



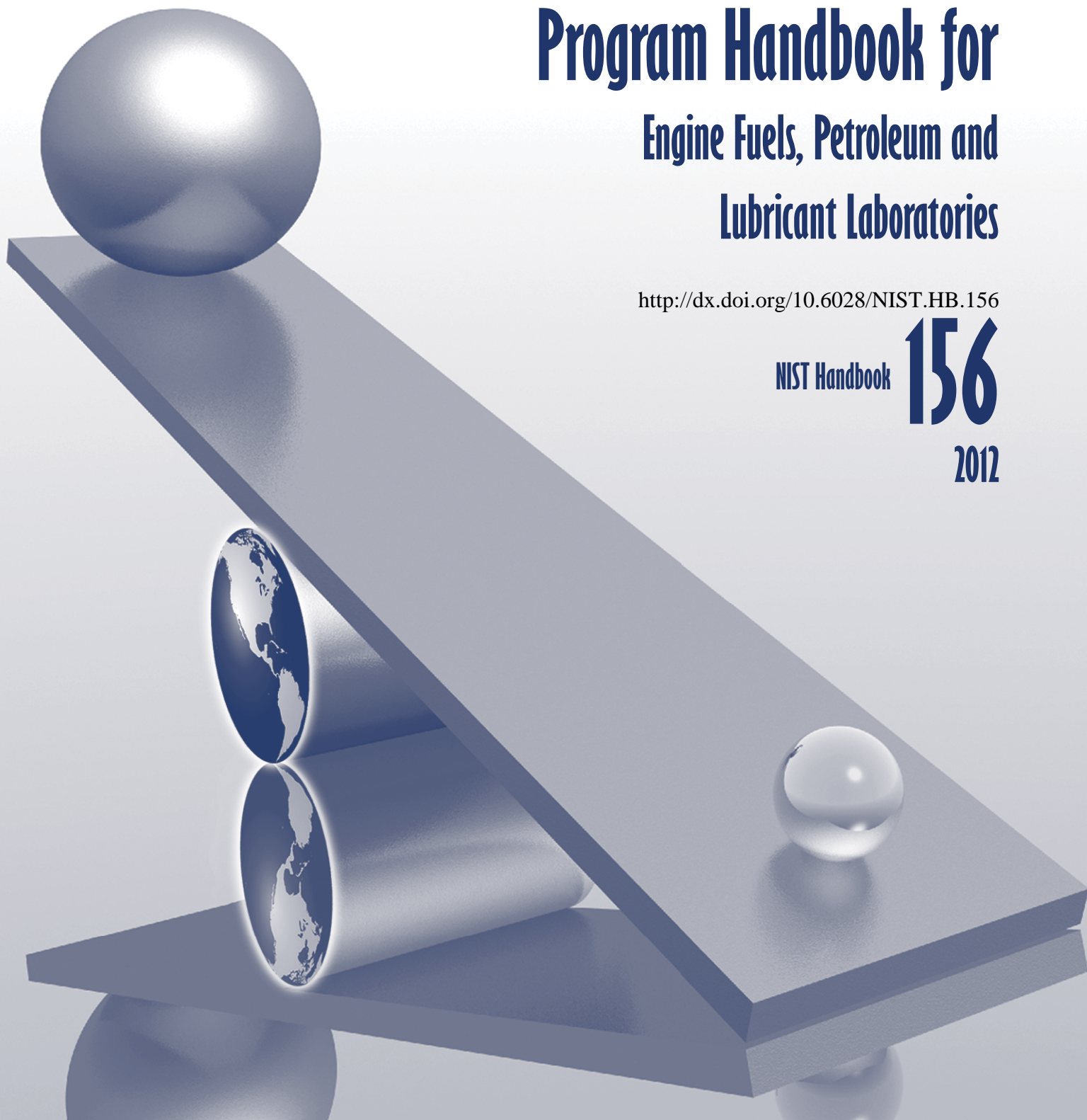
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**National Institute of
Standards and Technology**
U.S. Department of Commerce

Program Handbook for Engine Fuels, Petroleum and Lubricant Laboratories

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NIST
**National Institute of
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PROGRAM HANDBOOK

for Engine Fuel, Petroleum and
Lubricant Laboratories

NIST Handbook - 156

2012 Edition

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Program Handbook for Engine Fuel, Petroleum Products and Lubricants Laboratories

Introduction

This handbook provides guidance on how to establish a management system for Fuel Quality Laboratories (FQL) using the International Organization for Standardization (ISO), International Electrotechnical Commission (IEC) ISO/IEC 17025:2005, “General Requirements for the Competence of Testing and Calibration Laboratories (17025).” This handbook includes standards similar to those in 17025 and NIST Handbook 143 “State Weights and Measures Metrology Laboratories – Program Handbook” except that 17025 and Handbook 143 impose mandatory requirements. This handbook is intended as guidance, so the “shalls” in the handbooks on which it is based have been replaced with “shoulds” to make it clear that the text is only recommended. Implementation Notes are shown in *italics* to provide examples of a way that users may implement the recommendations.

If a laboratory conforms to Section 3.0 “Management Guidelines” and Section 4.0 “Technical Guidelines” of this handbook, it may be eligible for accreditation. If accreditation by NIST’s National Voluntary Laboratory Accreditation Program (NVLAP) is sought, a request must be made to NVLAP to establish a testing laboratory accreditation program.¹ Section 3.0 “Management Guidelines” and Section 4.0 “Technical Guidelines” in this handbook follow the organization and numbering system of 17025, Handbook 143, and NIST Handbook 150 “National Voluntary Laboratory Accreditation Program (NVLAP) - Procedures and General Requirements” (2006) so that a laboratory (and assessors) can cross reference those standards. Also, some notes on laboratory safety are provided to provide a foundation for ensuring a safe working environment in an FQL. This basic information should be viewed only as an introduction to the subject and not as an exhaustive treatment of a model safety program. In addition to seeking the latest general guidance from state and federal safety officials, experts in laboratory safety and safe fuel handling procedures should be consulted.

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¹ See Section 2 “LAP Establishment, Development and Implementation” in NIST Handbook 150 “National Voluntary Laboratory Accreditation Program (NVLAP) – Procedures and General Requirements” (2006)¹ for information on establishing NVLAP accreditation.

Preface

This handbook is for use by fuel quality laboratories (FQL) in developing and improving the management and technical systems that govern their operations. It is also for use by government managers who are considering the establishment of an FQL² and must decide whether or not to contract for laboratory testing services, and if contracting, how to manage these external laboratories. Laboratory customers, regulatory authorities, and accreditation bodies may also use it as a basis upon which to judge the competence of laboratories.

The function of most FQLs may include the testing of fuels (of all types including biofuels and petroleum based), and other petroleum products including brake fluids and lubricants. Regardless of whether or not an FQL is operated by state government personnel, or contracted out to a private laboratory, it is an integral element of an engine fuel inspection program. Its main purpose is to satisfy the testing requirements as described in the laws and rules of the regulating agency. Over the years, the National Conference on Weights and Measures (NCWM) has provided guidance³ on establishing or upgrading an FQL using ASTM International (ASTM) standards and test methods.

ASTM was formerly known as the American Society for Testing and Materials. ASTM Subcommittee D02 on Petroleum Products and Lubricants is responsible for developing engine fuel specifications and is comprised of representatives from a broad group of stakeholders that are classified as producers, users, general interest and consumers. The stakeholders are the petroleum industry, biofuel and other alternative fuel industries, automotive manufacturers, and regulating agencies, testing laboratories, and other interested stakeholders. This representation ensures that test procedures have been reviewed by each segment of the testing community and laboratory results obtained utilizing these procedures will be widely accepted. These guidelines have been incorporated into this handbook with some modifications for clarity or to update the terminology.

This handbook is intended for State Directors:

² See the following references for planning a new laboratory: John E. Nunemaker, "Planning Laboratories: A Step by Step Process" *American Laboratory* March 1987, 19 (4), 104-112 and Jerry Koenigsberg, "Building a Safe Laboratory Environment" *American Laboratory* June 1987, 19 (9), 96-106. There is no better way to understand the complexities of testing than to visit a state with an active program. Several States, such as Arkansas, California, Florida, Georgia, Maryland, North Carolina, Missouri, Michigan, Washington and Tennessee (a contractual laboratory) have active programs and may be willing to host tours of their facilities. Interested parties are encouraged to make such a visit. A list of state weights and measures directors is available at www.nist.gov/owm on the Internet.

³ See http://ts.nist.gov/WeightsAndMeasures/fuels_and_lubricants.cfm

- who suspect that substandard fuels are available for sale in their state because the fuels are not being subjected to quality testing, and who want to gather evidence of existing fuel quality;
- who have been asked to establish a new quality testing program and want to determine the approximate cost and effort to set up a laboratory, whether in-house or on contract; and
- who operate ongoing quality testing programs using either an in-house FQL or a contract laboratory, and who want to determine whether the laboratory meets current national and international standards for laboratory quality.

Quality assurance/quality control programs were originally devised to give statistical verification of analytical results; however, they have now evolved to become the standard management model for laboratories. Safety processes, chain-of-custody procedures, sampling procedures, sample distribution procedures, and documentation of each step of a process has to be integrated into the quality management system of every FQL so that regulatory agencies, the regulated industry, and consumers may have confidence in the results obtained from the testing laboratory.

If an FQL (whether public or private) fulfills the recommendations of this handbook, it meets both the technical competence guidelines and management system guidelines that are necessary for it to consistently deliver technically valid test results.

NOTICE: THIS HANDBOOK DOES NOT ADDRESS ALL SAFETY ISSUES ASSOCIATED WITH OPERATING AN FQL. IT IS RECOMMENDED THAT THE FQL ESTABLISH ITS OWN ENVIRONMENTAL HEALTH AND SAFETY POLICIES AND PROCESSES INCORPORATING REGULATIONS AND BEST PRACTICES AS APPROPRIATE.

1.0. History

NIST collaborates with the National Conference on Weights and Measures (NCWM) (www.ncwm.net) and other organizations to develop technical publications, including handbooks that may subsequently be referenced or adopted into law by the states. In 1984, the NCWM adopted Section 2.20. “Gasoline and Oxygenate Blends” of the Uniform Regulation for the Method of Sale of Commodities requiring that engine fuels containing alcohol be labeled to disclose to the retail purchaser that the fuel contains alcohol. The member states of the NCWM deemed this action necessary since engine vehicle manufacturers were qualifying their warranties with respect to some gasoline-alcohol blends, engine fuel users were complaining to weights and measures officials about fuel quality and vehicle performance, and American Society for Testing and Materials International (ASTM) had not yet finalized quality standards for oxygenated (which includes alcohol-containing) fuels. While a few officials expressed the belief that weights and measures officials should not cross the line from quantity assurance programs to programs regulating quality, most state directors were persuaded that the issue needed immediate attention.

An Engine Fuels Task Force was appointed by the Chairman of the NCWM in 1984 to develop mechanisms for achieving uniformity in the evaluation and regulation of engine fuels. The Task Force developed the Uniform Engine Fuel Inspection Law⁴ and the Uniform Engine Fuel Regulation to accompany the Law. The Uniform Law required sellers to register the fuels they offer for sale and certify that they conform to the appropriate ASTM standards designated in the accompanying regulation.

In 1992, the NCWM established the Petroleum Subcommittee under the Laws and Regulations Committee. The subcommittee recommended major revisions to the regulation that were adopted at the 80th NCWM in 1995. The scope of the regulation was expanded to include all engine fuels, petroleum products, and automotive lubricants; its title was changed accordingly. The fuel specifications and method of sale sections were revised to address the additional products. Other changes included expansion of the definitions section and addition of sections on retail storage tanks, condemned product, registration of engine fuels designed for special use, and test methods and reproducibility limits.

In 2005, the Petroleum Subcommittee (renamed the Fuel and Lubricants Subcommittee in 2008) provided recommendations on establishment and upgrade of state FQLs and in 2007, the NCWM made recommendations for fuel specifications, methods of sale, and testing for gasoline-ethanol mixtures and biodiesel. Between 2008 and 2012, additional amendments were made to the regulation to reflect the continuous evolution in engine fuels being offered for sale. This handbook provides recommendations for the management quality system of the FQL, whose measurements underpin these specifications and methods of sale.

⁴ NIST Handbook 130 “Uniform Laws and Regulations in the areas of legal metrology and engine fuel quality” at <http://www.nist.gov/pml/wmd/pubs/hb130-12.cfm>

1.1. Program Summary

State fuels, petroleum products and lubricants laboratories (fuel quality laboratories or FQL) enforce standards to ensure equity in the buying and selling of engine fuels. As part of its program to encourage a high degree of technical and professional competence in such activities, the National Institute of Standards and Technology (NIST) Office of Weights and Measures (OWM) has developed performance quality management standards for voluntary use by FQLs. The requirements in Section 3.0 “Management Guidelines” and Section 4.0 “Technical Guidelines” in this handbook follow the organization and numbering system of NIST Handbook 143 and Section 4. “Management Requirements for Accreditation” and Section 5. “Technical Requirements for Accreditation” in NIST Handbook 150: 2006 “National Voluntary Laboratory Accreditation Program (NVLAP) - Procedures and General Requirements Clauses” so that a laboratory can cross reference that standard without having to develop a cross reference matrix. The requirements in Section 3.0. “Management Guidelines” and Section 4.0. “Technical Guidelines” in this handbook are also consistent with the requirements of ISO/IEC 17025:2005. Specific recommendations that clarify and amplify the requirements for an FQL, including major safety, environmental health, sampling, and chain-of-custody considerations are also included in this handbook.

1.2. Scope of a Fuel Laboratory Program

The scope of the fuels, petroleum products and lubricants testing capability is the set of test methods contained in ASTM Standards for Petroleum Products, Lubricants, and Fossil Fuels (Section 1.3.1. “ASTM Standards and Other Resources.” See also 4.4. “Test Methods and Method Validation”), including test methods for rating engine, diesel, and aviation fuels and related standards and test methods from the Society of Automotive Engineers (SAE) International. Careful consideration should be given to the selection of laboratory test procedures to be performed by an FQL, since these selections will affect instrument costs, number of personnel, timeliness of sample results, and confidence in results. As previously mentioned, ASTM and SAE specifications and test methods are recognized standards for engine fuels and lubricants and should be the primary choice for test procedures. The laboratory facility and equipment selected for a fuel quality laboratory is scalable so that the test capabilities can meet the needs as well as the resources of a jurisdiction. Full scale laboratories capable of performing the complete range of tests over all products require substantial capital investments and significant levels of staffing. However, a fuel quality program, even if limited in scope, is still a valuable public service because even a minimal regulatory presence in the marketplace will protect consumers and sellers alike. A fully equipped FQL may not be justified or possible for most jurisdictions given the size of their marketplace or budget limitations. For most jurisdictions the resources available for establishing a fuel quality laboratory may fall between a basic and full level of test capabilities. For example, a smaller state that receives most of its fuel from businesses in a state that has strong fuel quality laws and a comprehensive regulatory program may decide to implement a limited fuel quality program. Such a program may focus on responding to consumer complaints and to carrying out marketplace sampling to screen the most common fuels offered for sale to consumers.

Some states use handheld screening devices to monitor some aspects of the fuel and then send suspect samples to a contract laboratory for testing to ASTM standards, using those results to

determine if enforcement action is justified. Also, a jurisdiction that does not have fuel quality program may receive consumer complaints about suspected fuel quality problems. If state law allows it, that jurisdiction may want to implement a basic program that relies on sending fuel samples to an FQL in another state (or to a private contract laboratory) to initiate marketplace surveillance and to develop a base line of compliance. The results found in preliminary testing can be used to seek additional resources, if justified, to grow the program to include regular fuel sample collection from retailers and possibly the acquisition of a laboratory that could meet the needs of the jurisdiction. Another jurisdiction may want to develop an intermediate capability laboratory that tests samples to determine if they conform to most of the critical fuel specifications contained in the ASTM standards. To define its limited scope this jurisdiction may conduct marketplace sampling to identify the most common quality failures. Alternatively, they might reach out to states with existing fuel quality programs to identify failures found in other programs and initially implement the field sampling and laboratory tests to focus on those high risk areas. Later, this intermediate FQL could expand or modify its laboratory test equipment and capabilities to encompass a fuller range of quality specifications based on its experience and if financially feasible.

