understanding of the goals of the monitoring program is key to its success. The educational level of the employees, and their possible religious beliefs maybe a significant issue, resulting in a refusal to participate. The details of employee communications should be considered early in planning the program. It is important to determine the form of the communications and the content. The individual characteristics of the plant facility will contribute to the style of the communication and help to determine the means of informing the workers via individual letters, notification by the facility medical staff, use of charts and graphs, etc. In some cases, collective bargaining issues may also have an impact on how such communications are made, and whether other individuals are informed of the results.

It is also important to note that biological monitoring results of occupational exposure to a specific chemical, like air monitoring, are considered to be exposure monitoring, not medical results.[8] As such, they can be communicated to personnel who were not monitored but similarly exposed; however, some parameters that are measured in a biological monitoring program (e.g., ß -2-microglobulin in the case of cadmium exposure) are not unique to a specific exposure and might be a result of a pre-existing medical condition. Careful consideration therefore must be given when communicating results to employees who were not monitored, taking care to protect the privacy of those who were monitored.

13.4.5.1 Purpose and Nature of the Program

Employees should be thoroughly briefed on the purpose and nature of the biological monitoring program. Once the program is communicated properly, most employees will support it, albeit grudgingly in some cases, especially if they consider the program to be in their own best interests. Understanding the reasons behind the program and what is being monitored can benefit employee behavior. Employee awareness that program results can pinpoint improper handling of chemicals may improve conformance with proper procedures.

Initially, employees might incorrectly interpret a biological monitoring program as a means of using them as "guinea pigs" or "canaries." This misconception can usually be corrected by an educational session, possibly conducted jointly by an industrial hygienist and an occupational physician or nurse. An educational session gives the employees an opportunity to ask questions of knowledgeable individuals who are usually considered trustworthy. It is essential that the industrial hygienist and occupational physician be completely honest and straightforward during the session, especially with the difficult questions, and not be reluctant to admit a lack of knowledge when appropriate.

13.4.5.2 Employee Questionnaire

Because of potentially confounding factors, possible interferences, and non-occupational exposure, it is recommended that employees in both the exposed and control groups complete a questionnaire aimed at identifying these factors. This frequently can be done as part of a normal medical exam or conducted when the sample is collected. Good examples of questionnaires can be found in the OSHA chemical standards, such as those for cadmium[5] and asbestos.[9]

13.4.6 Concurrent Exposure Monitoring

Concurrent air monitoring should routinely be conducted as part of a biological monitoring program. The timing of the air monitoring may vary, however, depending on whether the exposure is short or long term. For instances in which biological monitoring is a measure of acute exposure, it is important that air monitoring be done very close in time to the biological monitoring, preferably covering the identical exposure period.

When the effect being evaluated reflects a chronic exposure, the time overlap between biological and air monitoring is not as critical. In this case, air monitoring must be sufficiently detailed to adequately characterize the overall level of exposure and be able to differentiate between dissimilar employee populations.

Measuring other routes of exposure, such as dermal or oral, can be attempted, but procedures for doing so are less well developed than for air monitoring.

13.4.7 Program Plan

As a final step before beginning a biological monitoring program, the considerations above (Sections 13.4.1 through 13.4.6) should be documented in a brief program plan. The program plan should also address reporting of results and follow-up actions (see Section 13.7). This will help ensure that all individuals are working together with a common understanding. It is even more important to do this if a facility has not conducted any biological monitoring in the past.

13.5 Sample Collection

13.5.1 Timing and Frequency

As in air monitoring, collection of samples is key to producing meaningful data. Samples that are not collected in the proper time frame might give misleading results. Specifications for the timing of sample collection are usually included in the supporting information of the regulation or the documentation of the BEI.

For some chemicals, the compound(s) and/or its metabolite(s) may be rapidly eliminated from the body. In such cases, the elimination kinetics will dictate when the biological sample should be collected: during the shift, at the end of the shift, at the end of the workweek, etc. For these materials, samples must be collected at the same time relative to the work shift to provide comparable data.

Chemicals that are not rapidly metabolized or eliminated will accumulate in the body (e.g. lead, PCBs, etc.). For these types of materials the timing of sample collection is not as critical because the samples will reflect exposure over long periods of time.

To attempt to get a measure of true occupational exposure, samples should be collected prior to exposure to establish "baseline" data. For rapidly metabolized materials this can be accomplished simply by sampling immediately prior to the beginning of the work shift. Baseline data for slowly metabolized materials may only be available by sampling at the beginning of employment or prior to a new work assignment.

It is difficult to give specific guidance on the number of samples and the frequency of sampling campaigns. Ideally, every individual in the exposed population should be sampled. There can be significant differences between individuals' metabolisms, so selecting a few individuals to be representative of a larger population is not appropriate, as is the case with air sampling. Populations exposed to chemicals that are rapidly metabolized will require more sampling (every day for 3–5 consecutive days) than populations exposed to chemicals that are slowly metabolized (a single sample from each individual used to indicate cumulative exposure).

If biological monitoring has not been done previously, more samples are likely to be collected at the beginning until a database can be accumulated that will indicate the uniformity of the population or certain individuals or job tasks that receive greatest exposure. Also, sampling frequency might be dictated by regulations if measured concentrations exceed critical values. For example, the OSHA Lead Standard requires that any employee with a blood lead level >50 µg/dL must be resampled within 2 weeks.[4,10]

13.5.2 Containers, Preservation, Holding Times, and Shipping

Proper sample containers and preservatives are necessary to avoid contamination or possible loss of analyte(s). In most cases, containers and preservatives are specified in the regulations or the documentation supporting the BEIs. The laboratory selected for sample analysis will frequently supply the proper containers with the preservative already added. The laboratory should also supply detailed written instructions on the proper use of the sample containers, timing of the sample, and shipping precautions. Immediately before collecting samples it is important to make adequate preparations such as removing contaminated clothing and washing the hands or skin. When collecting samples it is important to collect them using only the specified container(s), avoiding any intermediate collection vessels or sample transfers that have not been designated specifically in the sampling plan. Extra steps or apparatuses increase the possibility of sample contamination, especially for metals or non-metabolized organic compounds.

Blood samples are usually collected in sterile tubes containing the anticoagulant and fitted with a top compatible with the analyte of interest. As a rule of thumb, the volume of blood

drawn should be equal to 2.5 times the volume required for analysis. Urine samples are usually collected in large mouth cups made of plastic or glass. Breath samples can be collected in glass bulbs, inert gas sampling bags, on solid sorbents, or exhaled directly into the analysis instrument (e.g., the measurement of blood alcohol).

Holding times and storage conditions are different for each chemical/determinant. Metabolites and organic analytes generally have shorter holding times than metals. Some analytes might require refrigeration immediately after sample collection. Storage conditions also apply to the time the samples spend in shipment to the analytical lab, sometimes requiring overnight shipment on ice. Again, the regulations or documentation supporting the BEIs usually contain guidance on holding times and storage conditions. Breath samples can present special problems because of the possibility of the analyte condensing or reacting with the walls of the sample container. These difficulties can be reduced by minimizing sample holding time and analyzing the samples immediately if possible. To account for the possible effect of storage times and conditions, quality assurance samples (see Section 13.6) should be prepared or obtained at the same time the samples are collected, and they should be stored and shipped with the samples.

13.5.3 Qualifications of Personnel

Since biological monitoring involves collection of biological samples, it is important for personnel involved in sample collection to be under the supervision of qualified personnel. A medical doctor (MD), registered Medical Technician (MT), or registered nurse (RN) should qualify. Invasive sampling procedures required for blood or fat samples must be performed only by personnel with adequate training and may not be delegated to others.

13.5.4 Documentation

Documentation of the sampling conditions and the work environment is required to allow a meaningful interpretation of the data. It is especially important to note whether the sampling was done as part of a routine effort to monitor normal working conditions or if unusual events occurred during the work period. Any pre- or post-sampling questionnaires, shipping documents, and analytical lab report forms should be archived as well. Many testing laboratories are also medical laboratories and provide an analysis request form with spaces for the following: patient information, sample collection information, name of the representing physician, specimen description, name of the person collecting the specimen, and description of the tests requested. In lieu of preprinted laboratory forms, a custom form can be developed that should include the items above and address sample chain-of-custody.

13.5.5 Safety Considerations

All personnel — employees and employers — must be familiar with OSHA regulations on bloodborne pathogens.[1] Hazards associated with handling of biological fluids must be explained to each employee during training sessions. Methods to minimize exposure (such as engineering and work practice controls, personal protective equipment, housekeeping, and proper labeling) must be part of the overall program.

13.6 Sample Analysis

13.6.1 Laboratory Certification, Licenses, or Permits

Under the Clinical Laboratory Improvement Act (CLIA) of 1988, any laboratory analyzing biological samples must obtain a CLIA Certificate. Laboratories performing biological monitoring analyses will require a High Complexity Testing Certificate. Laboratories can be accredited by the College of American Pathologists (CAP), by individual states, or by any other accrediting agency approved by CLIA.[11]

If there is a need to analyze biological samples for drugs, National Institute on Drug Abuse (NIDA) certification may be required for drug testing of certain occupations (e.g. truck drivers, airline pilots, etc.) but it is not necessary for all situations.[12] For many laboratories, CAP drug testing accreditation ensures quality testing without the high expense of the annual NIDA certification. The laboratory must be licensed by the federal Nuclear Regulatory Commission (NRC) to analyze for radioactive material.[13] Some state regulatory agencies may also require licenses.

13.6.2 Quality Assurance Program

The laboratory should have implemented a formal QA program. The program should describe the procedures used to ensure high quality data (e.g., personnel qualifications and training, documentation of laboratory procedures, instrument calibration and maintenance) and the control measures taken to monitor and, when necessary, to improve the laboratory's results (e.g., QC samples, control charts, resolution of deficient performance).[14–17] If requested, the laboratory should be willing to provide a copy of its QA manual.

13.6.3 Performance — Accuracy, Precision, and Limit of Detection

To allow for meaningful interpretation, the data must have accuracy and precision sufficient to allow samples from unexposed individuals to be clearly distinguished from regulatory levels or BEIs. A laboratory should have an established QA/QC protocol to ensure the validity of the results. A laboratory should be able to specify its criteria for accuracy and precision of results and how they are applied to each set of samples analyzed.

