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Standard Practice for In-Service Monitoring of Lubricating Oil for Auxiliary Power Plant Equipment[1]

This standard is issued under the fixed designation D6224; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

A more systematic approach to monitoring auxiliary power plant equipment can help to minimize the high cost of oil changes and unplanned shutdowns. These avoided costs must be balanced against the cost of sampling and laboratory testing.

This practice is designed to help the user evaluate the condition of the lubricant through its life cycle by carrying out a meaningful program of sampling and testing of oils in use. This practice is performed in order to collect data and monitor trends which suggest any signs of lubricant deterioration and to ensure a safe, reliable, and cost-effective operation of the monitored plant equipment.

1. Scope*

1.1 This practice covers the requirements for the effective monitoring of mineral oil and phosphate ester fluid lubricating oils in service auxiliary (non-turbine) equipment used for power generation. Auxiliary equipment covered includes gears, hydraulic systems, diesel engines, pumps, compressors, and electrohydraulic control (EHC) systems. It includes sampling and testing schedules and recommended action steps, as well as information on how oils degrade.

NOTE 1—Other types of synthetic lubricants are sometimes used but are not addressed in this practice because they represent only a small fraction of the fluids in use. Users of these fluids should consult the manufacturer to determine recommended monitoring practices.

1.2 This practice does not cover the monitoring of lubricating oil for steam and gas turbines. Rather, it is intended to complement Practice D4378.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
D257 Test Methods for DC Resistance or Conductance of Insulating Materials
D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
D665 Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water
D892 Test Method for Foaming Characteristics of Lubricating Oils
D893 Test Method for Insolubles in Used Lubricating Oils
D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils
D974 Test Method for Acid and Base Number by Color-Indicator Titration
D1169 Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids
D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

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[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand is the direct responsibility of Subcommittee D02.C0.01 on Turbine Oil Monitoring, Problems and Systems.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard

D1401 Test Method for Water Separability of Petroleum Oils and Synthetic Fluids
D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
D1533 Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration
D2272 Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel
D2273 Test Method for Trace Sediment in Lubricating Oils
D2422 Classification of Industrial Fluid Lubricants by Viscosity System
D2668 Test Method for 2,6-*di-tert*-Butyl- *p*-Cresol and 2,6-*di-tert*-Butyl Phenol in Electrical Insulating Oil by Infrared Absorp
D2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
D2982 Test Methods for Detecting Glycol-Base Antifreeze in Used Lubricating Oils
D3427 Test Method for Air Release Properties of Hydrocarbon Based Oils
D3524 Test Method for Diesel Fuel Diluent in Used Diesel Engine Oils by Gas Chromatography
D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4378 Practice for In-Service Monitoring of Mineral Turbine Oils for Steam, Gas, and Combined Cycle Turbines
D4739 Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration
D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration
D6810 Test Method for Measurement of Hindered Phenolic Antioxidant Content in Non-Zinc Turbine Oils by Linear Sweep Voltammetry
D6971 Test Method for Measurement of Hindered Phenolic and Aromatic Amine Antioxidant Content in Non-zinc Turbine Oils by Linear Sweep Voltammetry
D7155 Practice for Evaluating Compatibility of Mixtures of Turbine Lubricating Oils
D7414 Test Method for Condition Monitoring of Oxidation in In-Service Petroleum and Hydrocarbon Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry
D7546 Test Method for Determination of Moisture in New and In-Service Lubricating Oils and Additives by Relative Humidity Sensor
D7464 Practice for Manual Sampling of Liquid Fuels, Associated Materials and Fuel System Components for Microbiological Testing
D7647 Test Method for Automatic Particle Counting of Lubricating and Hydraulic Fluids Using Dilution Techniques to Eliminate the Contribution of Water and Interfering Soft Particles by Light Extinction
D7669 Guide for Practical Lubricant Condition Data Trend Analysis
D7687 Test Method for Measurement of Cellular Adenosine Triphosphate in Fuel, Fuel/Water Mixtures, and Fuel-Associated Water with Sample Concentration by Filtration
D7720 Guide for Statistically Evaluating Measurand Alarm Limits when Using Oil Analysis to Monitor Equipment and Oil for Fitness and Contamination
D7843 Test Method for Measurement of Lubricant Generated Insoluble Color Bodies in In-Service Turbine Oils using Membrane Patch Colorimetry
D7978 Test Method for Determination of the Viable Aerobic Microbial Content of Fuels and Associated Water—Thixotropic Gel Culture Method
E1064 Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration
F311 Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters
F312 Test Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters

2.2 *ISO Standard:*[3]

ISO 3448:1992 Industrial liquid lubricants—ISO viscosity classification
ISO 4406:1999 Hydraulic fluid power—Fluids—Method for coding the level of contamination by solid particles

2.3 *SAE Standards:*[4]

J300 Engine Oil Viscosity Classification
J306 Automotive Gear Lubricant Viscosity Classification

3. Significance and Use

3.1 This practice is intended to help users, particularly power plant operators, maintain effective control over their mineral lubricating oils and lubrication monitoring program. This practice may be used to perform oil changes based on oil condition and test results rather than on the basis of service time or calendar time. It is intended to save operating and maintenance expenses.

3.2 This practice is also intended to help users monitor the condition of mineral lubricating oils and guard against excessive component wear, oil degradation, or contamination, thereby minimizing the potential of catastrophic machine problems that are more likely to occur in the absence of such an oil condition monitoring program.

3.3 This practice does not necessarily reference all of the current oil testing technologies and is not meant to preclude the use of alternative instrumentation or test methods that provide meaningful or trendable test data, or both. Some oil testing devices and sensors (typically used for screening oils that will be tested according to standard methods) provide trendable

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[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096, http://www.sae.org.

indicators that correlate to water, particulates, and other contaminants but do not directly measure these.

