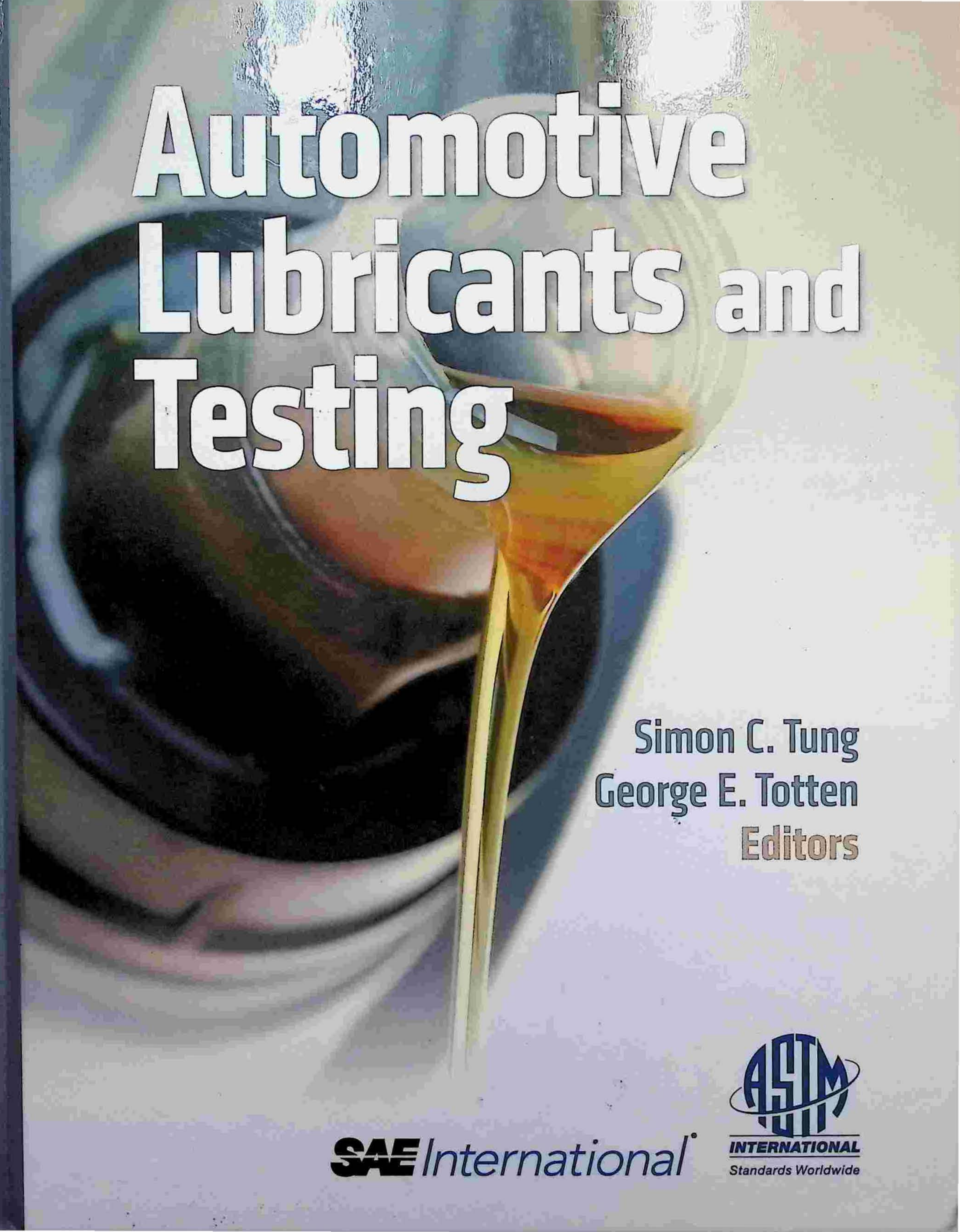

Automotive Lubricants and Testing

Simon C. Tung
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Editors

SAE International

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22

Analysis of In-Service Automotive Engine Oils

Jim C. Fitch

22.1 INTRODUCTION

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Although there are a few isolated exceptions, condition monitoring of passenger car motor oils (PCMOs) has not yet emerged as a strong market. There are several understandable reasons for this. One is the fact that most car owners are not interested in paying a premium to extend engine life. Most car owners seem to be satisfied with the current engine life expectancy. This is evidenced by the fact that less than 10 % of PCMOs in use are synthetic formulations despite their widely promoted benefits.

Unlike commercial and industrial applications, in which machine owners often run equipment to their end of useful life, car owners are more commonly enticed to sell earlier for newer models. After all, why invest in engine life extension when the benefit of the investment would only be gained by the next owner of the vehicle?

Sampling is another impediment. Automobiles are not fitted with convenient oil sampling valves, nor are these valves easy to retrofit on engines. The only practical alternative is to obtain a sample from the dipstick port by drop-tube vacuum sampling or from the oil pan drain port. Neither of these locations is suitable for obtaining a representative sample.

The other factor is the cost and turnaround time of getting the data. Although laboratory automation has increasingly enabled basic tests to be performed quickly and with minimal cost of labor, routine oil analysis is still expensive for personal car owners. Some instruments are actually an integration of several conventional oil analysis sensors and often include viscometry, molecular spectroscopy, and atomic spectroscopy, typically with no needed glassware or sample preparation steps. So too, many new onboard sensors have been introduced that monitor key oil properties in real time. They displace the need for oil sampling and can alert the car owner to the optimal timing of an oil change or the presence of aberrant oil properties and wear metals.

22.2 OIL SAMPLING METHODS

Oil sampling is a critical component of a high-quality and effective oil analysis program regardless of the machine involved. Errors in obtaining a representative sample impair all further oil analysis efforts. There are two primary goals in obtaining a representative oil sample [1,2]:

1. *Maximize data density:* Samples should be taken in such way that meaningful properties of the oil can be extracted with sufficient discriminant validity; restated, we want the most meaningful information per millilitre of oil possible. This information relates to such properties as cleanliness and dryness of the oil, depletion of additives, and the presence of wear particles being generated by the engine. For example, sampling oil from a cold engine would fail to capture a representative concentration of particles, sludge, water, and other contaminants prone to stratification by gravity and time.
2. *Minimize data disturbance:* Samples should be extracted in such a way that the concentration of information is uniform; consistent; and unaltered by the sampling process, hardware used, or location. For example, it is important to make sure that the sample does not become contaminated during the sampling process. This can occur by using dirty sample bottles, unclean or unflushed sampling valves, and other exposures that alter the target properties of the sample.

To ensure good data density and minimum data disturbance in oil sampling, one should consider the following factors, each of which is discussed in detail later in the chapter [1,2]:

- *Sampling location:* In engines, live zone sampling will produce the best results. Sampling from dipstick ports or oil pan drain ports should be avoided.
- *Sampling procedure:* The quality of the procedure by which a sample is drawn is critical to the success of oil analysis.
- *Sampling hardware:* The hardware used (valves, pumps, probes, tubing, pressure regulators, etc.) to extract the sample should not disturb sample quality but should aid it. It should be clean, easy to use, rugged, and cost-effective.
- *Sample bottle:* The type, size, and cleanliness of the oil sample bottle help to ensure that a representative sample is achieved.

22.2.1 Sampling from Oil Pressure Line

When a sample is taken from a line in a circulating system, it is referred to as a live zone sample. On an automobile engine, the best location for obtaining such a sample is on the pressure line between the pump and the filter (Figure 22.1). As previously noted, for most engine types, this is not an easy option for locating a sampling valve,

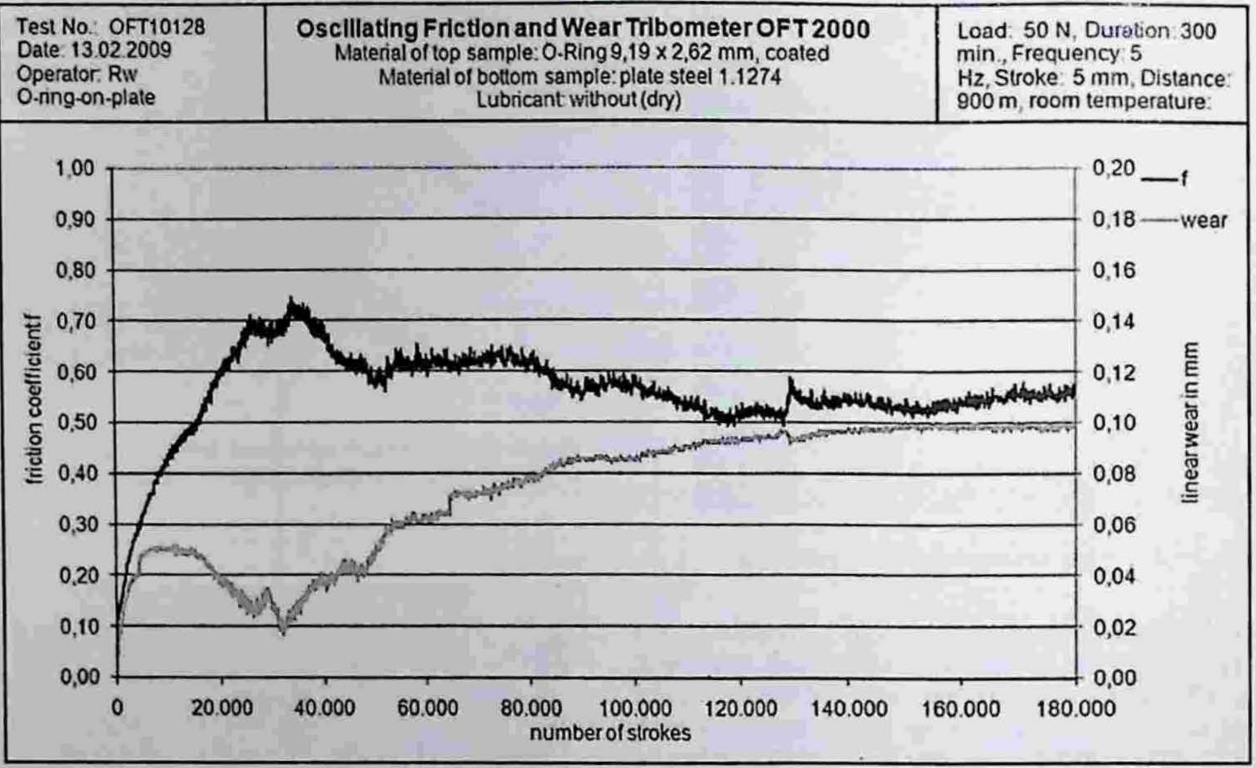

Figure 21.51—Development of friction coefficient and linear wear over number of strokes in oscillating model simulation of an o-ring-seal against a steel plate.

