# CHAPTER 10

# PETROLEUM LABORATORY OPERATIONS

## Section I. Safety During Laboratory Operations

#### GENERAL PRECAUTIONS

All petroleum products present fire hazards for personnel who handle them. However, petroleum laboratory personnel, who work with chemicals in close quarters, are exposed to additional dangers. The chemicals handled may be toxic, corrosive, explosive, flammable, irritant, or carcinogenic. Safe and efficient laboratory operations depend on the observance of well-established safety practices and a thorough knowledge of testing procedures. The testing procedures usually involve using equipment and materials that are potentially dangerous. Injury to personnel and damage to equipment by fire, chemicals, dangerous pressures and vacuums, or misuse of equipment can be avoided by alert and responsible laboratory technicians. Each laboratory should have operating procedures that include all safety considerations. Strict observance of established safety, care, and handling procedures will allow laboratory personnel to perform their duties in a safe and hazard-free environment. All laboratory personnel should be familiar with safety awareness and communication issues concerning substances used or tested at the facility. Also, Material Safety Data Sheets for those substances should be on hand. Some of the more common safety precautions that should be observed during laboratory operations are as flows:

• Do not consume any food or beverages in the laboratory or storage rooms.

• Check for leaks in the oxygen, gas or vacuum systems by listening for hissing sounds, checking unexplained drops in pressure and applying soapy water to the joints and fittings.

• Always have someone else with you in the laboratory when performing test procedures.

• Pay attention to the test in progress. If it is necessary to leave the laboratory or to leave a test in progress, request assistance from another technician or notify the supervisor. Make sure no safety hazard will result from youbaence.

• Maintain a professional manner in the laboratory. Any unnecessary diversions can result in increased hazards and unsatisfactory test results.

• Do not attempt to perform tests simultaneously unless each test can be given the required attention.

• Whenever in doubt concerning any operation, consult qualified authority for advice.

• Do not attempt unauthorized shortcuts to save time, as they generally are not in accordance with safe laboratory procedures or valid test results.

• Be prepared for any emergencies that may arise. Be familiar with the proper action to take in event of emergencies.

• Protective gear (gloves, goggles, and approved lab coat) should be worn when working with chemicals or fuels in the laboratory.

• Wear hearing protection when working around loud machinery (air compressor, generator, and so on).

• When ending daily operations, make a thorough and orderly check of laboratory, equipment, and facilities to ensure that no hazards may develop during the time the laboratory is unattended. Inspections should be performed when the laboratory is unoccupied for an extended length of time.

## PREVENTING FIRES

Fire probably represents the greatest single hazard in the laboratory. Laboratory personnel must be aware of the potential sources of fire and work conscientiously when handling combustible materials or supplies and samples that may form flammable vapors. Chapter 2 of FM 10-67-1, discuses the properties of petroleum and their inherent fire hazards, safety procedures, and fire fighting practices. This should be required reading for anyone associated with petroleum products. Specific to the petroleum laboratory, the following fire prevention rules, at a minimum, should be observed to prevent personal injury and equipment damage.

DO:

• Inspect the laboratory for adequate ventilation.

• Ventilate the laboratory and storeroom to prevent the accumulation of fumes and vapors.

• Check seals, tags, pressure gages, and hoses of fire fighting equipment periodically to make sure they are properly serviced and ready for use.

• Inspect any apparatus you will be working with to ensure that it is fixed firmly in place.

• Check burner tubing frequently to ensure that it is not faulty.

• Check electrical wiring frequently. Look for loose or defective connections or frayed insulation.

• Keep volatile liquids and flammable products away from direct (engine exhausts, open flames, and direct sunlight) and indirect (circuit breakers, switches, and electric motors) sources of heat.

• Make certain there is no open flame or exposed heating element nearby when pouring highly volatile liquids.

• Use glass beads or porcelain fragments, to prevent boiling over or splattering of liquids when heating.

• Use flammable liquids near a source of ignition ONLY if the test procedures require it. • Move flammable debris away from hazardous areas as soon as possible.

• Set hot liquids aside to cool in covered containers before discarding

• Break burned matches, or dispose of them in an ash receiver before discarding them in a refuse container.

• Discard organic products in authorized containers.

• Keep oily rags in a metal, airtight, closed container.

• Immediately clean the area of a spill with absorbent material. Dispose of this absorbent material according to the installation waste disposal plan.

• Perform ohm tests every two years on all static grounds in the laboratory. (Maximum resistance is 25 ohms.)

• Inspect to see that the funnel and funnel base of the filtration apparatus have electrical continuity and are grounded.

• Store chemicals that are hazardous, when near one another, in separate areas. (Oxidizers must be kept separate from flammable and corrosives.)

## DO NOT:

• Smoke in the laboratory or associated areas where chemicals are handled or stored.

• Leave open flames or heating elements unattended.

• Discard organic products (hot or cooled) in sinks or drains.