3.4 This practice is intended for mineral oil products, and not for synthetic type of products, with the exception of phosphate esters fluids typically used in power plant control systems.

4. General Properties of Lubricating Oils

4.1 In general, lubricating oils are designed to reduce friction and wear, provide cooling, control deposits, and combat the effects of contamination. A base oil's lubricating properties are enhanced by selected additives. Different machines have different lubricant additive requirements, some of which are described in this section. Proper lubrication minimizes or precludes contact between metal surfaces and reduces component wear.

4.2 *Gear Oils*—The primary requirement of gear oils is that they prevent wear and minimize other forms of damage such as pitting and scuffing by maintaining a lubricant film between the moving surfaces.

4.3 *Hydraulic Oils*—A hydraulic fluid is required to transmit hydraulic pressure and energy, minimize friction and wear in pumps, valves, and cylinders, and protect metal surfaces against corrosion. To obtain optimum efficiency of machine operation and control, the viscosity of the oil should be low enough to minimize frictional and pressure losses in piping. However, it also is necessary to have a sufficiently high viscosity to provide satisfactory wear protection and minimize leakage of the fluid. High-viscosity index fluids help to maintain a satisfactory viscosity over a wide temperature range. The anti-wear properties of high-quality hydraulic oils usually are improved by suitable additives. Since the clearances in pumps and valves tend to be critical, it is important to provide adequate filtration equipment (full flow or bypass, or both) to maintain a minimum particle content and thus minimize wear. The antioxidant additives in the hydraulic oil should give the oil good oxidation stability to avoid the formation of insoluble gums or sludges; the oil should have good water separation properties, and, because air may be entrained in the system, the oil should have good air-release properties and resistance to foaming. Similarly, good rust protection properties will assist in keeping system metals in satisfactory condition.

4.4 *Diesel Engine Oils*—In addition to the typical role of lubricating oils which is to lubricate, clean, cool and seal, diesel engine oils are formulated to provide protection from acids and disperse soot particles that are created during the combustion process. Diesel engine oils are compounded with alkaline additives to neutralize the sulfuric acids that are produced when the diesel fuel is combusted. They are also compounded with dispersant/detergents to keep the engine clean and the by-products of combustion (fuel soot) suspended. The combination of wear regimes found in the diesel engine require the lubricants to have high levels of anti-wear additives to protect the engine from wear during the most severe condition. Multi-grade lubricants (high viscosity index) are often employed in diesel engine lubricants that are required to operate over a wide temperature range.

4.5 *Turbine Oils or Circulating Oils, or Both*—These oils provide satisfactory lubrication and cooling of bearings and gears (for example, in auxiliary turbines, pumps and gearboxes as circulating oils). They also can function as a governor hydraulic fluid. The oil must have a viscosity high enough to maintain a sufficiently thick film of oil on load-bearing surfaces, but low enough to minimize energy losses while providing adequate cooling. These oils are recommended where the degree of loading on bearings and gears is less than in gear oil applications. Turbine or circulating oils, or both, have excellent oxidation resistance and contain rust inhibitors; they are often referred to as rust and oxidation inhibited (R&O) oils. They can also contain additives to improve water separability and decrease foaming tendency.

4.6 *Compressor Oils*—In addition to possessing the correct viscosity for satisfactory bearing and cylinder lubrication, particularly for air compressors, very good oxidation resistance is required to avoid degradation of the lubricant in the presence of heated air. This is particularly important for mineral oils where discharge temperatures are high, since carbon and oxidized oil deposits may autoignite if exposed continuously to temperatures above 148 °C. The fire potential that exists under these conditions make low volatility and high auto-ignition values equally or more important than high-flash or fire points. In compressor lubrication, condensed water is present frequently. For this reason, the oil must possess properties that ensure that the oil rather than water wets the metal surfaces. Also, to avoid the accumulation of water-in-oil emulsions in the after coolers, the water should separate out rather than form an emulsion.

4.7 *Electrohydraulic Control (EHC) Fluids*—Triaryl phosphate ester EHC fluids are inherently fire-resistant and maintain this property throughout their service life. The very low vapor pressure and chemical nature of these fluids result in high flash point, fire point, and autoignition temperature. EHC fluids should be continuously purified using bypass systems to maintain acid number, moisture, and particulates at low levels. Moisture can cause hydrolysis of EHC fluids which results in elevated acid number. Components constructed of copper and lead alloys should be avoided. These fluids are chemically different from mineral oils; consequently, the interpretation of test results will be significantly different. The fluid supplier should be consulted if there is a question about interpretation of analytical results.

5. Operational Factors Affecting the Service Life of Oils

5.1 *New Oil Quality and Suitability for Intended Use*—Use of high-quality oils that meet recognized standards (such as manufacturer military specifications *and* OEM specifications) is the best assurance of potentially long service life. Careful oil storage is important to prevent the degradation of the lubricant while in storage or being dispensed. Accurate labeling of lubricant containers is vital to ensure proper identification.

5.1.1 Viscosity is the most important characteristic of an oil. Oil load bearing and lubricating properties are related to its viscosity. The use of oil with incorrect viscosity can increase wear rates, heat build-up, and lube degradation. In extreme cases, the use of oils with incorrect viscosities can result in rapid catastrophic failures.

5.1.2 Oils that meet the equipment manufacturers' requirements should be used. For situations where the manufacturer simply offers a generic viscosity classification without specific performance criteria, the user should consult the equipment manufacturer, lubricant suppliers, and experts in the field of lubrication.