21.7 CONCLUSIONS

The tribology of precision systems in automotive applications differs significantly from the teachings of classic mechanical engineering in some areas. The small size of the parts amplifies the effects of roughness, bearing geometry, and bearing design. The often high-power density of precision assemblies also creates thermal problems, especially when polymer materials are used. The very small volumes of the lubricants used and their mutual interactions with the friction materials assume tremendous significance and can lead to damage in production parts. Loss of lubricant by migration is a typical problem that generally only occurs in precision mechanical systems.

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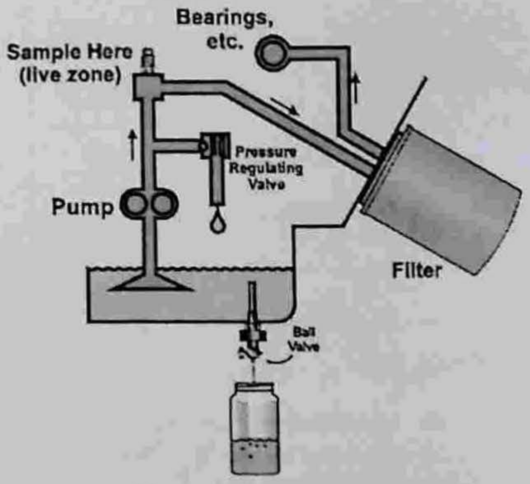


Figure 22.1—Sampling location on an engine wet-sump circulating system.

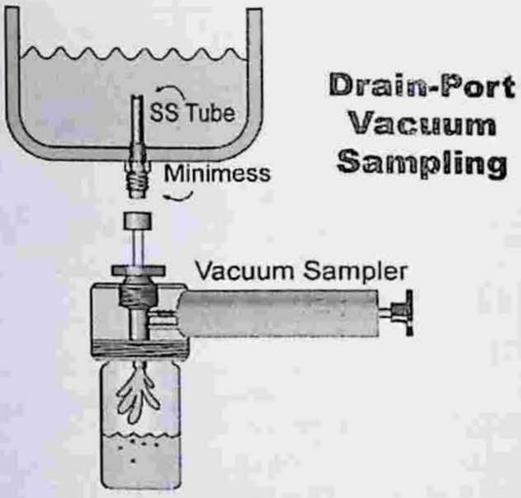


Figure 22.2—Drain port sampling aided by a vacuum pump.

although with special hardware and adapters such valves can be installed successfully. There are measures that can be taken during the sampling process that improve the quality and effectiveness of the live zone sample. The following is a summary of recommended sampling practices [1,2]:

1. Sample from turbulent zones where the fluid is moving and the oil is well mixed (i.e., from circulating fluid).
2. Sample after the oil has completed its primary function(s). In an engine, the oil that lubricates cylinders, gearing, and bearings falls by gravity back to the sump and is quickly picked up by the suction strainer and pump. Contaminants and wear debris introduced by the engine are infused in this fluid. Sampling from a valve between the pump and the filter during engine operation enables a live zone sample to be obtained.
3. Sample engines during typical working conditions and while the engine is running and the fluid is hot. Try not to sample after an oil change, filter change, after long-idle times, or at some time when the fluid would not represent typical conditions.

22.2.2 Drain Port Sampling

The most basic method for sampling is to remove the drain plug from the bottom of the sump, allowing an amount of fluid to flow into the sample bottle. For many reasons, this is not an ideal sampling method or location. Most important is the fact that bottom sediment, debris, and particles (including water) enter the bottle in concentrations that are not representative of what is experienced near or around where the oil lubricates the engine. Because of this stratification, the drain port sampling method should be avoided if at all possible (except for the purpose of inspecting for free water, coolant, and sediment).

Drain port sampling can be greatly improved by using a short length of stainless steel tubing, extending inward and up into the active moving zone of the sump (Figure 22.1) [1–3]. In many cases, this ball valve and tube assembly can be threaded into the drain port without interfering with the use of the plug for periodic draining of the oil.

A third option is called drain port vacuum sampling. With this method, a minimess or similar valve is installed as previously described using an inward-directed pilot tube, but instead of fluid passing into a sample bottle by gravity, it is assisted by a vacuum sampler (Figure 22.2) [1–3]. This is particularly helpful when the oil is highly viscous (cold temperature) or otherwise difficult to sample by gravity alone. During the sampling process, the connector on the end of the plastic tube of the vacuum pump is threaded onto the minimess valve. A vacuum is produced by the pump pulling oil downward into the sample bottle.

22.2.3 Drop-Tube Vacuum Sampling

One of the most common methods for sampling an engine sump is to use the drop-tube vacuum sample method. With this method, a tube is inserted through the dipstick port and lowered into the sump cavity. For the best results, the tube should be cut approximately 10 in. longer than the dipstick and inserted approximately 0.24 in. shorter than the dipstick [3]. This sampling method has several drawbacks and should be avoided if other sampling methods, as previously described, can be used instead. The following is a summary of the risks and problems associated with drop-tube vacuum sampling:

- *Tube location:* A tube that is inserted into a dipstick port is extremely difficult to control. The tube's final resting place is hard to predict, resulting in samples being taken from different locations each time. There is always a risk of the tube actually going all of the way to the bottom of the sump where debris and sediment are picked up (not representative of circulating fluid).
- *Drop-tube contamination:* There is considerable concern that the tube will scoop up debris from the sides of the casing as it is being inserted. Also, the tube itself may be contaminated because of poor cleanliness control during handling and storage.
- *Large flush volume:* The drop-tube method substantially increases the volume of fluid that must be flushed through the sampling hardware (in advance) to obtain a representative sample.
- *Machine intrusion:* The drop-tube method is intrusive. The engine must be invaded to draw a sample. This intrusion introduces the risk of contamination, and there is always the concern that the engine might not be properly restored to run-ready condition before startup.

22.2.4 Sampling Bottles and Hardware

An important factor in obtaining a representative sample is to make sure the sampling hardware is completely flushed before obtaining the sample. This is usually accomplished using a spare bottle to catch the purged fluid. It is important to flush the dead sampling pathway volume five to ten times before obtaining the sample. All hardware that the oil comes into contact with is considered dead volume and must be flushed, including

- Sampling ports, valves, and adapters,
- Probe-on sampling devices,
- Adapters for using vacuum sample extraction pumps,
- Plastic tubing used for vacuum pumps (this tubing should not be reused to avoid cross-contamination between oils).

There is an assortment of sampling bottles that are commonly used in oil analysis. An appropriate bottle needs to be selected for the application and the tests that are planned. The following parameters should be considered when selecting sample bottles [1–3]:

- *Size:* There are several different sizes of sample bottles that are available. They vary from 50-mL (or ~2 oz of fluid) to 200-mL bottles. The most common bottle size is 100 mL, which is generally suitable for automotive engine oil analysis.
- Another consideration in selecting the bottle size is the fact that the entire volume of the bottle should not be filled with oil during the sampling process. Only a portion of the sample bottle should be filled. The unfilled portion, called the ullage, is needed to allow proper fluid agitation by the laboratory to restore even distribution of suspended particles and water in the sample. It is advised to only fill the bottle approximately three-quarters full.
- *Material:* Modern sample bottles are made of polyethylene terephthalate (PET) plastic because of (1) their chemical compatibility with most base oils and additives, (2) the fact that they are clear, (3) their strength (fracture resistance), (4) their wide availability, and (5) their low cost. The primary disadvantage of using PET is the risk that the sample bottles will melt or become soft when sampling high-temperature engine oils (above 200°F).
- *Cleanliness:* One of the most important considerations in selecting a sample bottle is to make sure it is sufficiently clean [4,5]. The bottle's required cleanliness level should be determined in advance. The bottle supplier should provide a certificate of cleanliness that is based on random testing of the bottles per ISO 3722. Bottles can be classified according to their contribution to the particle count into the following cleanliness categories:
  - *Clean:* Fewer than 100 particles, greater than 10 µm/mL fluid.
  - *Super clean:* Fewer than 10 particles, greater than 10 µm/mL fluid.
  - *Ultraclean:* Less than 1 particle, greater than 10 µm/mL fluid.

The selection of the bottle cleanliness depends heavily on the target cleanliness of the fluids being sampled. Engine oils sampled for particle counting should generally use bottles 10 times cleaner than the fluid target cleanliness for the same volume. A clean or super clean sample bottle is typically sufficient for most automotive oil analyses.

22.2.5 Oil Sampling Frequency

The objective of oil analysis, like engine condition monitoring in general, is incipient (early) detection of root causes or impending failure conditions. The engine and oil will generally give off silent alarms when problems first occur. In time, as the severity increases, these alarms are no longer silent and even the most rudimentary condition monitoring methods can reveal the problem. Of course, at this point, a great deal of damage may have already occurred. It is also likely too late to arrest the problem on the run; the engine may require a major repair or need to be rebuilt.

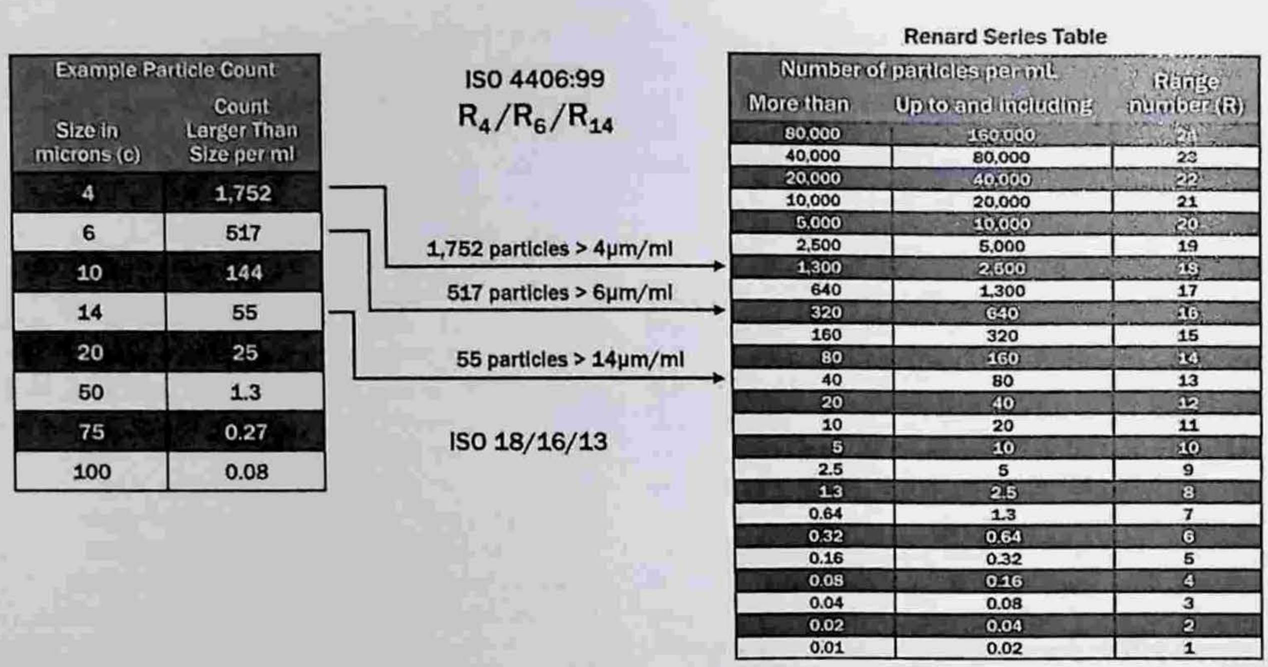
One of the greater benefits of oil analysis is its sensitivity to these silent alarms and the detection of incipient failures and faults. The method of doing this successfully is discussed further in this chapter. Scheduled sampling intervals are common in oil analysis. The sample frequency is typically just before the drain intervals for gasoline and diesel engines [3]. However, it is important that the interval be consistently maintained based on engine run time (e.g., hours or kilometres). Loosely regulated drain intervals (and sampling intervals) will result in erratic data behavior (untrendable) unless mathematically normalized.