• Store oily rags in cabinets or drawers.

## MODULAR, MOBILE AND AIR MOBILE LABORATORIES

The following procedures should be observed when operating in the modular, mobile or airmobile laboratories.

• Purge the laboratory prior to entering. If the purge system is inoperative, open all the doors and vents, and wait at least 30 minutes before entering the laboratory.

• Ground the laboratory and generator.

• Remove all waste drums and sample cans from the area when setting up or taking down the nets from the laboratory and equipment. (The nets generate static electricity.)

• Do not store empty or full sample cans in the laboratory. Store empty or full sample cans in a protective area, at least 50 feet away from laboratory and generators.

• Test and calibrate the gas alarm system, as required.

#### FIRE EXTINGUISHERS

Trained personnel may use solid water streams, water sprays, and water fogs to control or extinguish fires in specific situations. One of the most immediate means of extinguishing a fire is with the use of a fire extinguisher. The Army uses both portable hand extinguishers and wheeled units. Portable hand fire extinguishers are effective only in a fire's earliest stages. Portable hand fire extinguishers, except pump-tank units, are available in different sizes and types. The pumptank unit uses water or an antifreeze solution (usually calcium chloride with corrosion inhibitors). Wheeled fire extinguishers offer more flexibility because they have longer hoses and greater capacities. Procedures for extinguishing fires should be posted in the laboratory. Personnel should be thoroughly drilled in the areas of fire prevention and proper, quick response to the sighting of a fire. They should also be familiar with the nature of petroleum fires and with the fire extinguishing equipment in the laboratory. Fire extinguishers and other fire fighting equipment should be within easy reach but safe from fire. In order for the extinguisher to be an effective tool, the following general rules must be followed.

• Know the location of and types of fire extinguishers on hand in the laboratory, and the classes of fires they are intended for.

• Inspect the laboratory facility monthly to ensure that all extinguishers are in their designated places, are readily accessible, are not damaged, and the nozzles are not clogged

• When using the extinguisher, follow the manufacturer's instructions exactly

• Recharge extinguishers immediately after use.

• All fire extinguishers must be tested.

• Examine all fire extinguishers, at least once a year or more often, depending on local regulations.

• Ensure that pressure-type extinguishers are tested hydrostatically.

• Testing should be performed on all fire extinguishers IAW current directives.

## TYPES OF FIRE EXTINGUISHERS

The following paragraphs describe the more common types of fire extinguishers and their properties.

Soda-Acid Extinguisher. The soda-acid extinguisher is the most common type of watersolution extinguisher that uses gas pressure as the expellent. The chemicals in the extinguisher are sodium bicarbonate (baking soda) and sulfuric acid. The sodium bicarbonate is in water-solution form in the extinguisher, and the acid is contained in a loosely stoppered glass bottle. When someone inverts the extinguisher, a chemical reaction produces carbon dioxide that builds up pressure and expels the water. Use this extinguisher type for Class A fires only.

Antifreeze Extinguisher. The antifreeze extinguisher contains a calcium chloride solution charge. The expellant is gas from carbon dioxide cartridges or from a chemical reaction. The operator charges the extinguisher by inverting it and bumping it on the floor or by squeezing a valve lever. Use this type of extinguisher for Class A fires.

Loaded-Stream Extinguisher. The loadedstream extinguisher is charged with an alkali-metal salt solution and other salts. Potassium salts are part of the charge. The way the agent works on a fire differs with the class of the fire. It puts out class A fires immediately and helps keep them from starting again. The way it works on small class B fires is unclear. The agent produces no smothering vapor, but there seems to be a chemical reaction that tends to hold down combustion.

Carbon Dioxide Extinguisher. The carbon dioxide extinguisher comes in many sizes. The charge of liquid carbon dioxide under 800 to 900psi pressure is released by a hand valve at the top of the unit. A tube runs from the top to the bottom of the unit. This tube allows the release of only liquid carbon dioxide until the extinguisher uses about 80 percent of its charge. Gaseous carbon dioxide then flows until the charge exhausts. The charge flows in a high-velocity stream, and a horn or flaring nozzle keeps it from being diluted. When the operator releases the charge and it enters the horn, the chilling effect turns about 30 percent of the charge into dry ice or snow. Cooling of the gas as it expands causes this. Carbon dioxide dilutes air in class B fires. It works well on class C fires because it is not a conductor

Dry Chemical Extinguisher. The drychemical fire extinguisher is available in a wide range of sizes. The chief agent is sodium bicarbonate powder with additives that produce water repellency and free flow. The expellant is carbon dioxide, nitrogen, or compressed air. The extinguisher puts out the fire by smothering it. It works well on class B and C fires. Purple K Extinguisher. The purple K extinguisher is a dry chemical extinguisher using the extinguishing agent potassium bicarbonate (KHCO3), commonly called purple K. Carbon dioxide gas discharges the purple K in a wide stream from a low-velocity nozzle. This fire extinguisher works by smothering and is designed for use on class B and C fires. Purple K is highly corrosive. Purple K extinguishers usually have a 20pound capacity.