5.1.3 When fresh, unused lubricants are received, it is advisable to obtain typical test data from the oil supplier. Upon receipt of the first oil charge, take a sample of oil to confirm the typical test data.

5.1.3.1 Because systems usually contain some residual oil, (whether from a previous charge or a flush) the baseline sample to be used for condition monitoring comparisons should be taken after the new oil is charged and circulated for up to 24 h (depending on the size of the reservoir and turnover time).

5.1.3.2 This baseline should be the reference sample for the physical and chemical properties of the fluid, and for future comparisons with in-service oil information. **This is most important!** Recommended tests for new oil are given in the schedules of this practice. (**Warning—**Physical and chemical properties of lubricants after installation may not match results obtained for new oil as received from the supplier.) (**Warning—**Storage conditions affect the shelf life of lubricants. Manufacturing shelf life recommendations should be followed. If no shelf life guidance information is available and the lubricants is greater than two years old, the lubricant manufacturer should be consulted to confirm suitability for use.)

5.1.4 Manufacturer shelf life recommendations should be observed. Oils should be stored to preserve their original quality and prevent contamination. Stored oils may be tested to ensure and document their quality, cleanliness, and continued suitability for their intended use. It is suggested that oil manufacturers' recommendations be followed when storing lubricants to ensure maximum product life.

5.1.5 Make-up oils should normally be of the same type, quality, and manufacturer. Available formulations may change over a period of time. Lubricant incompatibility can arise from mixing differing base stocks and additive packages and should be avoided. When oils must be mixed, testing should be performed in an attempt to determine compatibility in accordance with Practice D7155. Consideration should be given to consulting the lubricant supplier(s) and equipment manufacturer prior to mixing oils.

5.2 *Deterioration of Oils in Service*—Air (oxygen), elevated temperatures, metals, and water (moisture) are present to some extent in lubricating systems. Record these factors that promote lubricant degradation. Lubricant deterioration occurs by one or more of the following processes:

5.2.1 *Oxidative Degradation*—This process occurs as the result of chemical changes brought about by oxygen in the atmosphere and proceeds by a chain reaction that is with the presence of water, heat, and certain metals. The results of oxidation can consist of AN increase, viscosity (KV) increase), or sludge and varnish deposits, or a combination thereof as end-products within the lubricating system.

5.2.2 *Thermal Degradation*—This process occurs in the absence of oxygen and at much higher temperatures. Typically, temperatures of more than 300 °C may cause the hydrocarbon molecule to crack and produce various degradation species. Some of these species are low molecular weight by-products that evaporate or burn (producing a noticeable "burnt" smell in the oil) and other degradation species are high molecular weight by-products that form sludge and varnish.

5.2.3 *Lubricant Deposits and Sludge (Lacquering/Varnishing)*—As lubricants degrade either through thermal and mechanical forces, they may produce submicron, high molecular weight, polar insoluble particles. These particles may agglomerate, become insoluble in the oil, as it is non-polar in nature, and adsorb onto the metal parts of a lubricating system. Some highly refined base oils used in the manufacturing of lubricants (API Group II and above) may be less tolerant to the presence of these degradation by-products due to their high purity and lower solvency. Base oils with proper formulation can result in low deposit tendency oils. Deposits (such as varnish) could be very costly to an equipment operator, as they may deposit on bearing and turning gear surfaces increasing wear, settle in servo-valves causing valves to stick and seize, coat heat exchangers lowering their performance and form in reservoirs acting as a catalyst to further degradation. If deposits are found in the system, analysis can be performed on the deposit to identify possible root causes. The insoluble polar compounds (soft contaminants) may be removed by the use of varnish separation technologies and through carefully performed high velocity flushes.

5.2.3.1 When these conditions of lubricants deposits and sludge occur, dramatic reductions in the viscosity of the oil are possible and the flash point of the resulting fluid can also change.

5.2.4 *Hydrolysis*—Hydrolysis is a mode of degradation in the presence of moisture. This is very important for phosphates (and other esters) and may also have an effect on additive systems in oil-based products. The major characteristic of hydrolysis is the generation of corrosive acids in the fluids.

5.2.5 *Loss of Additives*—Additives are used to protect the oil and enhance its performance abilities. When these additives are depleted as a result of service, the performance of the oil will be reduced as a consequence of oil oxidation, foaming, excessive wear, or premature rusting.

5.2.6 *New Oil Make-up Rate*—Addition of new oil is required in nearly every system to make up for losses due to leakage, filter changes, or other maintenance. Monitor the amount and frequency of added make-up oil, since they play a very significant part in determining the life of a system oil charge.

5.3 *Contamination*—Contamination of lubricating oils occurs both from outside and from within the system. Common types of contamination are: debris introduced at initial startup or after an overhaul, lubricant degradation byproducts, component wear debris, airborne particulates, and water (moisture). Contamination is often the most significant factor affecting oil service life. Contamination of oil is a valid reason to change oil and flush to restore system cleanliness.

5.3.1 *Condition of Equipment on Start-up*—Oil system contamination prior to start-up usually consists of preservatives, paint, moisture, rust particles, and construction debris such as dust, dirt, or welding spatter. Whenever practical, flushing the system before starting operation is recommended. Fluid cleanliness should be brought to a level of one to two ISO FDIS 4406.2 classes below warning levels before beginning operation. If flushing is not performed, oils should be tested soon after startup or repair to verify their cleanliness.

5.3.2 *External Contamination-Solids*—Solid contamination consists of any material small enough to pass through bearing seals and vents or which can be introduced with make-up oil. From whatever source, contamination must be dealt with by monitoring oil condition and using purification devices such as filters and centrifuges on a regular basis.