22.3 OIL TESTING AND ANALYSIS

In used oil analysis, testing plans are defined based on the specific questions that need to be answered about the lubricant, the engine, and the operating conditions. Because there are many possible tests, these questions need to be given careful consideration to optimize results and keep costs streamlined. Because lubricant testing involves specific costs to the vehicle owner, the value gained in getting specific answers to the questions must be weighed against these costs. The following are examples of questions that may need to be answered [3]:

- *Lubricant type/quality questions:*
  - Is the specified lubricant being used?
  - Have two incompatible lubricants been mixed?
- *Lubricant health/condition questions:*
  - Have any critical additives become depleted or impaired?
  - Has the base oil been damaged by oxidation, hydrolysis, or thermal degradation?
  - Has the oil's viscosity changed because of evaporation, shear, contamination, or various chemical reactions?
  - What is the residual life of the lubricant?
- *Lubricant contamination questions:*
  - Has the lubricant been cross-contaminated (mixed) with other vehicle fluids?
  - Has the lubricant become contaminated with fuel, soot, dirt, water, or coolant?
- *Wear and fault detection questions:*
  - Is there incipient evidence of abnormal wear?
  - Which engine surface is generating the wear?
  - What is the tribological, chemical, or mechanical cause of the wear?
  - How severe (advanced) or threatening is the wear?
- *Maintenance, operations, and commissioning questions:*
  - Does a filter need to be changed?
  - Is there internal or external leakage?
  - Is there evidence of abnormal operating temperatures, pressures, or duty cycle?
  - Has the engine been improperly repaired or rebuilt?

In its simplest and most basic form, lubricant analysis is performed to improve the quality of lubrication maintenance decisions. When well designed and implemented,



Example Particle Count	
Size in microns (c)	Count Larger Than Size per ml
4	1,752
6	517
10	144
14	55
20	25
50	1.3
75	0.27
100	0.08

Renard Series Table

Number of particles per mL		Range number (R)
More than	Up to and including	
80,000	160,000	24
40,000	80,000	23
20,000	40,000	22
10,000	20,000	21
5,000	10,000	20
2,500	5,000	19
1,300	2,600	18
640	1,300	17
320	640	16
160	320	15
80	160	14
40	80	13
20	40	12
10	20	11
5	10	10
2.5	5	9
1.3	2.5	8
0.64	1.3	7
0.32	0.64	6
0.16	0.32	5
0.08	0.16	4
0.04	0.08	3
0.02	0.04	2
0.01	0.02	1

Figure 22.3—Under ISO 4406:99, a sample is given a fluid cleanliness rating using the above table. To do this, the number of particles greater than three size ranges—4, 6, and 14 µm—are determined in the equivalent of 1 mL of sample. In the above example, the particle count distribution shown in the table on the left translates to an ISO 4406:99 rating of 18/16/13.

many of the questions listed above can be answered without excessive expense or complexity. This is best accomplished by directing the testing program around three important categories of lubricant analysis:

1. *Fluid properties analysis:* This category of oil analysis deals with the assessment of the chemical, physical, and additive properties of the oil. Its primary goals are to define the remaining useful life (RUL) of the lubricant as well as to confirm that the correct lubricant is currently in use. It can also detect improperly mixed lubricants (e.g., a motor oil and a differential gear lube).
2. *Contamination analysis:* Contaminants are foreign energy or substances that enter a lubricant and engine from the environment or are generated internally. Contamination compromises engine reliability and promotes premature lubricant failure. Oil analysis can be effective at ensuring that goal-driven targets for contamination control are maintained.
3. *Wear debris analysis:* When components wear, they generate debris in the form of small particles (typically from 1 to 100 µm in size). The lubricant is usually the first recipient of this wear debris because of its close proximity to the frictional surfaces where the debris was formed. Monitoring and analyzing the generated debris enables technologists to detect and evaluate abnormal conditions such that effective and timely maintenance decisions can be made and implemented.

22.3.1 Review of Common Used Oil Analysis

22.3.1.1 PARTICLE COUNTING

Particle counting is a common oil analysis test that reports the number of particles above specified size ranges (in micrometres) per fluid volume (usually per 1 or 100 mL). Also, particle concentration and distribution data may be expressed in terms of ISO 4406:99 Cleanliness Codes (Figure 22.3). Particles can be manually counted using optical microscopy (ISO 4407 and ASTM F312-97). In this method, an aliquot of fluid is passed through a membrane. Afterward, particles on the membrane are manually counted under a microscope. The method is similar to the patch test procedure discussed in Section 22.3.1.8. There are also commercial methods available that enable membranes to be optically scanned and digitally analyzed for particle size, count, and shape (ISO 16232-7). This method is referred to as particle micropatch imaging.

Most laboratories use automatic particle counters, which can report a particle count or ISO Code in just a couple of minutes. The two methods are laser optical (ISO 11500 and ASTM D7647) particle counters and pore-blockage (BS 3406) particle counters. Optical particle counters direct a laser light source at passing particles in the sensor cell (Figure 22.4). The amount and frequency of light blockage is measured by a photodiode. This signal is converted to particle size and count by the use of standardized calibration methods [4,6].

Because in-service engine oil is heavily loaded with soot (a combustion byproduct), the ability to use optical methods has limited application. However, a modified method using particle resuspension has been met with considerable success. This method filters the particle from the oil using a standard patch test procedure (5-µm pore size). Next, the filtered particles are backwashed into a superclean hydraulic fluid (or similar fluid). The soot particles pass through the membrane so that only those particles resuspended in the hydraulic fluid (>5 µm) are seen by the particle counter [4].

Pore-blockage particle counters use calibrated screens through which the sample flows during a test (Figure 22.5). The profile of the pressure rise or flow decay, caused by particle blockage of the screen's pores, is measured. This

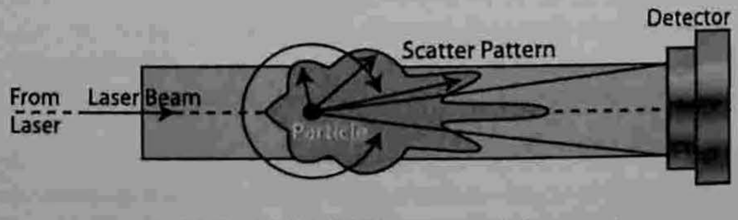


Figure 22.4—Light-blockage particle counter.

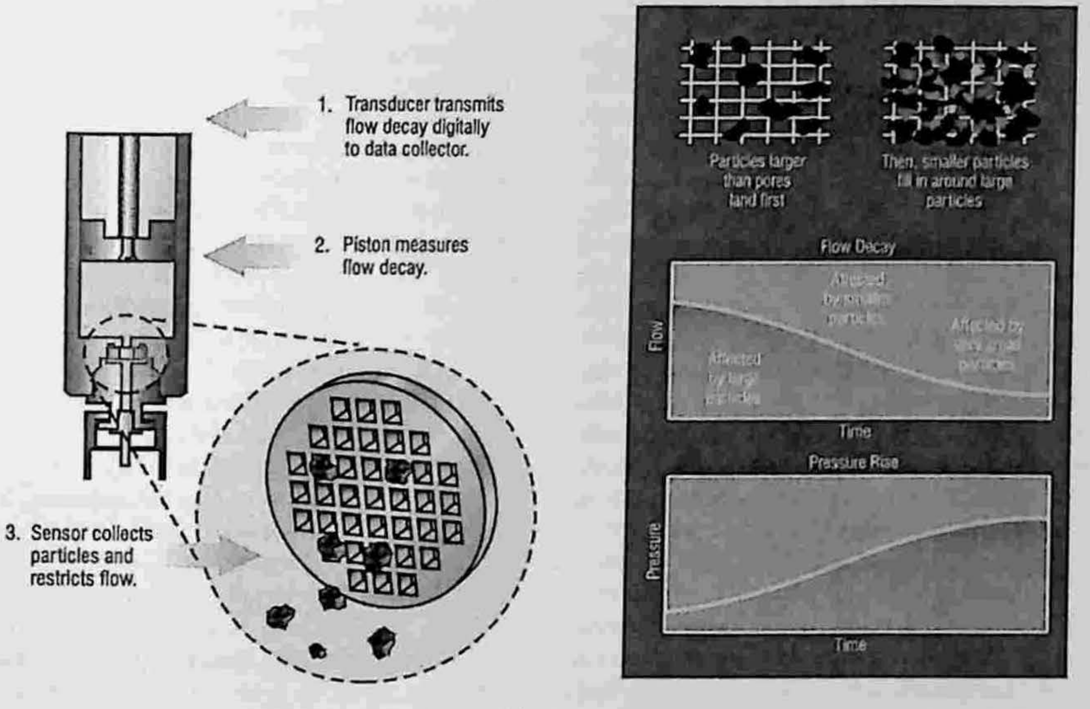


Figure 22.5—Pore-blockage particle counter.

profile is mathematically converted to an estimated particle count or ISO Code on the basis of calibration standards. Pore blockage is popular with laboratories testing engine oils because it is not hampered by soot [7,8].

Some modern optical particle counting technologies also have the ability to characterize particle shape. This is referred to as direct image particle counting (ASTM D7596). With this added information, interpretation of the source, type, and severity of the particles can be estimated [9,10].

Summary of applicable particle count standards: ISO 11171, ISO 4406, ISO 11500, ASTM D7647, ASTM D6786, FTM 3012, FTM 3010, ISO 4407, BS 3406, ISO/DIS 21018, ISO 16232-7, and ASTM D7596.