## METHODS OF EXTINGUISHING PETROLEUM FIRES

The following methods may be used to extinguish petroleum fires, depending on the class of the fire. Table 10-1 illustrates the National Fire Protection Association's four classes of fire, their source and extinguising agent(s).

• Water and Water Fog. Water should be used as an absolute last resort for extinguishing petroleum fires. Keep in mind that water is heavier than petroleum, and could cause the fuel to float thus spreading the fire. It is more effective as a cooling agent.

Class of Fire	Source of Fire	Extinguishing Agent
Α	Combustibles, such as: wood, paper, grass,	Water
	brush, rubbish.	
В	Liquid, such as: gasoline, and other fuelsl-so	Air-diluting agent, such as carbon-d
	vents, lubricants, paints, oils, and similab-su	oxide; or a smothering agent, such as
	stances that do not leave embers.	foam or a fire blanket.
С	Electrical equipment, such as motors,	Air-diluting agent, such as carbon-d
	switches, and transformers.	oxide or a smothering agent, such as
		foam or a fire blanket. DO NOT use
		an agent that is a conductor of electri
		ity.
D	Combustible materials and other chemicals,	Use a smothering agent, such as foam

Table 10-1. Classes of Fire

such as: sodium, potassium, titanium, and	or a fire blanket.
phosphorous. DO NOT allow water to come	
in contact with these substances, if ignited.	

• Blankets. Blankets are mainly used to put out a fire on a person's clothing. Wet blankets can sometimes be used effectively to help smother a fire at a vapor leak or a vent.

• Carbon Dioxide. As opposed to water, carbon dioxide can be used on most petroleum fires.

• Sand. Sand can be used to cover or absorb liquids for fire prevention. Sand is most useful in coping with fires caused by small quantities of flammable liquids and greases on the floor.

WARNING: Do not use water for extinguishing oil fires because it will spread the fire. Water is also a conductor of electricity and should not be used on electrical fires

## HANDLING CHEMICALS

Many of the chemicals in the laboratory are dangerous. Any action associated with these chemicals should be done safely. Broken or damaged containers must be handled with caution to prevent exposure of personnel to the hazards that may be involved with a particular chemical. The following safety precautions need to be observed by all personnel handling chemicals.

Personal Safety. To prevent personal injury in the laboratory, the following safety measures should be employed.

• Wear rubber gloves when you handle acids or bases.

• Wear goggles when it is necessary to break up chemicals, or when you handle quantities of corrosive liquids.

• Exercise caution when handling a 30 percent or stronger solution of hydrogen peroxide, to prevent contamination. Wash the area thoroughly with water if skin becomes containated.

• Do not handle mercury with your bare hands.

• Do not taste laboratory chemicals.

• Smell a chemical only when it is necessary and then, only by wafting a small amount of vapor with the hand, toward the nose.

• If any acid contaminates your skin, rinse the contaminated area thoroughly with water. The affects of strong acidic solution may be lessened by applying sodium bicarbonate (baking soda) after rinsing the area with water. Consult a physician immediately.

Storing Chemicals. To prevent personal injury when storing chemicals in the laboratory, the following measures should beneployed.

• Make sure that every container and bottle is properly labeled.

• Store heavy and large containers of chemicals on or as near to the floor as possible.

• Do not fill a container with material other than that indicated on the label.

• Store a standard solution of an acid or a base to avoid contamination by atmospheric CO<sub>2</sub> (see Figure 10-1, page 10-6).

• Do not store oxidizing agents with reducing agents.

• Do not store acids and bases together; they will react with each other.

• Do not place bottles containing acids or alkalis on high shelves or on top of equipment. Store them on low shelves so they can be easily reached.

• Store caustic soda solution and sulfuric acid in strong glass containers, never in galvanized iron drums.

• Keep all sample containers capped or plugged at all times, except when pouring out test portions. Always replace the same cap or stopper in the container from which it was removed.

• Hold the stopper of a reagent bottle between two fingers of the pouring hand when pouring from a bottle. Never lay the stopper on a surface that might be touched by personnel or their garments. • Keep reagent bottles stoppered tightly, and dry reagent bottles before replacing them on the shelf.

• Wipe up any acid that spills or splashes on benches, tables, or floors.

• Dispose of all unlabeled and contaminated chemicals IAW local environmental regulations or SOPs.



## Figure 10-1. Preparation of a carboy for storage of acidic and basic solutions

Handling Chemicals. To prevent personal injury and damage to surrounding areas while handling chemicals, the following measures should be employed.

• Always pour acid into water, especially sulfuric acid. Never pour water into acid.

• Use Pyrex glassware when diluting acids. Ordinary glassware may be broken by the heat generated from the mixture of acid and atter.

