2005 Petroleum Laboratory Guidelines Update Proposal

2.6.6. Basic Engine Fuels, Petroleum Products and Lubricants Laboratory
(Added 1994; Amended 200X)
(Developed by the Petroleum Subcommittee)

Introduction

The petroleum fuels and lubricant laboratory is an integral element of an inspection program and is generally developed to satisfy the testing requirements as described in the laws and rules of the regulating agency. This document outlines the basic facets of such a laboratory and can be used as a model to initiate or upgrade a program. Since a testing program is of little value unless recognized standards and methods are utilized, this description of a model laboratory has been developed under the assumption that recognized ASTM International and SAE International standards and test methods have been incorporated into the laws, rules, and policies of the regulating agency.

This document provides sufficient information to investigate cost associated with the development of a fuels and lubricant laboratory. Information pertaining to facility needs, recommended ASTM test procedures, test equipment, and the number of personnel required for staffing has been included. Hidden costs associated with the unique working environment of laboratories are often overlooked during initial evaluations; therefore sections have also been included dealing with quality assurance, safety, and hazardous materials.

Laboratories may be required to perform additional analysis outside the purview of consumer regulations, e.g., analyses pertaining to environmental regulations or tax fraud investigations. This document will not address those areas specifically; however, information presented here may assist in the determination of general costs and requirements.

State-Operated or Contract

The decision to operate a State testing laboratory, to enter into a contractual agreement with a private testing laboratory, or to have a hybrid of the two depends on a variety of factors: the scope of the program, funding sources, political climate, etc. The question is often asked: “Is there a point at which it is cheaper for a State to operate its own fuels laboratory?” The Motor Fuel Task Force assembled in 1984 concluded that a program testing 6000 samples per year (500 samples per month) is the minimum level to justify building and equipping a fuel laboratory.

Consideration must be given to the time required for the laboratory to complete the analyses. The value of any inspection program is diminished if laboratory turnaround time is so great that the product is consumed before the results of an analysis are known. If a contract laboratory is chosen, analysis time and should be given consideration during negotiations to ensure an effective program. Because of the hazardous nature of fuels, transportation can be difficult and costly should be factored into the decision. A State-owned laboratory should be assured the proper resources, e.g., a full staff and well maintained instruments, to be able to meet satisfactory turnaround time.

Laboratory Facility

A testing laboratory 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 for 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.

The actual design of the laboratory is dependant upon the products which will be tested. For example, if the octane or cetane number is to be determined, special considerations must be made for foundation and utilities.

Special considerations should be given to the following:

1. Sufficient ventilation to ensure that workers are not unduly exposed to gasoline fumes and 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, eyewash stations, etc.

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 fuel testing can be performed in accordance with ASTM requirements. 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, vacuum pump 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 retained evidence, reference fuel and excess fuel and lubricant after analysis. Depending on the number of samples, this may consist of a properly ventilated storage area with locking storage cabinets and 55 - gallon drums, to a flammable storage room and several 500 - gallon 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 products tested and the estimated sample flow. The following space listing is for a small laboratory capable of testing approximately 6000 fuel 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. Office, bathroom facilities, conference room, etc. (as required). 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 m^2 [750 ft^2])
3. General laboratory (70 m^2 [750 ft^2])
4. Distillation laboratory (37 m^2 [400 ft^2])
5. Shipping and receiving (includes preparation area for empty sample containers) (37 m^2 [400 ft^2])
6. Flash point laboratory (19 m^2 [200 ft^2])
7. Shop area (23 m^2 [225 ft^2])
8. Storage for supplies (23 m^2 [225 ft^2])
9. Secured, cooled, and ventilated sample and flammable storage area (23 m^2 [225 ft^2]). (Insulation and a dedicated ventilation and cooling system should be considered for this room.)

Total square footage (exclusive of item 1) -- 30 m^2 (3225 ft^2). Including offices, bathroom facilities, hallways, etc., the total building size may exceed 372 m^2 (4000 ft^2). 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 place 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 dependant upon the tests which will be performed. However, if work is limited to viscosity measurement an additional 37 m^2 (400 ft^2) should be sufficient.

Tests and ASTM Test Procedures

Careful consideration should be given to the selection of laboratory test procedures since these selections will affect instrument costs, number of personnel, timeliness of samples, and confidence in results. As previously mentioned, ASTM and SAE specifications and test methods are universally recognized standards for fuels and lubricants and should be the primary choice for test procedures. The ASTM Subcommittee D02 on Petroleum Products and Lubricants is responsible for developing specifications and test procedures and is generally comprised of representatives from the petroleum industry, automotive manufactures, and regulating agencies. 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.