5.3.3 *External Contamination-Liquid*—Coolant leaks, moisture or steam condensation, or introduction of improper lubricating oils can compromise the oil. Accumulated water promotes oil degradation as well as interfering with lubrication. Contamination with an improper lubricant is not easily corrected without a complete oil change. An oil monitoring program may be used to monitor and identify contaminants likely to be encountered in service.

5.3.4 *Internal Contamination*—Contaminants include wear debris, oil degradation products, and microbial growth. The types of internal contaminants will vary by equipment type and oil type; the rate of generation will be highly dependent on the equipment operating conditions. The analysis methods employed must be able to identify expected wear debris and degradation products. Testing frequencies should be sufficient to account for operating conditions.

6. Sampling

6.1 *General*—When taking lubricant samples from storage tanks or equipment in service, it is important that the extracted sample is representative and is taken from a specified location(s) to monitor the properties of the lubricant. The following are some suggested guidelines for proper sampling technique and sample handling techniques. (See also Practice D4057.) The user should have written procedures to insure that samples are taken consistently according to good maintenance practices.

6.1.1 *Microbiological Testing*—When sampling in order to perform microbiological testing, refer to Practice D7464 for guidance on sample collection and handling.

6.2 *Representative Sampling*—A representative sampling location is a sampling location that supports repeatable and representative lubricant sampling to monitor the health of the equipment and the properties of the lubricant. Collect oil samples when machines or equipment are running at normal operating temperatures, loads, pressures and speeds. To ensure that insoluble material is suspended evenly throughout the system. A fluid sample is probably not representative if: *(1)* the system fluid is hot while the sample is cold, *(2)* the fluid in the system is one color or clarity in an in-line sight glass while the sample is a different color or clarity, and *(3)* the fluid viscosity of the reservoir fluid is different from that of the sample when both are at the same temperature. Samples should be taken in the same manner each time to allow reliable trending of oil properties.

6.2.1 It should be noted that on occasion a sample may be requested which will not be representative. At that time, sampling instructions, as specified by the requestor, must be followed. For example, a sample might be taken off the top or the bottom of a tank to check for contamination. In all cases, the sample point should be marked on the sample container.

6.3 *Sampling Location*—Assign sample location upstream of filters and downstream of machine components such as bearings and gears to obtain the best data. The sampling downstream of the filters is only advised to determine the efficiency (beta-ratio) of filters or filtration systems.

6.4 *Flushing Procedures*—Always flush a sample line before a sample is taken and flushing will be achieved by flushing properly the sampling valves, devices, and hardware thoroughly prior to taking oil samples.

6.4.1 The flushing is usually accomplished using a spare container/bottle to catch the purged fluid. It is important to flush 5 to 10 times the dead space volume before obtaining the sample.

6.4.2 All hardware that the oil comes into contact with is considered dead space and must be flushed, including, system dead-legs, sampling ports, valves and adapters, probe on sampling devices, adapters for using vacuum sample extraction pumps, as well plastic tubing used for vacuum pumps (this tubing should not be reused to avoid cross-contamination between oils).

6.4.3 After opening the sample port and flushing, fill the sample container/bottle to approximately 50 % of its capacity with the oil—leave enough void to allow shaking of the sample prior to testing. In case of flash point testing, for most accurate testing, reduce the head space volume.

6.5 *Documenting Sampling Procedures*—To ensure that each sample is taken in the same manner and from the same point, the operator defines and documents oil sampling procedures for each system such as: *(1)* tools needed, *(2)* line flushing requirements, *(3)* sampling locations, *(4)* sampling methods, *(5)* safety requirements, and *(6)* sample bottle labeling.

6.5.1 It should be noted that on occasion a sample may be requested which will not be representative. At that time, sampling procedures, as specified by the requestor, must be followed. For example, a sample might be taken off the top or the bottom of a tank to check for contamination. In all cases, the sample point should be marked on the sample container/bottle.

6.6 *Oil Sampling Frequency*—Take oil samples at a specified frequency; this will ensure that any problems are identified early. Sampling frequencies will be set specifically for each machine or piece of equipment, since each is unique in its intended performance, condition, locality, operating environment, and maintenance schedule.

6.7 *Oil Sample Container/Bottles*—Take oil samples in oil sample containers or bottles which should be:

6.7.1 *Clean*—If in doubt about its cleanliness, use another sample container/bottle. If this is not possible, flush it out with the fluid to be sampled. Take special care to ensure that sample containers/bottles for water separability, particle count, and wear debris testing are clean.

6.7.2 *Resistance to the Material Being Sampled*—For example, fire-resistant phosphate ester fluids will dissolve certain plastics. (This includes the liner in bottle caps.) To verify the sample container/bottle's resistance, if time permits, allow the sample to stand in the sample container/bottle and observe its effects. Aluminum foil or polytetrafluoroethylene (PTFE) make good, resistant cap liners.

6.7.3 *Appropriate for Required Handling*—Sample containers/bottles with leaking tops and glass sample containers/bottles improperly protected are not suitable for shipment. Stringent packaging requirements must be followed if shipment is to be made by air.

6.7.4 *Appropriate for the Analyses Required*—As an example, some plastic sample containers/bottles may not be acceptable for flash point testing (per Test Method D92) because volatile materials may leak through the container/bottle walls. Use sample containers/bottles of either glass or polyethylene for wear debris analysis samples (to avoid material leaching).

NOTE 2—Some lubricant suppliers and commercial testing laboratories provide sample containers/bottles that meet all these requirements. Use these whenever possible. If frequent samples are taken, an adequate supply of containers/bottles should be kept.

NOTE 3—For samples intended for microbiological testing, see Practice D7464. Although the guidance provided in Practice D7464 is nominally for fuel sample collection, the procedures provided are equally applicable to turbine oils.