• Never heat mercury in an open container and never shake more than 20 millimeters of mercury in a glass container. If a spill occurs, ensure adequate ventilation.

• Hold the container cap in your free hand when pouring a sample from a container or bottle. Never place the cap or stopper on a counter, as it may contaminate the sample. Clean up the mercury and sulfur together and put them in a suitable container for disposal IAW local environmental regulations.

• If any chemical is spilled or splashed on the body, immediately wash the contaminated area thoroughly with water.

• If a strong solution of tetraethyl lead is spilled, cover the spill with dry chloride of lime, CaOC1<sub>2</sub>... Sand or other noncombustible absorbent material may also be used. Wait 5 minutes for reaction to be completed. Flush off with water and wash area with soap and water. If the solution is spilled on clothing, remove clothing and discard contaminated articles. Do not attempt to wash contaminated clothing for reuse. Collect all contaminated absorbent materials and place them into a suitable container for disposal IAW local environmental regulations or SOPs. Contact the local environmental office for further guidance on spill reporting, cleanup, and disposal procedures.

• Make certain that a supply of dilute (18%) acetic acid is available when a doctor test or alkali wash is being performed. Use the dilute acid freely on any part of the body, except the eyes,

that may be contaminated with doctor or caustic solution. If doctor or caustic solution should contaminate the eyes, immediately wash out with water and report to hopital.

WARNING: Mercury is a poisonous material that may enter the body by ingestion, inhalation, or skin absorption. Mercury has such density, high surface tension, and low viscosity that pouring without splashing and spilling is almost impossible. When mercury is poured, always use a funnel and make the transfer over spill trays. If a mercury spill occurs, make sure there is adequate ventilation. Do not vacuum or sweep the area as this will disperse mercury throughout the laboratory. Cover the spill with sulfur. Clean up the mercury and sulfur together and discard them in a suitable container. All spills must be reported to the local environmental office.

#### SUBSTITUTE SOLVENTS

Field conditions may require the substitution of certain solvents. In this case, the commonsense rule of "like dissolves like" should be used. When in doubt as to the correct solvent to substitute, consult the ASTM test method. For example, toluene could possibly be substituted for benzene in a solvent capacity, but n-hexane would not serve the purpose.

#### HANDLING EXCESS CHEMICALS

Contaminated chemicals are useless. Do not place spatulas and other objects in chemical containers, as the spatula may contain foreign matter that will cause contamination. Similarly, if excess chemicals or samples are removed from a container, do not put it back into the container. All used chemicals should have well-established rules for disposal.

## HANDLING SOLUTIONS

Prepare a chromic acid cleaning solution by slowly adding 800 milliliters of concentrated sulfuric acid to 500 milliliters of a saturated solution of potassium di chromate and water. Prepare the solution in a sink, using a Pyrex container or equivalent glassware. Although chromic acid is

more effective as a cleaning agent when it is heated, precaution should be taken to avoid boiling the solution. If the solution develops a greenish color, it is useless and should be discarded.

WARNING: Handle the solution with extreme care to avoid personal injury. A face shield and rubber gloves should be used. Chromic acid is a powerful oxidizing agent.

To increase the rate of solution, use a hotplate, not an open flame. Heat the solution in a Pyrex container or equivalent glassware under a fume hood. If a water-free solution is not necessary, dissolve the solute in a small quantity of water before adding it to the warm alcohol. (Alcoholic solutions have toxic and flammable properties.)

WARNING: Never use high-temperature ovens to heat volatile fluids. An explosion may occur and injure personnel.

## CONTROLLING PRESSURE AND VACUUM

The following safety precautions should be observed by all personnel while operating the air/vacuum systems.

• Handle cylinders of compressed gas with particular care. Never allow cylinders to drop or bounce or to come in contact with fire, sparks, or electrical circuits, as explosions may result.

(Because compressed-gas cylinders are made of steel, such explosions have the same destructive effect as a bomb burst.)

• Make sure that all store cylinders are capped, are supported to prevent rolling or falling, and are stored away from heat.

• Always store and transport cylinders in an upright position.

• Never put oil or grease on the valves of cylinders or pressure regulators (pressurized oxygen and oil can create an explosion).

• Do not exceed the pressure or temperature that has been designated as the safe upper limit for

the apparatus or equipment being used, Do not use a cylinder without a regulator.

• Do not use faulty copper, plastic, or rubber tubing when performing operations requiring pressure or vacuum.

• Make sure that glass vacuum apparatus is properly shielded when it is in use.

• Release pressure cautiously when using gasoline bombs for tests. The bomb plug may stick momentarily and then blow out suddenly.

• Always wear goggles when opening air valves that are close to the face.

• Store propane cylinders in the propane storage locker, away from heat or ignition sources.

The following safety precautions should be observed by all personnel handling chemical containers.

• Make sure that chemical containers having vent caps are inspected, and containers that do not have vent caps are vented periodally.