Limit of detection (LOD) and limit of quantitation (LOQ) of the analyte in a particular sample matrix (e.g., cadmium in urine) depend on the method being used for the analysis and can vary from laboratory to laboratory (see Chapter 8 of this manual for further discussion of LOD and LOQ). Many laboratories do not distinguish between LOD and LOQ. Consult the laboratory for its LOD/LOQ and how it is determined for the particular analyte and matrix involved in your biological monitoring program. As a general guide, the LOD or LOQ should be no more than one-tenth the regulatory level or exposure index.

13.6.4 Safety Considerations

All laboratory personnel, such as the specimen-receiving technicians and laboratory analysts, should be familiar with the OSHA requirements on bloodborne pathogens.[1] Hazards associated with handling of biological fluids must be explained to each employee during training sessions. Methods to minimize exposure (such as engineering and work practice controls, personal protective equipment, housekeeping, and proper labeling) must be part of the overall program. Compliance with the OSHA laboratory chemical hygiene standard[18] helps in meeting these objectives.

13.7 Quality Assurance/Quality Control

13.7.1 General Considerations

This section deals with QA/QC considerations external to the laboratory, from the point of view of the sample submitter. It is not intended to take the place of a laboratory's internal QA/QC program. As with any other type of sampling or monitoring, some level of QA/QC is necessary to provide a degree of confidence in the results obtained. Because of the type of samples used in biological monitoring, the QA/QC procedures are somewhat different than those commonly in use for air monitoring. The type of QA/QC samples used, however, fall into the same general categories of knowns, spiked samples, blanks, and duplicates.

13.7.2 Availability of Standard Reference Materials

NIST provides a number of biological standards containing a variety of toxic materials at known concentrations.[19] These materials are all certified and include acceptable ranges around the reference value. The reference materials for urine are normally supplied as a dried material requiring only the addition of high purity water to reconstitute the sample. Once in liquid form, they can then be submitted to the laboratory as if they were a routine sample. In some cases, dried blood specimens are similarly available.

The following facilities, in addition to NIST, are among those that provide urine and blood containing known concentrations of contaminants. This is done either as part of an inter-laboratory proficiency testing program or as a service. [**NOTE:** This is not a comprehensive list. Inclusion or exclusion of facilities, however, does not necessarily represent an endorsement by AIHA®.]

Centre de Toxicologie du Quebec
Le Centre Hospitalier de l'Universite Laval
2705 Blvd. Lurier
Quebec, Quebec G1V 4G2
Canada
(418) 654-2100
http://www.inspq.qc.ca/ctq/default.asp?Page=1&Lg=en

Kaulson Laboratories, Inc.
691 Bloomfield Ave.
Caldwell, NJ 07006
(201) 226-9494
http://www.kaulsonlab.com/

Biorad Laboratories
Quality Control Division
9500 Jeronimo Road
Irvine, CA 92618
(800) 854-6737
http://www.bio-rad.com/

Utak Laboratories, Inc.
25020 Avenue Tibbitts
Valencia, CA 91355 (800) 235-3442
http://www.utak.com/

Accurate Chemical and Scientific Corp.
300 Shames Dr.
Westbury, NY 11590
(516) 333-2221
http://www.accuratechemical.com/

Reference materials for breath analysis are not commercially available. Standard gas mixtures can be purchased, but they do not contain the potentially interfering species in human breath. Standard gas samples can be used to spike breath samples by volumetric addition, which can provide an indication of the accuracy of the analysis.

13.7.3 Preparation of Spiked Samples

Because of difficulties in accurately spiking biological samples with known concentrations of chemicals, it is usually preferable to purchase known samples (see above). If samples of known concentration are not available, spikes should be prepared by qualified laboratory personnel

only, such as analytical chemists. Spiked biological samples should be generated with routines similar to those used for preparing spikes of other liquid media.

The least difficult media to handle is urine. Standard concentrations of metals in urine can be prepared from commercial concentrated standards for atomic absorption spectroscopy, using urine as the final diluent. If possible, the volume of the spiking solution added should be less than 1% of total solution volume. In some cases it will be necessary to choose the contaminant species carefully to be sure it remains soluble at the urine's pH. Solubility is usually not a problem if an aliquot of the spiked urine is transferred to a standard sample container with preservative immediately after it is spiked.

Because of the limited volume of available material, at least when compared with urine, the techniques for spiking blood samples are somewhat different. Spikes can be made using a microsyringe directly into the vacutainer tube in which the blood was collected. To calculate the quantity of analyte to add, the amount of blood in the tube can be estimated to within 5% by filling a spare tube with water to an equivalent level and then measuring the amount of water in a graduated cylinder. To have the minimum effect on the sample, the spiking solution should be of minimum volume with a correspondingly higher concentration (e.g., 10–20 µL spiked into 5–10 mL of blood).

The actual concentration of the material of interest should be determined for a portion of the nonspiked sample as well. This will allow the determination of the recovery from the spiked sample. Ideally, spikes should be submitted in duplicate unless the reproducibility of the analysis is known.

Spiked samples submitted to the laboratory should be prepared over a range of concentrations (e.g. 0.1, 0.5, and 1 times the regulatory level or BEI). To be a reliable indicator of method performance, the spiked sample concentration should be at least 2 to 5 times greater than the nonspiked sample. If the amount added (spiked) is equal to or less than the amount already present in the sample, then the inherent variability in the analysis of the spiked and nonspiked sample can have a disproportionately large effect on the calculated percent recovery. Spikes at low levels (0.1) are best prepared using samples from the control population for which the background concentration is expected to be lowest.

13.7.4 Blanks and Duplicates

Blanks and duplicates are the easiest forms of QC tests available. True blanks, the matrix containing everything but the analyte of interest, for biological monitoring do not exist; however, samples from the control population are sometimes considered to be blanks. In most manufacturing facilities, samples from nonexposed individuals can be obtained from the office staff, workers in areas remote from the area of concern, samples collected from nonexposed individuals during routine physicals, or from the medical staff. Although these are not blanks in the true sense of unexposed sampling media, they may be the best substitute available for biological monitoring studies. In the case of very rapidly metabolized compounds (half-life of 2–3 hours or less), preshift samples from an individual may be the best control sample for that individual.

Duplicate samples can be prepared by splitting a sample from an exposed individual. This is easily done for urine samples if the volume is adequate. If the samples are prepared without a preservative, after collection, the sample can simply be split into two containers. Alternatively, the submitter may be asked to donate an additional sample. For blood collection, a second tube can usually be drawn with little difficulty.

13.7.5 Frequency of Quality Assurance Samples

The frequency of submitting samples of known concentration, blanks, and duplicates normally reflects the availability of the samples and the ease and expense of obtaining them. Duplicates

will normally be obtained most easily, followed by blanks. Commercially available or specially prepared spikes can be difficult to obtain and may not be used as frequently.

On a routine basis, at least one set of duplicates, and preferably a blank as well, should accompany each set of samples submitted to the laboratory. This allows for a recurring evaluation of the reproducibility of the laboratory's testing. Because of the difficulties in preparing the samples, spikes may be used less frequently; however, the routine submittal of either known spikes or purchased standard reference materials should be a part of a periodic evaluation of the laboratory. Depending on the numbers of samples submitted and the criticality of the analytical results, the submission of spikes may be done at a frequency that varies from monthly to annually.

13.7.6 Corrective Action

If some data points are suspected to be erroneous, it might be possible for the lab to reanalyze the sample(s) in question. This might not be possible if the lab discards samples soon after analysis or if the sample is totally consumed in the analysis. If the parameter being measured is used to assess chronic exposure and the half-life of the parameter is very long compared with the time elapsed since the sample was taken, another sample can be collected from the individual for analysis.

Poor results on QA samples or unexpected results from the control population might require the basic design of the study to be reconsidered. Consult documentation for the BEIs or the analytical lab for "normal" (non-occupationally exposed) concentration ranges.

Elevated control values may be caused by contaminated sampling equipment, inadequate analytical method, exposure from unanticipated sources, or — especially in the case of a nonspecific parameter such as ß-2-microglobulin for Cadmium — confounding effects possibly unrelated to the compound of interest. Recoveries from reference or spiked samples should generally fall in the range of 75%–125% of the nominal value. Values outside this range can be caused by an inadequate analytical method, sample decomposition (especially if the analyte is an organic compound), or an improper spiking technique.

13.7.8 Proficiency Testing

There are a variety of proficiency testing programs similar to the Proficiency Analytical Testing (PAT) program for air sample analysis administered by AIHA. Participation in an approved proficiency program (run by the College of American Pathologists [CAP], the New York State Department of Health, or the Wisconsin State Laboratory of Hygiene) for Blood Lead is mandatory for laboratories conducting analyses in support of the requirements for testing under the OSHA lead standards.[4,10] The "OSHA List of Laboratories Approved for Blood Lead Analysis" is available from OSHA[20] and is updated periodically. Each laboratory is graded for twelve months and with greater than 89% acceptable sample results, the laboratory is approved. In 2009 there were over 190 laboratories on the OSHA-approved list.

A number of other inter-laboratory testing programs are available for analytes other than Lead. The CAP has proficiency testing for several trace metals in urine, serum, and blood. The Centre de Toxicologie du Quebec (referenced in section 13.7.2) conducts inter-laboratory studies on arsenic, lead, mercury, chromium, selenium, fluoride and cadmium, among others. Samples are provided bimonthly. The participating laboratory is provided with both a bimonthly and annual report of the laboratory's results. The Finland Institute of Occupational Health runs a program that includes several organic solvent metabolites: mandelic acid, methylhippuric acid, phenol, phenylglyoxylic acid, trichloroacetic acid, and 2,5-hexanedione.

Although the PAT program is aimed at providing quality assurance for analysis of a limited number of materials (asbestos, cadmium, lead, zinc, silica and a small number of organics) using air sampling media, it is required that laboratories in the program participate in those

areas in which they routinely do analyses. Similarly, a laboratory conducting biological monitoring should also participate in a proficiency testing program for those materials for which it routinely conducts analyses, provided that they are available. Customers of a laboratory should request summaries of the lab's performance in the testing program to help assess the lab's accuracy and precision. If not done as a matter of course by the laboratory, participation in proficiency testing, when available, should be included as part of your contract for services.