1.3. References

In Section 1.3.1. “ASTM Standards and Other Resources” several ASTM standards are specifically referenced. The dates of adoption of the standards are omitted because they undergo updates and revisions on a regular basis. Laboratories should adopt and use the latest version of the referenced standard. These ASTM fuel specifications and testing procedures form the nucleus for an engine fuels testing laboratory with law enforcement and consumer protection responsibilities. The list is not intended to be all inclusive or exclusive. The standards and test methods listed here do not exclude other ASTM procedures (or standards from other organizations) that are designed for the purpose and that give comparable results. Each specification or test method includes a reference section that points the reader to other related documents that are relevant and which will provide additional and connected information. The significance of each of these tests is explained in the specifications. Some of the test procedures listed make provisions to allow the use of automated equipment. Such equipment is usually more expensive than manual test equipment. However, the increased cost can be recovered in a high production laboratory by reduced labor costs. Asterisks after test methods indicate a preferred method (based on advice provided by experts in this area) due to cost or ease of implementation.

1.3.1. ASTM Standards and Other Resources

1.3.1.1. Spark Ignition Engine Fuel

- a. ASTM D4814 – Standard Specification for Automotive Spark-Ignition Engine Fuel.
- b. ASTM D86 – Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure.
- c. Octane (Antiknock Index).
 - i. Research: ASTM D2699 – Standard Test Method for Research Octane Number of Spark-Ignition Engine Fuel.

- ii. Engine: ASTM D2700 – Standard Test Method for Engine Octane Number of Spark-Ignition Engine Fuel.
- iii. Antiknock Index = (Research + Engine)/2.
- d. Vapor Pressure.
 - i. ASTM D4953 – Standard Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method).
 - ii. ASTM D5190 – Standard Test Method for Vapor Pressure of Petroleum Products (Automatic Method).*
 - iii. ASTM D5191 – Standard Test Method for Vapor Pressure of Petroleum Products (Mini Method).*
 - iv. ASTM D5482 – Standard Test Method for Vapor Pressure of Petroleum Products (Mini Method—Atmospheric).*
- e. Oxygenate Content.
 - i. ASTM D4815 – Standard Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C1 to C4 Alcohols in Gasoline by Gas Chromatography.
 - ii. ASTM D5599 – Standard Test Method for Determination of Oxygenates in Gasoline by Gas Chromatography and Oxygen Selective Flame Ionization Detection.
 - iii. ASTM D5845 – Standard Test Method for Determination of MTBE, ETBE, TAME, DIPE, Methanol, Ethanol and tert-Butanol in Gasoline by Infrared Spectroscopy.
- f. Sulfur Content.

Sulfur Content – due to environmental law and regulations, the sulfur limits shown in D4814 may be significantly higher than permitted. The detection limit and precision of each method should be considered when selecting a test method. Alternative test methods D5453 or D7039 may be used if they are correlated to D2622.

 - i. ASTM D2622 – Standard Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry.
 - ii. ASTM D5453 – Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence.
 - iii. ASTM D7039 – Standard Test Method for Sulfur in Gasoline and Diesel Fuel by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry.

1.3.1.2. Diesel Fuel

- a. ASTM D975 – Standard Specification for Diesel Fuel Oils.
- b. ASTM D93 – Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester.
- c. ASTM D86 – Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure.

- d. ASTM D1298 – Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method.
- e. Sulfur Content. (The appropriate test method is dependent upon the grade.)
 EPA performance-based test method approach adopted in the diesel sulfur rule, multiple sulfur test methods are allowed. Methods qualify for approval provided they meet specified performance criteria under the Performance Based Testing Criteria in 40 CFR 80.584.
 - i. ASTM D2622 – Standard Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry.
 - ii. ASTM D3120 – Standard Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry.
 - iii. ASTM D4294 – Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry.
 - iv. ASTM D6920 – Standard Test Method for Total Sulfur in Naphthas, Distillates, Reformulated Gasolines, Diesels, Biodiesels, and Engine Fuels by Oxidative Combustion and Electrochemical Detection.
- f. Cloud Point.
 - i. ASTM D2500 – Standard Test Method for Cloud Point of Petroleum Products.
 - ii. ASTM D5771 – Standard Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method).
 - iii. ASTM D5772 – Standard Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method).
 - iv. ASTM D5773 – Standard Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method).
- g. Water and Sediment.
 ASTM D2709 – Standard Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge.
- h. Cetane.
 ASTM D613 – Standard Test Method for Cetane Number of Diesel Fuel Oil. (NOTE: Tests using this method may be costly and the availability of test providers limited. An alternative approach is to use the test methods in D4737 for routine testing and use D613 as the referee method in cases where the test results found with D4737 are in dispute. While D4737 is not the definitive method, it can be used to estimate the Cetane number if tests using D613 are not available.)
 ASTM D4737 – Standard Test Method for Calculated Cetane Index by Four Variable Equation.*
 Alternative Cetane Tests.
 ASTM D6890 – Standard Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber.

ASTM D7170 – Standard Test Method for Determination of Derived Cetane Number (DCN) of Diesel Fuel Oils—Fixed Range Injection Period, Constant Volume Combustion Chamber Method.

i. Lubricity.

ASTM D6079 – Standard Test Method for Evaluating Lubricity of Diesel Fuels by the High-Frequency Reciprocating Rig (HFRR).

j. Viscosity.

ASTM D445 – Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity).

1.3.1.3. Kerosine

a. ASTM D3699 – Standard Specification for Kerosine.

b. Flash Point.

ASTM D56 – 05 Standard Test Method for Flash Point by Tag Closed Cup Tester.

c. Distillation.

ASTM D86 – Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure.

d. Sulfur Content.

i. ASTM D2622 – Standard Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry.*

ii. ASTM D4294 – Standard Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry.

iii. ASTM D5453 – Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence.

iv. ASTM D7039 – Standard Test Method for Sulfur in Gasoline and Diesel Fuel by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry.

e. Color.

ASTM D156 – Standard Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method).

1.3.1.4. Aviation Turbine Fuel

a. ASTM D1655 – Standard Specification for Aviation Turbine Fuels.

b. Flash Point.

ASTM D56 – Standard Test Method for Flash Point by Tag Closed Cup Tester.

c. Distillation.

ASTM D86 – Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure.

- d. Water Reaction.
ASTM D1094 – Standard Test Method for Water Reaction of Aviation Fuels.
- e. Freezing Point.
ASTM D2386 – Standard Test Method for Freezing Point of Aviation Fuels.
- f. Thermal Oxidation Stability.
D3241 – Standard Test Method for Thermal Oxidation Stability of Aviation Turbine Fuels.

1.3.1.5. Engine Oil

Viscosity.

- a. SAE J300 – Engine Oil Viscosity Classification.
- b. Kinematic Viscosity.
ASTM D445 – Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity).
- c. Cold Cranking Simulator.
ASTM D5293 – Standard Test Method for Apparent Viscosity of Engine Oils Between – 5 °C and – 35°C Using the Cold-Cranking Simulator.
ASTM D4684 – Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature.
- d. D4683- Standard Test Method for Measuring Viscosity of New and Used Engine Oils at High Shear Rate and High Temperature by Tapered Bearing Simulator Viscometer at 150 °C.
- e. D4741 Standard Test Method for Measuring Viscosity at High Temperature and High Shear Rate by Tapered-Plug Viscometer.
- f. D5481 Standard Test Method for Measuring Apparent Viscosity at High-Temperature and High-Shear Rate by Multi-cell Capillary Viscometer.

1.3.1.6. Gear Oil

Viscosity.

- a. SAE J306 – Automotive Gear Lubricant Viscosity Classification.
- b. Kinematic Viscosity.
ASTM D445 – Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity).
- c. Brookfield Viscosity.
ASTM D2983 – Standard Test Method for Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer.
- d. Oil Pumpability.
ASTM D4684 Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature.

1.3.1.7. Automatic Transmission Fluid

- a. Kinematic Viscosity.
ASTM D445 – Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity).
- b. Brookfield Viscosity.
ASTM D2983 – Standard Test Method for Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer.

1.3.1.8. Sampling

- a. ASTM D5842 – Standard Practice for Sampling and Handling of Fuels for Volatility Measurement.
- b. ASTM D4057 – Standard Practice for Manual Sampling of Petroleum and Petroleum Products.
- c. ASTM D4306 – Standard Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination.
- d. ASTM D4177 – Standard Practice for Automatic Sampling of Petroleum and Petroleum Products.
- e. ASTM D4418 – Standard Practice for Receipt, Storage, and Handling of Fuels for Gas Turbines.

1.3.1.9. Quality Assurance Guidelines

- a. ASTM D6792 – Standard Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories.
- b. ASTM D6299 – Standard Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance.

1.3.2. Biofuels

1.3.2.1. Ethanol for Blending

- a. ASTM D4806 – Standard Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel.
- b. D5501-Standard Test Method for Determination of Ethanol Content of Denatured Fuel Ethanol by Gas Chromatography.

1.3.2.2. Gasoline-Ethanol Mixtures: 51 % to 83 % Blends

- a. ASTM D5798 – Standard Specification for Ethanol Fuel Blends for Flexible-Fuel Automotive Spark-Ignition Engines.

- b. Ethanol/Methanol Content.
D5501-Standard Test Method for Determination of Ethanol Content of Denatured Fuel Ethanol by Gas Chromatography.
- c. Sulfur Content.
ASTM D5453 – Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence.
- d. Acidity.
ASTM D1613 – Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products.
- e. Gum Content – Solvent-washed and Unwashed.
ASTM D381 – Test Method for Gum Content in Fuels by Jet Evaporation.
- f. pH_e.
ASTM D6423 – Test Method for Determination of Ph_e of Ethanol, Denatured Ethanol Fuel and Fuel Ethanol.
- g. Inorganic Chloride Content.
ASTM D7319 Test Method for Determination of Total and Potential Sulfate and Inorganic Chloride and Fuel Ethanol by Direct Injection Suppressed Ion Chromatography or ASTM D7328 Test Method for Determination of Total and Potential Inorganic Sulfate and Total Inorganic Chloride in Fuel Ethanol by Ion Chromatography Using Aqueous Sample Injection.
- h. Copper.
ASTM D1688 – Test Methods for Copper in Water (as modified by Section 8.1.8.1. in D5798).
- i. Water Content.
ASTM E203 – Test Method for Water Using Volumetric Karl Fischer Titration or ASTM E1064 Test Method Water in Organic Liquids by Coulometric Karl Fischer.
- j. Vapor Pressure.
ASTM D4953 – Standard Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method), ASTM D5190 – Standard Test Method for Vapor Pressure of Petroleum Products (Automatic Method)* or ASTM D5191 – Standard Test Method for Vapor Pressure of Petroleum Products (Mini Method).*

1.3.2.3. Biodiesel (B100) and Biodiesel Blends – The following standards apply to biodiesel, but not necessarily to biomass-based diesel.

- a. ASTM D975 – Standard Specification for Diesel Fuel Oils.
- b. ASTM D396 – Standard Specification for Fuel Oils.
- c. ASTM D7467 – Standard Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20).

- d. ASTM D6751 – Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels (typically used to control pure biodiesel (B100) quality prior to blending with conventional diesel type fuels).

Safety Related Standard/Regulation

1.3.3. References on Safety

- 1.3.3.1.** NFPA 45: Standard in Fire Protection for Laboratories Using Chemicals – 2011, Next Edition: 2015.
URL: <http://www.nfpa.org/aboutthecodes/AboutTheCodes.asp?DocNum=45&cookie%5Ftest=1>
- 1.3.3.2.** NFPA 30: Flammable and Combustible Liquids Code – 2008, Next Edition: 2012.
URL: <http://www.nfpa.org/aboutthecodes/AboutTheCodes.asp?DocNum=30&cookie%5Ftest=1>
- 1.3.3.3.** NFPA 55: Compressed Gases and Cryogenic Fluids Code – 2010, Next Edition: 2013.
URL: <http://www.nfpa.org/aboutthecodes/AboutTheCodes.asp?DocNum=55>
The Code of Federal Regulations (CFR) are available online through the “Federal Register” search engine at:
<http://www.gpo.gov/fdsys/search/submitcitation.action?publication=CFR>
- 1.3.3.4.** Code of Federal Regulations – 40 CFR Parts 260 - 300, Protection of Environment – Environmental Protection Agency (EPA).
 - Use the “Federal Register” Search Engine, see link above.
- 1.3.3.5.** EPA Resource Conservation and Recovery Act 40 CFR Parts 124, 260-268, 270-272, 280.
 - Use the “Federal Register” Search Engine, see link above.
- 1.3.3.6.** EPA Toxic Substances Control Act, applicable regulations in Title 40 CFR, Parts 700-763, 790-799.
 - Use the “Federal Register” Search Engine, see link above.
- 1.3.3.7.** Clean Air Act (EPA), applicable regulations in 49 CFR Parts 25, 50-53, 60-61.
 - Use the “Federal Register” Search Engine, see link above.
- 1.3.3.8.** Clean Water Act (EPA), applicable regulations in 49 CFR Parts 25, 104-117, 129, 131, 135-136.
 - Use the “Federal Register” Search Engine, see link above.
- 1.3.3.9.** Comprehensive Environmental Response, Compensation, and Liability Act (EPA) 40 CFR Parts 700-763, 790-799.
 - Use the “Federal Register” Search Engine, see link above.
- 1.3.3.10.** Occupational Safety and Health Act (OSHA), applicable regulations in 29 CFR Part 1910.

- Use the “Federal Register” Search Engine, see link above.

1.3.3.11. Hazardous Materials Transportation Act (DOT), applicable regulations in 49 CFR Parts 172-177.

- Use the “Federal Register” Search Engine, see link above.

Other Safety Related Resources

1.3.4. Other Safety References

- a. National Safety Council.
URL: <http://www.nsc.org/Pages/Home.aspx>
- b. Department of Transportation Safety Community.
URL: <http://www.dot.gov/safety.html>
- i. Department of Transportation – HAZMAT Safety Community.
URL: <http://www.phmsa.dot.gov/hazmat>
- ii. Safety Training and Outreach from the Pipeline and Hazardous Materials Safety Administration (PHMSA).
URL: <http://www.phmsa.dot.gov/hazmat/training-outreach>

2.0. Terms and Definitions

For the purposes of this handbook, the terms and definitions given in this handbook, NIST Handbook 150 and the relevant ASTM standards apply (additional terms are defined in Section 5.0 “Glossary”).

2.1. ASTM Interlaboratory Crosscheck Program or National Exchange Group – Interlaboratory comparison programs that operate under the auspices of ASTM.⁵

2.2. Biofuel – Liquid fuels and blending components produced from biomass feedstocks, used primarily for transportation (definition from U.S. Energy Information Administration – <http://www.eia.gov/tools/glossary/?id=renewable>).

2.3. Biodiesel – the monoalkyl esters of long chain fatty acids derived from plant or animal matter that meet the registration requirement for fuels and fuel additives under 40 CFR part 79 and the requirements of ASTM D6751-07b (“Standard specification for biodiesel fuel blend stock (B100) for middle distillate fuels”).⁶ Some engine manufacturers have already specified

⁵ See <http://www.astm.org/STATQA/> (downloaded June 6, 2012).

⁶ Code of Federal Regulations (CFR) Title 16: Commercial Practices, Part 306 – Automotive Fuel Ratings, Certification and Posting, downloaded 10 July 2012 from <http://ecfr.gpoaccess.gov/cgi/t/text/text-idx?c=ecfr&rgn=div5&view=text&node=16:1.0.1.3.30&idno=16>

that biodiesel must meet ASTM D6751 as a condition of meeting their warranty requirements.

2.4. Biomass-based Diesel – a diesel fuel substitute produced from non-petroleum renewable resources that meet the registration requirements for fuels and fuel additives established by the EPA under 42 U.S.C. 7545. It includes fuel derived from animal wastes, including poultry fats and poultry wastes, and other waste materials, or from municipal solid waste and sludges, and oils derived from wastewater and the treatment of wastewater. The term does not include biodiesel as defined above. (This means that biomass-based diesel does not necessarily meet the ASTM D6751 standard, but it should meet ASTM D975.)

2.5. Chain of Custody (CoC) – the chronological documentation or paper trail, showing the seizure, custody, control, transfer, analysis, and disposition of evidence, physical or electronic. The chain of custody requires that from the moment the evidence is collected, every transfer of evidence from person to person be documented and that it be provable that nobody else could have accessed that evidence. It is best to keep the number of transfers as low as possible. Chain of custody is used in most chemical sampling situations to maintain the integrity of the sample by providing documentation of the control, transfer, and analysis of samples. Chain of custody is especially important in environmental work where sampling can identify the existence of contamination and can be used to identify the responsible party.

2.6. Chemical Hygiene Plan – required by the “Laboratory Standard” 29 CFR 1910.1450. A document that covers general lab safety, chemical hazards, Personal Protective Equipment (PPE), safety equipment, certification programs, safety responsibilities, exposure procedures, and other occupational health topics.

2.7. Evacuation Plan – A document that outlines the steps to be taken when evacuating the laboratory during an emergency.

2.8. Hazard (Occupational Safety and Health Administration [OSHA]) – Any facility, location, equipment, tool, job, task, or an action that presents a potential for harm.