New instrumental methods are often introduced to facilitate testing. Chemical methods have been devised to replace or screen physical methods which 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 physical parameters may be drawn with confidence. A new laboratory is cautioned to refrain from investing in this instrumentation and the laboratory expertise necessary to perform the test procedures until they are approved by ASTM. Screening methods have been employed by State laboratories to maintain or increase sample coverage. Screening procedures are a deviation 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 screen sample exceeds a predetermined parameter, the sample is analyzed using the proper ASTM procedure. Screening should be discouraged as a means to increase sample coverage. Strategies, such as selective sampling and testing, should be employed as a means for effective regulation.

Following are references to ASTM and SAE specifications and testing procedures which form an effective nucleus for a testing laboratory with regulatory responsibilities. ASTM test methods listed here do not necessarily exclude other ASTM procedures that are designed for the purpose and that give comparable results. The significance of each of these analyses is included in the ASTM specifications. Some of the test procedures listed make provisions to allow the use of automated equipment. Such equipment is usually more expensive. However, the increased cost can be recovered in a high production lab by reduced labor costs. The Asterisks after test methods indicate a preferred method due to cost or ease of implementation.

Spark Ignition Engine Fuel Specifications – D 4814

1. Distillation	D 86
2. Octane (Antiknock Index)	
Research	D 2699
Motor	D 2700
3. Vapor Pressure	
Dry Method	D 4953
Automatic Method	D 5190*
Mini Method	D 5191*
Mini Method - Atmospheric	D 5482*
4. Oxygenate Content	
GC with TC or FID	D 4815
GC with OFID	D 5599
Infrared Spectroscopy	D 5845

5. Sulfur Content (Due to environmental law and regulations, the sulfur limits shown in D 4814 may be significantly higher than specified. The detection limit and precision of each method should be considered when selecting a test method.)

X-Ray Spectrometry	D 2622
Microcoulometry	D 3120
Ultraviolet Fluorescence	D 5453
6. Water Tolerance	D 6422
7. Workmanship	D 4814

Diesel Fuel Specifications – D 975

1. Flash Point	D 93
2. Distillation	D 86

3. Sulfur Content (The appropriate test method is dependent upon the grade. The forth-coming reduction in sulfur content by EPA starting in June, 2006 will require equipment with lower detection limits and better precision.)

X-Ray Spectrometry	D 2622
Microcoulometry	D 3120
X-Ray Fluorescence	D 4294
5. Cloud Point	
Manual Method	D 2500
Stepped Cooling (Automatic)	D 5771
Linear Cooling Rate (Automatic)	D 5772
Constant Cooling Rate (Automatic)	D 5773
6. Water and Sediment	D 2709
7. Cetane	D 613
8. Lubricity	D 6079

Kerosene Specifications – D 3699

1. Flash Point	D 56
2. Distillation	D 86
3. Sulfur Content	
X-Ray Spectrometry	D 2622
X-Ray Fluorescence	D 4294*
Ultraviolet Fluorescence	D 5453
4. Color	D 156
5. Water and Sediment	D 1796

Aviation Turbine Fuel - D 1655

1. Flash Point	D 56
2. Distillation	D 86
3. Water Reaction	D 1094
4. Freeze Point	D 2386

Motor Oil – SAE J300

1. Kinematic Viscosity	D 445
2. Cold Cranking Simulator	D 5293

Gear Oil – SAE J306

1. Kinematic Viscosity	D 445
2. Brookfield Viscosity	D 2983

Automatic Transmission Fluid

1. Kinematic Viscosity	D 445
2. Brookfield Viscosity	D 2983

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 which may prove to be a better long-term investment when the cost 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.

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 which are consulted for this information. Generally, these departments have a particular protocol for developing an information management system.

Office Equipment and Supplies

No listing is given since needs are determined by the program’s scope. However, the costs of items such as desks, filing cabinets, computers, forms, and miscellaneous office supplies must be considered when planning an initial budget.

Quality Assurance/Quality Control

The previous sections have addressed structural aspects of an engine fuels testing laboratory: building requirements, testing procedures, and analytical instruments. The management system for a laboratory is as unique as the structural requirements. Quality assurance/quality control programs were originally devised to give statistical verification of analytical results; however, they are now evolving to become the standard management model for laboratories. Chain of custody procedures, sample retention procedures, sample distribution procedures, and documentation of each step has been integrated into the quality assurance program.

ASTM has developed two documents which provide quality assurance guidelines for a petroleum laboratory. They are ASTM D 6792, Quality System in Petroleum Products and Lubricants Testing Laboratories and ASTM D 6299, Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance. The first document, D 6792, provides a guide to the essential aspects of a quality assurance program. It includes such issues as sample management, record management, accurate test data, proficiency testing, corrective actions, and training. The second document, D 6299, describes in great detail methods to assure test precision and accuracy.

Another source of information in establishing a quality assurance program is the International Organization for Standardization (ISO) model quality assurance program, ISO 9000. There is no accreditation program specifically for State testing laboratories, and ISO 9000 accreditation is currently quite expensive; however, the ISO 9000 is an excellent model to use in developing a management system.