• Keep containers of volatile liquids as cool as possible. Exercise caution in releasing any pressure that may have formed in the container. Always release the pressure gradally.

• Remove caps or stoppers periodically to vent the vapor. (The practice of venting containers of volatile liquids does not apply to those samples collected for vapor pressure tests.)

• Vent separator funnels frequently when shaking volatile liquids. Always wrap the funnel

with a rag when shaking an extremely volatile liquid.

Other laboratory equipment that requires safe handling procedures are discussed below.

• Glassware. Wash the apparatus with solvent, soapy water, tap water, or distilled water, and then allow to dry. If necessary use a cleaning solution. When glassware is prepared for storage, the stopcocks should be free of grease. Store Teflon stopcocks, or their equivalent, loosely in the barrel and keep them free of grease. Wrap glass stopcocks with a strip of paper for long-term storage to avoid fusing of surfaces such as glass stoppers in flasks.

• Crucibles. Mark crucibles permanently with ink manufactured for this purpose. India ink or pencil markings cannot withstand the high temperature crucibles may be exposed to and cannot be used for this purpose.

• Pipets. Before using, inspect pipets for damage to the tip. Use a suction bulb, not the mouth, to draw the liquid up to a point above the etched line. Remove the bulb and rub thumb across the orifice, letting the meniscus drop to a position where its lowest point coincides with the etched line. Touch the tip to a smooth surface to remove any remaining drop. Allow liquid to drain into an appropriate container. Touch the tip to the side of the glass to ensure complete drainage. Pipets marked TD should not have the remaining drops blown out upon drainage.

• Spatulas. Use steel spatulas only when necessary. A small, clean sheet of paper will often serve the same purpose and can be disposed of after use.

• Centrifuge and Centrifuge Tubes. Do not open the centrifuge enclosure while the centrifuge is in operation. Centrifuge tubes positioned opposite each other must not differ in weight by more than 1 gram.

• Separatory funnels. Hold separatory funnels with the palm of the left hand, securing the stopper firmly in place. Use the right hand to operate the stopcock. When the stopcock is opened for venting, the outlet should be directed away

from the operator for safety reasons. Remove the stopper for rapid draiage.

• Corks. Bore corks on a soft wood surface after softening the cork with a roller. When inserting glass tubing or thermometers into corks, wrap the glass in a cloth and hold it as near to the cork as possible. Use water or glycerin as a lubricant. Fire-polish edges of glass tubing to avoid injury to laboratory personnel. Use corks in preference to rubber stoppers because corks are easier to bore and are less reative.

• Thermometers. The mercury in thermometers may contain bubbles. To remove the bubbles, carefully heat the bulb so that the mercury line slowly extends the length of the instrument; then slowly cool the thermometer.

• Ventilation and Fume Hoods. Make sure that the laboratory is ventilated adequately and that fume hoods are operating properly. Use fume hood when working with toxic vapors. Leave the area immediately if a material that gives off toxic vapors is spilled. Return to the area only after it has been ventilated, or if fresh-air respirators have been obtained.

#### CONTROLLING FUMES

The following safety precautions are presented to aid in controlling toxic fumes.

• Make sure the laboratory is properly ventilated and the fume hoods are operating properly. The supply room, where chemicals are stored, must also be adequately ventilated.

• Always work in a fume hood when using benzene, aniline, bromine water, or other materials producing toxic vapors.

• Perform all gas alarm system tests and calibrations as specified to ensure proper operation of system.

• If any material is spilled which gives off toxic fumes, all personnel should leave the area immediately and return only after the area has been adequately purged, or after suitable breathing equipment has been obtained.

#### ELECTRICAL SAFETY

The following electrical safety precautions apply to all operators and maintenance personnel for the petroleum laboratories.

• Equipment producing a tingle sensation should be reported promptly for repair.

• Keep the use of extension cords to a minimum and the cords as short as possible.

• Be sure insulation and wire size are adequate for the voltage and current to be carried.

• Work on electrical devices should be done after the power has been disconnected or shut off, and suitable precautions taken to keep the power off during the work. • If it is necessary to work on "live" electrical equipment, the person doing so must be fully knowledgeable and have a second person present who is trained in first aid. Never work on "live" equipment alone.

• Wear safety glasses or a face shield where sparks or arcing may occur.

• Never use metallic pencils or rulers, or wear rings or watches when working on electrical equipment.

• Avoid using or storing flammable liquids near electrical equipment.

# Section II. Laboratory Analysis Reporting

#### GENERAL

Laboratory administrative procedures discussed in this section are not mandatory. They are included only as suggested methods of accounting for samples received for the following purposes:

- Testing.
- Initiating the analysis reports.

• Assigning routine and special tests to laboratory technicians.

• Recording test results.

• Reporting test results to requesting agencies with recommendations for use, reclamation, downgrading, or other dispition.