2.9. Hazardous Waste Management Plan – A document that helps provide compliance with various EPA regulations, providing detailed instructions for the safe disposal of laboratory reagents and hazardous substances.

2.10. Incident – any unplanned event resulting in, or having the potential for injury, ill-health, damage or other loss.⁷

2.11. Material Safety Data Sheets (MSDS) – a form with data regarding the properties of a particular substance. An important component of workplace safety and product stewardship, it is intended to provide workers and emergency personnel with procedures for handling or working

⁷ <http://education.qld.gov.au/strategic/eppr/health/hlspr005/definitions.html>

OSHA does not define incident, but see hazard as a base for defining any incident or near miss <http://e-osh.blogspot.com/2008/10/definition-of-hazard-risk-incident.html>.

with that substance in a safe manner, and includes information such as physical data (e.g., melting point, boiling point, flash point, etc.) toxicity, health effects, first aid, reactivity, storage, disposal, protective equipment, and spill-handling procedures. OSHA requires that MSDS be available to employees for potentially harmful substances handled in the workplace under the Hazard Communication regulation, and is also required to be made available to local fire departments and emergency planning officials. The American Chemical Society defines Chemical Abstracts Service Registry Number (CAS numbers) to provide a unique number for each chemical, and CAS numbers are used internationally in MSDSs.

2.12. Occupant Emergency Plan – A document that lists the people and agencies to call (and contact telephone numbers and email information is included) during an emergency.

2.13. Respirator Program –A program that ensures compliance with 29 CFR 1910.134, and covers the use, care, and fitting of respirators.

2.14. Risk – the statistical chance of danger from an event; an evaluation of the severity and likelihood of an event. OSHA requires that risks be reported when they are associated with medical conditions or exposure.

2.15. Stop Work Authority – the right, obligation, authority, and responsibility to stop any work or actions that are unsafe to personnel, equipment, or that if continued may damage the environment.

3.0. Management Guidelines

3.1. Organization

3.1.1. The laboratory or the organization of which it is part should be an entity that can be held legally responsible.

Implementation Note: *this applies to the laboratory, whether a public (governmental) or private entity.*

3.1.2. It is the responsibility of the laboratory to carry out its testing activities in such a way as to meet the requirements of this handbook and to satisfy the needs of the customer, the regulatory authorities, and other organizations.

3.1.3. The laboratory management system should cover work carried out in the laboratory's permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

3.1.4. If the laboratory is part of an organization performing activities other than testing, the responsibilities of key personnel in the organization that have an involvement or influence on the testing activities of the laboratory should be defined in order to identify potential conflicts of interest.

Implementation Note: *A Fuel Quality Laboratory (FQL) is a testing laboratory. If the laboratory seeks to be recognized as a third-party laboratory, it should be able to demonstrate that it is impartial and it and its personnel are free from any undue commercial, financial, and other pressures, which might influence their technical judgment. The third-party testing laboratory should not engage in any activities that may endanger the trust in its independence of judgment and integrity in relation to its testing activities.*

3.1.5. The laboratory should:

- 1) have managerial and technical personnel who, irrespective of other responsibilities, have the authority and resources needed to carry out their duties, including the implementation, maintenance and improvement of the management system, and to identify the occurrence of departures from the management system or from the procedures for performing tests, and to initiate actions to prevent or minimize such departures (see also 4.2 “Personnel”);
- 2) have arrangements to ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work;
- 3) have policies and procedures to ensure the protection of its customers’ confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results;
- 4) have policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment or operational integrity;
- 5) define the organization and management structure of the laboratory, its place in any parent organization, and the relationships between quality management, technical operations and support services;
- 6) specify the responsibility, authority and interrelationships of all personnel who manage, perform, or verify work affecting the quality of the tests;
- 7) provide adequate supervision of testing staff, including trainees, by persons familiar with methods and procedures, purpose of each test, and with the assessment of the test results;
- 8) have technical management, which has overall responsibility for the technical operations and the provision of the resources needed, to ensure the required quality of laboratory operations;
- 9) appoint a member of staff as quality manager (however named) who, irrespective of other duties and responsibilities, should have defined responsibility and authority for ensuring that the management system related to quality is implemented and followed at all times; the quality manager should have direct access to the highest level of management at which decisions are made on laboratory policy or resources;

- 10) appoint deputies for key managerial personnel (see Implementation Note);
- 11) ensure that its personnel are aware of the relevance and importance of their activities and how they contribute to the achievement of the objectives of the management system; and have policies and procedures to ensure the safety of its laboratory personnel and health of the environment in the conduct of its laboratory testing, including stop work authority.

Implementation Note: *Individuals may have more than one function and it may be impractical to appoint deputies for each function. The "Laboratory Standard," 29 CFR 1910.1450, requires a designated Chemical Hygiene Officer.*

3.1.6. Top management should ensure that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.

3.2. Management System

3.2.1. The laboratory should establish, implement and maintain a management system appropriate to the scope of its activities. The laboratory should document its policies, systems, programs, procedures, and instructions to the extent necessary to assure the quality of the test results. The system's documentation should be communicated to, understood by, available to, and implemented by the appropriate personnel.

Implementation Note: *Environmental health and safety is an integral part of the laboratory management system. Safety should be of primary consideration for the laboratory. All FQLs should have a safe work environment; safety must be inherent in the organization's culture.*

Objectives should include:

- *Achieving an injury-free work place;*
- *Promoting a healthy workplace and mitigating significant health risks;*
- *Eliminating spills and environmental incidents; and*
- *Identifying and mitigating key environmental risks.*

Managerial and administrative controls are one of the basic methods of controlling hazards.

Hazard Evaluation⁸ – Management and staff should analyze the significance of potential hazards through risk analysis or hazard evaluation. Proactive safety protocols prepared for new projects or hazardous methods can systematically identify the risks and plan what steps can be taken to mitigate the risks. A risk control assessment that fully addresses these issues and evaluates any alternatives should be the basis for a systematic plan for projects in the laboratory. Hazard

⁸ <http://www.fda.gov/ScienceResearch/FieldScience/ucm173343.htm>

evaluation includes such factors as the following:

- *Identification of health and physical hazards associated with the material or procedures and the ramifications of exposure;*
- *Estimating the probable exposure by:*
 - *Considering the quantity and form of material;*
 - *Determining the distribution and degree of exposure; personnel exposed.*
- *Determining stability, compatibility, and storage issues;*
- *Assessing the availability and use of various controls, including Personal Protection Equipment (PPE), engineering controls, and managerial/administrative controls; and*
- *Reviewing regulatory issues such as waste or shipping, cleaning up spills, and contamination control.*

3.2.2. The laboratory's management system policies related to quality, including a quality policy statement, should be defined in a quality manual. The overall objectives should be established, and should be reviewed during management review. The quality policy statement should be issued under the authority of top management. It should include at least the following:

- 1) the laboratory management's commitment to good professional practice and to the quality of its testing in servicing its customers;
- 2) the management's statement of the laboratory's standard of service;
- 3) the purpose of the management system related to quality;
- 4) a requirement that all personnel concerned with testing activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work;
- 5) the laboratory management's commitment to comply with this handbook and to continually improve the effectiveness of the management system; and
- 6) the laboratory management's commitment to the safety of their employees and the health of the environment.

Implementation Note: *Safety for everyone present in the FQL and the health of the environment should be part of the quality policy statement. The quality policy statement should be concise and may include the requirement that tests should always be carried out in accordance with stated methods and customers' requirements. When the test laboratory is part of a larger organization, some quality policy elements may be in other documents.*

3.2.3. Top management should provide evidence of commitment to the development and implementation of the management system and to continually improving its effectiveness.

3.2.4. Top management should communicate to the organization the importance of meeting customer requirements as well as statutory and regulatory requirements.

3.2.5. The quality manual should include or make reference to the supporting procedures including technical procedures. It should outline the structure of the documentation used in the management system.

3.2.6. The role and responsibilities of technical management and the quality manager, including their responsibility for ensuring conformance with this handbook, should be defined in the quality manual. The procedures to follow when test methods are updated; that is, the protocol that a quality manager must follow to ensure that all personnel are updated and trained to ensure they run the most current methods correctly should be included in the quality manual. Also, if updates require instrument retrofits, the procedures for making sure that instruments conform to the changes in the test methods should also be included.

3.2.7. Top management should ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented.

3.2.8. Top management should provide evidence of commitment to the safety of the laboratory employees and the health of the environment while operating the testing laboratory.

3.3. Document Control

3.3.1. General

The laboratory should establish and maintain procedures to control all documents that form part of its management system (internally generated or from external sources), such as regulations, standards, other normative documents, test methods, as well as drawings, software, data (including digital and paper documents) specifications, instructions, and manuals.

Implementation Note: *In this context, "documents" could be policy statements, procedures, specifications, calibration tables, charts, text books, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic, or written. The control of data (including digital and paper documents) related to testing is covered in 4.4.7. "Control of Data." The control of records is covered in 3.15. "Control of Records" and 3.16. "Technical Records."*

3.3.1.(a) The FQL should document a Chemical Hygiene Plan, a chain-of-custody plan and procedure, a Hazardous Waste Management Plan, an Evacuation Plan, an Occupant Emergency Plan, applicable Material Safety Data Sheets, and, if applicable, a Respirator Program.

Implementation Note: *Each laboratory's Environmental Health and Safety documents should include, but are not limited to the following:*

- *Chemical Hygiene Plan⁹ or Program: The "Laboratory Standard," 29 CFR 1910.1450, requires a Chemical Hygiene Plan and designated Chemical Hygiene Officer. The basic tenets of safe laboratory work will be found in the Chemical Hygiene Plan or Program. This document helps provide compliance with the "Lab Standard," 29 CFR 1910.1450. Information in this document covers general laboratory safety, chemical hazards, Personal Protective Equipment (PPE), safety equipment certification programs, special SOPs for highly hazardous work, safety responsibilities for various personnel, procedures following exposure to a chemical, and other occupational health topics. Every FQL should have this reference.*
- *Hazardous Waste Management Plan or Program: This document helps provide compliance with various Environmental Protection Agency regulations. The document provides detailed instruction for the safe disposal of laboratory reagents and hazardous substances. Every FQL should have this reference.*
- *Evacuation Plan: This document outlines the steps taken when evacuating the building during an emergency. If the laboratory is co-located with other tenants in a common building, this document may cover all tenants and, therefore, will not be particular to the laboratory. Every FQL should have this reference. (This plan may be combined with the Occupant Emergency Plan at some facilities.)*
- *Occupant Emergency Plan: This document outlines whom to call during an emergency (e.g., building managers, local fire, police, and emergency medical teams). The plan may also provide questions to ask when receiving a telephone bomb threat, or what to do during an earthquake or severe weather. Every laboratory should have this reference.*
- *Respirator Program: This program ensures compliance with 29 CFR 1910.134 – the Respirator Standard. This program covers the use, care, and fitting of respirators. The first choice for protection from respiratory hazards is engineering controls (e.g., fume hoods). However, if the lab decides that it wants or needs to provide the extra protection of respirators the Respirator Standard is followed.*
- *Program to Implement Requirements on Department of Transportation (DOT) Shipment of Dangerous Goods: As mandated by DOT, the shipment of hazardous chemicals requires detailed packaging and labeling. The FQL may have a work instruction or standard procedure devoted to these requirements. NOTE: most private shippers require its users to have employees that are trained and/or certified for hazardous materials.*
- *Program to maintain Material Safety Data Sheets (MSDS) – This is a form provided by the manufacturer or other party, with data regarding the properties of a particular substance. An important component of workplace safety and product stewardship, the*

⁹ There are many good chemical hygiene plans that can be used as models on the internet. Some of the best are for educational organizations. See for examples: <http://ehs.mit.edu/site/>, <http://www.cheme.cornell.edu/cheme/facilities/labsafety/index.cfm>, <http://www.fda.gov/ScienceResearch/FieldScience/ucm173343.htm>.

MSDS is intended to provide workers and emergency personnel with procedures for handling or working with that substance in a safe manner, and includes information such as physical data (melting point, boiling point, flash point, etc.) toxicity, health effects, first aid, reactivity, storage, disposal, protective equipment, and spill-handling procedures. OSHA requires that MSDS be available to employees for potentially harmful substances handled in the workplace under the Hazard Communication regulation and is also required to be made available to local fire departments and emergency planning officials. The American Chemical Society defines Chemical Abstracts Service Registry Number (CAS numbers) to provide a unique number for each chemical; these CAS numbers are also used internationally in the MSDS. Documents may be involved in an administrative hearing or even in a court proceeding, and therefore should be preserved by the best means possible. Documents must be retained in accordance with administrative record retention policies and must be preserved until legal or regulatory proceedings, including appeals, are terminated.

3.3.2. Document Approval and Issue

3.3.2.1. All documents issued to personnel in the laboratory as part of the management system should be reviewed and approved for use by authorized personnel prior to issue. A master list or an equivalent document control procedure identifying the current revision status and distribution of documents in the quality system should be established and be readily available to preclude the use of invalid and/or obsolete documents.

3.3.2.2. The procedure(s) adopted should ensure that:

- 1) authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed;
- 2) documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements;
- 3) invalid or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use; and
- 4) obsolete documents retained for either legal or knowledge preservation purposes are suitably marked.

3.3.2.3. Management system documents generated by the laboratory should be uniquely identified. Such identification should include the date of issue and/or revision identification, page numbering, the total number of pages or a mark to signify the end of the document, and the issuing authority(ies).

3.4. Document Changes

3.4.1. Changes to documents should be reviewed and approved by the same function that performed the original review unless specifically designated otherwise. The designated

personnel should have access to pertinent background information upon which to base their review and approval.

3.4.2. Where practicable, the altered or new text should be identified in the document or the appropriate attachments.

3.4.3. If the laboratory's documentation control system allows for the amendment of documents pending the reissue of the documents, the procedures and authorities for such amendments should be defined. Amendments should be clearly marked, initialed and dated. A revised document should be formally reissued as soon as practicable.

3.4.4. Procedures should be established to describe how changes in documents maintained in computerized systems are made and controlled.

3.5. Review of Requests, Tenders, and Contracts

3.5.1. The laboratory should establish and maintain procedures for the review of requests, tenders, and contracts. The policies and procedures for these reviews leading to a contract for testing should ensure that:

- 1) the requirements, including the methods to be used, are adequately defined, documented, and understood (see 4.4.2. "Selection of Methods");
- 2) the laboratory has the capability and resources to meet the requirements; and
- 3) the appropriate test method is selected and is capable of meeting the—customers' requirements (see 4.4.2. "Selection of Methods").

Any differences between the request or tender and the contract should be resolved before any work commences. Each contract should be acceptable both to the laboratory and the customer.

Implementation Note: *The request, tender, and contract review should be conducted in a practical and efficient manner, and the effect of financial, legal, and time schedule aspects should be taken into account. For internal customers, reviews of requests, tenders and contracts can be performed in a simplified way. The review of capability should establish that the laboratory possesses the necessary physical, personnel, and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the tests in question. The review may also encompass results of earlier participation in interlaboratory comparisons or proficiency testing and/or the running of trial test programs using samples or items of known value in order to determine uncertainties of measurement, limits of detection, confidence limits, etc.*

3.6. Records of Reviews

Records of review including any significant changes should be maintained. Records should also be maintained of pertinent discussions with a customer relating to the customer's requirements or the results of the work during the period of execution of the contract.

Implementation Note: *For review of routine and other simple tasks, the date and the identification (e.g., the initials) of the person in the laboratory responsible for carrying out the contracted work are considered adequate. For repetitive routine tasks, the review need be made only at the initial inquiry stage or on granting of the contract for ongoing routine work performed under a general agreement with the customer, provided that the customer's requirements remain unchanged. For new, complex, or advanced testing tasks, a more comprehensive record should be maintained.*

3.6.1. The review should also cover any work that is subcontracted by the laboratory.

3.6.2. The customer should be informed of any deviation from the contract.

3.6.3. If a contract is to be amended after work has commenced, the same contract review process should be repeated and any amendments should be communicated to all affected personnel.

Implementation Note: *This handbook may be applied to private or public (governmental) FQLs contracted for petroleum testing by government.*

3.7. Subcontracting of Tests¹⁰

3.7.1. When a laboratory subcontracts work, whether due to unforeseen reasons (e.g., workload, need for further expertise or temporary incapacity) or on a continuing basis (e.g., through permanent subcontracting, agency or franchising arrangements), this work should be placed with a competent subcontractor. A competent subcontractor is one that, for example, complies with this handbook for the work in question.

3.7.2. The laboratory should advise the customer of the arrangement in writing, and when appropriate, gain the approval of the customer, preferably in writing.

3.7.3. The laboratory is responsible to the customer for the subcontractor's work, except in the case where the customer or a regulatory authority specifies which subcontractor is to be used.