One excellent method to evaluate the performance of a laboratory is to compare the results obtained with other laboratories. ASTM has developed an Interlaboratory Crosscheck Program to achieve this goal. Samples are periodically sent to participating labs for analysis. The results are submitted to the summarizer and statically compared to other participating laboratories. The summarized results are then compared to the published precision statements. Coded summary reports (to maintain confidentiality) are sent to each participant. The program includes automatic transmission fluid, aviation turbine fuel, engine oil, gear oil, gasoline and diesel fuel as well as other products.

ASTM operates a National Exchange Group (NEG) to distribute fuels among participating laboratories and provides a statistical report of the results. There are three subgroups of the NEG: the Motor Fuel Exchange Group, the Diesel Fuel Exchange Group, and the Aviation Gasoline Exchange Group. Of the three types of participation, only two will concern a state laboratory: a member laboratory receives monthly samples and agrees to participate in special method research; and a "quarterly participant" receive two sets of samples every 3 months but is not bound to run special tests. The NEG will provide a means for assessment of quality at the national level. There are also regional groups which provide similar quality assessment exchange programs: Appalachian, Atlantic, Great Lakes, Mid-Continent, Northwest, Pacific Coast, Rocky Mountain, Texas Regional and LA Gulf Coast, Sabine, and Texas City-Houston Subgroups.

Safety Program

A laboratory can be an extremely hazardous work environment, so safety must be integrated into all operations of a laboratory. The Occupational Safety and Health Administration (OSHA) established a requirement effective January 1, 1991, for laboratories to develop a Chemical Hygiene Plan (29 CRF 1910.1450). The guidelines for the Chemical Hygiene Plan were left intentionally general so that an organization's plan could be customized for unique situations in individual laboratories. The Chemical Hygiene Plan details an organization's responsibilities for safety training, supply and maintenance of safety equipment and personal protective equipment, monitoring employee's exposure level to hazardous chemicals, medical consultation and examination, and availability of documents addressing safety procedures and emergency response. The Chemical Hygiene Plan is required to be reviewed annually which provides a format to plan and track improvements.

Reference documents are an essential part of an effective safety program. Safety procedures should accompany and complement testing procedures to ensure an employee is performing functions in an acceptable manner. Emergency response manuals address hazardous or potentially hazardous situations. Proper procedures for handling large spills, evacuation of work areas, and employees who have been overexposed to hazardous materials are typically found in the emergency response manual. Material Safety Data Sheets (MSDS) contain pertinent information regarding the hazards of chemicals and the necessary precautions. These documents should be distributed to employees or located in an easily accessible location.

Coordination with local fire and hazmat (hazardous material) departments is essential to ensure rapid emergency response. A chemical inventory and a diagram of the laboratory space is often requested by these departments to expedite their response. Periodic review of the chemical inventory will ensure unnecessary chemicals will be disposed of in a timely manner.

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 which 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 the safety of the laboratory.

Personal safety equipment should be provided to all laboratory personnel. Eye protection, lab coats/aprons, and gloves will provide minimum protections. If the use of a fume hood is not practical and an employee is exposed to petroleum or chemical fumes, organic respirators should be provided to minimize exposure. 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. Due to the explosive nature of gasoline, even safety equipment needs to be evaluated for safety; for example, explosion-proof motors should be installed to evacuate fumes from a hood. Eyewash stations, fire extinguishers, emergency shower, and fire blankets should all be placed strategically for maximum protection.

In the event of a spill, 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 absorb 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.

Hazardous Waste

Testing laboratories generate quantities of hazardous waste. Waste chemicals from various analyses and residual samples must be stored and disposed in an appropriate manner. 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. Some companies provide disposal services which recycle products. This type of service is usually less expensive and provides protection from future "cradle to grave" liabilities. Therefore, waste materials should be segregated to take advantage of recycling services.

Personnel

The staffing requirements for a testing laboratory will be dependent on the number of samples, the number of tests performed on the samples, and the testing instruments chosen. The staff recommended here will be suitable for a fuels testing 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

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 operation of the laboratory.

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.

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 5 years of engine operation is necessary to develop an expertise. To expedite this process, engine operators should periodically attend training workshops and regional exchange group meetings. 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.

Concluding Note

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 Caroline, Missouri, Michigan, Washington and Tennessee (a contractual laboratory) have active programs and are willing to host tours of their facilities. Interested parties are encouraged to make such a visit.

References:

John E. Nunemaker, "Planning Laboratories: A Step by Step Process" *American Laboratory* March 1987, 19 (4), 104-112.

Jerry Koenigsberg, "Building a Safe Laboratory Environment" *American Laboratory* June 1987, 19 (9), 96-106.