#### PETROLEUM SAMPLE TAG FILE

The petroleum sample tag should be detached (at the perforation) from the sample container at the time of receipt. The laboratory number is assigned serially from the laboratory logbook, and preliminary information about the sample is recorded in the logbook. This information includes the sample number, date sampled, unit submitting sample, laboratory number, grade of product, specification, source of sample, quantity represented, date received, date test started, date test completed, and marks.

## PETROLEUM LABORATORY ANALYSIS REPORT

Recording Analyses. The senior laboratory technician transfers information on the sample tag to the heading of a DA Form 2077 (see Figure 10-2). The sample tag is then returned to the card file.

The technician refers to the product specification to determine the tests to be performed and records limits on the DA Form 2077 work copy. He then sends this information by informal memorandums to the technicians. The memorandums are identified by the laboratory number of the analysis report. Work copies of all analysis reports in process are arranged by laboratory report number and attached to a clip board kept in a central location. Notebooks may be kept by laboratory technicians to record test data. When a test or a series of identical tests are completed, the technicians turn in results to the senior technician. The senior technician reviews each complete work copy for completion of required tests and test accuracy. The results are recorded on the work copy. He then makes a recommendation for disposition and gives it to the laboratory NCOIC for review/approval. The NCOIC reviews the test results, compares them with use limits for the product, and decides

whether the product represented by the sample can be safely used for its intended purpose. If all use limits have been met, the product is approved for use. The approval is indicated on the bottom of the report with or without a time limitation, as appropriate.

Processing. A typed report is prepared from the approved work copy. The senior technician sends the report, including enough copies to satisfy interested agencies, to the requesting agency. The technician also provides a copy for the laboratory. The original is forwarded to the requesting agency. A carbon copy of the typed report, the work copy, and the sample tag are fastened together and placed in a permanent file for future reference.

PETROLEUM PRODUCTS LABOR For use of this form, see FM 10-67-2; the	Proponent ag	ANALYSI ency is TRA	S REPORT		REPORT NO.	
PRODUCT NOMENCLATURE AND TYPE THERINIF ENEL AVIATIO	ON G	PADE	JP-8	SPEC MI	L-T-83	133 D
SAMPLE SUBMITTED BY Installation	LEE	(A 23	801	AMT PROD SAMPLE REPRESE	NTS	
MANUFACTURER OF SUPPLIER OF PRODUCT	, .	11 010		SOURCE OF SAMPLE - Truck. Ture TRK 310	k, Aircratt, etc.)	
SAMPLE TAKEN BY (Name)			TEM NO.	NSN	DATE SAMPLE	TAKEN
QUAL NO. BATCH NO.	FILL DATE		DLVR	4/30-01-031-5816	DATE SAMPLE	REC
	FUEL	BULK STORA	3E 🗌 R	OUTINE SURVEILLANCE	15 AUG	XX
CED MAN DETRI I AR	FUEL	PACKAGED	Pf	ROCUREMENT ORIGIN	DATE TESTS S	
SEAMAN PEIRE CAD	ALLIE	D PRODUCTS		ROCUREMENT	DATE TESTS C	
FORT LEE, VA 25001		R EFFECTIVEN		FECIAL	16 AUG	XX
TEST	SPECQUAL	RESULT		TEST	SPEC/QUAL	RESULT
1. GRAVITY 'API/SOCR 60°/60°F	37-51	43.0	27. WATER AND SED	MMENT % VOL MAX		
a. MID			28. F5II % VOL		<u> </u>	
b. BOT	Carlos - Auto	904	a.	MID ROT		
	C + 0	- 300	<i>"</i>	AVG	10-15	./30
3. COLOR VISUAL	RPT	w/w	29. PARTICULATE CO	DNTAMINANT MGS/GAL		
a. HELLIGE (Colorimeter)			30. THERMAL STAB	ILITY INCHES HG		
b. ASTMANAR/SAYB	RPT	-10	a.	PREHEATER RATING		
C. SAYB AFTER HEAT MIN			31. SULFIDES (Tank W	ater BTMS)		
4. ODOR			32. WATER SEPARO		70	85
5. DISTILLATION I BP C	RPT	139.0	33. % ASH PLAIN/SU			
a. 10 % REC- MAX C	205	172.0	34 % LEAD		+	
	ROT	211.0	35. % PHOSPHOROS	· · · · · · · · · · · · · · · · · · ·		
	RPT	2490	37. BURNING TEST (	(6 hrs)	1	
	300	262.0	38. KIN CS-CS- AT -	ZO C MAX	8.0	7.0
f. % RECOVERED	RPT	98.0	a. KIN CS-SSU AT	r 'F		
g. % LOSS	1.5	1.4	た. KIN CS/SSU AT	r 'F		
h. % RESIDUE	1.5	.6	KIN CS/SSU AT	r 'F		
i. 10% + 50% EVAP <sup>1</sup> F MIN			d SSFAT	°F		
6. ENGINE RATING O.N. MOTOR METHOD			e. VISCOSITY INC		+	
			40 PRECIPITATION			
C BMB SUPER CH METHOD			41. SEPARATION %	MAX		
d. CETANE NUMBER /INDEX MIN	RPT	46.0	42. ACID NO/BASE N	IO MAX		
7. RVP (PSI)			43. CHANNEL PT	°F MAX		
8. GUM EXISTENT MG/100 ML MAX	7.0	5.0	44 SAPONIFICATION	N NO MAX		ĺ
GUM (Wash) MG/100 ML MAX			45. DIELECTRIC STR	RENGTH KV MIN		ļ
GUM POTENTIAL MG/100 ML MAX	ļ	ļ	46. FOAM SEQ 1. ML	S MAX (TND/STAB)		
PRECIPITATE MG/100 ML MAX			d. SEQ 2. MLS M	AX (TND/SIAB)		
9. TELTML (MUGM/GAL) MAX			47 PENETRATION I	INWORKED 77"E	+	
10. DRIDATION STABILITY MINUTES			4. PENETRATIO	N WORKED 77°F	-	
12. SULFUR BY LAMP BOMB % MAX			48. DROP PT/MELT	PT <sup>°</sup> FMIN		
13. FREEZING PT C MAX	-47	-53	49. CORR AND OXID	ATION STAB		
14. CORROSION COPPER STRIP 2 NR @ 1000	I MAX	16	50. SWELLING SYN	RUBBER %		
15. AROMATICS % VOL MAX	Ļ		51. LOW TEMP STAE	BILITY		
15. OLEFINS % VOL MAX			52. SALT SPRAY TES	ST		
17. SMOKE POINT MM MIN			53. WORK STABILIT	×		·.··
18. SMOKE VOLAT INDEX MIN	1		55 THICKENER TYP	>F		
	38.0	34.0	56. THICKENER COM		+	
21. CLOUD POINT <sup>2</sup> F MAX	0000	<u> </u>	57. CORROSION PRO	OTECTION		
22. POUR POINT <sup>2</sup> F MAX		1	58. REMOVAL			
23. WATER REACT INTERFACE RATING MAX	16	16	59. APPARENT VISC	AT <sup>'</sup> F		
. SEPARATION VOLUME CHANGE MAK	rpt/rpt	1/0	a. SHEAR RATE	POISES	- , .	20
24. CARBON RESIDUE % WT MAX	. <u> </u>	<u> </u>	60. SED CONTAM.		15	3.0
25. WATER % VOL MAX	+		62 OTHER Constit		is min	00
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Figure 10-2. DA Form 2077