3.7.4. The laboratory should maintain a register of all subcontractors that it uses for tests and a record of the evidence of conformance with this handbook for the work in question.

3.8. Purchasing Services and Supplies

3.8.1. The laboratory should have a policy and procedure(s) for the selection and purchasing of services and supplies it uses that affect the quality of the tests. Procedures should exist for the purchase, reception, and storage of reagents and laboratory consumable materials relevant for the

¹⁰ *When proficiency testing or interlaboratory comparisons are conducted, the normal operations and operators of the tests should participate; the proficiency testing or interlaboratory comparisons should not be subcontracted if the normal operation or test is not subcontracted.*

tests.

3.8.2. The laboratory should ensure that purchased supplies, reagents, and consumable materials that affect the quality of tests are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the tests concerned. Records of actions taken to check compliance should be maintained.

3.8.3. Purchasing documents for items affecting the quality of laboratory output should contain data describing the services and supplies ordered. These purchasing documents should be reviewed and approved for technical content prior to release.

Implementation Note: *The description may include type, class, grade, precise identification, specifications, drawings, inspection instructions, other technical data including approval of test results, the quality required, and the management system standard under which they were made.*

3.8.4. The laboratory should evaluate suppliers of critical consumables, supplies, and services that affect the quality of testing and should maintain records of these evaluations and list those approved.

Implementation Note: *This should include (but not be limited to) such items as chemical reagents, personal protective equipment, and hazardous waste disposal service providers.*

3.9. Service to the Customer

3.9.1. The laboratory should be willing to cooperate with customers or their representatives in clarifying the customer's request and in monitoring the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other customers.

Implementation Note: *Such cooperation may include: providing the customer or the customer's representative reasonable access to relevant areas of the laboratory for the witnessing of tests performed for the customer; and preparing, packaging, and dispatching of test items needed by the customer for verification purposes. Most customers value the maintenance of good communication, advice, and guidance in technical matters, and opinions and interpretations based on results. Communication with the customer, especially in large assignments should be maintained throughout the work. The laboratory should inform the customer of any delays or major deviations in the performance of the tests.*

3.9.2. The laboratory should seek feedback, both positive and negative, from its customers. The feedback should be used and analyzed to improve the management system, testing activities, and customer service.

Implementation Note: *Examples of the types of feedback include customer satisfaction surveys and review of test reports with customers.*

3.10. Complaints

The laboratory should have a policy and procedure for the resolution of complaints received

from customers or other parties. Records should be maintained of all complaints and of the investigations and corrective actions taken by the laboratory (see also 3.14. “Preventive Action.”)

3.11. Control of Non-conforming Tests

3.11.1. The laboratory should have a policy and procedures that should be implemented when any aspect of its testing, or the results of this work, do not conform to its own procedures or the agreed requirements of the customer. The policy and procedures should ensure that:

- 1) the responsibilities and authorities for the management of non-conforming work are designated and actions (including halting of work and withholding of test reports certificates, as necessary) are defined and taken when non-conforming work is identified;
- 2) an evaluation of the significance of the non-conforming work is made;
- 3) corrective action is taken immediately, together with any decision about the acceptability of the non-conforming work;
- 4) where necessary, the customer is notified and work is recalled; and
- 5) the responsibility for authorizing the resumption of work is defined.

Implementation Note: *Identification of non-conforming work or problems with the management system or with testing activities can occur at various places within the management system and technical operations. Examples are customer complaints, quality control, instrument calibration, checking of consumable materials, staff observations or supervision, test report and calibration certificate checking, management reviews, and internal or external audits.*

3.11.2. Where the evaluation indicates that the non-conforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the corrective action procedures given in 3.14. “Preventive Action” should be promptly followed.

3.12. Improvement

The laboratory should continually improve the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions, and management review.

3.13. Corrective Action

3.13.1. General

The laboratory should establish a policy and procedure and should designate appropriate authorities for implementing corrective action when non-conforming work or departures from the policies and procedures in the management system or technical operations have been

identified.

Implementation Note: *A problem with the management system or with the technical operations of the laboratory may be identified through a variety of activities, such as control of non-conforming work, internal or external audits, management reviews, feedback from customers, and from staff observations. Corrective action may also arise from OSHA inspections.*

In these instances, it may be necessary to clarify the nature of the work performed by the FQL in contrast to other chemical laboratories, such as research and development laboratories in order to achieve compliance with regulations as well as maintain laboratory efficiency without sacrificing safety.

An FQL normally follows routine processes for large numbers of samples, so the sample containers, for example, may contain less information than found in R&D labs, and reference may have to be made back to data logs and other documents to obtain all information that would ordinarily be entered on one-off labels such as are routine in an R&D lab. Whatever the outcome of a regulatory inspection be certain (1) of what the inspector or auditor considers full compliance; (2) of what recourse the laboratory management has in suggesting alternative responses for compliance; (3) to record back and forth discussions and any final resolution; and (4) to share the outcome with peer FQLs for future improvements and use.

3.13.2. (Root) Cause Analysis

The procedure for corrective action should start with an investigation to determine the root cause(s) of the problem.

Implementation Note: *Cause analysis is the key and sometimes the most difficult part in the corrective action procedure. Often the root cause is not obvious and thus a careful analysis of all potential causes of the problem is required. Potential (root) causes could include customer requirements, the samples, sample specifications, methods and procedures, staff skills and training, consumables, or equipment and its calibration.*

3.13.3. Selection and Implementation of Corrective Actions

Where corrective action is needed, the laboratory should identify potential corrective actions. It should select and implement the action(s) most likely to eliminate the problem and to prevent recurrence. Corrective actions should be to a degree appropriate to the magnitude and the risk of the problem. The laboratory should document and implement any required changes resulting from corrective action investigations.

3.13.4. Monitoring of Corrective Actions

The laboratory should monitor the results to ensure that the corrective actions taken have been effective.

3.13.5. Additional Audits

Where the identification of nonconformities or departures casts doubts on the laboratory's compliance with its own policies and procedures, or on its compliance with this handbook, the laboratory should ensure that the appropriate areas of activity are audited in accordance with

3.17. “Internal Audits” as soon as possible.

Implementation Note: *additional audits often follow the implementation of the corrective actions to confirm their effectiveness. These audits should be necessary only when a serious issue or risk to the business is identified.*

3.14. Preventive Action

3.14.1. Needed improvements and potential sources of nonconformities, either technical or concerning the management system, should be identified. When improvement opportunities are identified or if preventive action is required, action plans should be developed, implemented, and monitored to reduce the likelihood of the occurrence of such nonconformities and to take advantage of the opportunities for improvement.

3.14.2. Procedures for preventive actions should include the initiation of such actions and application of controls to ensure that they are effective.

Implementation Note: *Preventive action is a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints. Apart from the review of the operational procedures, the preventive action might involve analysis of data, including trend and risk analyses, and proficiency testing results.*

3.14.3. The laboratory should evaluate its policies and procedures for hazard control on at least an annual basis, in a proactive approach, using preventive action.

Implementation Note: *Hazard Evaluation and Control¹¹ Hazard control and hazard evaluation is a proactive approach to personnel and environmental safety. The three basic procedures to control hazards are the following:*

- *Management and administrative controls,*
- *Technical and engineering controls, and,*
- *Personal Protective Equipment (PPE).*

However, nothing replaces good work practices for ensuring the safest work environment possible. Management/administrative and technical/engineering controls are the preferred method of hazard mitigation as they reduce or remove the hazards, and provide protection in the event of an error in work practices. The use of PPE is the least desirable way to control

¹¹ <http://www.fda.gov/downloads/ScienceResearch/FieldScience/UCM092174.pdf>

hazards. PPE only reduces exposure to the hazard; the hazard is still present. PPE can fail during use and only offer temporary protection. It is critical the proper PPE is assigned for the hazard. PPE also has certain physical limitations in use and can even cause injury to the user if not used properly. Supervisors and employees should analyze the significance of potential hazards associated with laboratory operations through risk analysis or hazard evaluation. Proactive safety protocols prepared for new projects or very hazardous methods can systematically identify the risks and plan what steps can be taken to mitigate the risks.

Physical Hazards – Physical hazards are those caused by direct interaction with the mechanics of the work environment.

Potential physical hazards in the workplace include the following:

- *extreme hot and cold temperatures,*
- *noise,*
- *electricity,*
- *needles, blades (sharps), and*
- *electric and magnetic fields (EMF).*

Other physical hazards include:

- *injuries from slips, trips, and falls,*
- *cuts, hot surfaces, chemicals (fumes and caustic burns)*
- *falling and other moving objects,*
- *dusty environments, and*
- *Poor ergonomic work practices.*

In the Laboratory Standard, OSHA defines physical hazards to mean a chemical that is the following:

- *combustible liquid,*
- *compressed gas,*
- *explosive,*
- *flammable,*
- *organic peroxide,*

- oxidizer,
- pyrophoric,
- unstable (reactive), or
- water reactive.

Thus, gasoline and similar liquid chemicals are types of physical hazards under the Laboratory Standard. Technical and engineering controls, meaning the source of the hazard is modified by a permanent solution, mitigate most physical hazards. Examples include the installation of fume hoods, tempering the air by heating or cooling, ensuring that Ground-Fault-Circuit-Interrupter (GFCI) receptacles are in place, providing floor mats to reduce the chance of slipping, and separate storage for combustible and flammable compounds. Physical hazards are the number one cause of laboratory accidents. Improper lifting techniques, slips, trips and falls, and cuts make up the vast majority of laboratory accidents. Proper housekeeping, paying attention to office and laboratory surroundings, and other good working practices can minimize all of these accidents. OSHA addresses some of the physical hazard issues in the general sections in 29 CFR 1910. The National Fire Protection Agency covers laboratories in NFPA 45 Standard on Fire Protection for Laboratories Using Chemicals (2011 Edition). The NFPA covers issues with flammability in NFPA 30 Flammable and Combustible Liquids Code, and compressed gases in portable cylinders in NFPA 55.

Chemical Hazards

Chemical hazards can enter and harm the body by four main routes:

- absorption through the skin;
- inhalation;
- injection; and
- ingestion.

Chemical hazards cause harm in seven different ways:

- catching fire;
- explosive or reactive;
- corrosive;
- irritant;

- *causing chronic organ damage over time;*
- *causing an allergic reaction; and*
- *causing genetic or reproductive harm.*

A chemical's potential for harm is affected by its properties, (e.g., solid, liquid, or gas). Some questions to ask: if it is a solid, what size are the pieces – micron sized particles, granules, or large chunks? What is the temperature of the chemical? How easily is the chemical absorbed through the skin? Is it toxic? Does it persist in the environment, or is it easily dissipated?

The hazardous properties of many of the chemicals used in the laboratory have been extensively studied by the National Toxicology Program (NTP), the International Agency for Research on Cancer Monographs (IARC), and the American Conference of Government Industrial Hygienists (ACGIH). ACGIH has established Threshold Limit Values (TLVs), for many chemicals. The TLV is an 8-hour time-weighted average (TWA) believed to be the average concentration most workers can be exposed during an 8-hour workday, day after day, five days per week, without harmful effects. Short-term exposure limits (STEL) establishes for materials that are more toxic, the maximum concentration employees can be exposed to for periods up to fifteen minutes that should not be exceeded at any time during a workday. Ceiling (C) is a maximum concentration never to be exceeded. OSHA adapted many of the recommendations of the ACGIH and listed the chemicals and their permissible exposure limits for the TWA in the Limits for Air Contaminants Table (Table Z-1) in 29 CFR 1910.1000.

One of the easiest ways to gather information about the chemical hazards of a compound is to read the Material Safety Data Sheet (MSDS). No employer may allow the use, handling, or storage of a controlled hazardous product in a workplace unless the product carries a label, a material safety data sheet, and the worker has received the training and information to carry out the work entrusted to him safely. Every laboratory is required to have a MSDS library containing an MSDS sheet for every chemical in their inventory. Additionally, the MSDSs are readily found on the websites of most suppliers. Examples include the following: <http://www.vwrsp.com> or <http://www.fishersci.com> or <http://hazard.com/msds/> or <http://www.ilpi.com/msds/index.html>.

In addition to general safety guidelines, OSHA has standards for chemicals in various sections of 29 CFR 1910.1; these include formaldehyde, benzene, benzenidine, arsenic, lead, cadmium, and methylene chloride.

Respiratory Hazards – *There are three types of respiratory hazards:*

- *oxygen-deficient air;*
- *particulate contaminants; and*
- *gas and vapor contaminants.*

In the laboratory, particulate, gas, and vapor contaminants are the most probable. The most preferred way of dealing with these hazards is through engineering controls, as these will remove or mitigate the hazard.

Petroleum laboratories might use the following ventilation controls:

- *use of directional airflow in the rooms;*
- *frequent air exchanges in the rooms;*
- *use of chemical hoods;*
- *storage cabinets; and*
- *weighing hoods.*

Use the hood often to mitigate respiratory hazards. This ubiquitous source of protection is one of the best defenses in the laboratory. The analyst should use the following procedures when working in the hood:

- *place the sash down low enough to protect the face and neck;*
- *always work at least six inches inside of the hood;*
- *never block the rear air vents;*
- *keep the amount of materials used in the hood at a minimum;*
- *dissuade fellow employees from disturbing the air patterns when the hood is in use; and*
- *test hoods annually.*

Other ways to mitigate respiratory hazards include the following:

- *use of vacuum systems near dust producing operations;*
- *higher number of air exchanges in laboratories than in normal office settings; and*
- *not recycling laboratory air through office portions of the building.*

The following cases may warrant the use of respirators:

- 1) *Laboratory exposure to respiratory hazards may not be controlled using the usual ventilation devices in instances where the Threshold Limit Values or level for safe exposure is very small. An employee may want additional protection even though the respiratory hazard has been mitigated to levels considered safe by OSHA or other*

governing bodies. Any use of a respirator requires that the laboratory have a Respiratory Protection Program as defined by 29 CFR 1910.134. Care is taken to assign the employee a respirator designed to control his particular potential exposure. Under the OSHA Respiratory program, the employee must be medically fit to wear a respirator, trained about the hazard, instructed how to use a respirator, and fit-tested.

- 2) Respirators are commonly used in the laboratory for dust control in grinding operations. Typical respirators use P100 filters. Those involved with hazardous waste consolidation may be fitted with half mask respirators and organic acid vapor cartridges.*

General Safety Guidelines

While accidents or serious mishaps in the laboratory are rare, it is extremely important to be prepared and know what to do in case of an emergency. Laboratories are outfitted with specialized equipment and kits for chemical spills, fires, and personal injuries. Written procedures specifying what one should do in emergencies are available. Requirements for emergency evacuations can be found in the NFPA Life Safety Code and OSHA 29 CFR 1910.38. A chemical inventory and a diagram of the laboratory space are often requested by local emergency fire and rescue departments to expedite their response.

Maintenance

- Predictive Maintenance – an approach that determines when maintenance is needed based on the actual equipment condition and data on past performance.*
- Preventive Maintenance – any planned maintenance designed to extend equipment life and avoid unscheduled maintenance outages. The goal is to prevent equipment failure rather than react to it.*
- Reactive Maintenance – the practice of waiting until an equipment failure occurs and then repairing the equipment.*
- Reliability-Centered Maintenance – a systematic approach to evaluate a facility's equipment and develop a cost-effective approach to maintaining its reliability, by focusing attention on the higher-priority components first.*

See 1.3.3. “References on Safety” and 1.3.4. “Other Safety Related Resources.”

3.15. Control of Records

3.15.1. General

3.15.1.1. The laboratory should establish and maintain procedures for identification, collection, indexing, access, filing, storage, maintenance, and disposal of quality and technical records. Quality records should include reports from internal audits and management reviews as well as records of corrective and preventive actions.

3.15.1.2. All records should be legible and should be stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss. Retention times of records should be established.

Implementation Note: *Records may be in any media, such as hard copy or electronic media. Documents, no matter what the content, may be involved in a regulatory action or court proceeding and, therefore, should be preserved by the best means possible. Preservation should be effective until any legal or regulatory proceedings are terminated.*

3.15.1.3. All records should be held secure and in confidence.

3.15.1.4. The laboratory should have procedures to protect and back up records stored electronically and to prevent unauthorized access to or amendment of these records.

3.16. Technical Records

3.16.1. The laboratory should retain records of original observations, derived data, and sufficient information to establish an audit trail, calibration records, staff records, and a copy of each test report issued, for a defined period. The records for each test should contain sufficient information to facilitate, if possible, identification of factors affecting the uncertainty and to enable the test to be repeated under conditions as close as possible to the original. The records should include the identity of personnel responsible for the sampling, performance of each test, and checking of results.