22.3.1.2 VISCOSITY

Kinematic viscosity is a measure of a fluid's internal (molecular) resistance to flow (shear) under gravitational forces. It is determined by measuring the time, in seconds, required for a fixed volume of fluid to flow a known distance by gravity through a capillary within a calibrated viscometer at a closely controlled temperature (ASTM D445 or ISO 3105). This value is converted to standard units such as centistokes (cSt) or square millimetres per second (mm^2/sec). Viscosity reporting is only relevant when the temperature at which the test was conducted is also reported (e.g., 12 cSt at 100°C).

Viscosity affects engine operation, energy losses, and the oil film thickness in bearings, cylinders, valves, cams, gearing, and other frictional zones. Therefore, its measurement and trending is critical to used engine oil analysis. Even modest changes in viscosity may adversely influence the lubricant's performance and stability, possibly causing metal-to-metal contact and accelerated wear [11]. Change in a lubricant's viscosity is also a common symptom of a host of other problems. As such, a viscosity trend excursion may be the first symptom of a far more serious problem.

The rate of viscosity change from oil oxidative degradation depends on the presence of pro-oxidation stressing agents that are in the oil. This can be sharply intensified in internal combustion engines. These include heat, water contamination, agitation, oil pressure, acidic combustion byproducts, metal particles (metal catalysts), entrained air, and degraded remnants of previously used oil. Oil oxidation causes viscosity to increase from synthesis and polymerization. Given enough time, oxidation can transform an oil into a tar-like substance.

A change in viscosity can result from a host of other root causes such as the following [3] (Figure 22.6):

- Excessive amounts of contamination such as water, fuel, glycol, and solvents;
- Hydrolysis (from water contamination) of ester-based synthetic lubricants (some API Group V base oils);
- Volatilization of the light ends of the basestock (increases the viscosity);
- Topping up with the incorrect viscosity grade; and
- Severe mechanical shearing of the oil (lowers viscosity).

Applicable viscosity standards: ASTM D445, SAE J300, ISO 3105, ASMT D6971, ASTM D6810, ASTM D7590-09, and ISO 3448.

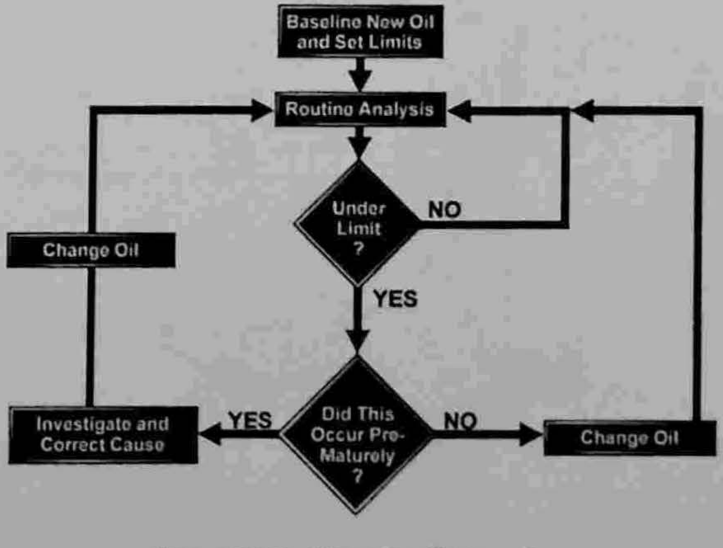


Figure 22.6—Viscosity diagnostics.

22.3.1.3 BASE NUMBER

Primarily for engine crankcase applications, the base number (BN) measures the reserve alkalinity of an oil. Engine oils are formulated with overbased additives (high alkalinity) such as calcium sulfonate, which can neutralize the acids that enter an oil from combustion blowby, base oil oxidation, and environmental contamination. Once the reserve alkalinity has depleted through normal consumption (neutralization) as the oil ages, the oil can subsequently become highly corrosive to bearings, valves, pistons, and cylinder walls as acid ingression continues.

The BN test is performed by colorimetric or potentiometric titration (usually potentiometric because of the opaqueness of used engine oil) (Figure 22.7). During the test, the alkaline oil is titrated (neutralized) with hydrochloric (HCl; preferred for used oil analysis) or perchloric acid (risk of false positives on alkalinity). Results are reported in milligrams of potassium hydroxide (KOH) per gram of oil. The result is actually a derived value that represents the volume of KOH that is required to neutralize the volume of BN reagent acid (HCl) that is required to neutralize the reserve alkalinity of the sample being tested. In this way, the units are balanced, (i.e., 1 unit of BN neutralizes 1 unit of acid number [AN]).

BN numbers trend steadily downward as the oil's reserve alkalinity is depleted by the progressive neutralization of acids from combustion and base oil oxidative. A rapid change in BN may be caused by one or more of the following [3]:

- Burning high-sulfur fuel,
- Abnormal fuel dilution,
- Poor combustion,
- Excessive blowby,
- Hot running conditions,
- Severe oxidation,
- Overextended drain interval,
- Wrong oil addition, and
- Glycol contamination.

If BN numbers change rapidly, the root cause should be determined and corrective actions should be taken.

Applicable standards: ASTM D974, ASTM D2896, ASTM D4739, and ASTM D5984-96.

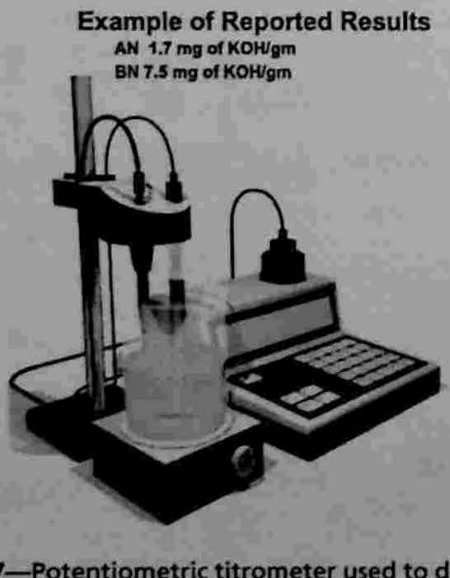


Figure 22.7—Potentiometric titrometer used to determine ANs and BNs.

22.3.1.4 ACID NUMBER

This method is primarily used for non-crankcase industrial lubricants, although many laboratories will perform BN and AN tests on gasoline and diesel engine oils. AN is a measure of the acid concentration of the oil. It does not measure acid strength (like pH). AN is a titration test method, and results are expressed as the volume (milligrams) of KOH required to neutralize the acidic components in 1 g of sample oil. The reported unit is milligrams of KOH per gram of oil. AN can be quantified by colorimetric (color change) or potentiometric (electrical voltage change) titration methods (Figure 22.7). For dark-colored engine oils, the latter method should be used.

As motor oil ages, becomes contaminated with combustion blowby, or oxidizes, small amounts of organic acids begin to accumulate in the oil causing the AN to increase. These acidic constituents will initially be neutralized by the overbase detergent and other overbase formulation constituents. For diesel engine motor oils with BNs starting at 9–10, the BN will trend downward to 5–5.5 before the AN starts to rise. By the time the BN falls to 3–3.5, the AN will have risen by 1 to 1.5 (indicating increasing corrosion risk) [3].

A high AN typically indicates the oil's useful life has expired and it needs to be changed. For mineral oils and many synthetics, an AN above 4.0 is highly corrosive, risking attack of metal surfaces leading to pitting, etching, and permanent damage. Strong acids can enter an oil from external contamination sources; these include sulfuric, nitric, HCl, hydrofluoric, and phosphoric acids. Corrosive damage risk is increased in the presence of water contamination, which strengthens the corrosive potential of acids.

A slow increase in AN over a long period of use is considered normal for certain lubricants. However, once an upper limit is reached, the oil will need to be changed. Rapid change in AN may be due to one or more of the following events:

- Severe oxidation of oil,
- Depletion of overbase additives, or
- Large makeup of an incorrect oil such as antiwear hydraulic oils or gear oils that have high baseline AN values

Applicable standards: ASTM D664, ASTM D974, ASTM D1534 (transformer oils), and ASTM D3339.

22.3.1.5 FOURIER TRANSFORM INFRARED SPECTROSCOPY

Fourier transform infrared spectroscopy (FTIR) is a method that provides a rapid means to simultaneously monitor the molecular components of multiple oil parameters. In one common instrument configuration, a fixed thickness of oil (path length) is applied to the FTIR instrument's test cell through which infrared energy is passed. Numerous oil properties, additives, and contaminants absorb infrared energy at particular infrared spectral bands (similar to frequency). A fast Fourier transform is applied to create a wavelength spectrum of attenuated (absorbed and reflected) infrared energy (attenuated total reflectance [ATR] cell) or transmitted infrared energy (transmission cell).

The spectrum of the used oil is generally compared to the baseline of the new oil for analysis to identify certain contaminants (e.g., soot, water, glycol, and fuel) and certain oil degradation products (e.g., oxides, nitrates, and sulfates)

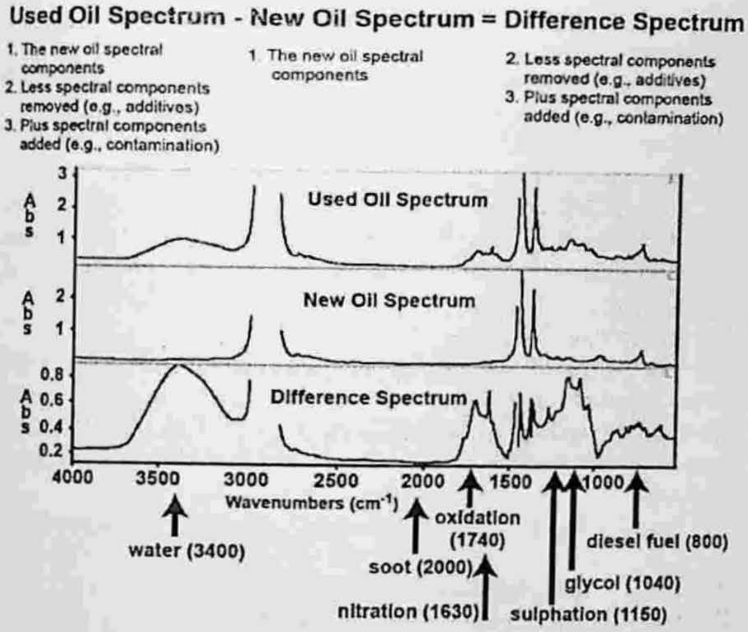


Figure 22.8—Spectral subtraction method for infrared spectroscopy.

and additives (e.g., zinc dialkyldithiophosphate [ZDDP] and phenols) (Figure 22.8) [3].

Infrared spectroscopy is unique in that it assesses the constituent components of the oil sample at the molecular level. This information is very useful when the existence of compounds such as additives and oxidation byproducts (which are often difficult to measure and trend by other means) are of interest.