6.8 *Sample Labeling*—Properly label a sample container/bottle in order to track the history of a particular piece of equipment. The equipment must be identified uniquely. Labels should include the following information as appropriate:

6.8.1 Customer name,

6.8.2 Site (or plant name),

6.8.3 Location (unit number, tank number, compartment number, and so forth),

6.8.4 Equipment serial number (or other ID),

6.8.5 Oil and machine service hours,

6.8.6 Date sample taken,

6.8.7 Type of fluid sampled,

6.8.8 Sampling point,

6.8.9 System operating temperature and temperature of oil at sampling point,

6.8.10 Type of purification system (filters, centrifuge, and so forth),

6.8.11 Make-up (volume) since last sample was taken, and

6.8.12 Coolant additives.

6.9 *Sampling of New Oil Deliveries*—Thoroughly clean all sampling devices before use to avoid cross-contamination.

6.9.1 Take samples representative of the fluid being examined but obtained from the point(s) most indicative of gross contamination by debris and water, that is, just above the bottom of the drum or tanker compartment bottom.

6.9.2 When consignments of oil are in drums, sample them in accordance with Practice D4057.

6.9.3 In cases where the product is suspected of being non-uniform, sample a larger number of drums. Where contamination is suspected, there may be no alternative to sampling every drum.

6.9.4 For bulk consignments, sample each tanker compartment. If these are clear of debris and water, then the samples can be combined for subsequent laboratory analysis of the consignment. The user may decide to perform a limited number of tests on individual compartment samples; a composite sample may be tested for other properties.

6.9.5 From tanker deliveries, sample individual tanker compartments. The sample should be taken preferably from the outlet of the flexible pipework or at least from the tanker bottom valve manifold. This is important because the tanker contents can become contaminated by residual material left in the bottom valve manifold. This can occur particularly when different products are being carried in separate compartments or previous deliveries of a different product have been made to other locations without subsequent adequate cleaning and flushing. Dead leg piping should always be drained and flushed prior to sampling.

6.9.6 Bottom samples (if desired) must be collected by either a tube or thief sampler (for example, Bacon bomb). These samplers permit collection of settlings on the bottom of the containers without introducing false contamination by scraping the container lining or wall.

6.10 *Preservation of Sample and Analysis of Oil Samples*—It is generally advised to ship the oil samples immediately to the oil analysis laboratory, as ideally, oil should be analyzed within as soon as reasonably achievable after being sampled. If oil samples are stored for an extended period of time, this may result in a non-representative sample.

6.10.1 If the samples are to be retained for extended periods of time, special arrangements should be made in agreement with the oil analysis laboratory to ensure that the integrity of the sample is not compromised. The special arrangement may include storing in dark amber glass bottles in an ambient temperature area as the longer an oil sample is stored in the container/bottle, the more oxidation products will be generated.

6.10.2 Store the sample(s) away from strong light and as close to room temperature as possible.

7. Examination of New Oil on Delivery

7.1 Experience has shown the need for standardizing procedures and acceptance criteria for the sampling, examination, and acceptance of incoming supplies of lubricating oil. It is essential that personnel responsible for sampling and testing have the necessary experience and skills, and that scrupulous attention to detail be applied at all times to avoid erroneous results.

7.2 It is equally essential that all incoming supplies of oil be adequately monitored to guard against incorrect or contaminated material being delivered. This information can be used from quality control of in-coming fluid as well as future fluid reference data. The cleanliness of the delivery container should be noted; if the container is dirty on the outside, there may be particulate contamination of the oil inside. Particulate contamination can also be a problem when the lubricant comes in contact with dirty or poorly maintained equipment. Final filtration while filling equipment may be used in lieu of or in addition to particle counting. The final filter should be as fine or finer than the lubrication system filter of the equipment being filled.

7.3 Sampling of incoming supplies should be in accordance with proper sampling procedures (see Section 6).

7.4 All samples should be immediately examined for appearance.

7.5 Testing schedule guidelines for various types of new oils are provided in Table 1. With drums, tests should be completed on a composite (or bulked) sample before the oil is used in service. Individual samples should be retained until the bulk sample is passed as satisfactory.

7.6 With tanker deliveries, the additional tests to be completed before the tanker is discharged can only be judged from the risk involved by the acceptance of non-specification product. That is, can the charge be readily recovered and corrected before passing into service if the subsequent tests indicate this is necessary.

7.7 Handling and dispensing methods contribute to the required health and cleanliness specifications of the lubricant. All sources and opportunities of contamination must be avoided.

7.8 The product specifications for new oils should be clearly communicated between the user and supplier. If a sample of oil fails to meet the product specifications agreed upon by user and supplier, the sample should be retested to verify the initial test

TABLE 1 Guidelines for Sampling and Testing New Oils[A]

NOTE 1—Legend–R = Recommended; O = Optional.

NOTE 2—An infrared spectrum may be obtained for new oil to ensure that the oil is not contaminated and to provide a baseline for comparison to spectra of in-service oil.