Implementation Note: *Technical records are accumulations of data (see 4.4.7. “Control of Data”) and information, which result from carrying out tests, and that indicate whether specified quality or process parameters are achieved. They may include forms, contracts, work sheets, work books, check sheets, work notes, control graphs, external and internal test reports, customers’ notes, papers, and feedback.*

3.16.2. Observations, data, and calculations should be recorded at the time they are made and should be identifiable to the specific task.

3.16.3. When mistakes occur in records, each mistake should be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records should be signed or initialed by the person making the correction. In the case of records stored electronically, equivalent measures should be taken to avoid loss or change of original data.

3.17. Internal Audits

3.17.1. The laboratory should periodically, and in accordance with a predetermined schedule and procedure, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the management system and this handbook. The internal audit program should address all elements of the management system including the testing activities. It is the responsibility of the quality manager to plan and organize audits as required by the schedule and requested by management. Such audits will be carried out by trained and qualified

personnel who are, wherever resources permit, independent of the activity to be audited.

3.17.2. When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity (integrity) of the laboratory's test results, the laboratory should take timely corrective action, and should notify customers in writing if investigations show that the laboratory results may have been affected.

3.17.3. The area of activity audited, the audit findings, and corrective actions that arise from them should be documented.

3.17.4. Follow-up audit activities should verify and record the implementation and effectiveness of the corrective action taken.

3.17.5. The laboratory should audit its safety and environmental health programs as part of its internal audit.

Implementation Note: *Laboratory safety and environmental health should be assessed as part of the internal audit. Items that are inspected and evaluated during this inspection should include but not be limited to:*

- *environmental (e.g., temperature, lighting, noise, tripping hazards, stable shelving, and odors);*
- *housekeeping (e.g., neat storage areas, clear aisles, safe storage, trash disposal);*
- *fire safety (e.g., emergency evacuation plan, emergency lights, clearance of combustibles from open flames and hot equipment);*
- *electrical (e.g., switch cover plates used, no power cord frays, permanent wiring used, GFCI used in wet areas, circuit breakers identified);*
- *general laboratory safety (e.g., no food or drink in the laboratory, proper storage of chemicals, proper facility for solvent storage, no excess chemicals, certified hoods and storage cabinets, proper use of hoods, labels on chemicals and refrigerators, hazard signage on laboratory doors, proper management of carcinogens, peroxide labels used, no mercury contamination, pipetting devices used, equipment is grounded, equipment operated under pressure inspected, glass under pressure taped);*
- *compressed gas safety (e.g., gas cylinders are labeled, secured, properly transported, and properly stored, correct regulators used);*
- *Personal Protective Equipment (e.g., laboratory coats, safety glasses, goggles, face shields, gloves, closed toe shoes, respirators, flushing eyewashes, and safety showers, maintained first aid kits); and*
- *waste management, including infectious, hazardous, select agent, radioactive, recycled, and universal wastes (e.g., regulated wastes properly managed, labeled, proper*

disposition, and documentation)

3.18. Management Reviews

3.18.1. In accordance with a predetermined schedule and procedure, the laboratory's top management should periodically conduct a review of the laboratory's management system and testing activities to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. The review should take account of:

- the suitability of policies and procedures;
- reports from managerial and supervisory personnel;
- the outcome of recent internal audits (see 3.17 "Internal Audits;")
- corrective and preventive actions;
- assessments by external bodies;
- the results of interlaboratory comparisons or proficiency tests;
- changes in the volume and type of the work;
- customer feedback;
- complaints;
- recommendations for improvement; and
- other relevant factors, such as quality control activities, resources, and staff training.

Implementation Note: *A typical period for conducting a management review is once every 12 months. Results should feed into the laboratory planning system and should include the goals, objectives and action plans for the coming year. A management review includes consideration of related subjects at regular management meetings.*

3.18.2. Findings from management reviews and the actions that arise from them should be recorded. The management should ensure that those actions are carried out within an appropriate and agreed timescale.

3.18.3. The laboratory safety and environmental health programs should be part of the management review.

Implementation Note: *The management and administrative controls in place for safety (including environmental safety) should be reviewed. These types of controls are for example:*

General Safety Guidelines¹² and rules such as, but not limited to the following:

- *never work alone in the laboratory (use a “buddy system”);*
- *never mouth-pipette;*
- *wear safety glasses or goggles at all times in the laboratory;*
- *practice personal hygiene rules, (e.g., wash hands before leaving the laboratory);*
- *no eating or drinking in the laboratory;*
- *use Personal Protective Equipment (PPE);*
 - *do not wear laboratory coats outside the laboratory area;*
 - *wear closed toe, sturdy shoes;*
- *practice good housekeeping techniques;*
 - *keep walkways clear;*
 - *label and date all containers.*

See the Chemical Hygiene Plan for guidance identified for the local laboratory.

Emergency Preparedness¹³

- *Know laboratory policies and procedures.*
- *Read the laboratory's chemical plan and evacuation plan to determine the steps needed in different emergencies. The emergency response plan and occupant emergency plan can provide useful information. These documents provide the following information:*
 - *who to contact in an emergency,*
 - *when and how to clean up a chemical spill,*
 - *where the MSDS are located, and*
 - *when one should use a fire extinguisher.*

¹² <http://www.ehs.cornell.edu/lrs/manual/ch4.cfm>
<http://www.osha.gov/SLTC/laboratories/index.html>

¹³ <http://www.fda.gov/ScienceResearch/FieldScience/ucm173344.htm>

- *If clarification is needed on any of these or other emergency procedures, ask a supervisor or chemical hygiene officer.*
- *Be familiar with laboratory surroundings.*
- *From the laboratory workbench, know where:*
 - *the nearest exit(s);*
 - *eyewash fountain;*
 - *safety shower;*
 - *MSDS;*
 - *fire extinguisher; and*
 - *first aid kits are located.*
- *It is a good practice to identify at least two or more exits in case one is inaccessible. Before working in a different location, identify the location of all safety equipment. If anyone cannot locate any of the above-mentioned items, ask a supervisor or a co-worker for assistance.*

Practice Drills – *The best way to understand something thoroughly is to run through a mock exercise or drill. Consider instances where one may need to walk from a hood to the eyewash station if one's eyesight is impaired due to acid splashed in the eye. Know how to activate the eyewash as well as the time required for a good rinse. This exercise can also be applied to an accident that would need the use of safety showers.*

Emergency (e.g., flood, workplace violence) and Fire – *Be aware of the nearest exits. In the event of a fire, alert others to the situation. Trained, authorized personnel only use fire extinguishers provided in the laboratory.*

Injury – *If someone is injured, assess the situation; assist if possible. Immediately leave the area, close the doors, and leave by the nearest exit. Alert responsible fire commanders if anyone is still inside the building. Follow the laboratory's evacuation plan and any special procedures in the plan (e.g., some facilities arrange for supervisors to meet with their group outside to account for any potential missing employees). Fire drills should be held at least annually.*

Chemical Spill Kits – *Be familiar with chemical spill kits contents and the procedures for cleaning-up spills. Document the spill, the personnel exposed during the incident, and the clean-up procedure. Chemical spill clean-up procedures depend upon the type and amount of the chemical spilled. Acid and base spills can be neutralized, rendering them non-hazardous, while solvent and toxic chemical spills need to be absorbed and disposed as hazardous waste.*

***Hazardous Waste Disposal** – Periodic review of the chemical inventory will ensure unnecessary chemicals will be disposed of in a timely manner.*

4.0. Technical Guidelines

4.1. General

4.1.1. Many factors determine the correctness and reliability of the tests performed by a laboratory. These factors include contributions from:

- human factors (4.2. “Personnel”);
- accommodation and environmental conditions (4.3. “Accommodation and Environmental Conditions”);
- test methods and method validation (4.4. “Test Methods and Method Validation”);
- equipment and its calibration and maintenance (4.5. “Equipment”);
- measurement traceability (4.6. “Measurement Traceability”);
- sampling (4.7. “Sampling”); and
- the handling of test samples (4.8. “Handling of Samples”).

4.1.2. The extent to which the factors contribute to the total uncertainty of measurement differs considerably between (types of) tests. The laboratory should take account of these factors in developing test methods and procedures, in the training and qualification of personnel, and in the selection and calibration of the equipment it uses.

4.2. Personnel

4.2.1. The laboratory management should ensure the competence of all who operate specific equipment, perform tests, evaluate results, and sign test reports. When using staff that is undergoing training, appropriate supervision should be provided. Personnel performing specific tasks should be qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

Implementation Note: Staffing Requirements – *The staffing requirements for an FQL will be dependent on the number of samples, the number of tests performed on the samples, and the testing instruments chosen. The staff recommended below will be suitable for a laboratory with moderate automation (auto-sampler for the gas chromatograph, automated RVP instrument, etc.) running approximately 6000 to 8000 samples per year.*

- 1 - Laboratory Administrator
- 2 - Chemists

- 2 - CFR Engine Operators
- 2 - Laboratory Technicians
- 1 - Clerk

Laboratory Administrator – The laboratory administrator should have strong management skills and familiarity with laboratory operations and chemical techniques. The administrator's responsibilities include the development and implementation of the quality assurance program, safety program, and hazardous waste program, as well as providing guidance for the daily operations of the laboratory.

Chemists – The chemists should have a strong chemistry background and familiarity with instrumental techniques. In addition to normal analytical responsibilities, chemists should assist with the review of analytical results by technicians. Chemists also can assist in the development and implementation of the quality assurance, safety, and hazardous waste programs.

Engine Operators – The engine operators are the most difficult positions to fill. The ideal operator will have petrochemical experience with a mechanic's background since the majority of the engine maintenance will be performed by the operators. The petroleum industry estimates approximately five years of engine operation is necessary to develop an expertise in engine operation. To expedite this process, engine operators should periodically attend training workshops.

Laboratory Technicians – Laboratory technicians should have laboratory experience and a familiarity with scientific methods. Cross training of these individuals is an effective means of maintaining an even workflow through the laboratory. In some technical areas (e.g., nondestructive testing), it may be required that the personnel performing certain tasks hold personnel certification. The laboratory is responsible for fulfilling specified personnel certification requirements. The requirements for personnel certification might be regulatory, included in the standards for the specific technical field, or required by the customer.

The personnel responsible for the opinions and interpretation included in test reports should, in addition to the appropriate qualifications, training, experience and satisfactory knowledge of the testing carried out, also have:

- relevant knowledge of the technology used for the manufacturing of the items, materials, products, etc. tested, or the way they are used or intended to be used, and of the defects or degradations which may occur during or in service;
- knowledge of the general requirements expressed in the legislation and standards; and
- an understanding of the significance of deviations found with regard to the normal use of the items, materials, products etc., concerned.

4.2.2. The management of the laboratory should formulate the goals with respect to the education, training, and skills of the laboratory personnel. The laboratory should have a policy and procedures for identifying training needs and providing training of personnel. The training program should be relevant to the present and anticipated tasks of the laboratory. The effectiveness of the training actions taken should be evaluated.

Implementation Note: *The most effective safety tool is thorough training of employees. Each new employee should be trained with the Chemical Hygiene Plan, safety procedures, emergency response manual, and MSDS's. Subsequent review sessions should be scheduled to ensure familiarity of individual responsibilities and actions. Educational videos are available specifically addressing laboratory safety that can assist in the training process. Hands-on training should be utilized to demonstrate the proper use of fire extinguishers, fire blankets, and other safety equipment in the laboratory. An effective safety program will produce aware employees who can suggest enhancements to improve the safety of the laboratory.*

4.2.3. The laboratory should use personnel who are employed by, or under contract to, the laboratory. Where contracted and additional technical and key support personnel are used, the laboratory should ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's management system.

4.2.4. The laboratory should maintain current job descriptions for managerial, technical, and key support personnel involved in tests.

Implementation Note: *Job descriptions can be defined in many ways. At a minimum, the following should be defined:*

- *the duties and responsibilities with respect to performing tests;*
- *the duties and responsibilities with respect to the planning of tests and evaluation of results;*
- *the duties and responsibilities for reporting opinions and interpretations;*
- *the responsibilities with respect to method modification and development and*
- *validation of new methods;*
- *expertise and experience required;*
- *qualifications and training programs;*
- *managerial duties.*

4.2.5. The management should authorize specific personnel to perform particular types of sampling, test, to issue test reports, to give opinions and interpretations, and to operate particular types of equipment. The laboratory should maintain records of the relevant authorization(s),

competence, educational and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel. This information should be readily available and should include the date on which authorization and/or competence is confirmed.

4.2.6. All laboratory personnel should receive appropriate annual training in environmental health and safety.

4.3. Accommodation and Environmental Conditions

Implementation Note: *Environmental health¹⁴ comprises aspects of human health, including quality of life, that are determined by interactions with physical, chemical, biological, and social factors in the environment. It also refers to the theory and practice of assessing, correcting, controlling, and preventing those factors in the environment that may adversely affect the health of present and future generations (from the Pew Environmental Health Commission).*

Chemical releases into the environment can have an adverse effect on human health and may result in human disease and environmental damage. Many of the chemicals used can have an adverse environmental effect. Two sources of pollution from the laboratories are air contamination from building exhausts and waste disposal. Laboratory exhaust may not be uniformly regulated (check with state and local environmental regulatory agency), but waste disposal is closely regulated.

Hazardous substances are used every day in the laboratory. The previous Implementation Notes on safety describe some of the programs designed to mitigate the hazards associated with the use of hazardous substances. When a task is completed, the left-over chemicals, reagents, and media should be disposed in accordance with the various regulations designed to protect the environment from hazardous waste.

The Environmental Protection Agency is the Federal Agency most responsible for promulgating laws designed to protect the environment. Occupational Safety and Health Administration (OSHA) and the Department of Transportation (DOT) have also promulgated regulations regarding environmental health as it applies to the laboratories. The state, county or public utility district will likely have additional requirements for proper waste disposal.

Chain of Custody – *Each state should have established state-wide minimum physical security standards for its facilities including building security, protection of official samples, visitor control, document security, controlled substances (Schedule I and II), alcohol (95 % and absolute), and certain laboratory items such as rare metals, certified reference materials, test weights, and syringes (1 mL to 20 mL).*

Controlled Areas – *The following areas are designated as controlled areas within the laboratory and need additional protective measures to ensure the integrity of the security interest involved:*

- *solvent storage area;*

¹⁴ <http://www.fda.gov/downloads/ScienceResearch/FieldScience/UCM092174.pdf>

- *alcohol storage area;*
- *sample storage area;*
- *document room;*
- *computer room; and*
- *mail room*

For these controlled areas, the following additional protective measures are provided:

- *Access is limited to only those employees who need access in the performance of their official duties.*
- *Entrances are secured at all times or monitored by an authorized employee or security guard.*
- *Doors are equipped with high security locks or card readers with alarm contacts. High security locks are keyed "separately" from the building master key system. Card readers are keyed "alike."*
- *Controlled areas are cleaned only during normal working hours and under the supervision of an authorized employee or security guard.*
- *Locks or their combinations are changed if the key or combination has been compromised, if the area has been discovered unsecured or unattended, or when an employee no longer needs access due to transfer, termination, or retirement.*

4.3.1. Laboratory facilities for testing, including but not limited to energy sources, lighting, and environmental conditions, should be such as to facilitate correct performance of the tests. The laboratory should ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any measurement. Particular care should be taken when sampling and tests are undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of tests should be documented.

Implementation Note: *Security of Samples from point of collection, and in the Laboratory – For purposes of litigation, regulatory agencies must be able to prove the legal integrity (e.g., chain of custody) of all samples and data introduced as evidence. This means it is necessary to have an accurate written record to track the possession, handling, and location of samples and data from collection through reporting. Verification of who has possessed the samples and data and where the samples have been is easier if you follow chain-of-custody procedures. Good training resources and model forms are available at www.epa.gov/eogapti1/coc/.*

Since there is no way to know in advance which samples and data may be involved in litigation,

you should always follow chain-of-custody procedures whenever samples and data are collected, transferred, stored, analyzed, or destroyed. A secure chain of custody, combined with the use of proper analytical methods and techniques, is necessary for a legally defensible reporting of the sample.