Oil contamination by water, soot, glycol, incorrect make-up oils, and chemical solvents can all be monitored using FTIR. The analyst or instrument software looks for discrepancies in spectral features, or bands, at specific wavenumbers (WN or cm^{-1}) (Figure 22.9) [11–13]. Soot produces a broadband spectrum shift so a wavenumber where no other significant spectral activity occurs is selected for soot measurement (typically 2000 WNs). Wavenumbers refer to the number of spectral waves per centimetre of path length.

Overall lubricant health can also be monitored using FTIR. Oil degradation caused by oxidation and nitration results in unique wavenumber-specific absorption features (e.g., centering at 1750 and 1630 cm^{-1}). A decrease in absorption at spectral bands relating to certain additives can also be observed.

Applicable standards: ASTM D7214-07a, ASTM D7412-09, ASTM D7414-09, ASTM D7415-09, ASTM D7416-09, ASTM D7418-07, and ASTM E2412-04.

22.3.1.6 FERROUS DENSITY

A significant increase in the population of large (>5 μm) ferrous particles usually suggests the presence of an abnormal wear condition and should serve as a warning of impending engine failure. Several methods are available for determining the concentration of large ferrous debris. The severity of the wearing event is generally proportional to step changes in generation rate of large particles. The ferrous density instruments report results in different measurement units established by the instruments' manufacturer. These instruments are not currently standardized, although they are widely used.

Contamination, poor lubrication, and adverse mechanical conditions are the usual causes of high ferromagnetic particles. In automotive engines as well as most other types of machinery, at least one surface in a frictional pair is typically ferrous (iron or steel), and it is usually the surface most critical to reliability. For this reason the monitoring of ferrous density in used lubricants can provide valuable engine health information and an early warning to failure. The need for ferrous density readings is further magnified by the fact that elemental analysis becomes less accurate with larger size particles (>5 μm), which is usually the critical size range in monitoring and detecting impending failure [3].

Some ferrous density instruments use powerful rare-earth magnets to separate ferromagnetic particles from all other particles. Once these particles are separated, their density or concentration can be estimated using various means (optical, particle count method, magnetic flux method, Hall effect, etc.) [14]. It should be noted that some ferrous particles are generally not magnetic. These include corrosion debris (red-iron oxide) and high-alloy stainless steel. Figure 22.10 shows the measurement units for direct reading ferrography, which is perhaps the most commonly used ferrous density instrument by commercial laboratories.

Parameter	Wavenumber (cm^{-1})
Oxidation:	
• Mineral oil	1750
• Organic ester	3540
• Phosphate ester	815
Sulfation	1150
Nitration	1630
Soot	2000
Water:	
• Mineral oil	3400
• Organic ester	3625
Glycol	880, 3400 1040, 1080
Fuel dilution:	
• Diesel	800
• Gasoline	750
• Jet fuel	795-815
Phenol oxidation inhibitors	3650
ZDDP antiwear/antioxidant	980

Figure 22.9—Common spectral search areas used in infrared spectroscopy.

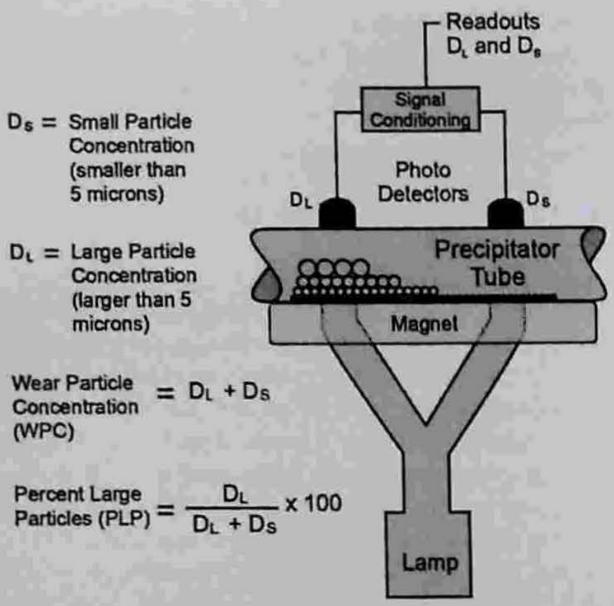


Figure 22.10—Direct reading ferrography.

An increase in the generation of ferrous particles can be brought about by several factors, including

- Load changes (e.g., hauling conditions, mountain terrain);
- Eccentric shaft loads caused by imbalance or misalignment;
- Insufficient lubrication caused by wrong lubricant, starvation, additive depletion, or lubricant degradation;
- Contamination by particles, water, air, coolant, fuel, solvents, etc.; and
- Component fatigue and wear.

The plot in Figure 22.11 shows three engines with different abnormal wear conditions as measured with direct reading ferrography [15]. The wear index is a cumulative score for the severity (large particles) and density (number of particles) of ferromagnetic wear debris.

Applicable standards: Specific to equipment supplier.

22.3.1.7 MICROSCOPIC CONTAMINANT AND WEAR PARTICLE IDENTIFICATION

When abnormal wear metals have been identified by other methods, including particle counting, elemental spectroscopy, or ferrous density analysis, a common and important

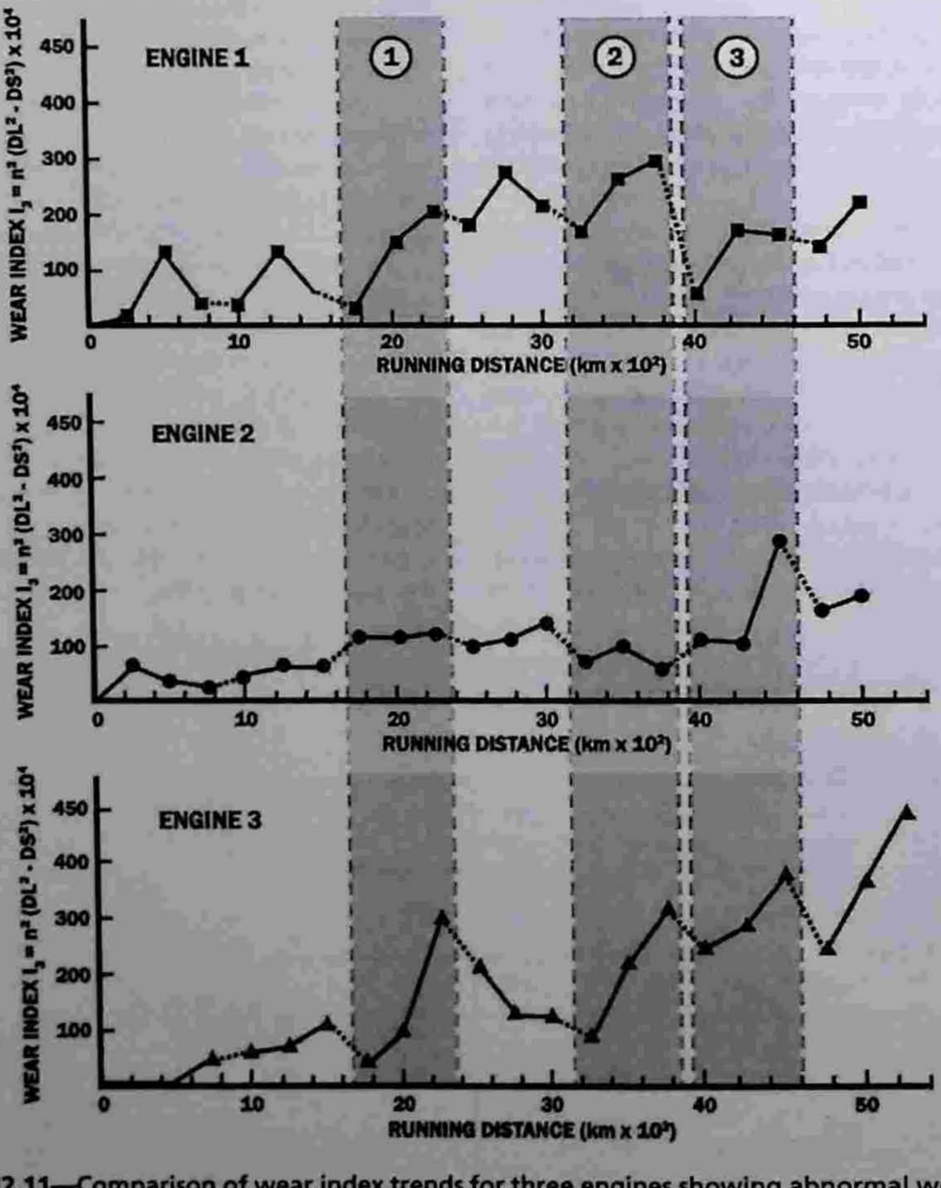


Figure 22.11—Comparison of wear index trends for three engines showing abnormal wear of the following components (1) cam and tappet wear (engine 1); (2) valve spring, piston pins, and tappets wear (engines 1 and 2); and valve spring wear (engines 1 and 3).

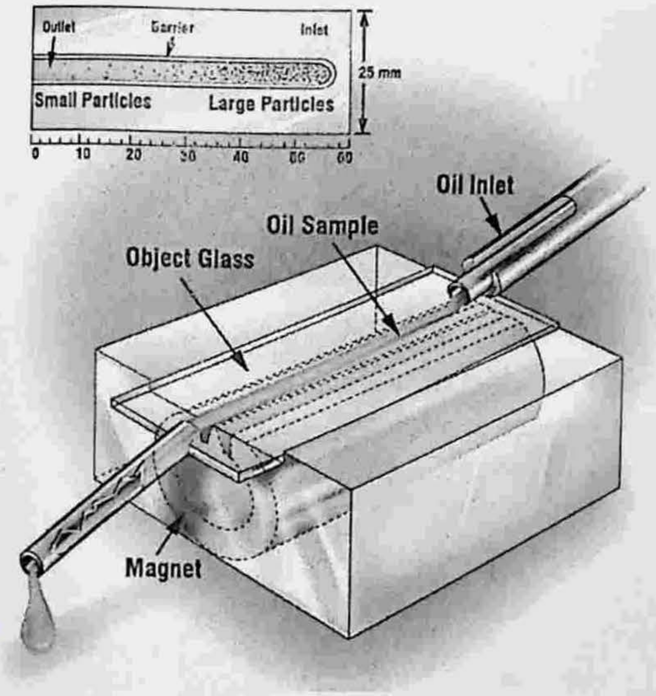


Figure 22.12—Particles are pinned down to the ferrogram by a high-gradient magnetic field.

exception test that should follow is microscopic particle examination and identification. The most common version of the procedure is referred to as analytical ferrography [14]. Analytical ferrography involves the analysis of debris deposited onto a ferrogram slide (aided by a magnet) or alternatively a filtergram membrane (Figure 22.12).