Test	Common Methods	Gear Oils	Hydraulic Oils	Diesel Engine Oils	Turbine/ Circulating Oils	Air Compressor Oils[B]	EHC (PO_4 Esters)	EHC (Mineral Oils)
Appearance[C]	Visual	R	R		R	R	R	R
Viscosity, (40 °C)	D445	R	R	O[D]	R	R	R	R
Viscosity (100 °C)	D445			R				
Acid number	D664 D974	R	R		R	R	R	R
Water	D6304[E] D95/D7546	O[F]	O[F]	R[F]	O[F]	O[F]	R	O[F]
Antioxidants/Oxidation inhibitor	D2668				O	O		
	D6971[G]				O[G]	O[G]		
Oxidation stability (RPVOT)	D2272				O	O		
API gravity or density	D1298 D4052						O	
Flash point (COC)	D92			O				
Water separability	D1401		O		O			
Particle counts	Equipment Manufacturer's Method		R				O	O
Base number	D974 D2896 D4739			R				
Electrical resistivity	D257/D1169						R	
Elemental Analysis	D5185[H]	O	O	O	O	O	O	O

[A] Tests which are performed on in-service oils for trending purposes should also be performed on new oils for baseline information.
[B] Does not include refrigeration (chiller) oils.
[C] Appearance includes observations such as color, clarity, odor, and sediment.
[D] A diesel engine oil must be tested at 40 °C and 100 °C if it is necessary to determine whether it is the proper multi-grade oil.
[E] Test Method D6304 Procedure C is recommended for lube oils.
[F] Recommended if oil is not clear and bright.
[G] Test Method D6971 is not recommended for zinc-based lubricating oils.
[H] Another spectrochemical method such as rotating disk electrode (RDE), atomic absorption (AA), or x-ray fluorescence (XRF) may be substituted for the ICP method.

result. A resample should be taken and tested if needed to verify that the sample is representative of the shipment. If the retest or resample still fails to meet product specification, an investigation should be made to determine whether the problem is due to transportation, handling, or product formulation. The user must recognize that *typical values* are not the same as *purchase specifications*.

8. Significance of Tests

8.1 In determining the condition of the oil and equipment for continued service, important properties of in-service oils include:

8.2 *Viscosity*—Most commercial turbine oils are sold under ISO (International Standards Organization) viscosity classification system. Typical turbine fluids fall into ISO VG-32, VG-46, VG-68 and higher viscosity grades corresponding to 32 mm^2/s, 46 mm^2/s, 68 mm^2/s at 40 °C (Classification D2422). The viscosity of gear oils is classified using either ISO 3448 or D2422 viscosity classification for industrial gear oils or SAE J306 for automotive gear oils. The viscosity of diesel engine oils is classified according to SAE J300. The viscosity (for example, for multi-grade oils) can be measured at 40 °C and 100 °C in order to calculate the viscosity index and determine that the correct oil has been used. The main purpose for checking the viscosity of in-service oil is to determine if the correct oil is being used and to detect contamination. In extreme cases, in-service oils will experience a significant increase in viscosity due to thermal or oxidative degradation. Contamination can cause the viscosity to either increase or decrease, depending on the contaminant. Emulsified water and diesel fuel soot will increase the viscosity, while diesel fuel, Freon, or solvents will decrease the viscosity. Dissolved water in phosphate ester fluids can reduce the fluid viscosity slightly. Contamination from a different lubricant can change the viscosity of the oil in either direction. The method normally used for viscosity determinations is Test Method D445.

8.3 *Acid Number*—The test most used to indicate the extent of oxidation is the acid number (Test Method D664 or D974). With phosphate esters, acidity is most frequently an indication of hydrolysis. Many rust inhibitors used in lubricating oils are acidic and contribute to the acid number of the new oil. An increase in acid number above the value for new oil indicates the presence of acidic oxidation products or, less likely, contamination with acidic substances. The acid numbers determined by these two test methods are not identical and only loosely correlate; a single method should be used consistently. The use of Test Method D974 on aged phosphate ester fluids which have significantly darkened in color, and especially those which have been dyed prior to use, is not recommended.

8.4 *Water Content*—If a mineral oil is clear and bright, the amount of dissolved water present is of little significance. Most mineral oils will remain clear with up to 75 µg/g water at room temperature. Phosphate ester fluids can hold more than 1000 µg/g water at ambient temperature and still be clear and bright. The presence of water determined by screening methods (such as the hot plate splatter test for mineral oils) may be confirmed using a standard test method. Adequate lubrication cannot be maintained by an oil which contains a significant quantity of water. The analytical range for Test Method D95 is 0.05 % to 25 % and the range for Test Method D6304 is 50 µg/g to 1000 µg/g. Other methods (such as Test Methods D1533 and E1064) are available for measuring the water content in oils.

8.5 *Antioxidants Level*—The measurement of antioxidant concentration is important for monitoring the oxidation of industrial lubricants and their remaining useful life. Existing practices for measuring the concentration of phenolic (or amine) antioxidants include infrared spectrometry Test Method D2668 and linear sweep voltammetry Test Method D6971 or D6810.

8.5.1 *FTIR*—The Fourier Transform Infrared (FTIR) by Test Method D7414 is a refined infrared spectroscopy method, which can be used to monitor the remaining antioxidants blended into the oil. It can also be used to monitor the increase in oxidation products as the oil degrades. Each antioxidant is a specific chemical substance and will absorb infrared light at particular wavelengths and absorptivities; some antioxidants may not be detectable by infrared spectroscopy. (Test Method D2668 may be used for antioxidants if the wavelengths and absorptivities are known.)

8.5.2 *Linear Sweep Voltammetry*—Voltammetry is an electrochemical test technique, which can be used for measuring many antioxidant additives. The technique applies a voltage ramp through a 3 electrode sensing system and measures the current flow that occurs when the applied voltage equals the oxidation potential of the antioxidant. The potential at peak current is diagnostic for the antioxidant, and the amplitude of the peak is proportional to the concentration of the antioxidant. Antioxidants such as hindered phenols and amines can be measured by Test Method D6971 or D6810.