13.8 Reporting Results

13.8.1 Interpretation

Results should be reported in a timely manner to those individuals being monitored. Monitoring reports should state the value found in the biological monitoring sample, the regulatory limit or BEI, the value expected in a normal non-occupationally exposed population, and the laboratory's limit of detection. Additional information on limit of quantitation, accuracy, and precision can also be reported, but these can be a source of confusion to the layperson and can focus attention away from the most important issue — comparison of the individual's value to the BEI or regulatory limit. Consideration should also be given to reporting summary results to individuals who were not monitored but who were similarly exposed.

If results differ markedly from those expected based on air monitoring data, there should be discussions with the employee to determine if significant exposure may occur dermally, via ingestion, or non-occupationally. An occupational physician who is familiar with the chemical of interest and/or the plant operations can often aid in the interpretation of the results and provide the proper perspective for interpreting "abnormal" results. Other factors to consider when interpreting results include regional variations across the country, age, sex, and information received via the employee questionnaires such as hobbies.

13.8.2 Follow-up

The results of a biological monitoring program might call for a variety of follow-up actions. As with air monitoring, control measures such as engineering controls or personal protective equipment may be justified if measured concentrations approach established limits. Data from individuals that exceed a regulatory limit or BEI may justify further medical tests and/or removal from the job. These actions must be coordinated with the facility's occupational medicine group and might even be required if it is determined that most of the exposure occurs off the job.

OSHA has established a minimum period of 30 years past employee retirement for retention of monitoring records. A summary of results obtained on quality assurance samples should also be maintained as an aid in determining the significance of results.

13.9 References

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Chapter 14
Quality Assurance Guidelines for Microbiological Samples

by Mary Eide

14.1 Introduction

The information in this chapter will aid laboratories and analysts in meeting the challenges unique to analysis of samples containing microorganisms. The purpose of the analysis is in some cases to identify the microorganism, but also to determine concentration in bulk material or air samples. Laboratories performing these analyses should be accredited through the AIHA-Laboratory Accreditation Programs, LLC (AIHA-LAP, LLC) Environmental Microbiological Laboratory Accreditation Program ((EMLAP).[1] They should also follow the policies of AIHA-LAP, LLC.[2] This program is intended for accreditation of microbiological laboratories specializing in the analysis of microorganisms commonly detected in air (e.g., spore trapping), surface (e.g., tape lifts, swabs, wipes), and bulk (e.g., dust, liquids, building materials) samples collected from schools, hospitals, offices, industrial, agricultural and other work environments.

14.2 Facilities

The laboratory needs to have the proper facilities. The facility should meet the requirements of the appropriate and most current biosafety level guidelines, as defined by CDC/NIH[3], WHO[4] and AIHA.[1] The laboratory should have a documented routine monitoring program to verify they have adequate contamination control. The laboratory must have proper facilities for biological and chemical storage and disposal of waste.

14.3 Equipment

The laboratory should utilize a microscope/magnification system suitable for performing the methods in use at the laboratory (e.g., capable of the magnifications required). The alignment should be documented each day of use for each microscope/magnification system used. The laboratory should have a reference library appropriate to the Fields of Testing FoT(s) to be accredited.

14.3.1 Non-fluorescence Microscopy

Analysis of microbiological samples by non-fluorescence microscopy should include the following equipment:

14.3.1.1 A compound optical microscope having a high magnification (e.g., 100x) liquid immersion objective having a numerical aperture (n.a.) of at least 1.25; or,

14.3.1.2 An optical microscope having a theoretical or calculated point to point resolution at 0.34 µm or better. The resolution is calculated as follows: 1.22 x 0.55 µm/ [condenser n.a. + objective n.a.]; or,

14.3.1.3 A magnification system having a measured optical resolution of 0.34µm or better. For example, the optical resolution may be measured with resolution target testing slides.

14.3.1.4 Each non-fluorescence microscope should have an ocular micrometer which is checked annually with a stage micrometer.

14.3.2 Fluorescence Microscopy

A microscope used for fluorescence microscopy should have a non- immersion objective of at least 40X magnification, and should be used in conjunction with oculars of at least 10X magnification.

14.3.3 Culturable FoT(s)

14.3.3.1 The laboratory should have a Class II biological safety cabinet (BSC) whose performance has been certified by a NSF accredited field certifier according to NSF Standard 49 field requirements (or national equivalent outside the U.S.).[5] Annual certification is required and needs to be documented.

14.3.3.2 The laboratory should have a steam sterilizer (autoclave) with functioning temperature and pressure gauges or a contract with a biohazard waste disposal company for the disposal of potentially viable waste.

14.3.3.3 Laboratories with steam sterilizers should use indicators to document successful sterilization with each use.

14.3.3.4 Laboratories with steam sterilizers should use biological indicators (e.g. spore strips or ampoules) with each use or at least once a week, whichever is less to document the sterilization process.

14.3.3.5 The laboratory should have incubators, refrigerators and freezers with temperature settings appropriate for the scope of work performed at the laboratory.

14.4 Personnel

The laboratory should conform to the personnel requirements as specified in Module 2A, Section 2A.5.2 (and all sub-sections), and to the requirements as detailed in the following sections. In all cases, training records for degreed laboratory staff should include a copy of the transcript or diploma from an accredited college/university, and a copy of all training courses pertinent to their job.

14.4.1 Technical Manager

Qualifications of the Technical Manager in addition to those in Module 2A are:

14.4.1.1 The Technical Manager should be experienced in the selection and use of bioaerosol, surface, fluid and raw material sampling methods and in sample processing for the quantification and identification appropriate to the FoTs of mesophilic and thermophilic bacteria, and mesophilic, xerophilic, thermotolerant fungi (molds and yeasts), and fungi identified by spore trap collection methods.

14.4.1.2 Training records for the Technical Manager should include documentation of ability to identify genus/group of fungi from spore trap analysis and genus/species of fungi that are reported. Bacterial identification training records should document training of relevant diagnostic procedures (e.g., gram stain, oxidase, biochemical reactions) as appropriate to the FoT(s). Legionella training records must include documentation of relevant diagnostic procedure (ability to recognize presumptive colonies, confirmation using DFA, latex agglutination, or molecular methods).

14.4.2 Laboratory Analytical Staff

The environmental microbiological program should distinguish the two titles below for those conducting analytical procedures within the laboratory.

14.4.2.1 Laboratory technicians should have a high school diploma or General Education Development (GED). During this required training period, the trainee should perform work (and have work reviewed prior to release) under the direct supervision of a qualified technician, analyst and/or the Technical Manager. Technicians may function in the same manner as analysts for Air - Direct Examination (spore trap) analysis after completion of six (6) months documented on the job training and demonstrated proficiency. For all other analyses, technicians may function in the same manner as analysts after one (1) year documented on the job training and demonstrated proficiency. All technicians should demonstrate continued ability to produce reliable results through accurate analysis of proficiency testing samples, quality control samples, and quality assurance samples.

14.4.2.2 Laboratory Analysts should have a bachelor's degree in a physical or biological science. Analysts should have three (3) months of documented training for Air - Direct Examination (spore trap) and six (6) months of documented on-the-job training functioning for all other analyses as an analyst trainee. During the required analyst training period, the trainee should be under the direct supervision of another qualified analyst and/or the Technical Manager. During this period, the trainee should have all work reviewed prior to release by another qualified analyst and/or the Technical Manager. All analysts should have demonstrate continued ability to produce reliable results through accurate analysis of proficiency testing samples, quality control samples, and quality assurance samples.

14.4.3 Training Records

Training records for technicians and analysts should include documentation of ability to identify genus/species of fungi and genus/group of fungi that are reported. Bacterial identification training records should document training of relevant diagnostic procedures (e.g., gram stain, oxidase, biochemical reactions). Legionella training records must include documentation of relevant diagnostic procedure (ability to recognize presumptive colonies, confirmation using DFA, latex agglutination, or molecular methods). All analysts and technicians should have demonstrated ability to produce reliable results through accurate analysis of certified reference materials (CRMs), proficiency testing samples or in-house quality control samples. This demonstration should be performed and documented at a minimum of every six (6) months.

14.5 Analytical Methods

For quantitative testing procedures, the laboratory should establish and verify the minimum reporting limit(s) and linear ranges annually. This must be completed and documented for each test and matrix. Records should be stored along with these quantitative test records. The laboratory should have written Standard Operating Procedures (SOPs) for the following:

14.5.1 Processing and analysis of samples

14.5.2 Determining analytical sensitivities for each quantitative or semi-quantitative method

14.5.3 Appropriate retention, waste treatment and disposal of environmental microbial samples

14.5.4 The identification of fungi and/or bacteria

14.5.5 Identification of fungal spores and structures

14.5.6 Biosafety and decontamination for the applicable FoT(s).

14.5.7 Additionally there are requirements for air fungal direct examination FoT that the analytical methods should include a description of sample trace analysis, scope magnification, counting rules, percentage of trace analyzed and calculations.

14.6 Quality Assurance/Quality Control

Routine quality assurance/quality control procedures should be an integral part of laboratory procedures and functions. The laboratory Quality Assurance program should address the elements in AIHA-LAP, LLC Module 2A, Section 2A.4.2.1 and should also include the following additional elements specific to microbiological analyses.[1]

14.6.1 General Elements

14.6.1.1 Compliance with acceptable quality assurance and quality control guidelines for microbiology laboratories, such as APHA-AWWA-WPCF guidelines in *Standard Methods for the Examination of Water and Wastewater, The Manual of Environmental Microbiology*, or equivalent national guidelines for foreign laboratories.

14.6.1.2 To assess precision, intra-analyst analyses should be completed at a minimum of five (5) percent, or at least one (1) each month samples are received, whichever is greater.

14.6.1.3 To assess accuracy, inter-analyst analyses should be completed at a minimum frequency of five (5) percent or at least one (1) each month samples are received, whichever is greater.

14.6.1.4 The laboratory should use control charts or quality control databases to compare intra- and inter-analyst analysis performance to established control limits.

14.6.1.5 The laboratory should ensure quality control of culture media and analytical reagents per lot number for appropriate sterility, microbial growth and/ analytical reactions. Records should be maintained. Acceptance criteria should be documented.