Using an optical microscope (typically bichromatic with bottom and top lighting), the particle morphology (shape), color, size, reflectivity, surface appearance, edge detail, angularity, and relative concentration provide the analyst with clues about the nature, severity, and root cause of the contaminant ingression or wear problem [14]. Scanning electron microscopy can also be used to examine particles as well as their elemental composition using an energy-dispersive spectroscopy feature. Ferrograms can be heated on a hot plate to 330°C to help reveal the composition of particles. The heat can alter the color of the particle by forming an oxide film that can help reveal its composition. The following describes how some particles change in color from heat exposure according to composition differences [15]:

- *Copper alloy (bushings):* Yellow before and after heat treatment.
- *Aluminum alloy (piston, bearings):* Heat treat does not affect.
- *Stainless steel:* Slight straw color when heated.
- *Lead/tin babbitt (bearings):* White color, mottled blue, purple when heated.
- *Copper/lead babbitt (bearings):* Yellow color; becomes yellow with blue/purple mottling when heated.
- *Low-alloy steel (connecting rod, valve springs):* Exposed to progressive heating, light tan → straw color → blue/violet → pale blue.
- *Cast iron (cylinders, crankshaft, camshaft):* yellow-brown.

Although largely a qualitative technique, the analyst typically reports the presence and concentration of wear particles, friction polymers, dirt and sand, fibers, and other solid contaminants on a scale of 1–10 or 1–100 to describe the severity or concentration of the debris field. Descriptive text and photomicrographs usually accompany the enumerated values to clarify conclusions and recommend corrective measures.

It is important to determine the root cause of impending engine failure and abnormal wear problems so they can be eliminated to preclude recurrence. By combining information from analytical ferrography with other lubricant analysis and maintenance technology evaluations, the analyst attempts to answer the following questions:

- Where in the engine does the contaminant or wear debris originate?
- What is causing it?
- How severe or threatening is it (residual life)?
- Can the condition be mitigated or arrested without downtime or production loss?

Applicable standards: ASTM D7684 (patch ferrography) and ISO 16232 7&8.

22.3.1.8 *Patch Test*

This method is similar to the microscopic contaminant and wear particle identification previously described. Using a vacuum pump (either manual or electric), a small amount of sample is pulled through a porous membrane (typically ~5 μm) to enable suspended particles to become deposited on the membrane's surface. A solvent is used to rinse any residual oil from the surface of the membrane. Afterward, the membrane can be visually inspected for overall particle density and color. If an abnormal debris field is encountered, the membrane can be placed under a top-lit microscope for detailed analysis and characterization of the particles (Figures 22.13 and 22.14) [3].

Many analysts estimate the fluid's ISO Code (ISO 4406:99) on the basis of the overall appearance of particles, sometimes using comparator standards. One such comparator standard used in the patch analysis of aviation fuels is ASTM D2276, which is also easily adapted to lubricants. However, unlike optical particle counters, patch testing allows particle shape, color, edge detail, and organic particles to be inspected. Unlike analytical ferrography, patch testing is a relatively inexpensive screening procedure and can easily be performed in the field.

Applicable standards: ASTM D7670 and SAE ARP 4285.

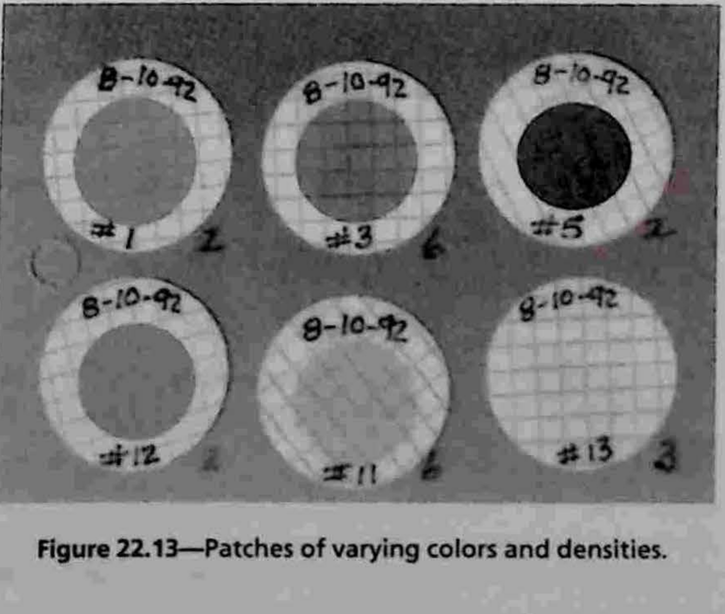


Figure 22.13—Patches of varying colors and densities.

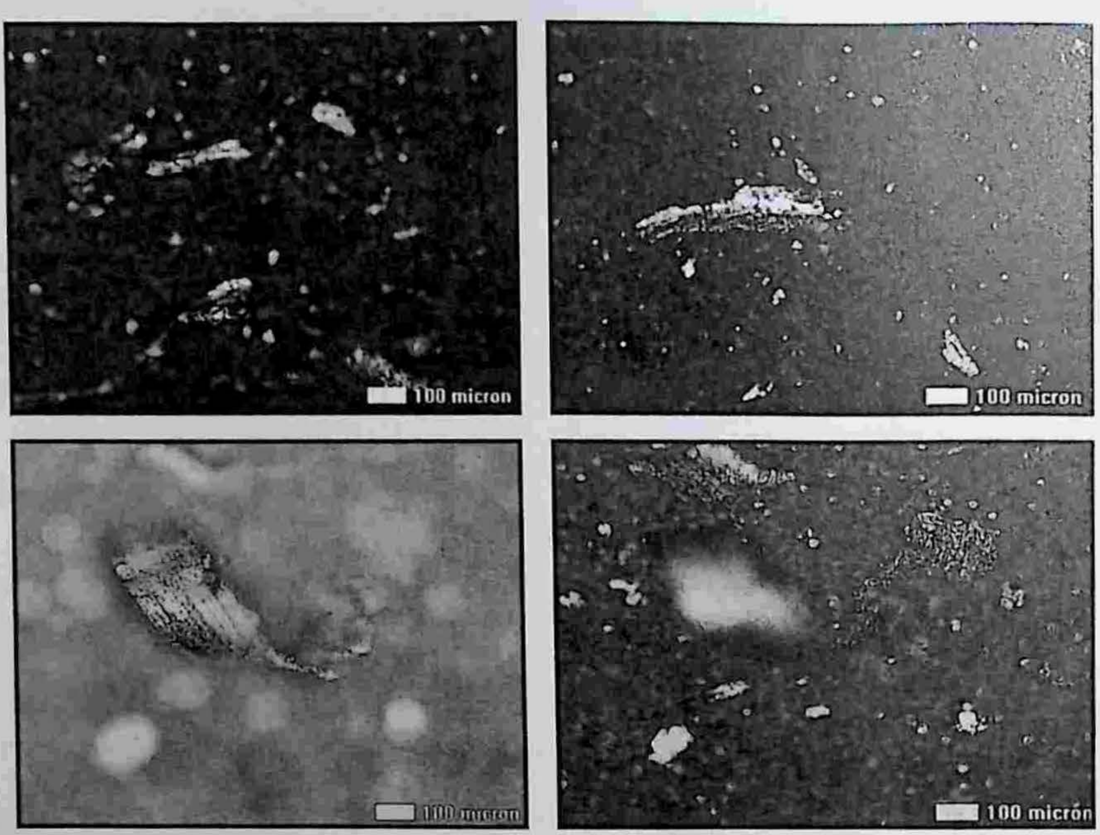


Figure 22.14—Patch images under magnification.

22.3.1.9 WATER CONTENT BY KARL FISCHER TEST

The Karl Fischer test is a titration method to measure the water concentration in oil. It is usually performed after the sample is screened with the hot plate "crackle test" or by FTIR. Karl Fischer reports water concentration in percentage or parts per million (ppm) of the "total" water (free, emulsified, and dissolved) in the oil sample.

In the volumetric test, the oil is titrated with a standard iodine-containing Fischer reagent to an electrometric end point. The accuracy of the test is affected by the presence of sulfur-containing additives such as detergents, antiwear agents, rust inhibitors, and antiscuffing agents. Many laboratories prefer the coulometric method combined with co-distillation to eliminate the risk of additive interferences [16].

Water corrodes iron and steel surfaces, accelerates corrosion, depletes and degrades additives, promotes base oil oxidation, and alters lubricant film strength. Large amounts of water form persistent emulsions in motor oils that join with insoluble oxidation products to form sludge and may significantly impair engine reliability. In addition, free water may cause the formation of hard, brittle deposits on bearing surfaces.

The following are common causes of elevated water content in engine oil:

- Oil cooler leak,
- Atmospheric condensation,
- Intermittent running conditions, and
- Cold-temperature service.

Applicable Karl Fischer standards: ASTM D1744-volumentric (withdrawn but still in active use) and ASTM D6304-98-coulometric.

22.3.1.10 ELEMENTAL SPECTROSCOPY

Elemental spectroscopy quantifies the presence of dissolved and some undissolved inorganic materials by element in the oil. Nearly all elemental spectrometers used today for oil analysis are of the atomic emission type. One such instrument works by exposing the sample to extreme temperatures generated by arcing electrodes commonly referred to as rotating disc electrodes (RDEs).

Another common method uses an argon plasma torch to vaporize the sample. This is known as an inductively coupled plasma (ICP) spectrometer. The extreme heat excites the atoms in the sample causing them to emit energy in the form of light. Atomic elements emit light at specific frequencies. The spectrometer quantifies the amount of light generated at these frequencies to estimate the concentration of each element (iron, lead, tin, etc.) in parts per million by weight.

Atomic emission spectroscopy is particle-size limited. Dissolved metals and suspended particles up to approximately 2 μm are detected with high accuracy. The accuracy diminishes (lower recovery) as particle size increases up to 5 μm. Elemental concentrations can be greatly understated for particles larger than 5 μm.

Increasing or decreasing elemental concentration can signal a change in the generation of wear debris, the ingress of contaminants, and the addition or depletion of additives. The table in Figure 22.15 generally categorizes the common elements observed with oil analysis as wear, contamination, or additives.

The following should be considered when diagnosing nonconforming elemental data [3,11]:

- An increase in the concentration of elements such as iron, copper, chromium, tin, aluminum, and lead suggests that abnormal wear is occurring. Further steps should include analysis of the debris with complementary testing to determine its severity, nature, origin, and root cause.
- Increasing concentrations of silicon, sodium, boron, calcium, and magnesium can signal the ingress of

Element	Wear	Contamination	Additive
Iron (Fe)	X	X	
Copper (Cu)	X	X	X
Chromium (Cr)	X		
Tin (Sn)	X		
Aluminum (Al)	X	X	
Lead (Pb)	X		
Silicon (Si)		X	X
Sodium (Na)		X	X
Boron (B)		X	X
Calcium (Ca)		X	X
Magnesium (Mg)		X	X
Zinc (Zn)	X		
Phosphorous (P)		X	X
Molybdenum (Mo)			X
Potassium (K)		X	

Figure 22.15—Common elements and their sources.

contamination. A lock-step increase in silicon and aluminum typically suggests dirt ingress, although silicon is also the primary element found in antifoaming additives (silicone), which can cause confusing results. Increasing levels of sodium and boron may signal the ingestion of glycol-based coolant. Calcium and magnesium are often present when hard water is ingested (e.g., from cooling system leaks), but they are also common elements found in engine oil additives.