Implementation Note: *An FQL requires a unique building designed to accommodate laboratory instruments ranging from a delicate gas chromatograph to octane engines capable of producing severe vibrations. In addition, extremely flammable liquids will be stored and tested throughout the facility. Obviously, the facility design must minimize the chances of explosion and fire and also be capable of withstanding the forces of an explosion. National Fire Protection Association (NFPA) 45, "Standard on Fire Protection for Laboratories Using Chemicals" should be reviewed with contractors to ensure minimum standards are met.*

Special considerations should be given to the following:

- 1) Sufficient ventilation to ensure that workers are not unduly exposed to gasoline fumes or other toxic vapors.*
- 2) Fume hoods and exhaust systems in laboratory areas.*
- 3) Drain lines resistant to acid and petroleum products.*
- 4) Traps to prevent petroleum products from entering the sewer system.*
- 5) Special foundations for ASTM/Cooperative Fuel Research Committee (CFR) engines. It is recommended that sufficient foundations for future expansion be installed during initial construction.*
- 6) Necessary safety equipment, such as fire blankets, fire extinguisher, and eyewash stations appropriate for the laboratory environment.*
- 7) Automatic fire extinguishing system for laboratory areas. The extinguishing system's design should include considerations regarding the susceptibility of laboratory instruments to damage when exposed to water or dry chemicals.*
- 8) An adequate heating, ventilation, and air conditioning (HVAC) system to handle excess heat generated by distillation instruments and octane engines.*
- 9) A properly designed and sized electrical system.*
- 10) The laboratory's design must ensure that all fuels testing can be performed in accordance with ASTM requirements. This consideration is especially important for the CFR engines. Volume 05.04 of the Annual Book of ASTM Standards contains valuable information regarding the design of a knock-testing laboratory.*
- 11) Automatic hydrocarbon monitors to warn of critical accumulation of explosive vapors.*

Several fixed equipment items are necessary for the laboratory's operation, including:

- 1) Air compressor and piping of sufficient size to supply the entire laboratory's needs.*
- 2) Gas and water piped to all areas of the laboratory.*
- 3) Storage area for excess fuel after analyses. Depending on the number of samples, this may consist of a properly ventilated storage area with 208 L (55 gal) drums to several 1892 L (500 gal) storage tanks. (Larger tanks may be needed if they are to supplement the program's vehicle's needs.)*

The size of the laboratory will depend upon the needs of the agency and the scope of the fuels testing laboratory (The State of Minnesota reports that they entered into an arrangement with a local fuel company for the company to accept excess fuel after analyses into their waste fuel piping systems for reclamation. This has greatly reduced that state's need for any significant excess fuel storage area). The following space listing is for a small laboratory capable of testing approximately 6000 samples per year. Some space requirements, such as those for octane testing, may seem large, but it is strongly recommended that two additional engine foundations be installed during initial construction.

- 1) Offices, bathroom and storage facilities, conference room and other workspaces (as needed). No space requirements are listed as this must be determined by the user based on program needs and local building codes.*
- 2) Octane laboratory – designed for four engines (75 m² [750 ft²])*
- 3) General laboratory (70 m² [750 ft²])*
- 4) Distillation laboratory 37 m² [400 ft²])*
- 5) Shipping and receiving (includes preparation area for empty sample containers) (37 m² [400 ft²])*
- 6) Flash point laboratory (19 m² [200 ft²])*
- 7) Shop area (23 m² [225 ft²])*
- 8) Storage for supplies (23 m² [225 ft²])*
- 9) Secured, cooled, and ventilated sample and flammable storage area (23 m² [225 ft²]). (Insulation and a dedicated ventilation and cooling system should be considered for this room.)*

Total square footage (exclusive of item 1) – 30 m² (3225 ft²). Including offices, bathroom facilities, hallways, etc., the total building size may exceed 372 m² (4000 ft²).

It is not necessary to isolate each testing operation into separate laboratories. However, because of the noise generated, it is recommended that the test engines (octane and Cetane) be placed in a separate room.

If lubricant testing is to be performed, the size of the general laboratory will need to be increased. The amount of increase is dependent upon the tests that will be performed. However, if work is limited to viscosity measurement an additional 37 m² (400 ft²) should be sufficient.

4.3.2. The laboratory should monitor, control and record environmental conditions as required by the relevant specifications, methods and procedures or where they influence the quality of the results. Due attention should be given, for example, to biological sterility, dust, electromagnetic disturbances, radiation, humidity, electrical supply, temperature, and sound and vibration levels, as appropriate to the technical activities concerned. Tests should be stopped anytime the environmental conditions jeopardize the results of the tests.

4.3.3. There should be effective separation between neighboring areas in which there are incompatible activities (e.g., office and lunch rooms separated from laboratory.) Measures should be taken to prevent cross-contamination.

4.3.4. Access to and use of areas affecting the quality of the tests should be controlled. The laboratory should determine the extent of control based on its particular circumstances.

4.3.5. Measures should be taken to ensure good housekeeping in the laboratory. Special procedures should be prepared where necessary.

4.3.6. The laboratory should have special (isolated) foundations for the installation of ASTM/Cooperative Fuel Research Committee (CFR) engines. The laboratory's design must ensure that all fuels testing can be performed in accordance with ASTM requirements. This consideration is especially important for the CFR engines¹⁵.

Implementation Note: *Automatic hydrocarbon monitors to warn of critical accumulation of explosive vapors are excellent safety devices. Hoods should be tested annually.*

Hazardous Waste – *FQLs generate small quantities of hazardous waste. Used oil from CFR engines and waste chemicals from various analyses must be stored and disposed in an appropriate manner.*

The local Hazardous Waste Manager must have a hazardous waste program document providing guidance on proper waste disposal through detailed step-by step instructions.

Hazardous waste results from the conduct of research and analytical testing, unused or out of date hazardous chemicals and their containers, samples, empty containers that previously held a

¹⁵ Volume 05.04 of the Annual Book of ASTM Standards contains valuable information regarding the design of a knock-testing laboratory.

toxic chemical, batteries, waste oils, TLC plates and packed columns, asbestos, mercury, items contaminated with hazardous chemicals, and material generated during spill clean-up operations. Prior to disposal, one must stop to determine if any waste generated is hazardous.

Definitions, critical in interpreting the waste regulations and understanding how to manage the waste on-site, can be found in 40 CFR 261 Subparts C and D. It is imperative everyone understands what chemicals are listed on EPA's "P" list; these wastes are segregated from other waste streams. Chemicals listed on EPA's "P" list of acute hazardous wastes are more highly regulated and call for additional management and expense.

Contact the Hazardous Waste Manager and consult the local Hazardous Waste Management Plan for any question regarding waste prior to disposal (e.g., determining if waste is hazardous). Annual hazardous waste training should be given for all waste generators. The majority of regulations for storage, disposal, and documentation of hazardous materials may be found in EPA's SARA Title III, 40 CFR 1500.

Additional regulations and permits may be required by state, county or municipal agencies. Familiarity with the regulations will be advantageous when considering the design of the laboratory. Specific expenses related to hazardous waste disposal will often be determined by local regulations and the availability of hazardous waste handlers.

4.4. Test Methods and Method Validation

4.4.1. General

The laboratory should use appropriate methods and procedures for all tests within its scope. These include sampling, handling, transport, storage and preparation of items to be tested, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of test data.

The laboratory should have instructions on the use and operation of all relevant equipment, and on the handling and preparation of items for testing, where the absence of such instructions could jeopardize the results of tests. All instructions, standards, manuals, and reference data relevant to the work of the laboratory should be kept up to date and should be made readily available to personnel (see 3.3. "Document Control") Deviation from test methods should occur only if the deviation has been documented, technically justified, authorized, and accepted by the customer.

Implementation Note: *International or national standards, or local specifications that contain sufficient and concise information on how to perform the tests, do not need to be supplemented or rewritten as internal procedures if these standards are written in a way that they can be used as published by the operating staff in a laboratory. It may be necessary to provide additional documentation for optional steps in the method or additional details.*

4.4.2. Selection of Methods

The laboratory should use test methods, including methods for sampling that meet the needs of the customer, which are appropriate for the tests it undertakes. Methods published in

international, regional, or national standards should preferably be used. The laboratory should ensure that it uses the latest valid edition of a standard unless it is not appropriate or possible to do so. When necessary, the standard should be supplemented with additional details to ensure consistent application. When the customer does not specify the method to be used, the laboratory should select appropriate methods that have been published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment. Laboratory-developed methods or methods adopted by the laboratory may also be used if they are appropriate for the intended use and if they are validated. The customer should be informed as to the method chosen. The laboratory should confirm that it can properly operate standard methods before introducing the tests. If the standard method changes, the confirmation should be repeated. The laboratory should inform the customer when the method proposed by the customer is considered to be inappropriate or out of date.

4.4.3. Laboratory-developed Methods

The introduction of test methods developed by the laboratory for its own use should be a planned activity and should be assigned to qualified personnel equipped with adequate resources. Plans should be updated as development proceeds and effective communication between all personnel involved should be ensured.

4.4.4. Non-standard Methods

When it is necessary to use methods not covered by standard methods, these should be subject to agreement with the customer and should include a clear specification of the customer's requirements and the purpose of the test. The method developed should have been validated appropriately before use.

Implementation Note: *For new test methods, procedures should be developed and documented before tests are performed, and should contain at least the following information:*

- 1) appropriate identification;*
- 2) scope;*
- 3) description of the type of item to be tested;*
- 4) parameters or quantities and ranges to be determined;*
- 5) apparatus and equipment, including technical performance requirements;*
- 6) reference standards and reference materials required;*
- 7) environmental conditions required and any stabilization period needed;*
- 8) description of the procedure, including:*

- *affixing of identification marks, handling, transporting, storing and preparation of items,*
 - *checks to be made before the work is started,*
 - *checks that the equipment is working properly and, where required, calibration and adjustment of the equipment before each use,*
 - *the method of recording the observations and results;*
 - *any safety measures to be observed;*
- 9) *criteria and/or requirements for approval/rejection;*
- 10) *data to be recorded and method of analysis and presentation;*
- 11) *the uncertainty or the procedure for estimating uncertainty.*

4.4.5. Validation of Methods

4.4.5.1. Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

4.4.5.2. The laboratory should validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation should be as extensive as necessary to meet the needs of the given application or field of application. The laboratory should record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

Implementation Note: *Validation may include procedures for sampling, handling and transportation. The techniques used for the determination of the performance of a method should be one of, or a combination of, the following:*

- *calibration using reference standards or reference materials;*
- *comparison of results achieved with other methods;*
- *interlaboratory comparisons;*
- *systematic assessment of the factors influencing the result;*
- *assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.*

For changes in the validated non-standard methods, their influence should be documented and, if appropriate, another validation should be carried out.

4.4.5.3. The range and accuracy of the values obtainable from validated methods (e.g., the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, should be relevant to the customer's needs.

Implementation Note: *Validation includes specification of the requirements, determination of the characteristics of the methods, a check that the requirements can be fulfilled by using the method and a statement on the validity. As method development proceeds, regular review should be carried out to verify that the needs of the customer are still being fulfilled. Any change in requirements requiring modifications to the development plan should be approved and authorized. Validation is always a balance between costs, risks, and technical possibilities. There are many cases in which the range and uncertainty of the values (e.g., accuracy, detection limit, selectivity, linearity, repeatability, reproducibility, robustness, and cross-sensitivity) can only be given in a simplified way due to lack of information.*

4.4.5.4. If screening techniques or devices are considered either for the field or for the laboratory, they should be subjected to the same ASTM interlaboratory cross checks that the laboratories undergo for a test period of at least [e.g., one year]. Potential device marketers should be requested to provide data for a much larger data set.

Implementation Note: *New instrumental methods are often introduced to facilitate testing engine fuels. Chemical methods have been devised to replace or screen physical methods that may enhance efficiency by reducing staff or analysis time necessary to perform physical methods. These methods are normally devised for a controlled environment, such as a processing plant, where the chemical components of the samples are generally known and a correlation between the chemical components and physical parameters may be drawn with confidence.*

A new FQL is cautioned to refrain from investing in this instrumentation and the laboratory expertise necessary to perform the test procedure until the test procedure has been approved through ASTM. Screening methods have been employed by state laboratories and governmental agencies to maintain or increase sample coverage. Screening procedures may be a variation of accepted ASTM procedures; certain sections of a procedure may be excluded or modified, such as chilling a sample to the appropriate temperature or accurately timing a distillation analysis.

When a screened sample exceeds a predetermined parameter, the sample must be analyzed using the proper ASTM procedure. Strategies, such as sampling from the batch of samples or only performing certain tests on all samples, should be employed in the laboratory as means for effective regulation.

4.4.6. Estimation of Uncertainty of Measurement

4.4.6.1. A laboratory performing its own calibrations should have and should apply a procedure to estimate the uncertainty of measurement for all calibrations.

4.4.6.2. When appropriate, testing laboratories should have and should apply procedures for estimating uncertainty of measurement. In certain cases, the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases, the laboratory should at least attempt to identify all the components of uncertainty and make a reasonable estimation, and should ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation should be based on knowledge of the performance of the method and on the measurement scope and should make use of, for example, previous experience and validation data.

Implementation Note: *The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as the requirements of the test method; the requirements of the customer; and the existence of narrow limits on which decisions on conformity to a specification are based. In those cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions (see 4.10. “Reporting the Results”).*

4.4.6.3. When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation should be taken into account using appropriate methods of analysis.

Implementation Note: *Sources contributing to the uncertainty include, but are not necessarily limited to, the reference standards and reference materials used, methods and equipment used, environmental conditions, properties and condition of the item being tested or calibrated, and the operator. The predicted long-term behavior of the tested and/or calibrated item is not normally taken into account when estimating the measurement uncertainty. For further information, see ISO 5725-1 “Accuracy (Trueness and Precision) of Measurement Methods and Results - Part 1: General Principles and Definitions” and the “Guide to the Expression of Uncertainty in Measurement” at <http://www.bipm.org/en/publications/guides/gum.html>.*

4.4.7. Control of Data

4.4.7.1. Calculations and data transfers should be subject to appropriate checks in a systematic manner.

4.4.7.2. When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage, or retrieval of test data, the laboratory should ensure that:

- 1) computer software developed by the user is documented in sufficient detail and is

suitably validated as being adequate for use;

- 2) procedures are established and implemented for protecting the data; such procedures should include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing; and
- 3) computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of test data.

Implementation Note: *Commercial off-the-shelf software (e.g., word processing, database and statistical programs) in general use within their designed application range may be considered to be sufficiently validated. However, laboratory software configuration/modifications should be validated as in 4.4.7.2.(1) under 4.4.7. “Control of Data.”*

Implementation Note: *Information Management System – No recommendations are made for an information management system. However, it should be noted that an information management system is an effective tool to manage data and statistical information when devising sampling strategies and when measuring the general effectiveness of a program. Minimum requirements for an information management system include a database server and database adequate to handle sample biographical and analyses information. A means to network technicians and staff to the information is necessary to facilitate transfer of information. Considerations for software security and equipment security (limited access to the database server) should be given to ensure the integrity of the data. Many departments have established information management centers that are consulted for this information. Generally, these departments have a particular protocol for developing an information management system.*

4.5. Equipment

4.5.1. The laboratory should be furnished with all items of sampling, measurement and test equipment required for the correct performance of the tests (including sampling, preparation of test samples, processing and analysis of test data). In those cases where the laboratory needs to use equipment outside its permanent control, it should ensure that the requirements of this handbook are met.

Implementation Note: *Laboratory Equipment and Supplies - Scientific instrumentation is typically more expensive than initially anticipated even when one has experience purchasing equipment. ASTM has approved methods utilizing automated instruments that may prove to be a better long-term investment when the costs of operating personnel are included. The cost of equipment and supplies change, therefore, providing estimates in this document would be of little value. Because of the relatively small demand for laboratory equipment, it is common to have only one source. However, when possible, obtaining competitive bids can reduce costs. Purchasing used equipment from other labs or vendors can provide a source of equipment at reduced costs.*

4.5.2. Equipment and its software used for testing and sampling should be capable of achieving the accuracy required and should comply with specifications relevant to the tests

concerned. Calibration programs should be established for key quantities or values of the instruments where these properties have a significant effect on the results. Before being placed into service, equipment (including that used for sampling) should be calibrated (see 4.6. “Measurement Traceability.”) or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications.

Implementation Note: *Calibrating chemical measurement equipment usually entails introducing samples of known quantities into the device to make sure it is giving the results desired. The known quantities should be in the same matrix as the unknown samples. The analyst can sometimes make up a known quantity sample for this purpose; at which point, it is extremely important to know the purity of the constituent materials used to generate the known quantity sample. Additionally, retained samples from participation in the ASTM International Interlaboratory Crosscheck Program or National Exchange Group are good sources for known quantity samples. Running the known quantity sample over time (in some instances, every time an unknown sample is run) will alert the FQL when a device is malfunctioning or part of the system is wearing out, etc. This is equivalent to running a control chart for the equipment to indicate when action on the device/system is needed. If multiple analysts use the same equipment, control charts for each operator should be maintained.*

4.5.3. Equipment should be operated by authorized personnel. Only up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) should be readily available for use by the appropriate laboratory personnel.

4.5.4. Each item of equipment and its software used for testing and significant to the result should, when practicable, be uniquely identified.