14.6.1.6 Acceptance criteria on 5% replicate and duplicate analysis, daily reference slide analysis (spore traps) and monthly reference culture analysis (all culturable FoTs) should be documented. Acceptance criteria should include:

a) Taxon identification acceptability
b) Taxon abundance ranking acceptability
c) Count or concentration acceptability determined statistically (quantitative QC analysis only)

14.6.2 Additional Laboratory Requirements for All Culturable FoTs

14.6.2.1 The laboratory should keep routine temperature documentation of refrigerators, freezers and incubators. Acceptance criteria should be documented.

14.6.2.2 The laboratory should maintain a microbial culture collection of common organisms relevant to the applicable FoT(s). Cultures should be from recognized sources when possible. Source and date of acquisition for each culture should be documented. Procedures for maintaining the cultures and using them for training and QC purposes should be available.

14.6.2.3 The culture collection should be used at least monthly to prepare blind cultures to be used as part of the routine QC program to monitor accuracy in culture identification.

14.6.2.4 Additional Requirements for Fungal Direct Examination Air FoTs

14.6.2.4.1 A slide collection should consist of field samples with various count levels and genera/groups of spores should be maintained and used as part of total spore analysis quality control. For each day of analysis, at least one slide from this collection should be reviewed by each analyst. Analysis should be consistent with the method for field samples. Slides should be reviewed on a rotational schedule such that a different slide is reviewed each day until the entire slide collection has been examined. The analysis of these slides should be incorporated into the daily QC plan. Acceptance criteria for spore concentration(s) for each reference slide should be stated. The upper and lower control limits should be statistically calculated based on three (3) standard deviations from the reference slide means.

14.6.2.4.2 For the Fungal Direct Examination Air FoT, the laboratory should participate in and have documentation of a round robin slide exchange of real samples consistent with the requirements of AIHA-LAP, LLC Policy Module 6. The following are additional requirements:

a) Analytical data should include raw counts and final concentrations for each fungal structure observed.
b) Acceptance criteria should be determined and take into account organism identification, ranking and quantification.
c) The traverse width or field of view to be used in calculations for each microscope should be documented at least annually, if applicable.

14.6.3 Proficiency Testing

Participation in AIHA PAT Programs, LLC's Environmental Microbiology Proficiency Analytical Testing (EMPAT) program or an equivalent proficiency testing program approved by AIHA-LAP, LLC is a prerequisite to qualification under the AIHA-LAP, LLC Environmental Microbiology Laboratory Accreditation Program (EMLAP). Laboratories in the EMLAP are required to analyze samples for those Fields of Testing (FoT)/Method(s) for which accreditation is sought, according to the approved EMLAP Scope/PT list maintained on the AIHA-LAP, LLC's website.[1]

Laboratories participating in an AIHA-LAP-approved proficiency testing program to seek accreditation for the EMLAP should conform to all proficiency testing requirements as outlined in this module.

14.7 Results

The laboratory's results should address the elements in Module 2A, Section 2A.5.10 and should also include the following additional elements:

14.7.1 Reports should include raw counts, which are actual counts without extrapolation or calculation.

14.7.2 For quantitative results, the analytical sensitivity should be stated in the final reporting units. The analytical sensitivity is the lowest concentration that can be detected, which for microbiological samples is 1 raw count per amount or portion analyzed and calculated, expressed in the reporting units.

14.7.3 For analyses utilizing multiple dilutions and/or varying percentages of sample and/or trace analyzed, the applicable analytical sensitivities should be reported.

14.8 Safety, Health, Environmental, and Transportation Regulations

Laboratories accredited under EMLAP are expected to follow all applicable federal, state, and local regulations regarding safety, health, environment or transportation. Potentially viable microbial waste should be collected in properly designated biohazard containers and disposed of properly, either by autoclaving, sterilizing, or incinerating, or by contracting with a biohazard waste disposal company. Failure to comply with applicable federal, state and/or local regulations regarding safety, health, environment or transportation may result in suspension, denial, or withdrawal of EMLAP accreditation.

14.9 References

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Chapter 15
Quality Assurance Considerations for Radioactive Samples

by Linda Youmans

15.1 Introduction

The analysis of radioactive samples (like any sample within the Scope of your AIHA® accreditation) involves the policies of AIHA® Laboratory Accreditation Programs, LLC[1] in addition to the policies of the U.S. Nuclear Regulatory Commission (NRC).[2] When evaluating the quality assurance needs in a radiological environment, the purpose is not to contradict or to lessen requirements, but to consider the challenges associated with that work that are "above and beyond" what is necessary for non-radiological sample handling. Depending upon the radiological environment, there may be no differences, or they may be significant. For example, a low level radiological sample may often be handled in the same way as a chemical hazard, but a highly radioactive sample could require containment, time restrictions, and handling tools that greatly affect the analysis process. As with biological and some chemicals, there may not be any NIST traceable standards so other reference materials need to be evaluated to meet quality control requirements.

15.2 Specific Challenges to Sampling and Analysis in Radiological Environments

Radiological environments, sample types, and overall design vary greatly. The challenge is that each laboratory evaluates potential differences and that the quality plan addresses results of this evaluation along with any contingencies/limitations. The following list of challenges is not all inclusive, but is representative.

15.2.1 Remote Containment and Radiological Worker Challenges

The use of remote containment systems when handling higher level radiological samples can be a big factor in quality during sampling or analysis. Performing work inside a glovebox or using a remote manipulator transforms a simple task such as replacing a cap, or pouring a solution, into an action requiring great skill.

Equipment operation itself may also change when adapted for radiological work. Instead of using a microscope eyepiece to view a sample, the worker may be required to view a sample through a camera lens, which often affects resolution. Radiological protective clothing/equipment (i.e. additional layers of gloves, finger dosimetry, respiratory protection, fresh air supply suits) affects worker agility/precision. Like the sampling/analytical method itself, the skills necessary to perform radiological work are greatly dependent on the individual and his/her experience.

15.2.2 Assumptions

When determining/evaluating method performance, differences between samples analyzed in radiological vs. non-radiological environments are often overlooked. Proficiency samples, not typically radiological, often bypass the radiological laboratory. Setup testing, or method development testing, is sometimes performed in a clean area and method detection limits, control limits, and overall performance are based on these test results.

15.2.3 Analytical Interferences

Common test methods often do not address interferences caused by radiological components. It is very important that this is evaluated before the method is approved for use and that steps are added to continue evaluation when necessary.

15.2.4 Waste

A novel dimension is added to waste handling when radiological components are included. Waste handling becomes not just "an end of the day activity". Maintaining cleanliness and removing clutter throughout the process prevents an accumulation of radioactive materials. Increased activity may invalidate current radiological postings and result in more stringent regulatory/handling requirements. Periodic housekeeping also decreases clutter. Just one of the dangers is that a crowded workspace can become an easy way to overturn a vial and release material outside of containment.

15.2.5 Sample Handling

Ensuring sample control is maintained may be more challenging when there are multiple resources required for sampling, shipment, receipt, and analysis. Radiological control personnel, accountability, and hazardous material transportation must all be given access to the sample for various purposes.

15.2.6 Practicality

For any method being setup in a radiological environment, consider the limitations before actually placing the method online. A method may be technically sound, yet need adjustments for real world implementation. Input from workers with field experience is highly recommended. Whenever possible, conduct a dry run and make adjustments accordingly before the method is implemented. A dry run is an analysis of analytical standards to assure that all radiological species are being detected.

15.2.7 Time

The factors listed above lead to an increase in sample processing time. Even without those factors to consider, a deliberate work pace is absolutely essential to safe handling of the radiological material and will add to processing time as well. The deliberate, slower pace is typically an improvement when considering quality. One exception is during performance of rapid time critical steps. Allowing for extra time seems contrary to ALARA (As Low As Reasonably Achievable) principles, which emphasize limiting radiological exposure. One should note however, from a safety perspective rushing is never advised. Instead, design and setup should be a deliberate, well thought out process that allows for safe, efficient movement.

15.2.8 Sample Receiving

Sometimes a laboratory receives a radioactive sample without warning. The laboratory should have policies in place to evaluate samples for this potential occurrence, such as having a Geiger counter in the sample receiving room, and other means of containment.

15.3 Quality Assurance Plan

Include quality concerns specific to radiological samples in the Quality Assurance Plan:

15.3.1 Outline the training/skill requirements of those working with specialized containment units or remote setups (glove boxes, manipulators, microscopes, e.g.).

15.3.2 Include or address all factors when performing Method Detection Limit (MDL), uncertainty, calibration, and other quality related calculations:

15.3.2.1 Performance testing is conducted in the actual environment where samples will be analyzed.

15.3.2.2 A separate calculation for radiological vs. non-radiological environments are developed OR both environments are considered in a single statistically valid calculation when reporting method uncertainty and MDLs.

15.3.2.3 Use of a cross section of trained personnel to obtain the values used in these calculations when possible.

15.3.2.4 Determination and continued evaluation of control limits either separately or using statistically valid data representing both sections of the laboratory.

15.3.2.5 Evaluating the differences in performing calibration/operation of portable equipment inside vs. outside the radiological environment. If a difference is noted, consider performing these actions inside the calibration/operation or otherwise accounting for any difference.

15.3.2.6 Analyze proficiency samples, round robins, or equivalent testing in both the radiological and non-radiological laboratories. This may be accomplished with dual samples, rotation between locations, or by other procedures outlined in Chapter 7 of this book.

15.3.2.7 It may not be practical to perform all testing inside the radiological environment because of waste or safety concerns. If not, the method by which radiological differences are evaluated must still be addressed in the quality assurance plan.

15.3.3 Pre-planning

Pre-planning is essential when handling radioactive samples. You should consider the following factors:

15.3.3.1 Design with efficiency of movement in mind. Think "practical" design as well as sound theory.

15.3.3.2 Obtain input from hygienists/laboratory analysts most familiar with radiological work.

15.3.3.3 Consider if some quality assurance samples can be dual purpose. For example, can a matrix spike also be used as a calibration verification? This may not be possible if quality is compromised, but should be considered as part of the development process.

15.3.3.4 Consider waste disposition as part of design, not as an afterthought. Again, do not assume that things will work as they do for non-radiological samples.

15.3.3.5 Plan a dry run in a radioactive environment/simulated radioactive environment.

15.3.3.6 Check with Federal, State, and Local laws to see if a special license is required to receive and handle radioactive materials for testing and what documentation you must have, along with regulations for radioactive sample disposal.