Recommendations for Disposition. If the product should not be used for its intended purpose, an alternative use must be considered and suitable recommendations made. The report should include the following information:

• Probable cause of product not meeting use limits.

• Nature and probable cause or source of contamination.

• Suggestions for preventing future contamination or deterioration.

• Recommendations for reclamation or other disposition of the product.

• On grade—Sample meets all specification requirements and can be retained for long storage.

• Suitable for use—Product is either deteriorated or contaminated to the point where one or more tests do not meet specification requirements but meet use limits (MILHDBK-200). Product should be used as soon as possible.

• Not suitable for use—Product fails to meet one or more use limits or specification requirement that has no use limit. Product must be downgraded or blended as recommended and retested before use.

#### TESTING

Correlation Testing. Correlation testing may be done by sending identical samples to two or more laboratories. These laboratories use the same apparatus in performing the tests. Correlation testing may also be done by having two or more technicians within a given laboratory perform tests on identical samples using the same apparatus under controlled conditions. Another way of testing is to have a single technician perform duplicate tests on identical material using the same apparatus under controlled conditions. Results should not differ by more than those specified in the test method. The first two procedures are checks on reproducibility, the third is a check on repea**lity**i

Equipment Calibration. Results of all tests are dependent on calibrated equipment. Most Army

equipment requiring calibration is listed in TB 43-180. Also, the calibration frequency and standard for test equipment is specified in the applicable test methods. Internally, the laboratory should verify calibration more frequently than TB 43-180 requires.

• C-level calibration procedures. C-level calibration will be performed by qualified laboratory personnel assigned to the labotary.

Analytical balance. The electric balance is a single-arm balance and has a weighing capacity of 100 grams. When the balance is provided with the manual taring accessories, the weighing capacity is increased to 150 grams. The precision (standard deviation) of the balance is plus or minus 0.05 milligrams; digital readability is 0.1 milligrams; and accuracy in the optical range is plus or minus 0.05 milligrams. ASTM E 319 procedures are recommended for evaluating performance and verifying the accuracy of the balance. These procedures determine the precision that a balance can compare known weight loads; that is, the built-in weights of the balance and a known weight load. Section 5, ASTM E 319, outlines procedures for preparing the balance for evaluation and section 8 outlines procedures for evaluating balance accuracy. A precision weight set (class S) is used to evaluate balance performance. The verification and evaluation of the balance are performed by operating personnel. The double beam balance may be evaluated using the above procedures. The standard deviation of the double balance is plus or minus 0.1 grams.