4.5.5. Records should be maintained of each item of equipment and its software, which is significant to the tests performed. The records should include at least the following:

- 1) the identity of the item of equipment and its software;
- 2) the manufacturer's name, type identification, and serial number or other unique identification;
- 3) checks that equipment complies with the specification (see Section 4.5.2. under 4.5. “Equipment.”);
- 4) the current location, where appropriate;
- 5) the manufacturer's instructions, if available, or reference to their location;
- 6) dates, results, and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;
- 7) the maintenance plan, where appropriate, and maintenance carried out to date; and

8) any damage, malfunction, modification or repair to the equipment.

4.5.6. The laboratory should have procedures for safe handling, transport, storage, use and planned maintenance of measuring equipment to ensure proper functioning and to prevent contamination or deterioration.

4.5.7. Equipment that has been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits, should be taken out of service. Equipment should be isolated to prevent its use or clearly labeled or marked as being out of service until it has been repaired and shown by calibration or test to perform correctly. The laboratory should examine the effect of the defect or departure from specified limits on previous tests and should institute the "control of non-conforming work" procedure (see 3.12. "Improvement.").

4.5.8. Whenever practicable, all equipment under the control of the laboratory and requiring calibration should be labeled, coded, or otherwise identified to indicate the status of calibration, including the date when last calibrated and the date or expiration criteria when recalibration is due.

4.5.9. When, for whatever reason, equipment goes outside the direct control of the laboratory, the laboratory should ensure that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

4.5.10. When intermediate checks are needed to maintain confidence in the calibration status of the equipment, these checks should be carried out according to a defined procedure.

4.5.11. Where calibrations give rise to a set of correction factors, the laboratory should have procedures to ensure that copies (e.g., in computer software) are correctly updated.

4.5.12. Test and calibration equipment, including both hardware and software, should be safeguarded from adjustments which would invalidate the test results.

4.5.13. The laboratory should be furnished with all items of administrative, engineering, and personal protection as required for the safe performance of the tests (including sampling, preparation of test samples, and processing and analysis of test data). This should include necessary safety equipment, such as fire blankets, fire extinguisher, eyewash stations, etc.

4.5.14. In those cases where the laboratory needs to use equipment outside its permanent control, it should ensure that the recommendations of this handbook are met.

Implementation Note: *Examples of Test Equipment – This list is for illustration purposes only. It is recommended that operating laboratories be contacted for recommendations for the latest equipment and technology used or needed to perform the various tests that the prospective lab intends to carryout. Current cost information is not provided because prices change frequently due to quantities requested, inflation, and other factors.*

Octane Testing

1 CFR Research Method Engine

1 CFR Engine Method Engine

1 Fuel Blending System

Humidity controller for CFR engines

Complete set of tools

Lift for removing cylinders

Supplies, spare parts, etc.

Distillation Testing

2 Explosion proof refrigerators 504 L (18 cu ft)

1 Mercury barometer

2 Mechanically refrigerated 4-unit distillation apparatus

1 Temperature-controlled bath

Automated distillation units may be substituted for the manual distillation units. The increased cost can be justified by a reduction in staff and an increase in precision.

Vapor Pressure (RVP) Testing

1 Grabner

1 McCleod gauge

1 Vacuum pump (2-Stage)

Sulfur Testing

1 X-Ray fluorescence analyzer

Oxygenate Testing

1 Gas chromatograph

Lead Testing

1 Atomic absorption instrument

Diesel-Kerosene Testing

2 Tag-closed cup flash testers

2 Pensky-Martens flash testers

10 Hydrometers for API gravity

1 Saybolt chronometer for color test

1 Cloud/Pour Point apparatus

Miscellaneous Items

100 Sample cases for sample transportation

1200 Sample containers

1 Oven for drying sample containers (glass)

1 7.6 liter/hour water still

1 Analytical balance

2 Bottle washers

PPE should be provided to all laboratory personnel. Eye protection, lab coats/aprons, and gloves will provide minimum protection. Determination of which equipment is necessary for handling particular chemicals can be found in the MSDS accompanying the chemicals. General laboratory safety equipment should be considered during the design or selection of a building. In addition to a good ventilation system, fume hoods should be provided where practical to isolate fumes from the laboratory. If the use of a fume hood is not practical and an employee is exposed to petroleum or other chemical fumes, organic respirators should be provided to minimize exposure.

Due to the explosive nature of gasoline, even safety equipment needs to be evaluated for safety; for example, explosion-proof engines should be installed to evacuate fumes from a hood. Eyewash stations, fire extinguishers, emergency showers, and fire blankets should all be placed strategically for maximum protection. In the event of a spill or fire, several safety items will prove useful. Activated charcoal, sold under a variety of names, is effective for absorbing small petroleum spills with the added benefit of quickly reducing vaporization. Other companies offer pads to quickly absorb spills. Similar products are offered to neutralize and adsorb acids and bases.

Safety signs should be posted at the entrance of each laboratory room listing possible hazards and restricted activities (e.g., No Smoking, Flammables, Eye Protection Required, etc.). These signs assist visitors and emergency response personnel to identify hazards quickly.

4.6. Measurement Traceability

4.6.1. General

All equipment used for tests, including equipment for subsidiary measurements (e.g., for environmental conditions) having a significant effect on the accuracy or validity of the result of a test should be calibrated before being put into service. The laboratory should have an established program and procedure for the calibration of its equipment.

Implementation Note: *Such a program should include a system for selecting, using, calibrating, checking, controlling and maintaining measurement standards and traceability, reference materials used as measurement standards, and measuring and test equipment used to perform tests.*

4.6.2. Specific Requirements

4.6.2.1. Tests

4.6.2.1.1. The program for calibration of equipment should be designed and operated so as to ensure that measurements made by the laboratory are traceable to the International System of Units (SI). When using external testing services, traceability of measurement should be assured by the use of calibration services from laboratories that can demonstrate competence,

measurement capability and traceability. The reports issued by these laboratories should contain the measurement results and/or a statement of compliance with an identified test methods (see also 4.10.3.2. under 4.10. “Reporting Results.”) Laboratories fulfilling the requirements of this handbook are considered to be competent.

Implementation Note: *Although an FQL is not a calibration laboratory, certain equipment within the laboratory should be calibrated by a laboratory meeting ISO/IEC 17025 requirements in these sections. The equipment requiring outside calibration is determined by whether a significant part of the measurement uncertainty is attributable to the result provided by this equipment. (See NOTE under 4.6.2.2.1. under 4.6.2.2. “Testing.”) For example, the laboratory balances and volumetric pipettes, etc. all probably significantly contribute to the final measurement uncertainty. Glass pipettes hold their calibration values and do not need to be recalibrated. Laboratory balances usually need recalibration.*

4.6.2.1.2. Some tests cannot currently be made strictly in SI units. In these cases, the test method should provide confidence in measurements by establishing traceability to appropriate measurement standards, such as:

- the use of certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material; and
- the use of specified methods and/or consensus standards that are clearly described and agreed by all parties concerned.

Participation in a suitable program of interlaboratory comparisons is required whenever possible.

Implementation Note: *Many of the tests by an FQL fall under this category.*

4.6.2.2. Testing

4.6.2.2.1. The requirements given in 4.6.2.1. “Tests” apply for measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from the calibration contributes little to the total uncertainty of the test result. When this situation arises, the laboratory should ensure that the equipment used can provide the uncertainty of measurement needed.

Implementation Note: *The extent to which the requirements should be followed depends on the relative contribution of the calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements should be strictly followed.*

4.6.2.2.2. Where traceability of measurements to SI units is not possible and/or not relevant, the same requirements for traceability to, for example, certified reference materials, agreed methods and/or consensus standards, are required as for calibration laboratories (see 4.6.2.1.2. under Section 4.6.2. “Specific Requirements”).

4.6.3. Reference Standards and Reference Materials

4.6.3.1. Reference Standards

The laboratory should have a program and procedure for the calibration of its reference standards. Reference standards should be calibrated by a body that can provide traceability as described in 4.6.2.1. “Tests.” Such reference standards of measurement held by the laboratory should be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated. Reference standards should be calibrated before and after any adjustment.

4.6.3.2. Reference Materials

Reference materials should, where possible, be traceable to the International System of Units (SI), or to certified reference materials. Internal reference materials should be checked as far as is technically and economically practicable.

Implementation Note: *If carefully managed, some of the sample sent in the ASTM International Interlaboratory Crosscheck Program or National Exchange Group can be retained for use by the FQL as internal reference materials for later use.*

4.6.3.3. Intermediate Checks

Checks needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials should be carried out according to defined procedures and schedules.

4.6.3.4. Transport and Storage

The laboratory should have procedures for safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity.

Implementation Note: *Additional procedures may be necessary when reference standards and reference materials are used outside the permanent laboratory for tests, calibrations or sampling.*

4.7. Sampling

4.7.1. The laboratory should have a documented sampling plan and procedures for sampling when it carries out sampling of substances, materials or products for subsequent testing. The sampling plan as well as the sampling procedure should be available at the location where sampling is undertaken. Sampling plans should, whenever reasonable, be based on appropriate statistical methods. The sampling process should address the factors to be controlled to ensure the validity of the test results.

Sampling is a defined procedure whereby a part of a substance, material or product is taken to provide for testing of a representative sample of the whole. Sampling may also be required by the appropriate specification for which the substance, material, or product is to be tested. In certain cases (e.g., forensic analysis), the sample may not be representative but is determined by availability. Sampling procedures should describe the selection, sampling plan, withdrawal, and preparation of a sample or samples from a substance, material, or product to yield the required information.

Implementation Note: *In general, the FQL receives samples from the field. It is the responsibility of the agent performing the field collection to use techniques that assure that the sample is representative of the whole, for example, of the whole storage tank, or of the blend that the pump is dispensing after washing the dispenser of previously pumped fuel. Thus, it is important for the sampling plan to be documented.*

4.7.2. Where the customer requires deviations, additions, or exclusions from the documented sampling procedure, these should be recorded in detail with the appropriate sampling data and should be included in all documents containing test results, and should be communicated to the appropriate personnel.

4.7.3. The laboratory should have procedures for recording relevant data and operations relating to sampling that forms part of the testing that is undertaken. These records should include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and diagrams, or other equivalent means to identify the sampling location as necessary and, if appropriate, the statistics the sampling procedures are based upon.

Implementation Note: *Sampling, including obtaining the samples from the field (see below) and maintaining the integrity and identification of samples throughout their processing are critically important. (See Implementation Note after 4.8.4. under 4.8 “Handling of Samples” below.)*

Selecting the Sampling Location: State regulations may dictate where the majority of samples will be collected. Some states require registration of the petroleum product with a known chemical profile before sale in the state. Thus, samples might be collected at a bulk storage facility or similar location. In other examples, the state is checking what is being offered for sale to the driving public; in these instances, sampling will occur at retail fueling locations, such as gas stations.

4.8. Handling of Samples

4.8.1. The laboratory should have procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of test and/or samples, including all provisions necessary to protect the integrity of the sample, and to protect the interests of the laboratory and the customer.

4.8.2. The laboratory should have a system for identifying test items. The identification should be retained throughout the life of the item in the laboratory. The system should be

designed and operated so as to ensure that items cannot be confused physically or when referred to in records or other documents. The system should, if appropriate, accommodate a subdivision of groups of items and the transfer of items within and from the laboratory.

4.8.3. Upon receipt of the test sample, abnormalities or departures from normal or specified conditions, as described in the test method, should be recorded. When there is doubt as to the suitability of an item for test, or when an item does not conform to the description provided, or the test required is not specified in sufficient detail, the laboratory should consult the customer for further instructions before proceeding and should document the discussion.

4.8.4. The laboratory should have procedures and appropriate facilities for avoiding contamination, deterioration, loss or damage of the sample during storage, handling and preparation. Handling instructions provided with the item should be followed. When items have to be stored or conditioned under specified environmental conditions, these conditions should be maintained, monitored, and recorded. Where a sample or a portion of an item is to be held secure, the laboratory should have arrangements for storage and security that protect the condition and integrity of the secured items or portions concerned. A sampling procedure and information on storage and transport of samples, including information on sampling factors influencing the test result, should be provided to those responsible for taking and transporting the samples. Keeping a test item secure can be for reasons of record, safety or value, or to enable complementary tests to be performed later.

Implementation Note: *The FQL must maintain the integrity and identification of samples and their results. This includes the:*

- *collection and unique identification of samples in the field,*
- *safe transport to the laboratory,*
- *secure storage of the samples in the laboratory,*
- *possible splitting of samples for multiple analyses,*
- *analysis of the samples,*
- *reporting test results, and*
- *Subsequent secure storage of samples if retention is required or desired.*

See chain-of-custody information at <http://www.epa.gov/eogaptil/coc/>.

4.9. Assuring the Quality of Test Results

4.9.1. The laboratory should have quality control procedures for monitoring the validity of tests undertaken. The resulting data should be recorded in such a way that trends are detectable and, where practicable, statistical techniques should be applied to the reviewing of results. This monitoring should be planned and reviewed and may include, but need not be limited to, the following:

- 1) regular use of certified reference materials and/or internal quality control using secondary reference materials;
- 2) participation in interlaboratory comparison or proficiency-testing programs;

- 3) replicate tests using the same or different methods;
- 4) retesting of retained items; and
- 5) correlation of results for different characteristics of an item.

Implementation Note: *The selected methods should be appropriate for the type and volume of the work undertaken.*

Implementation Note: *All FQLs should participate in interlaboratory programs to enable them to ensure integrity and accuracy of their test results. One such program is ASTM's Motor Gasoline Proficiency Testing Program that provides laboratories with a statistical quality assurance (SQA) tool, enabling them to compare, improve, and maintain, a high level of performance in the use of ASTM methods with other laboratories worldwide. Conducted three times annually, this program provides a different commercial sample, electronic report forms, and test instructions for each test cycle. A laboratory performs the tests that it normally conducts using the specified ASTM methods cited in the program. Samples, test instructions and data report forms are distributed electronically to each participant on the date samples are distributed. FQLs have approximately 8 weeks to submit test data with the final statistical summary reports being electronically distributed in approximately 25 business days. FQL can then use the statistical quality assurance information to monitor the strengths and weaknesses of their laboratory's performance in conducting tests and to satisfy proficiency testing elements of laboratory accreditation or to demonstrate the competence of the laboratory's testing program in enforcement actions.*

4.9.2. Quality control data should be analyzed and, where they are found to be outside pre-defined criteria, planned action should be taken to correct the problem and to prevent incorrect results from being reported.

4.9.3. FQLs should participate in the ASTM International Interlaboratory Crosscheck Program and/or the ASTM International National Exchange Group, as appropriate, as a means of assuring the quality of its test results.

Implementation Note: *When proficiency testing or interlaboratory comparisons are conducted, the normal operations and operators of the tests should participate; the proficiency testing or interlaboratory comparisons should not be subcontracted if the normal operation or test is not subcontracted.*

4.10. Reporting the Results

4.10.1. General

The results of each test, or series of tests carried out by the laboratory should be reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test methods. The results should be reported, usually in a test report, and should include all the information requested by the customer and necessary for the interpretation

of the test results and all information required by the method used. This information is normally that required by 4.10.2. “Test Reports,” and 4.10.2.1., or 4.10.3. “Opinions and Interpretations.” In the case of tests performed for internal customers, or in the case of a written agreement with the customer, the results may be reported in a simplified way. Any information listed in 4.10.2. “Test Reports” to 4.10.3. “Opinions and Interpretations,” which is not reported to the customer, should be readily available in the laboratory which carried out the tests. The test reports may be issued as hard copy or by electronic data transfer provided that the requirements of this handbook are met.

4.10.2. Test Reports

Each test report should include at least the following information, unless the laboratory has valid reasons for not doing so:

- 1) a title (e.g., Test Report);
- 2) the name and address of the laboratory, and the location where the tests were carried out, if different from the address of the laboratory;
- 3) unique identification of the test report (such as the serial number), and on each page an identification in order to ensure that the page is recognized as a part of the test report, and a clear identification of the end of the test report;
- 4) the name and address of the customer;
- 5) identification of the method used;
- 6) a description of, the condition of, and unambiguous identification of the sample(s) tested;
- 7) the date of receipt of the test sample(s) where this is critical to the validity and application of the results, and the date(s) of performance of the test;
- 8) if applicable, reference to the sampling plan and procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results;
- 9) the test results with, where appropriate, the units of measurement;
- 10) the name(s), function(s) and signature(s) or equivalent identification of person(s) authorizing the test report; and
- 11) where relevant, a statement to the effect that the results relate only to the items tested.

Test reports should also include the page number and total number of pages. It is recommended that laboratories include a statement specifying that the test report should not be reproduced except in full, without written approval of the laboratory.