15.3.4 Chain of Custody

Consider intermediate handlers and how sample control will be maintained during the entire sampling/analysis process. Consider personnel that might be a part of radiological screening, nuclear material accountability, transportation, and those assisting with packaging/removing packaging/placement in containment.

15.3.5 Customer Relations

Be proactive in addressing potential customer concerns and relations.

15.3.5.1 There will be an increase in the turnaround time relating to sampling and analysis. Pre-planning can reduce these times, but the customer should still be aware of anticipated differences.

15.3.5.3 The cost will probably increase when analyzing radiological samples and may even be vastly different between radiological samples with varying isotopes/activity levels. This is because the sampling/analysis typically requires more man-hours, there are additional waste disposal/handling costs, and/or often there are modifications required so that "off the shelf" equipment can be fitted to a radiological environment.

15.3.5.4 The uncertainty, MDL, and method limitations may be vary greatly, not only between radiological and non-radiological samples, but also among different types/levels of radioactivity. Information specific to the sample should be made available to the customer

15.4 References

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Chapter 16
Quality Assurance Considerations for Nanoparticles

by Mark D. Hoover, PhD, CHP, CIH and Keith P. Rickabaugh

16.1 Introduction

If the reader has read and understood the other chapters in this manual, they should have the foundation to successfully anticipate, recognize, evaluate, control, and confirm the appropriate development, management, evaluation, and application-specific limitations of a laboratory quality assurance (QA) program in a broad spectrum of situations.

The emerging and increasing development and use of numerous types of engineered nanomaterials for a myriad applications such as electronic, pharmaceutical, automotive, and aerospace presents challenges to the effective creation, conduct, and evaluation of a QA program. Challenges in the pharmaceutical industry, for example, include the fact that many historical and established drug formulations were developed using particles in the micrometer and larger size. With the introduction of drug constituents in the nanometer-size range, QA programs are being asked to consider how those nano-formulated materials may need to be collected, handled, or analyzed differently, or may respond in different ways from their larger or bulk materials.

Certain unique properties of engineered nanomaterials such as high surface area, enhanced surface area-to-mass ratios, reactivity, similarity to biological structures, and an associated lack of defined measurement methods may require adjustment of the QA program. As pointed out in the chapter on nanotechnology in the recent 3rd edition of the AIHA text on *The Occupational Environment: Its Evaluation, Control, and Management*, the unique properties of engineered nanomaterials may make them relatively more toxic than materials of larger size.[1] Further complicating the challenges of material characterization, the physical, chemical, and biological properties of nanomaterials may not be predictable from the knowledge of their behavior as larger particles or as a bulk material. Understanding those properties in relation to safety, health, and environmental concerns, as well as issues for materials performance and effective QA, is a work in progress. The nanotechnology chapter of the AIHA® text points to valuable experience and guidance developed by authoritative organizations such as the National Institute for Occupational Safety and Health.[2]

The intent of this chapter of the AIHA Laboratory Quality Assurance Manual is to advise sampling and laboratory personnel of special considerations to apply QA concepts for nanomaterial-related measurements. Guidance provided in sections 16.2 through 16.9 in this chapter build on the topic-based guidance provided in Chapters 2 through 9 of this book. Nanotechnology encompasses broad areas of ongoing and evolving research, development, and applications. Changes are underway that must be continually assessed.

16.1.1 Terminology

While precise definitions of nanotechnology are still somewhat variable, most standard definitions recognize the nanotechnology involves the science and engineering of matter at the nanoscale where properties may change with size or new properties may emerge. As defined by the National Nanotechnology Initiative, the term nanotechnology refers to an emerging area of technology development involving the understanding and control of matter at the

nanoscale, at dimension between approximately 1 and 100 nanometers (nm), where unique phenomena enable novel applications.[3] Nanostructured materials, also called nanomaterials, have external or internal features that fall within the nanosize scale but may be larger than 100 nm as a whole.

The reader is advised to be aware of differences in how terminology related to nanotechnology is being developed and used in organizations such as the International Organization for Standardization, Technical Committee 229 (Nanotechnologies). For example, according to ISO/TS 27687:2008, a nano-object is defined as material with one, two, or three external dimensions in the size range from approximately 1–100 nm.[4] In recognition of the fact that the precise definition of particle diameter depends on particle shape as well as how the diameter is measured, subcategories of nano-objects are (1) nanoplate, a nano-object with one external dimension at the nanoscale; (2) nanofiber, a nano-object with two external dimensions at the nanoscale with a nanotube defined as a hollow nanofiber and a nanorod as a solid nanofiber; and (3) nanoparticle, a nano-object with all three external dimensions at the nanoscale. Thus, nano-objects are commonly incorporated in a larger matrix or substrate referred to as a nanomaterial, and nano-objects may be suspended in a gas (as a nanoaerosol), suspended in a liquid (as a colloid or nanohydrosol), or embedded in a matrix (as a nanocomposite). Access to ISO definitions and associated standards is available through the ISO Online Browsing Platform (https://www.iso.org/obp/ui/).

As noted in the nanotechnology chapter of the AIHA text, the term ultrafine particle has traditionally been used by the aerosol research and occupational and environmental health communities to describe airborne particles smaller than 100 nm in diameter. As shown in Table 16.1, the terms naturally occurring ultrafine particle, incidental ultrafine particle, and engineered nanoparticles (ENPs) are sometimes used to differentiate among particles that are naturally occurring from sources such as volcanic eruptions, particles that are incidentally created during processes such as welding, and nanoparticles that are "engineered" (e.g., Figure 16.1).

Table 16.1 — Nanoparticle Types by Their Mode of Production

Nanoparticle Type	*Examples*
Naturally occurring (ultrafine)	Volcanic ash, sea spray, forest fire combustion products
Incidental (Ultrafine)	Welding fumes, diesel exhaust, combustion products from propane vehicles and direct-gas heaters
Engineered (Manufactured)	Nanotubes, nanoscale titanium dioxide, quantum dots

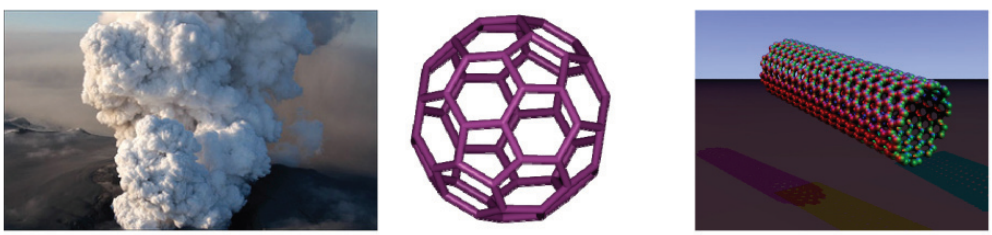

Figure 16.1 — Examples of (left) a dust plume including ultrafine particles from the Icelandic volcano, April 17, 2010, (Source: Wikimedia Commons); (center) a Buckminster Fullerene (Bucky Ball) composed of 60 carbon atoms (Source: Rice University); and (right) computer simulation of a carbon nanotube (Source: Wikimedia Commons)

16.1.2 A Perspective on Quality Assurance Needs and Gaps for Nanotechnology

As a starting point for delineating quality assurance considerations for the evolving field of nanotechnology, it is useful to examine the following list of eight needs, gaps, and opportunities recently developed from a critical review of nanoscale reference materials (RM) opportunities for environmental, health, and safety measurements.[5] The orienting considerations for RMs are reflective of the overarching challenges for quality assurance in general:

- Limited consensus on nanomaterials of concern: Various groups have prioritized nano-objects for development as "candidate RMs" but there is limited consensus;
- Lack of harmonized terminology: A lack of harmonized terminology hinders accurate description of many nano-object properties;
- Ill-defined properties of interest: Many properties identified for characterization are ill-defined or qualitative and hence are not traceable to fundamental units of measurement;
- Lack of standardized protocols: Standardized protocols are critically needed for characterization of nano-objects as delivered in relevant media and as administered to toxicological and other models;
- Inherent differences among characterization and measurement processes: Processes being used to characterize a nano-object must be understood because instruments may measure a given sample in a different way and artifacts or misinterpretation must be avoided;
- Need for harmonization of calibration and testing: Appropriate RMs should be used for both accurate instrument calibration and for more general testing purposes (e.g., protocol development and validation);
- Lack of clarity regarding the use of test materials: In situations where RMs are not available, there is a need to clarify the extent to which "representative test materials" that lack reference or certified values may be useful for hypothesis testing, toxicology testing, and inter-laboratory studies; and
- Need for interdisciplinary consensus: There is a need for consensus building within the nanotechnology and environmental, health and safety communities to prioritize RM needs and better define the required properties and (physical or chemical) forms of the candidate materials.

16.2 Personnel and Training

The design and conduct of personnel training for QA programs involving nanotechnology build on requirements for QA programs in general. The laboratory should conform to the personnel requirements as specified in Module 2A, Section 2A.5.2 (and all sub-sections) of the AIHA Laboratory Accreditation Policies, and to the requirements as detailed in the following sections. In all cases, training records for degreed laboratory staff should include a copy of the transcript or diploma from an accredited college/university, and a copy of all training courses pertinent to their job.[6]

The nanotechnology chapter of the AIHA text notes many sources for developing effective training programs for nanotechnology workers, including materials from the Occupational Safety and Health Administration (OSHA) and the National Institute for Occupational Safety and Health (NIOSH). Among those sources is the "Minimum Criteria" guidance of the National Institute of Environmental Health Sciences' Worker Education and Training Program (WETP), which has substantial results to corroborate its value. This guidance, which was updated in 2006, has provided the underlying principles for the creation, delivery and evaluation of training for over two million workers since the beginning of the program in 1987.The initial quality control for the program was developed through a participatory national technical workshop in 1990 and issued by the Program in 1991.[7] These original "Minimum Criteria" were updated in 1994 as the "Interpretive Guidance" to the "Minimum Criteria." The guidance has served as the quality control basis for the WETP training grants program to the present time. It was also adopted by OSHA as a non-mandatory appendix to the HAZWOPER standard.

The following Minimum Criteria recommendations should be applied to any training program created to deal with nanoparticles:

- Provide peer-to-peer training with hands-on activities whenever possible.
- Fill at least one-third of the training program hours with hands-on training.
- Avoid making computer-based training methods the sole form of training, although they can greatly augment the effectiveness and reduce the cost of training.
- Make sure proven adult-learning techniques are the core of all training.
- Precede all safety and health training with a needs analysis to ensure the appropriate knowledge, skills and attitudes are being transmitted.
- Follow all training with a proper evaluation to document that the knowledge, skills or attitudes were acceptably transmitted and that the trainee possesses the necessary abilities to perform the tasks.