- Numerous elements are used in a multitude of additives. For example, zinc and phosphorous are common in antiwear additives; sulfur, phosphorous, and molybdenum are common components of extreme pressure (e.g., from a differential lubricant) and friction modifier additives; and calcium and magnesium are frequent components of engine oil alkalinity improvers. Knowledge of the new oil baseline is critical to trending additive depletion with elemental spectroscopy.

Applicable standards: ASTM D4951 (ICP, additives); ASTM D5185 (ICP, additives, wear metals, and contaminants); ASTM D6595 (RDE, additives, wear metals, and contaminants) and ASTM D7303-06 (metals in grease by ICP).

22.3.1.11 FLASH POINT TEST AND FUEL DILUTION INSTRUMENTS

The flash point, when used to analyze used oils, can identify the presence of volatile molecules from fuel and other flammable contaminants. A lubricant's flash point is the lowest temperature at which an ignition source (small flame) applied to the oil's surface causes the vapors of the lubricant to ignite under specified conditions. The oil is said to have "flashed" when a blue flame appears and instantaneously propagates over the oil's entire surface. The oil flashes because a flammable mixture results when it is heated sufficiently, causing vapors to emerge and mix with oxygen in the air. The flash point temperature of an oil corresponds roughly to a vapor pressure of 3–5 mm Hg. Figure 22.16 shows the contaminants and other influencing conditions that can alter the flash point in used oils.

Many laboratories simply test up to a specified temperature (e.g., 20°C below the oil's normal flash point). If the oil flashes at this lower temperature it can be reliably assumed that it has been diluted with fuel or other low-boiling point flammable liquid. This pass/fail use of the flash point test reduces the time to perform the test as well as its cost. Those oils that fail can then be analyzed further to determine the specific flash point to estimate the total contaminant level (e.g., fuel contamination) [17].

This test is commonly used with engine oils (diesel, gasoline, and natural gas) and occasionally with natural gas compressor lubricants to detect excessive gas solubility. The closed-cup flash point test is the most widely used method for fuel dilution testing because of its sensitivity to low concentrations of fuel. Fuel or chemical dilution severely impairs the lubricant's effectiveness and can cause fire or explosion hazard [17].

	Decreases Flash Point	Increases Flash Point
Changes in oil chemistry	• Thermal cracking • Radiation (cracking by γ rays) • Microdieseling	• Polymerization
Additions to the oil	• Diesel fuel • Gasoline • Natural gas • Solvents • Wrong make-up oil • Water (instrument interference)	• Water • Coal dust • Glycol/antifreeze • Wrong make-up oil
Subtractions from the oil		• Thermal evaporation (boiling point off of light ends) • Vacuum dehydration

Figure 22.16—Contaminants and factors that influence flash point.

Applicable flash point standards: ASTM D92 (Cleveland open cup), ASTM D93 (Pensky-Marten closed cup), and ASTM D3828-97 (small-scale closed cup).

22.3.1.12 OTHER USED OIL ANALYSIS TESTS

The previously described tests represent the core analytical methods used in the analysis of in-service lubricating oils. There are many instruments and methods that are emerging onto the market that show considerable promise. Likewise, there are others that serve niche applications and still others that are slowly being phased out to make room for new technology. It is not possible to discuss all of these technologies and analytical methods. However, there are a few that fall outside of the mainstream that, for various reasons, are worth noting.

22.3.1.12.1 Optical Soot Meters

Whereas some laboratories commonly use infrared spectroscopy to estimate the percentage soot in engine oil (described previously), other laboratories prefer to use single-channel optical devices (either white light or infrared) to quantify the amount of nontransmitted light through a fixed path length or they use an ATR cell. The nontransmitted light fraction is defined as the soot load of the oil and is often reported as percentage soot. This is a nonstandardized test method.

22.3.1.12.2 Blotter Spot Testing

This simple test, also known as paper chromatography or radial planar chromatography, is used to examine soft insoluble suspensions in oil using blotter paper to which a small aliquot of sample is applied. Varnish and sludge-producing impurities will form distinct deposits and rings on the blotter paper as the oil wicks outward in a radial direction by capillary action. These impurities include carbon insolubles, oxide insolubles, additive degradation products, and glycol contamination. This is a good field and laboratory test and is nonstandardized.

22.3.1.12.3 Glycol Reagent Method

The Schiff's reagent method (ASTM D2982) is a colorimetric method for detecting trace amounts of glycol in lubricating oils. In this method, a solution of HCl and periodic acid is introduced to the oil to oxidize any glycol that may be present. The reaction produces an aldehyde, which in turn reacts with the Schiff's reagent, yielding a positive color change from colorless to pink/purple—the darker the color, the more glycol present.

22.3.1.12.4 Crackle Test

The crackle test is a noninstrument method of determining the presence of water in oil, and in some instances, estimating its concentration. The usual procedure is to place a couple drops of oil on the surface of a hot plate (320°F) and then visually examine the response of the oil to the heat. Oil with no free or emulsified water will become thin and spread because of the heat. Free and emulsified water will result is vapor bubbles and scintillation (crackling sound). With practice, the concentration of water can be estimated when compared to known levels of water. This is a nonstandardized method.

22.3.1.12.5 Fuel Dilution Meter

One other instrument of note is the fuel dilution meter that uses surface acoustic wave technology to detect and quantify fuel contamination in motor oil. It samples the "head space" in a bottle of oil on the basis of Henry's law to assess fuel vapor concentration. Fuel vapor concentration correlates to fuel dilution in the oil. This is a nonstandardized method.

22.4 SETTING LIMITS AND TARGETS

Oil analysis alarms serve as a "trip-wire" to tell the analyst that a threshold has been passed and that action is required. Some data parameters have only upper limits, such as particle counts or wear debris levels. A few data parameters use lower limits, such as BN, additive elements, flash point, oxidation stability, and FTIR (additive). Other data parameters such as viscosity and FTIR use upper and lower limits. These generally relate to important chemical and physical properties of the lubricant in which stability of these properties is desired.

Alarming techniques vary to fulfill the requirements of different oil analysis objectives. These techniques can be generally categorized as proactive and predictive alarms.

22.4.1 Proactive Alarms

Proactive alarms alert the user to abnormal conditions associated with root causes of engine wear, operating faults, and lubricant degradation. They are keyed to the proactive maintenance philosophy of setting targets and stabilizing lubricant conditions within those targets. A strategic premise of proactive alarms is that they be set to levels that will generate improvement over past performance (e.g., cleaner, dryer, etc.) or ensure that conditions are maintained to within levels that have previously been optimized relative to organizational objectives. Within the proactive domain, the following types of alarms and limits are used:

- *Goal-based targets:* Goal-based targets are used to reduce stress (e.g., contamination) on the oil and engine to extend service life (Figure 22.17).
- *Aging limits:* Aging limits alert car owners to the approaching end of the service life of the oil or engine component (Figure 22.18).

22.4.2 Predictive Alarms

Predictive alarms signal the presence of abnormal engine conditions or the onset of wear and failure. They are aligned with the goals of predictive maintenance (i.e., the early

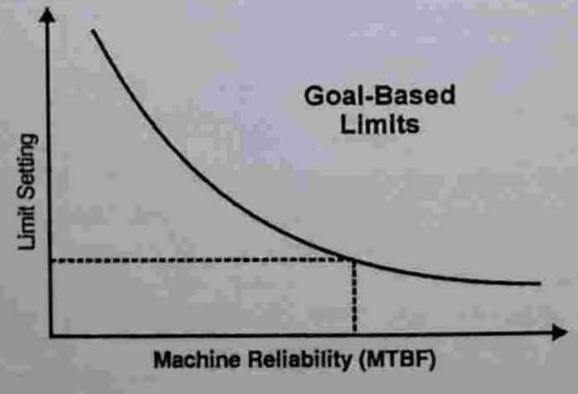


Figure 22.17—Goal-based limits are used on the oil and engine to extend service life.

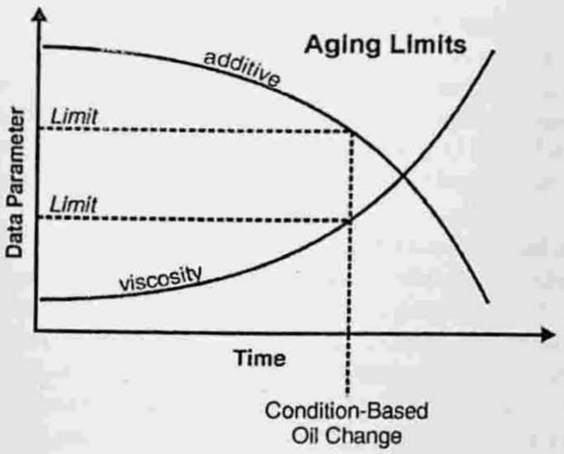


Figure 22.18—Aging limits alert to the approaching end of the service life of the oil or engine.

detection of engine failure symptoms as opposed to failure root causes [proactive maintenance]). Within the predictive domain, the following oil analysis alarming techniques can be used:

- *Rate-of-change-based alarms:* Rate of change alarms are typically set to measure properties that are being progressively introduced into the oil, such as wear debris (Figure 22.19). The add rate (change) can be calculated per unit of time, hours, cycles, etc. For example, a 100-ppm increase in iron over a period of 100 operating hours could be stated as 1 ppm/h operation. Rate-of-change limits are effectively applied to particle counting, elemental wear metals, ferrous density, and BN. It can also be effectively applied to monitor abnormal degradation of additives using elemental analysis, linear sweep voltammetry, and FTIR spectroscopy.
- *Statistical alarms:* This practice requires the availability of a sufficient quantity of historical data for the particular type of engine and type of service. A population mean and associated standard deviation are generated from the available data. The data from a sample of used oil are compared to the mean of the population. If the value falls within 1 standard deviation of the mean, then it is considered normal. If it falls outside of 1 standard deviation from the mean, but within 2 standard deviations, then it is considered a caution. If the result exceeds 2 standard deviations, the value is considered in critical alarm.

22.5 INTERPRETING AND APPLYING OIL ANALYSIS RESULTS

Interpreting oil analysis data requires an understanding of the specific oil analysis tests and an understanding of how these tests interrelate when oil and engine conditions change. Reference the table in Figure 22.20 during the discussion of the primary and secondary oil analysis indications for typical abnormal conditions.