4.10.2.1. In addition to the requirements listed in 4.10.2. “Test Reports,” test reports should, where necessary for the interpretation of the test results, include the following:

- 1) deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions;
- 2) where relevant, a statement of compliance/non-compliance with requirements and/or specifications;
- 3) where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer’s instruction so requires, or when the uncertainty affects compliance to a specification limit;
- 4) where appropriate and needed, opinions and interpretations (see 4.10.5. “Test Results Obtained from Subcontractors.”); and
- 5) additional information which may be required by specific methods, customers or groups of customers.

4.10.2.2. In addition to the requirements listed in 4.10.2. “Test Reports” and 4.10.2.1. reports containing the results of sampling should include the following, where necessary for the interpretation of test results:

- 1) the date of sampling;
- 2) unambiguous identification of the substance, material or product sampled (including the name of the manufacturer, the model or type of designation, lot code or serial numbers as appropriate);
- 3) the location of sampling (e.g., device or storage location where collected);
- 4) details of any environmental conditions during sampling that may affect the interpretation of the test results; and
- 5) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

4.10.3. Opinions and Interpretations

When opinions and interpretations are included, the laboratory should document the basis upon which the opinions and interpretations have been made. Opinions and interpretations should be clearly marked as such in a test report.

Implementation Note: *Opinions and interpretations should not be confused with inspections*

and product certifications as intended in ISO/IEC 17020 “Conformity Assessment -- Requirements for the Operation of Various Types of Bodies Performing Inspection” and ISO/IEC Guide 65 “General Requirements for Bodies Operating Product Certification Systems.” Opinions and interpretations included in a test report may comprise, but not be limited to, the following:

- *an opinion on the statement of compliance/noncompliance of the results with requirements;*
- *fulfillment of contractual requirements;*
- *recommendations on how to use the results; and*
- *guidance to be used for improvements.*

In many cases, it might be appropriate to communicate the opinions and interpretations by direct dialogue with the customer. Such dialogue should be documented on the report.

4.10.5. Test Results Obtained from Subcontractors

When the test report contains results of tests performed by subcontractors, these results should be clearly identified. The subcontractor should report the results in writing or electronically. When a test has been subcontracted, the laboratory performing the work should issue the test report to the contracting laboratory.

4.10.6. Electronic Transmission of Results

In the case of transmission of test results by telephone, telex, facsimile, or other electronic or electromagnetic means, the requirements of this handbook should be met (see also 4.4.7. “Control of Data.”).

4.10.7. Format of Reports and Certificates

The format should be designed to accommodate each type of test carried out and to minimize the possibility of misunderstanding or misuse. Attention should be given to the layout of the test report, especially with regard to the presentation of the test data and ease of assimilation by the reader.

4.10.8. Amendments to Test Reports

Material amendments to a test report after issue should be made only in the form of a further document, or data transfer, which includes the statement:

"Supplement to Test Report, serial number ... (or as otherwise identified)," or an equivalent form of wording. Such amendments should meet all the requirements of this handbook. When it is necessary to issue a complete new test report, this should be uniquely identified and should

contain a reference to the original that it replaces.

5.0. Glossary

The following section contains the definitions of a number of terms in the sense in which they are used throughout this handbook. Where applicable, definitions are taken from: 1) NIST Handbook 150 “NVLAP Procedures and General Requirements,” 2006 wherever possible; 2) “The International Vocabulary of Metrology – Basic and General Concepts and Associated Terms” (OIML V 2-200 VIM: 2010); or 3) NIST Handbook 143, “State Weights and Measures Laboratories – Program Handbook,” 2007 edition.

5.1. Accreditation – a formal Recognition that a laboratory is competent to carry out specific tests.

5.2. Accreditation Process – the process of demonstrating whether a laboratory is capable of fulfilling specified accreditation requirements.

5.3. Administrative Controls – procedures or rules that are to be followed to reduce the risks associated with a hazard for which engineering controls are not practical or possible. Although administrative controls can (and should) always be used to control employee exposure, they are prone to human error and cannot be relied upon to reduce exposure all the time. Examples of administrative controls include policies restricting access and signage.

5.4. Best Practice – a technique or methodology that has reliably led to a desired result.

5.5. Buddy System – A system of organizing employees into work groups in such a manner that each employee of the work group is designated to be observed by at least one other employee in the work group. The purpose of the buddy system is to provide rapid assistance to employees in the event of an emergency. It is important that the employee designated as observer not be subject to hazards in the work area.

5.6. Certified Reference Material – Reference material, characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

- **NOTE 1:** The concept of value includes qualitative attributes such as identity or sequence. Uncertainties for such attributes may be expressed as probabilities.
- **NOTE 2:** Metrologically valid procedures for the production and certification of reference materials are given in, among others, ISO Guide 34 “General Requirements for the Competence of Reference Material Producers” and Guide 35. “Reference Materials – General and Statistical Principles for Certification.”
- **NOTE 3:** ISO Guide 31 “Reference Materials -- Contents of Certificates and Labels.”

5.7. Ceiling (C) – the maximum concentration (exposure) of a toxic material never to be

exceeded. See also Threshold limit values (TLV) and Short-term exposure limit (STEL).

5.8. Chain of Custody (CoC) – refers to the chronological documentation or paper trail, showing the seizure, custody, control, transfer, analysis, and disposition of evidence, physical or electronic. The chain of custody requires that from the moment the evidence is collected, every transfer of evidence from person to person be documented and that it be provable that nobody else could have accessed that evidence. It is best to keep the number of transfers as low as possible. Chain of custody is used in most chemical sampling situations to maintain the integrity of the sample by providing documentation of the control, transfer, and analysis of samples. Chain of custody is especially important where sampling can identify the existence of contamination and can be used to identify the responsible party.

5.9. Chemical Hygiene Plan – required by the “Laboratory Standard” 29 CFR 1910.1450 under the Occupational Safety and Health Administration (OSHA). The document must cover general lab safety, chemical hazards, PPE, safety equipment, certification programs, safety responsibilities, exposure procedures, and other occupational health topics.

5.10. Corrective Action – an action taken to eliminate the causes of an existing nonconformity or other undesirable situation in order to prevent recurrence.

5.11. Customer – any person or organization that engages the services of a laboratory.

5.12. Engineering Controls – controls designed to eliminate or reduce exposure to a hazard through the use or substitution of engineered machinery or equipment. Examples of engineering controls include ventilation systems (fume hoods), sound-dampening materials to reduce noise levels, and safety interlocks.

5.13. Environmental health – comprises aspects of human health, including quality of life, that are determined by interactions with physical, chemical, biological, and social factors in the environment. It also refers to the theory and practice of assessing, correcting, controlling and preventing those factors in the environment that may adversely affect the health of present and future generations (from the Pew Environmental Health Commission).

5.14. Evacuation Plan – This document outlines the steps to be taken when evacuating the laboratory during an emergency.

5.15. Hazard – an object or situation that is potentially dangerous (i.e., an unsecured electrical cord might be a tripping hazard, or a finger-tight connection that is not in a properly vented area might start to leak into an enclosed space, leading to a flammable accumulation of hydrogen) OSHA defines a hazard as any facility, location, equipment, tool, job, task, or action that presents a potential for harm. In practical terms, a hazard often is associated with a condition or activity that, if left uncontrolled, can result in an injury or illness. Compare with Incident.

5.16. Hazardous Waste – any solid, liquid, semi-solid, or contained gaseous waste, which, because of its quantity, concentration, or physical, chemical, or infectious characteristics may:

- cause or significantly contribute to an increase in mortality or an increase in serious irreversible, or incapacitating reversible, illness, and
- pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed of, or otherwise managed.

5.17. Hazardous Waste Management Plan – This document helps provide compliance with various EPA regulations, providing detailed instructions for the safe disposal of laboratory reagents and hazardous substances.

5.18. Incident – an event that results in:

- a lost-time accident and/or injury to personnel,
- damage to project equipment, facilities or property,
- impact to the public or environment,
- an emergency response or should have resulted in an emergency response.

An incident is any unplanned event resulting in, or having the potential for injury, ill-health, damage or other loss.¹⁶

5.19. Inherent Safety Features – system design such that in both normal and emergency situations at least two failures must occur before injury, loss of life, or major equipment damage would result from the use of hazardous materials

5.20. Interlaboratory Comparisons – organization, performance and evaluation of tests on the same or similar items or materials by two or more laboratories in accordance with predetermined conditions.

5.21. Internal Assessment – the process of self-appraisal of a testing laboratory using specified general and technical criteria and checklists to evaluate compliance to Recognition (or accreditation) requirements; may be used as a management system review as well.

5.22. Laboratory – an organization that performs tests. When a laboratory is part of an organization that carries out activities additional to testing, the term "laboratory" refers only to those parts of that organization that are involved in the testing process. A laboratory's activities may be carried out at a permanent location, temporary, or remote location. A laboratory may be further defined as being a physical entity- that is, a testing facility that is separate and apart

¹⁶ <http://education.qld.gov.au/strategic/eppr/health/hlspr005/definitions.html>

OSHA does not define incident, but see a hazard as a base for defining any incident or near miss <http://e-osh.blogspot.com/2008/10/definition-of-hazard-risk-incident.html>.

physically from any other laboratory whether or not sharing common ownership, management, or management systems with any other laboratory(s).

5.23. Management System – system to establish policy and objectives and to achieve those objectives.

- **NOTE:** a management system of an organization may include different management systems, such as a quality management system, a financial management system, or an environmental management system. (ISO 9000:2000, 2.2.2.)

5.24. Management and Administrative Controls – procedures or rules that are to be followed to reduce the risks associated with a hazard for which engineering controls are not practical or possible. Although administrative controls can (and should) always be used to control employee exposure, they are prone to human error and cannot be relied upon to reduce exposure all the time. Examples of administrative controls include policies restricting access and signage.

5.25. Material Safety Data Sheets (MSDS) – a form with data regarding the properties of a particular substance. An important component of workplace safety and product stewardship, it is intended to provide workers and emergency personnel with procedures for handling or working with that substance in a safe manner, and includes information such as physical data (melting point, boiling point, flash point, etc.) toxicity, health effects, first aid, reactivity, storage, disposal, protective equipment, and spill-handling procedures. OSHA requires that MSDS be available to employees for potentially harmful substances handled in the workplace under the Hazard Communication regulation and is also required to be made available to local fire departments and emergency planning officials. The American Chemical Society defines Chemical Abstracts Service Registry Number (CAS numbers) to provide a unique number for each chemical and is also used internationally in the MSDS.

5.26. Measurement Assurance – a process to ensure adequate measurement results that may include, but is not limited to: 1) use of good experimental design principles so that the entire measurement process, its components, and relevant influence factors can be well characterized, monitored, and controlled; 2) complete experimental characterization of the measurement process uncertainty including statistical variations, contributions from all known or suspected influence factors, imported uncertainties, and the propagation of uncertainties throughout the measurement process; and 3) continuously monitoring the performance and state of statistical control of the measurement process with proven statistical process control techniques including the measurement of well-characterized check standards along with the normal workload and the use of appropriate control charts.

5.27. Measuring and Test Equipment (M & TE) – all of the measuring instruments, measurement standards, reference materials, auxiliary apparatus and instructions that are necessary to perform a measurement. This term includes measuring equipment used in the course of testing and inspection. In the context of this handbook, the term “measuring and test equipment” is taken to encompass “measurement instruments” and “measurement standards.” Moreover, a “reference material” is considered to be a type of “measurement standard.”

5.28. NVLAP – the NIST National Voluntary Laboratory Accreditation Program.

URL – <http://www.nist.gov/pml/nvlap/index.cfm>

5.29. Occupant Emergency Plan – This document outlines whom to call during an emergency.

5.30. On-site Assessment – systematic, independent, documented process for determining laboratory competence and for obtaining records, statements of fact or other relevant information by assessors at the laboratory facilities and other places where services are provided with the objective of determining the extent to which the criteria of this handbook are fulfilled.

5.31. Personal Protective Equipment (PPE) – clothing and/or equipment designed to protect workers from workplace injuries (e.g., face shields, safety glasses, hard hats, safety shoes, goggles, coveralls, gloves, vests, earplugs, respirators).

5.32. Proficiency Testing – the determination of laboratory performance by means of comparing and evaluating tests on the same or similar items or materials by two or more laboratories in accordance with predetermined conditions.

5.33. Preventive Action – an action taken to eliminate the cause of a potential nonconformity or other undesirable situation in order to prevent occurrence.

5.34. Pyrophoric – something that spontaneously ignites in air.

5.35. Quality Audit – a systematic and independent examination to determine whether quality activities and related results comply with planned arrangements and whether these arrangements are implemented effectively, and are suitable to achieve objectives.

5.36. Quality Control – the operational techniques and activities that are used to fulfill requirements for quality.

5.37. Quality Manual – a document stating the quality policy, management system, and quality practices of an organization. The quality manual may reference other laboratory documentation.

5.38. Recognition – the evaluation and issuance of a Certificate of Measurement Traceability and letter regarding the laboratory management system for state weights and measures metrology laboratories (not formal accreditation).

5.39. Reference Material (RM) – Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process.

- RM is a generic term. Properties can be quantitative or qualitative (e.g., identity of substances or species). Uses can include the calibration of a measurement system, assessment of a measurement procedure, assignment of values to other materials, and quality control. An RM can only be used for a single purpose in a given measurement.

5.40. Respirator Program – This program ensures compliance with 29 CFR 1910.134, and covers the use, care, and fitting of respirators.

5.41. Risk – the statistical chance of danger from an event; an evaluation of the severity and likelihood of an event. OSHA requires that risks be reported when they are associated with medical conditions or exposure.

5.42. Safety Culture – The assembly of characteristics and attitudes in organizations and individuals that establishes, as an overriding priority, that safety issues receive the attention warranted by their significance. It is the product of workers, managers, institutional values, attitudes, perceptions, competencies, and patterns of behavior that determine the commitment to, and the proficiency of, an organization's health and safety management.

5.43. Secondary Reference Material – See Reference Material.

5.44. Short-term Exposure Limit (STEL) – establishes for materials that are very toxic, the maximum concentration employees can be exposed to for periods up to 15 minutes that should not be exceeded at any time during a workday. See also Ceiling (C) and Threshold Limit Values (TLV).

5.45. Standard, Check (or control) – a standard that is used as part of a process measurement assurance program to provide a "check" on the process and standards to ensure that the standards, measurement results, and measurement processes are within acceptable statistical limits. See also Reference Material.

5.46. Standard, Intrinsic – intrinsic standards are based on well-characterized laws of physics, fundamental constants, or invariant properties of materials, and they make ideal stable, precise, and accurate measurement standards if properly designed, characterized, operated, monitored and maintained (NCSL, Traceability Resolution Meeting 1/25/96).

5.47. Standard, Primary – a standard that is designated or widely acknowledged as having the highest metrological qualities and whose value is accepted without reference to other standards of the same quantity. See also Certified Reference Material.

5.48. Standard, Reference – a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

5.49. Standard, Secondary – a standard whose value is assigned by comparison with a reference [primary] standard of the same quantity.

5.50. Standard, Working – a standard that is usually calibrated against a reference standard, and is used routinely to calibrate or check material measures, measuring instruments, or reference materials.

5.51. Standard Operating Procedure (SOP) – a procedure adopted for repetitive use when performing a specific measurement or sampling operation. It may be a standard method or one developed by the user.

5.52. Stop Work Authority – the right, obligation, authority, and responsibility to stop any work or actions that are unsafe to personnel, equipment, or that if continued may damage the environment.

5.53. Technical or Engineering Controls – controls designed to eliminate or reduce exposure to a hazard through the use or substitution of engineered machinery or equipment. Examples of engineering controls include ventilation systems (fume hoods), sound-dampening materials to reduce noise levels, and safety interlocks.

5.54. Threshold Limit Values (TLV) – an 8-hour time-weighted average (TWA) believed to be the average concentration most workers can be exposed during an 8-hour workday, day after day, five days per week, without harmful effects¹⁷ See also Short-term exposure limit (STEL) and Ceiling (C).

5.55. Traceability – property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty (VIM 2010 – 2.41).

5.56. Uncertainty – parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.

5.57. Vapor-Liquid Ratio Temperature (T-V/L 20) – a measure of the tendency for gasoline or blends to vaporize. The gasoline temperature at a V/L of about 20 °C can generally indicate the tendency to cause vapor lock.

5.58. Vapor Pressure (VP) – a measure of fuel volatility and, thus, drivability¹⁸. Fuels that vaporize too easily can result in vapor lock; fuels that do not vaporize easily may cause hard starting and poor acceleration when cold.

5.59. OWM – Office of Weights and Measures of the National Institute of Standards and Technology.

URL: www.nist.gov/owm

¹⁷ Committee on Acute Exposure Guideline Levels, Committee on Toxicology, National Research Council, “Acute Exposure Guideline Levels for Selected Airborne Chemicals,” Volume 8, National Academies Press, 2009.

¹⁸ Rand, Salvatore J., “Significance of Tests for Petroleum Products,” ASTM International, 2003