16.3 Uncertainty, Error, and Sources of Error

Follow current AIHA Industrial Hygiene Laboratory Accreditation Program (IHLAP) policies and refer to chapter 3 of this book for uncertainty, error, and sources of error. Given that the availability of standardized quality control samples for nano-related analyses is currently limited, inter-laboratory round robin testing following the policies outlined in Chapter 7 are an acceptable alternative.

16.4 Traceability

There are currently a limited number of certified reference standards for measuring nanoparticles. A list of nanoscaled reference materials can be found at http://www.nano-refmat.bam.de/en/ on the website of the German Federal Institute for Materials Research and Testing (BAM). The National Institute of Standards and Technology (NIST) has certified standards for soot, three sizes of gold nanospheres, carbon nanotubes and will characterize a sample of single-wall carbon nanotubes (SWCNTs) and provide a certificate for that sample.[8] A limited number of other nanoparticles may be available in the future, and information about their accessibility may be obtained at the nist.gov website.

16.5 Sampling and Analytical Procedures

As recommended in the nanotechnology chapter of the AIHA textbook, the selection of sampling and analytical procedures can benefit from a graded approach.[9] As illustrated in Figure 16.2, a desirable graded approach begins with prioritization of sampling needs based on process knowledge, and involves initial screening and detection of potential nanoparticle emissions, followed by comprehensive characterization and assessment of nanomaterials in locations of concern, and leading ultimately to the selection of sampling methods that are feasible and economical for routine monitoring and control.

NIOSH has developed, demonstrated, and is continually evolving a nanoparticle emission assessment technique (NEAT) which involves a practical combination of process knowledge, particle counting, and microscopy to conduct a semi-quantitative initial assessment of "suspected" emission sources compared to background particle concentrations, which can serve as a guide to a more detailed investigation using less portable, more expensive particle analyzers and techniques.[10–13] A combination of direct reading and time integrated sampling is best employed to more fully understand emissions and exposures within the workplace. This may range from basic industrial hygiene surveys, where portable instruments and personal sampling pumps and collection media are utilized,[14,15] to more detailed and complex investigations where further instrumentation and/or sampling equipment may be used in combination.[16–19] From a direct reading instrument perspective, a multimetric approach is highly recommended.[15,17] For specifically monitoring worker exposures, personal breathing zone samples are employed with subsequent analyses for specific markers of exposure and confirmatory microscopy.[14,18,20,21]

Level 0	Level 1	Level 2	Level 3
Prioritization of Sampling Needs	**Initial Screening and Detection**	**Comprehensive Characterization and Assessment**	**Routine Monitoring and Control**
•Process knowledge - Work flows - Anticipated or recognized hazards and potentials for exposure	•Process knowledge •Gross mass or activity counting •Optical particle counting •Condensation particle counting •Microscopy	•Composition - Elemental and chemical •Particle size - Physical, aerodynamic, thermodynamic, electrical mobility •Concentration - Peak, mean, variability •Biophysical factors - Shape, surface area, solubility •Other factors relevant to the assessment	•A necessary and sufficient subset of Level 1 and Level 2 methods for the material and situation of interest

Figure 16.2 — Illustration of a Graded Approach to Nanoparticle Sampling for Exposure Assessment and Control (Source: Adapted from Kulinowski and Lippy 2011[1] and Hoover 2011[9]).

NIOSH has also developed recommended exposure limits for carbon nanotubes[14] and for nano-titanium dioxide.[22] Analysis is performed using methods from the NIOSH Manual of Analytical Methods for the analyte in question, such as Carbon, elemental (NIOSH 5040)[24] for carbon nanoparticles, or Titanium (NIOSH 7300)[25] for titanium oxide nanoparticles, using modified sample digestion procedures.

The following ASTM methods, at the time of this book's publication, are available, with more methods in committee which will be published in the future:

- E2524 Standard Test Method for Analysis of Hemolytic Properties of Nanoparticles;
- E2834 Standard Guide for Measurement of Particle Size Distribution of Nanomaterial in Suspension by Nanoparticle Tracking Analysis (NTA);
- E2859 Standard Guide for Size Measurement of Nanoparticles using Atomic Force Microscopy; and
- E2864 Standard Test Method for Measurement of Airborne Metal and Metal Oxide Nanoparticle Surface Area Concentration in Inhalation Exposure Chambers using Krypton Gas Adsorption.[18]

The following ISO methods, at the time of this book's publication, are available, with more methods in committee which will be published in the future:

- ISO/TR 12802:2010 Nanotechnologies – Modeltaxonomic framework for use in developing vocabularies – Core concepts
- ISO/TS 17200:2013 Nanotechnology – Nanoparticles in powder form – Characteristics and measurements
- ISO/TS 11931:2012 Nanotechnologies – Nanoscale calcium carbonate in powder form – Characteristics and measurement
- ISO/TS 11937:2012 Nanotechnologies – Nanoscale titanium dioxide in powder form – Characteristics and measurement
- ISO/TS 16195:2013 Nanotechnologies – Guidance for developing representative test materials consisting of nano-objects in dry powder form
- ISO/TS 80004-7:2011 Nanotechnologies – Vocabulary – Part 7: Diagnostics and therapeutics for healthcare

- IEC/TS 62622 Ed. 1.0 en:2012 Nanotechnologies – Description, measurement and dimensional quality parameters of artificial gratings

There are Austrian, British, German, ISO, Swedish and ASTM standard test methods available for many different nanoparticle analytes at the American National Standards Institute (ANSI) webstore.[26] The National Cancer Institute's caNanoLab is a data sharing portal that provides nanotechnology characterization protocols and characterizations.[27]

The sampling and laboratory methods employed are typically dependent on one another as the sampling media, containers, and sample parameters must be compatible with the sample preparation and analysis methods used. The strategies used for both sampling and analysis should be clearly communicated to field and laboratory personnel. When creating a strategy to assess ENPs, some of the questions to be addressed are likely to include:

- Are there unique characteristics of the ENPs that can be used to differentiate the materials from incidental nanoparticles?
- Are there any nearby activities or processes that may release other ENPs or incidental nanoparticles?
- How should bulk samples of ENPs of concern be collected, handled, and applied for QA and ENP identification purposes?
- Can the levels of ENPs be quantified?
- Has the selected method been validated for the ENPs of interest?
- How will the state of validation influence the interpretation of results?
- How should the method sensitivity, specificity, and limits of detection and quantification be reported by the laboratory and handled by IH practitioners?
- How will total measurement uncertainly be assessed?
- Is there a suitable reference material available to challenge the laboratory methods being considered?
- What sampling parameters and lab procedure modifications are available and appropriate to achieve low enough reporting limits?
- Are the particles dispersed, agglomerated/aggregated, or embedded in matrix material?
- Is it acceptable to use indirect preparation methods for transmission electron microscopy analysis if samples are overloaded?
- How many laboratory and field blanks should be submitted and how should the results be applied to the air sampling data?
- Are special analytical reporting rules needed for endpoints such as particle counting or reporting of the state of particle aggregation or agglomeration needed for the material of interest?

16.5.1 Multiple Analytical Methods will Likely be Needed

In most cases, multiple sampling and analysis methods may be needed to evaluate the potential release of ENPs into the workplace and/or the environment. The selection of analytical methods to evaluate ENPs is not a trivial exercise. Figure 16.2 illustrated some of the characterization methods and characterization endpoints such as composition, particle size, surface area, etc., that may be relevant to meeting different sampling and analytical objectives. Determining whether a given ENP or interest is present at a very low concentration in the presence of other potentially interfering materials can be particularly challenging. Using multiple analytical methods such as particle counting along with electron microscopy can improve the likelihood of detecting and characterizing an ENP of interest.

Within a given analytical method there will likely be the need to tailor the details of the sample collection and analysis based on concentration differences in the presence or absence of interferences. Simultaneous samplings at multiple locations such as near the source of work activity, the worker breathing zone, "background," and various areas of interest may be prudent. Comparing results from multiple locations to each other and to data generated from direct-reading instruments can be useful to understand and index laboratory reported con-

centrations either qualitatively or semi-quantitatively. This may be especially important if the objective of a work place is to employ the As Low As Reasonably Achievable (ALARA) or control banding principles.

16.5.2 Preliminary Analytical Studies can be of Value

If possible, preliminary studies be performed on reference samples of ENPs to challenge existing methods for mass recoveries, interferences, and reproducibility prior to making recommendations for work place sampling. Depending on the results from a preliminary study, sample preparation and analysis methods can then be modified and tested to evaluate the appropriateness of using instrumental analysis methods.

Some general concerns when performing mass based laboratory analysis for bulk samples of ENPs include poor sample recovery issues when performing liquid filtrations (ENPs will likely pass through filters) and material loss during handling (e.g., dry materials in a ventilated hood). Furthermore, any material available for testing may be limited and the actual solubility of the ENPs may not be well documented.

Identification and differentiation of ENPs using electron microscopy (EM) techniques is often desired to specifically verify the presence of ENPs on collected samples. This is another area where it is important for field personnel to communicate with the laboratory. In order to perform EM analysis on a directly prepared filter (recommended to preserve "as sampled" particle characteristics), the particulates on a filter must be dispersed and have an appropriate particle loading. The particle loading on the filter should be heavy enough to readily detect particles of interest, but not so heavy that they overlap or touch each other so as to interfere with the analyses (1% to 5% by area on a filter is typically a "good" loading). For air samples, filter loadings will be directly dependent on sample volume, duration, workplace activities and conditions. Once an appropriately loaded sample is obtained, information regarding any ENPs such as the specific presence, abundance, size, shape, agglomeration state and particle associations can be evaluated. Field personnel should consider collecting samples of different volumes to increase the likelihood that at least some are not overloaded.