8.6 *Oxidation Stability (Rotary Pressure Vessel Oxidation Test, RPVOT)*—One of the most important properties of new oil containing oxidation inhibitor is its ability to resist oxidation while in service. This is accomplished by the addition of antioxidants. Measuring the concentration of the antioxidant additive or the ability of the additive to resist oxidation (as compared to the new oil) is an important factor in monitoring the degradation of industrial lubricants. The result of this measurement will offer the user a rating of *oxidation stability reserve*. Methods of measuring the resistance to oxidation are generally thermo-oxidative, catalytic degradation techniques such as Test Method D943 or D2272. Test Method D943 is generally reserved for new lubricant comparison and is too lengthy to be used for in-service monitoring. Test Method D2272, however, is widely used for in-service monitoring of oxidation stability reserve when compared to the new oil.

8.7 *Color*—New lubricating oils have colors ranging from light to moderately-dark. Darkening will occur in service but the change is usually slow over a period of time. Frequent checks for color are therefore useful as a quick on-the-spot test. A significant color change would be indicative that something has changed. A more detailed examination would be necessary to find the cause. Test Method D1500 is the standard method for defining the color of lubricants. Color darkening alone is not itself a cause for alarm (unless supported by additional tests).

8.8 *Gravity or Density*—This test only has significance with respect to contamination. As an example, phosphate ester EHC fluid cannot tolerate contamination with mineral oil. This test is of little value to determine the degree of degradation. The method normally used is Practice D1298 (hydrometer).

8.9 *Flash Point*—Lubricants must have flash points well above the minimum applicable safety requirements. Flash point is useful for detecting contamination with diesel fuel oil or low-boiling solvents. However, flash point is of little significance for determining the degree of degradation of in-service oils, since normal degradation has little effect on the flash point. The method in common use is Test Method D92 (Cleveland Open Cup).

8.10 *Insolubles*—Pentane insolubles (Test Method D893) may include oil-insoluble materials and some oil-insoluble resinous matter originating from oil or additive degradation, or both. Toluene insoluble materials may come from *(1)* external contamination, *(2)* fuel carbon and highly carbonized materials from degradation of fuel oil, and additives, and *(3)* engine wear and corrosion materials. A significant increase in insolubles indicates a potential lubrication problem. The measurement of insolubles can also assist in evaluating the performance characteristics of a new oil or in determining the cause of equipment failure.

8.11 *Insoluble Color Bodies*—This test can be a guide to end users on the formation of lubricant-generated, insoluble deposits. This test method extracts insoluble contaminants from a sample of in-service turbine oil onto a 47 mm diameter 0.45 µm thickness membrane patch. The color of the patch is then analyzed using a spectrophotometer and the results are reported as a ΔE value in the CIE LAB scale, as per Test Method A45 (Membrane Patch Colorimetry, MPC)

8.12 *Water Separability*—Water can get into lubrication systems due to oil cooler leaks, normal system breathing, and other means. Water adversely affects oils by acting with metals to catalyze oxidation. It also depletes water sensitive oil additives such as some types of rust inhibitors, and can cause rusting and corrosion. For some oils, water will settle to the bottom of the storage tank where it should be drained off as a routine operating procedure. Purification systems will also assist in removing the water. Unfortunately, if the oil has developed poor water separability properties (poor demulsibility), significant amounts of water may stay in the system and create problems. In addition to chemical effects on the oil and additives, the lubricating properties of the oil can be adversely affected. The water separability characteristics of an oil are adequately measured using Test Method D1401. This test determines the time required for significant amounts of water to separate from an oil-water emulsion. By design, water will not separate from some lubricating oils such as engine oils. The dispersant additive present in these oils disperses water as well as combustion residues, dirt, and oxidized compounds.

8.13 *Rust Evaluation*—Anti-rust protection provided by the lubricant is important for certain systems. Protection is required in areas of fluid flow, for surfaces covered by static drops of water, and for areas which are only occasionally splashed by the lubricant. New oil containing an anti-rust inhibitor additive must meet test requirements such as Test Method D665. In service, this additive can become depleted by *(1)* performing its proper function, *(2)* by removal with water, *(3)* by adsorption on wear particles and other solid debris, or *(4)* by chemical reaction with contaminants. In exceptional circumstances where alkaline or polluted water enters the system, additive removal will be much more rapid. Test Method D665 Procedure A (distilled water rust test) is usually adequate for determining a satisfactory level of anti-rust inhibitor for inland equipment. For marine usage, Test Method D665 Procedure B (seawater rust test) is recommended.

8.14 *Foaming Characteristics*—Foaming characteristics are measured by Test Method D892 which indicates both the tendency of the oil to foam and the stability of the foam after it is generated. This test may be useful in troubleshooting oil foaming problems in equipment. System foaming problems have three possible origins.

8.14.1 *Mechanical*—The system design can have a major impact on the foaming tendency of a lubricant.

8.14.2 *Antifoam Additive Depletion*—Anti-foam agents may be removed mechanically (due to fine filtration or centrifuging) because they are dispersed and not dissolved in the oil. They are known to absorb to filter fibers. They may also become less effective over time due to mechanical shearing.

8.14.3 *Contamination*—An attempt should be made to identify and remove the contaminants. In cases where this cannot be done adequately, this may be corrected with an addition of defoamant. This should only be carried out with advice from the lubricant supplier or qualified researcher and only after confirming performance on lab scale before treating the entire system.

8.15 *Air Release Properties*—Agitation of lubricating oil with air in equipment such as bearings, couplings, gears, pumps, and oil return lines may produce a dispersion of finely divided air bubbles in the oil. If the residence time in the reservoir is too short to allow the air bubbles to rise to the oil surface, a mixture of air and oil will circulate through the lubricating oil system. This may result in an inability to maintain oil pressure (particularly with centrifugal pumps), incomplete oil films in bearings and gears, and poor hydraulic system performance or failure. The time required for air entrained in the oil (in Test Method D3427) to reduce in volume to 0.2 % is recorded as the air bubble separation time.