• Manometer. The manometer is used to verify the accuracy of the RVP gauges. Mercury is used as the indicating fluid. The manometer is equipped with a double scale graduated in inches of mercury and psi. The scale has provisions for zero adjustment. The scale must be adjusted to the zero position prior to verifying gage accuracy. The accuracy of the manometer is verified by using a certified master gage with a range of zero to 15 psi graduated in increments of 0.1 psi and an accuracy of plus or minus 0.05 percent. Recommended A-level calibration frequency is 180 days.

• RVP gages. The RVP gages must be verified for accuracy after each test when deter-

mining vapor pressure of MOGAS. When determining the vapor pressure of aviation fuels (AVGAS and turbine engine fuel), operating personnel verify the gage for accuracy before and after each test. The accuracy of RVP gages is verified by using the manometer. When the gage reading and the manometer reading differ by 1 percent or less; that is, the gage correction factor is not greater than 0.05 psi for 5-pound gages or 0.15 psi for 15-pound gages, the gage is considered accurate. However, if the readings differ by more than 1 percent, the gauge is considered inaccurate and must be repaired oreplaced.

• Thermometers. The routine laboratory thermometers, ASTM 9 F, 12 F, and 58 F, have

scales including  $32^{\circ}F$  and are verified for accuracy

by determining the ice point. ASTM 18 F is verified at 100°F. ASTM 7 F, low distillation thermometer, is verified for accuracy at 200°F. ASTM 10 F, high range thermometer, is verified for accuracy at 212°F. ASTM 7 F, 10 F, and 18 F are verified for accuracy by direct comparison with a certified precision thermometer. The two certified precision thermometers (ASTM 64 F and 68 F) are certified by the A-level calibration facility. The precision thermometers must be certified at 360-day intervals. The error of the certified thermometers must not be more than the maximum scale error of the specification (ASTM E-1).

## Section III. Standard Publications and Forms

#### GENERAL

Publications that describe the acceptable military procedures for testing and evaluating the quality of a petroleum product are essential for use in a petroleum laboratory. The latest editions, with all change notices and current petroleum product specifications, must be on hand at all times to be used by laboratory personnel.

## MILITARY STANDARDIZATION HANDBOOK FOR FUELS, LUBRICANTS, AND RELATED PRODUCTS (MIL-HDBK-200)

This handbook provides general instructions and minimum procedures to be used worldwide by the military services in QS of US governmentowned fuels, lubricants, and related products. The procedures described in MIL-HDBK-200 include QS testing and use limits, minimum sampling and testing requirements, types of tests required on various petroleum products, and storage and transportation requirements.

#### FEDERAL TEST METHOD STANDARD NO. 791

FTMS No. 791 covers methods adopted for use by federal agencies in testing lubricants, liquid fuels, and related products. Only the federal test methods without adopted ASTM test standards are included in the publication. The federal standard has both alphabetic and numeric indexes of test methods. These list both the federal test designation and the corresponding ASTM test designation. New and revised federal test material and cancellation are issued as change notices by the General Services Administration. These are numbered consecutively and dated. Laboratories should retain all change notices until superseded by a reissue of the entire federal stadard.

ASTM STANDARDS 23, 24, 25, AND 47

The ASTM standards 23, 24, 25, and 47 are published annually and contain test methods for petroleum products.

# DFSCH 4120.1, REFERENCE LIST OF SPECIFICATIONS AND STANDARDS

This list shows all current petroleum specifications and is published periodically by the Defense Fuel Supply Center.

## DOD MANUAL 4140.25-M, PROCEDURES FOR THE MANAGEMENT OF PETROLEUM PRODUCTS

This manual covers procedures for management of DOD-owned petroleum stock.

## AR 715-27 (DLAM 4155.1), PETROLEUM PROCUREMENT QUALITY ASSURANCE MANUAL

This manual covers all areas of petroleum QA.

## FORMS

The following forms may be used in a petroleum laboratory.

• DA Form 285 (US Army Accident Investigation Report.

• DA Form 1804 (Petroleum Sample). See Figure 9-4.

• DA Form 2077 (Petroleum Products Laboratory Analysis Report). See Figure 10-2.

• DA Form 2404 (Equipment Inspection and Maintenance Worksheet).

• DA Form 2407 (Maintenance Request).

• DD Form 250 (Material Inspection and Receiving Report). See Figure 10-3.

• DD Form 250-1 (Material Inspection and Receiving Report - Continuation Sheet). See Figure 10-4.

• DD Form 314 (Preventive Maintenance Schedule and Record).

• DD Form 1425 (Specifications and Standards Requisition).

• SF 361 (Discrepancy in Shipmente Bort).

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Figure 10-3. DD Form 250

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Figure 10-4. DD Form 250-1