Prior to employing EM techniques, one of the first considerations should be to obtain representative specimens of ENPs. Examples of representative specimens can include project-specific bulk materials or air samples obtained immediately at the source of ENP generation or work activities. These representative specimens can serve as a reference sample for the evaluation of unique particle characteristics (e.g., appearance and chemistry) that can be used to categorize ENPs during the EM analyses of field survey samples. In the event that elemental analysis using EM methods is needed, transmission electron microscopy may be utilized on very thin and fragile support films to optimize x-ray count rates and minimize interferences. Evaluation of reference specimens can also be used to optimize EM analysis procedures such as selecting the correct magnification, imaging mode and defining signature characteristics of the ENPs. High resolution elemental mapping using scanning transmission microscopy (STEM) methods can also be useful in characterizing ENPs.

16.6 Sample Receipt and Handling

In addition to standard laboratory receipt and handling procedures, additional precautions may be prudent when working with ENPs. Prior to receipt, it would be favorable to review relevant documentation regarding anticipated hazards of the materials both from an aspect of protecting the worker and minimizing the likelihood of sample loss or degradation. Laboratory personnel should investigate Safety Data Sheets (SDS) rather than relying solely on them as it is probable that the information in this type of document may be insufficient to fully address the hazards related to the nanoscale properties of materials. For example, in 2011 NIOSH researchers reviewed forty-four MSDSs and reported that "the majority (67%) still provide insufficient data for communicating the potential hazards of engineered nanomaterials.[28]

16.6.1 Additional Sample Handling Considerations for ENPs

Below is a partial list of additional considerations when handling samples that contain ENPs. Many of them are health and safety considerations, such as contamination control, that contribute to effective quality assurance:

16.6.1.1 Open packages in an appropriate ventilated laboratory hood to protect workers from potential exposure from compromised containers or exterior deposits on sample containers/media.

16.6.1.2 Wet wipe the exterior of containers and place the containers/media received in secondary exterior containers (e.g., plastic bags) for transport to other areas.

16.6.1.3 Institute a spill clean-up program and have HEPA filtered vacuums available in sample preparation areas for immediate access and respond if a spill were to occur. Work with limited quantities of material at one time (only as much as necessary) to minimize potential hazard.

16.6.1.4 Keep work areas extremely clean and use good housekeeping procedures. If possible, have dedicated facilities for handling ENPs.

16.6.1.5 Clearly label each sample with verbiage or use another system denoting that samples are subject to any special handling procedures following any nanoparticle handling guidelines that are established.

16.6.1.6 When possible, work with liquid suspensions instead of dry powders and use disposable labware.

16.6.1.7 Limit access to ENP materials to only trained personnel. Store ENP materials in lockable, appropriately vented/filtered, flame resistant cabinets.

16.6.1.8 When handling and weighing dry powders containing ENPs, special low velocity filtered hoods designed specifically for ENPs should be considered in order to minimize release of nanoparticles (and sample loss) from air turbulence while still providing adequate worker protection. Use of a glove box or draft shields within a hood can also be considered for this purpose.

16.6.1.9 If sonication techniques are used to disperse ENPs in a suspension, cover and enclose containers to minimize aerosolization and potential loss of material.

16.6.1.10 Dispose of unneeded ENPs in an appropriate manner or return to the supplier following chain-of-custody guidelines.

16.7 Intra-laboratory and Inter-laboratory Testing

Follow current AIHA® IHLAP policies and refer to Chapter 7 of this book for policies and practice on inter-laboratory round robin testing. Communicate effectively to develop and apply agreed upon protocols. Consider video documentation of handling procedures.

16.8 Data Validation and Interpretation

In order to perform adequate data review and interpretation, it is first important to clearly establish and communicate the goals and purpose of any sampling and analytical study. Some examples of potential goals include:

- Evaluation of containments or engineering controls
- Comparing work practices or work activities
- Qualitative assessments for potential airborne release
- Risk modeling
- Verification of direct read measurements
- Comparison of one material to another

It is important to keep the project objectives in mind when evaluating data as the information gained from analyzing samples may not ideally meet the most rigorous QA requirements. However, the information gained from evaluation of differences in results from comparison samples may satisfy the needs of a particular project study. In many cases, the data should be qualified and results verified using other techniques. For instance, EM can be used to confirm presence of ENPs when evaluating results from mass based sampling and analysis methods.

It is likely, that analysis of samples involving ENPs may only be done on a limited basis by some laboratories and, as a result, the limited data available may not be amenable to statistical analysis. This can be further complicated by the fact that matrix spike and true duplicate analyses may not able to be performed on project specific ENPs due to limited amounts of material being available or that only "unique" samples were collected.

None-the-less, building upon well understood and established guidelines for evaluation of other substances such as asbestos or welding fumes should provide a robust framework from which QA/QC concepts can be applied. Any relevant anticipated or realized limitations regarding the quality of the results and the relevance to the method or laboratory accreditation should be communicated to end users and included as part of any written reports.

16.9 Reporting and Record keeping

Reporting and record keeping of results may not be as simple as giving the customers their results and retaining the analytical record. Because nanotechnology and the understanding of both material properties and performance and health and safety issues are evolving, more than traditional details of information may be needed to support both current and later reinterpretation, as well as to enable considerations of what is not known.

The ASTM International specification entitled *ISA-TAB-Nano* is an example of a nano-specific, spreadsheet-based format developed to facilitate the import/export of data on nanomaterials and their characterizations to and from nanotechnology resources.[29]

16.10 A "Nanoinformatics" Perspective for Building and Sustaining Improved QA

The following working definition is adapted from the Nanoinformatics 2020 Roadmap[30]:

"Nanoinformatics is the science and practice of determining which information is relevant to meeting objectives of the nanoscale science and engineering community, and then developing and implementing effective mechanisms to collect, validate, store, share, analyze, model, and apply the information, and then to confirm achievement of the intended outcome from use of that information."

16.10.1 Nanoinformatics Roles and Responsibilities

The determination of what information is relevant, the extent to which it must be reliable, and the confirmation thereof, is the essence of effective QA for nanotechnology. The decision-making, communication, and implementation roles and responsibilities for effective nanoinformatics are shared among information customers, information creators (especially those

who collect and analyze the sample), information curators (including the data-base designers and the record keepers), and the information analysts. All must understand, communicate, and fulfill their mutual needs.[31]

The promise of a nanoinformatics-informed approach is that, regardless of the primary purpose of the sample, the resulting information, including on how to conduct effective QA, can be understood and applied to advance both sustainable nanomaterial performance and nanotechnology health and safety.

16.10.2 Opportunities for Information Sharing to Build and Sustain Improved QA

To build and sustain improved QA, it is essential that personnel collaborate and share their information through entities such as the GoodNanoGuide[32], the National Nanotechnology Initiative signature initiatives (including the signature initiative on Nanotechnology Infrastructure (NKI)—Enabling National Leadership in Sustainable Design, and the signature initiative on Nanotechnology for Sensors and Sensors for Nanotechnology: Improving and Protecting Health, Safety, and the Environment)[33], the Nanomaterial Registry[34], the AIHA Nanotechnology Working Group[35], or NIOSH to name a few.

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Chapter 17
Author Index

Chapter 1 Andrew Teague, CIH

Mr. Teague is the Laboratory Director for Analytics Corporation, an industrial hygiene (AIHA-accredited) and environmental testing laboratory. He has over 32 years' experience in industrial hygiene monitoring and analytical chemistry, including development and evaluation of new IH sampling and analysis methods. He is a Past Chair of the AIHA® Sampling and Lab Analysis Committee. Mr. Teague has a Bachelor of Science Degree in Chemistry and Biology from Berry College. He is a Certified Industrial Hygienist in Chemical Aspects.

Chapter 2 Pam Kostle, CIH

Ms. Kostle received her M.S. degree in Occupational and Environmental Health (Industrial Hygiene) from the University of Iowa and a B.S. degree in medical technology, from Mount Mercy College, Cedar Rapids, Iowa. She retired from being Assistant Director of Environmental Health Division at the State Hygienic Laboratory (SHL) which provides comprehensive laboratory services for public health, environmental and terrorism response. Ms. Kostle is currently an Industrial Hygienist Specialist at the University Health Services/Occupational Health at University of Wisconsin-Madison. Ms. Kostle has served on the AIHA® LQAP Analytical Accreditation Board (AAB) from 2005-2010, including positions of Vice Chair, Chair and Past Chair. She continues to serve on the AIHA® LQAP AAB Policy Task Force. She has been a member of the AIHA® Sampling and Laboratory Analysis Committee since 2000, having served as Chair in 2007. She is a Certified Industrial Hygienist in the chemical aspects of industrial hygiene. Ms. Kostle has been recognized for her service and accomplishments including the AIHA® LQAP Past Chair Award, Mt. Mercy University Alumni Professional Achievement Award, and University of Iowa Outstanding Staff Award.

Chapter 3 Dr. Evan Floyd, PhD

Dr. Evan Floyd PhD, is an Assistant Professor in the Department of Occupational and Environmental Health at the University of Oklahoma. He has experience as an analytical chemist for Battelle Memorial Institute monitoring for nerve and blister agents in support of the Army's mission to destroy their aging chemical weapons stockpiles. He also practiced broad scope Industrial Hygiene with a consulting firm/ test lab. He completed his doctorate in Environmental Health Sciences, Industrial Hygiene from UAB in 2013 and focused on improving VOC exposure assessment by developing a new technique for desorbing sorbent samples. Dr. Floyd is continuing to develop this technique at OU and plans to adapt it for in-field prescreening of integrated VOC exposures.

Chapter 4 Periyasamy Subramanain, PhD, CIH

Dr. Periyasamy Subramanian received his PhD in Inorganic chemistry from the Indian Institute of Technology, Kanpur, India under the guidance of Dr. S. Sarkar. Dr. Subramanian currently serves as Senior Chemical Safety Specialist/Industrial Hygiene at the University of Iowa. As a Certified Industrial Hygienist in Chemical Aspects Dr. Subramanian has been a leader in collaborating with researchers and has contributed to more than forty peer reviewed publications in analytical chemistry and environmental and occupational health journals. His contributions include expertise in analytical techniques and on-line safety training courses for the university researchers to use via ICON for a wide range of topics from controlled substances to nanomaterials. During his career spanning over 20 years, he has had substantial impact on the development of technical information for sampling and analysis affecting the workplace

and leadership contributions to AIHA®, including past Chair of AIHA® Sampling and Laboratory Analysis Committee. Dr. Subramanian's has provided significant contribution to AIHA through key leadership roles within the AIHA® committees and local section. In addition to committee membership, leadership roles on committees, he also served as the contact for the AIHA Sampling and Laboratory Analysis Committee Industrial Hygiene Methods Exchange Network (IHMEN).