8.16 *Base Number*—New and used petroleum products (especially diesel engine lubricants) can contain basic additives. The relative amount of these materials can be determined by titrating with acids. This *base number* is a measure of the amount of basic substances in the oil and, hence, is a measure of additive depletion. Condemning limits must be empirically established. The base number methods do not provide equivalent test results; one method should be used consistently.

8.17 *Chlorine Content*—The chlorine content in phosphate ester EHC fluids can be determined via microcoulometry. Excessive chloride ion can cause electrochemical corrosion since the fluid flows through small openings at a rapid velocity.

8.18 *Resistivity*—Phosphate ester EHC fluids with low electrical resistivity can cause servo valve erosion.

8.19 *Mineral Oil Content*—The mineral oil content in phosphate ester EHC fluids must be minimized to preserve the fire-resistant properties.

8.20 *Glycol Content*—Leakage of glycol-based antifreeze into the crankcase is serious because the coolant tends to interfere with the lubricant and its ability to lubricate; it also promotes sludging. Ethylene glycol present in the coolant can increase varnish deposit formation in the crankcase due to glycol oxidation and the interaction between glycol and lubricant. Lubricant displacement, sludging, and deposit formation all lead to engine malfunction and possible seizure. It is important to detect glycol-base coolant contamination at low levels because early detection enables corrective measures to be taken to prevent leaking coolant from accumulating and seriously damaging the diesel engine.

8.21 *Fuel Dilution*—Some fuel dilution of the diesel engine oil may take place during normal operation. However, excessive fuel dilution is of concern in terms of possible performance problems.

8.22 *Particle Counts*—The most deleterious solid contaminants found in oil systems are those left behind when the system is constructed and installed or when it is opened for maintenance and repair. The need for proper cleaning and flushing of new or repaired oil systems is emphasized. In operation, there are few opportunities for solids to enter the lube oil system, although in very dusty areas where units may be outdoors, some solids can enter through improperly installed or operating vents as well as seals and air intakes. During operation, the equipment begins to accumulate significant amounts of particulates. Some may enter the system through the makeup oil when it is added. Fly ash may be drawn in with the air at bearing shaft seals. Other contaminants may be abrasive degradation and corrosion products developed in the system. Whatever the source, the presence of abrasive solids in the oil cannot be tolerated since they will promote scoring and damage to bearings and journals as well as causing malfunction and sticking of control mechanisms. These must be removed by the use of filters or centrifuge, or both. When the amount of makeup is low and the various filters and purifiers are operating satisfactorily, abrasive solids are generally removed before any damage is done. In a properly maintained system the particulate level presents no problem. Cleanliness of the system oil can be determined by gravimetric means (Practice F311 or Test Methods F312) or by particle counting (for example, Test Method D7647), the latter normally by means of electronic particle counters. Cleanliness levels can be represented by classification systems such as ISO 4406:1999. ISO 4406:1999 uses a numeric code to reference the number of particles larger than 4 μm/mL, 6 μm/mL, and 14 μm/mL of oil. ISO 4406:1999 assigns integer values to denote a range of particles whose upper limit doubles with each successive number. Desired cleanliness levels are sometimes designated by the equipment manufacturer or user. If a cleanliness level is not specified by the manufacturer, cleanliness in the range from ISO 16/14/11 to 18/16/13 (ISO 4406:1999) is usually considered satisfactory.

8.23 *Wear Particle Concentration (WPC)*—It has been shown that the separation and measurement of large and small wear particles is beneficial in the detection and diagnosis of related machine wear regimes. Techniques used to measure WPC should not be considered to be particle counts, rather a measurement of specific wear metal concentrations. Caution should be used when evaluating results since separation and measurement techniques may be alloy-specific. The wear particle concentration for a particular piece of equipment is monitored over time in order to detect a sudden increase in the wear trend and reduce the likelihood of catastrophic failure.

8.24 *Wear Debris Analysis (WDA)*—A microscopic analysis of wear debris can be used to describe a wear condition as normal rubbing wear, severe sliding wear, cutting wear, gear wear, or bearing wear. In addition, this debris analysis can reveal fibers, sand/dirt, lubricant degradation residue, red oxides, black oxides, and ferrous spheres. A rating can be given to each type of wear condition which is a combination of the size of the wear debris and the concentration of particles of the same type.

8.25 *Elemental Analysis*—Emission spectroscopy can be used to analyze for elements found in used lubricating oil. This analysis is generally limited to dissolved materials or particles smaller than about 8 μ. The elements found derive from wear debris, additives, and contamination. (See 9.5.)

8.25.1 Some wear on metal parts can be considered to be normal (although not desirable). Large amounts of metal contaminants usually indicate a serious machine problem. Since different machine parts are made from different metals, the presence of particular metals indicate which components are wearing. When a machine has been sampled several times (or when multiple machines of the same type have been tested), an evaluation is made to determine whether the metal concentrations are outside of the normal range. Samples from a piece of equipment that have metal concentrations increasing at an unusually high rate or outside of the normal range indicate that the equipment may have a problem. When no historical data is available, there is a chance that not enough significance will be given to a particular wear metal and its concentration.

8.25.2 Certain elements which are found as lubricant oil additives can also be analyzed to ensure that the appropriate additives are present and that there are no other inorganic additives which indicate that cross-contamination has occurred. An analysis should be performed on unused oil in order to establish a baseline for future comparisons.

8.25.3 Contaminants (such as dirt and water) in the oil can be carried throughout the machine and cause severe wear or corrosion. The presence of dirt can be detected by the presence of silicon (or aluminum). Inorganic constituents of fresh and treated water may be detected using elemental analysis. Examples of these may be: calcium from untreated water, sodium and magnesium from sea water, and potassium, sodium or