Chapter 5 Dr. Charles (Gus) Manning, PhD, CIH

Dr. Charles R. (Gus) Manning received his PhD in Analytical Chemistry from the University of Kansas. Since founding Assay Technology in 1981, his work has focused on the development of air sampling and analytical lab test technologies. He is a CIH and a Past Chair of the AIHA Committee on Sampling and Lab Analysis. He is currently the President and Technical Director of Assay Technology, a diffusive sampler manufacturer which operates an AIHA-accredited laboratory.

Chapter 6 Dave Sandusky, CIH

Mr. Sandusky is the Corporate Laboratory Director and also oversees the Quality Assurance Program at Forensic Analytical. He has over 26 years' experience in industrial hygiene and environmental laboratory operations. Mr. Sandusky has a Bachelor of Science Degree in Chemistry from the University of California at Berkeley, and a Master of Science degree in Chemistry from California State University at Hayward. He is a Certified Industrial Hygienist in Chemical Aspects.

Chapter 7 Keith R. Nicholson, MPH, CIH

Keith R. Nicholson, MPH, CIH, is Assistant Director of the Industrial Hygiene Chemistry Division and quality manager at the OSHA Salt Lake Technical Center. He is been an AIHA-LAP laboratory accreditation site assessor since 1994, and has served on and been chair of the AIHA Asbestos Analysis Committee (1990–1997). He was previously employed by DataChem Laboratories in Salt Lake City, Utah where he worked in various areas including laboratory operations, project management, safety, and quality management. He obtained his MPH with an industrial hygiene emphasis from the University of Utah, and is ABIH-certified in chemical aspects.

Chapter 8 Daniel J. Pastuf

Daniel J. Pastuf has a BS in Chemistry from Syracuse University, 1976. Mr. Pastuf has worked for 30 years in Environmental and Industrial Hygiene Laboratories primarily an Organic Analytical Chemist. He has done extensive Method Development in both GC and GC/MS. Mr. Pastuf is currently the Organics Manager at Galson Laboratories as well as the manager of the Equipment/Media/Pumps group. He is a past Chair of the Sampling and Laboratory Analysis Committee. His interests include Syracuse University sports, the New York Yankees and Giants, golf, and is a youth, High school, and Jr. college football official.

Chapter 9 Mary E. Eide

Mary Eide is retired from OSHA Salt Lake Technical Center where she worked for 33 years as a chemist developing 9 validated sampling and analytical methods, 101 partially validated methods and many studies. Ms. Eide was on the OSHA Forensics Team for 20 years assisting in investigations of major industrial accidents and fatalities. Ms. Eide is a Past Chair of the AIHA Sampling and Laboratory Analysis Committee. Ms. Eide has taught a PDC on air sampling strategies. Ms. Eide received the U.S. Secretary of Labor Exceptional Achievement Award both for her work on the Hexavalent Chromium Standard, and on Diacetyl and Acetoin.

Chapter 10 Jeffrey Cooper

Mr. Cooper directs Bureau Veritas' industrial hygiene laboratory. He has spent the last 14 years in a technical management role, overseeing all aspects of laboratory operations. These duties include technical and quality direction of laboratory analyses, evaluation of newly proposed methods, laboratory staffing, laboratory financials, laboratory timelines, professional development of personnel, and technological advancement of the laboratory. He has developed a wide range of analytical capabilities, due to his involvement in the industrial hygiene industry for more than 15 years. During this time, he has gained expertise in many techniques including gas chromatography (flame ionization, nitrogen-phosphorus, flame photometric, electron capture, thermal conductivity, and mass spectrometry detectors), high pressure liquid chromatography (HPLC), inductively coupled plasma atomic emission and mass spectrometry (ICP-AES and ICP-MS), polarized light microscopy (PLM), and transmission electron microscopy (TEM). He is well versed with many of the NIOSH, OSHA and EPA analytical methodologies. Mr. Cooper has also performed method validations that include creating atmospheres of contaminates to be collected. Some of the methods he has been involved with include a method for functional siloxanes using GC headspace analysis and extensive development work on a method for the analysis of butyltins by GC/FPD. Mr. Cooper has also evaluated many of the methods that are in use in the GC, HPLC, and metals departments. He belongs to the following professional affiliations: Member of the AIHA Technical Advisory Panel (TAP) in 2008 – 2010, Co-chair of the AIHA Pharmaceutical Round Robin Committee in 2009 – Present, and Member of the AIHA Sampling Analysis Laboratory Committee in 2010 – Present (Chair 2013-2014).

Chapter 11 Samuel D. Allen Iske, Jr., PhD, CIH, CSP

Dr. S. D. Allen Iske received his Bachelors of Science in Chemistry and Mathematics from Missouri Western State College and then completed his Doctorate in Chemistry from the University of Nebraska – Lincoln in Lincoln, Nebraska. He completed a Post-Doctoral appointment as a Visiting Research Associate at Ohio State University in Columbus, Ohio. He is a Certified Industrial Hygienist (CIH) and a Certified Safety Professional (CSP). Dr. Iske has had a working career for more than thirty years including work in both chemical and pharmaceutical private sector industry, pharmaceutical research in pulmonary drug delivery, OSHA State Consultant in Industrial Hygiene, and approximately nine years of teaching at the University of Central Missouri in the School of Environmental, Physical, and Applied Sciences, Safety Sciences area for both Industrial Hygiene and Safety courses. Dr. Iske is an active member in several professional organizations on the national and local levels. He has been a member of the National American Industrial Hygiene Association, American Society of Safety Engineers, ASTM International, Inc., and in the past American Chemical Society as well as the local chapters of Mid-America Section of AIHA, Heart of America Section of ASSE, and Kansas City Section of the ACS. He has served in leadership positions on both national and local committee. He serves as the Official Representative of the National AIHA to ASTM D-22 Technical Committee on Air Quality and to ISO (International Standards Organization) TC 146 Technical Committee on Air Quality. He also serves as a member of the US Technical Advisory Board and Delegate to ISO TC 146 on Subcommittees SC 2 Workplace and SC 4 General Aspects (Head Delegate). Dr. Iske is a Fellow of both the American Industrial Hygiene Association and ASTM International, Inc. and a member of Rho Sigma Kappa, UCM Chapter. Fellow Honor is the highest society honor bestowed on a member for leadership, professional service, technical contributions, and commitment.

Chapter 12 Martin Harper

Martin Harper is Chief of the Exposure Assessment Branch in the Health Effects Laboratory Division of NIOSH. His first degree is in Geology and he has a post-graduate qualification in analytical chemistry, a M.S. in earth sciences and a PhD in occupational health from the London School of Hygiene and Tropical Medicine. He is certified in the chemical practice of Industrial Hygiene and he is a Chartered Chemist, Fellow of the Royal Society of Chemistry. He has published several technical papers on asbestos sampling and analysis. He was a volunteer member of the AIHA Laboratory Quality Programs Committees between 1995 and 2001 and has since served on the Scientific Board of AIHA PAT, LLC.

Chapter 12 Donald Halterman

Donald Halterman holds an M.S. in Geology, concentrating on mineralogy and soil science. He has presented numerous talks on mineralogy, public health and safety, and forensics. He is currently an analytical chemist at the OSHA Salt Lake Technical Center analyzing asbestos, soil, and silica.

Chapter 13 Richard Mark O'Mara, CIH

Richard Mark O'Mara received his BS in Chemistry from Kansas Wesleyan in 1980 and his MS in Medicinal Chemistry from Purdue in 1990. He obtained his CIH in 1996 and was confirmed as an AIHA Fellow in 2009. He is a past Chair of the AIHA® Sampling and Laboratory Analysis Committee. He currently supervises Eli Lilly and Company's Corporate Industrial Hygiene Laboratory in Indianapolis, IN.

Chapter 15 Linda Youmans

Linda is a chemist and the Industrial Hygiene Laboratory Technical Director at Savannah River National Laboratory in Aiken, SC. She holds a Master of Science degree in Environmental Health from East Carolina University. Linda has 22 years of laboratory experience in the nuclear industry. She has held positions in water treatment, waste treatment, industrial hygiene, and other areas of process support. Linda is an active member of American Chemical Society (ACS) and the American Industrial Hygiene Association (AIHA). She is a board member of her local ACS section (Savannah River) and has held the role of National Chemistry Week Coordinator for the past six years. She is a member of AIHA's Proficiency Analytical Testing Board and Sampling and Laboratory Analysis Committee. She serves as vice chair for the Beryllium Health and Safety Committee, which promotes efforts to understand and prevent beryllium-induced conditions and illnesses and is primarily educational in mission. She is involved in many local educational outreach programs in her community.

Chapter 16 Keith Rickabaugh

Keith Rickabaugh is the Technical Director of materials and analytical services at RJ Lee Group. He has been working in the industrial hygiene field for over twenty years and has performed exposure measurements, analysis, and field surveys to evaluate the potential release of engineered nanoparticles in the work place for commercial, industrial and governmental organizations. He is an expert in the utilization of high resolution microscopy techniques to characterize particulates and materials and has a number of publications related to the environmental health and safety and sampling considerations when working with nano-enabled materials.

Chapter 16 Dr. Mark Hoover, PhD, CHP, CIH

Mark Hoover is a senior research scientist in the Division of Respiratory Disease Studies at the National Institute for Occupational Safety and Health in Morgantown, West Virginia. He coordinates the NIOSH Exposure Assessment Cross-sector Research Program, co-directs the NIOSH Center on Direct Reading and Sensor Technologies, and is a critical area leader in the NIOSH Nanotechnology Research Center. Mark is a past chair of the AIHA Nanotechnology Working Group and the AIHA Control Banding Working Group. He has a BS in Mathematics and English from Carnegie-Mellon University, MS and PhD degrees in Nuclear Engineering from the University of New Mexico, and is a certified health physicist and a certified industrial hygienist. His career has focused on establishing a technical basis for anticipating, recognizing, evaluating, controlling, and confirming appropriate management of hazards in the workplace and environment. He has chaired, co-chaired, or contributed to the development of many national and international standards and is author or co-author of more than 200 open literature publications.

AIHA® Laboratory Quality Assurance Manual, 5th edition

Edited by Andrew L. Teague, CIH and Mary E. Eide

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