22.5.1 Solid Particle Contamination

An alarm on particle contamination signals an increase in suspended particles due to such occurrences as the failure of the lube oil filter, abnormal ingestion of contaminants from the engine air induction system, contaminated new oil, or an increase in the internal generation of wear debris.

- *Primary test:*
    - *Particle count:* Use suitable automatic particle counting or microscopic particle counting methods. Report ISO code, or particle count (at different sizes in micrometres), or both.

	Lube/Machine Conditions													
Oil Analysis Tests	Particle Contamination	Wear Debris Detection	Wear Debris Analysis	Abnormal Viscosity	Moisture Contamination	Additive Depletion	Oxidation Stability	Glycol Contamination	Fuel Dilution	Soot Load	Alkalinity Reserve	Wrong Oil	Thermal Failure	Corrosive Conditions
Particle Count	P	S	S											
Viscosity				P	S		S	S	S	S		P	P	
AN/BN				S		S	P				P	S		P
FTIR Spectroscopy				S	P	P	P	S	P	P		P	S	S
Ferrous Density		P	S											
Analytical Ferrography	S		P											
RPVOT							P					S		
Moisture Tester					P									S
Elemental Spectroscopy	S	P	P		S	P		P	S			P		S
Flash Point Test				S					P			S	P	

Figure 22.20—Table of commonly used primary and secondary (confirming) oil analysis indicators.

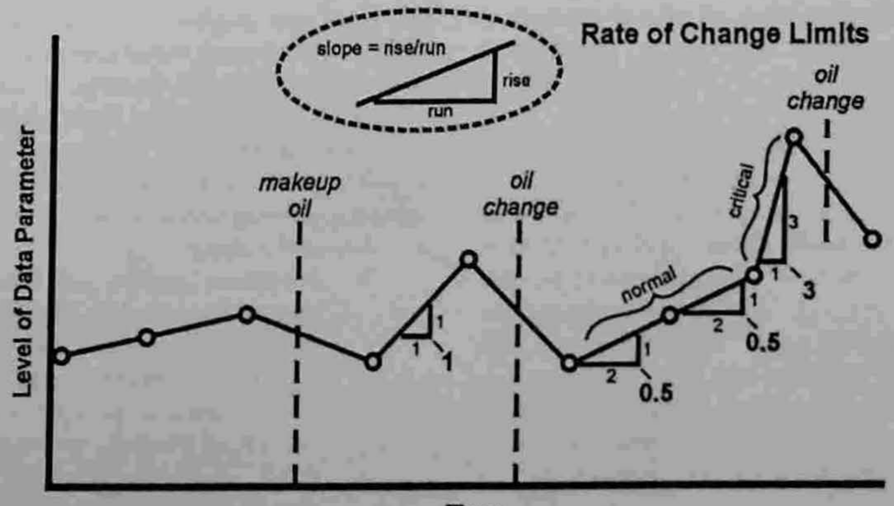


Figure 22.19—Trend-line slope is a visual indication of rate-of-change and parameter severity.

- *Secondary tests:*
  - *Elemental spectroscopy:* Often, when a particle count increases the elemental levels of the particles present increase. For dirt contaminated oil, silicon and aluminum typically increase. Likewise, wear metal elements may increase depending upon the metallurgy of the failing components.
  - *Wear particle identification:* Although expensive and time-consuming, examining individual particles onto a slide or membrane and viewing them under a microscope will reveal an increase in particle count and information on the particle source. Quantification of the particulate is limited with this technique.

22.5.2 Wear Debris Detection

When engines are operating abnormally because of lubrication failure, contamination, corrosive conditions, thermal distress, etc., a message is sent into the oil in the form of wear debris.

- *Primary tests:*
  - *Ferrous density:* Measuring an increase in the production of ferrous debris is a good indication of abnormal wear because so many engine components and frictional surfaces are constructed of steel or iron. These tests are biased against nonmagnetic wear debris generated from brass, bronze, aluminum, lead, tin, and stainless steel surfaces.
  - *Elemental analysis:* A rise in the level of elemental metals is an indication of the presence of an abnormal engine condition. By comparing results to component metallurgy records, the abnormal condition can often be qualified.
- *Secondary test:*
  - *Particle count:* Any wear that is generated will result in an increase in particle count. However, particle count reports no differentiation between ingested debris such as dirt and generated wear particles.

22.5.3 Wear Debris Analysis

When an abnormal wearing condition is encountered, it can be analyzed to provide an indication of the nature, severity, and root cause of the problem. This requires an investigation of the wear particles themselves along with a review of collateral information such as engine acoustics, exhaust smoke color, computer diagnostics, operational information, etc.

- *Primary tests:*
  - *Analytical ferrography:* This microscopic technique provides an abundance of information about the wear debris and the wearing event. By evaluating particle size, dimension, shape, and appearance, the analyst can often determine what wear mechanism generated the debris. By manipulating the particles with light, heat, and chemicals, the metallurgy of the particles can often be defined.
  - *Elemental spectroscopy:* When engine component metallurgy is known, elemental spectroscopy proves invaluable for localizing wearing components.
- *Secondary tests:*
  - *Ferrous density:* Trending an increase in the rate at which ferrous particles are generated provides important information about the severity of a failure event. Also, some ferrous density testers provide large and small ferrous particle differentiation. A rising "percentage large particles" reading (from direct reading ferrography) suggests increased severity. These devices are biased toward magnetic particles.
  - *Particle count:* The rate at which particle count increases is indicative of the severity of the problem. Also, most particle counters sort the particles by size range. An increasing generation of large particles suggests high urgency. Particle counters lack the ability to differentiate particles by type (dirt or wear).

22.5.4 Abnormal Viscosity

Viscosity can increase for several reasons. Oxidation, thermal failure, water/glycol contamination, soot loading, and wrong oil are the most common reasons. Fuel dilution, viscosity index improver (additive) shear-down, and base oil cracking all reduce viscosity.

- *Primary test:*
  - *Viscosity:* Viscosity is the "catch all" test for several abnormal lubricant or contaminant conditions. Changes in viscosity are often an early indicator of other problems that involve the need for an expanded scope of analysis.
- *Secondary tests:*
  - *AN:* If the increase in viscosity is associated with oxidative failure, the AN will typically increase. AN sometimes increases or decreases when the wrong oil (and wrong viscosity) has been added to the sump because of the influence of the additive package on the AN.
  - *FTIR spectroscopy:* When viscosity changes because of thermal failure, oxidation, fuel dilution, or glycol contamination, the FTIR spectrum tends to change at certain bands. If the wrong or mixed oil is being used, numerous spectral features will also tend to change.
  - *Flash point test:* When the oil has been contaminated with fuel or solvents, the flash point will drop.

22.5.5 Moisture Contamination

Moisture in all of its forms brings nothing but trouble to the lubricant and engine. It rusts iron and steel surfaces, promotes corrosion on other metal surfaces, and over time can destroy the lubricant. Moisture can enter the oil from many places, including coolant leaks and combustion blowby. It is common for moisture generated by engine combustion to condense in the crankcase because of wintertime and short-distance driving conditions.

- *Primary tests:*
  - *Moisture tester:* Water can be screened to approximately 500–1000 ppm with the crackle test depending on the exact procedure used and the type of oil. Quantification of the moisture content (if required) is best accomplished using the standard Karl Fischer titration procedure.
  - *FTIR spectroscopy:* FTIR serves as an effective screen for moisture above approximately 1000 ppm for mineral oils, depending on the instrument's signal-to-noise ratio. FTIR will also signal the

presence of glycol if leakage is from a cooler that uses glycol-based antifreeze.

- *Secondary tests:*
  - *Viscosity:* When high levels of water contaminate oil, an emulsion is often formed. The viscosity, as measured by traditional viscometers, will increase under these conditions.
  - *Elemental spectroscopy:* Often, metals accompany the ingested water. For example, hard water brings calcium. Salt water brings sodium. Water and glycol (coolant) bring sodium, boron, and potassium depending on the corrosion inhibitors used in the antifreeze. The source of the water can often be localized by examining the relative concentration of these trace metals.

22.5.6 Additive Depletion

Additives are among the most difficult parameters to measure using oil analysis. The additives exist as organic, inorganic, or organometallic compounds that improve base oil performance. Often, it is easier to assess the performance characteristic than the additive itself. Still it is possible to estimate the RUL of certain additives using conventional oil analysis techniques. New technologies have greatly expanded this capability.

- *Primary tests:*
  - *Elemental spectroscopy:* Many additives are organometallic compounds using zinc, phosphorous, magnesium, calcium, silicon, etc. These levels can be effectively assessed using elemental spectroscopy. The technique has two primary limitations:
    1. Additives can be decomposed and their constituent elements transformed into other molecules with no visible change in elemental concentration. These are often referred to as dead additives or additive floc; their mass still exists in the oil but they are no longer functional. For certain additives, a loss of elemental concentration of just 25 % is enough to merit concern for the remaining 75 % additive mass (that may be dead). Some additives lose mass by precipitation (drop out), water washing, particle scrubbing, filtration, etc.
    2. Many additives have elements similar to wear metals and contaminants. For example, dirt and some antifoaming agents appear on the oil analysis report as silicon.
  - *FTIR spectroscopy:* FTIR spectroscopy can to a certain extent measure the presence of active additive molecules, including organic molecules (see Figure 22.21) [18]. It is limited by (1) its poor ability to quantify additives in low concentrations and (2) interferences that might occur.
  - *BN:* The oil's reserve alkalinity (detergent additive) is measured using the BN test. As BN declines, the oils' ability to counteract the ingress of combustion acids is weakened. A loss of BN signals the need for an oil change or make-up oil.
  - *Blotter spot test:* The blotter spot test is extremely effective at assessing the condition of motor oil dispersancy.

22.5.7 Oxidation Stability

Oxidation stability is an indication of the oil's residual ability to resist oxidation. Some oil analysis tests measure the byproducts of oxidation whereas others attempt to measure the oil's ability to resist oxidation. Figure 22.22 plots the pre- and postoxidation physical and chemical oil properties that can be monitored by routine oil analysis [19].

- *Primary tests:*
  - *AN:* As an oil oxidizes, organic acids (formic, carboxylic, etc.) are produced. Measuring the concentration of these acids is a measure of oxidative damage that has occurred. Not all acids found in oil relate to oxidation.
  - *FTIR spectroscopy:* During oxidation, the oil's base molecules (hydrocarbon) are turned into ketones, aldehydes, carboxylates, and other transition molecules. Many of these new molecules have ester functional groups and can be measured with FTIR spectroscopy.
- *Secondary tests:*
  - *Viscosity:* As the oil oxidizes, its viscosity typically increases. As such, viscosity trending is not a good forecasting technique for oxidation stability but is rather a positive indication of the onset of oxidation.

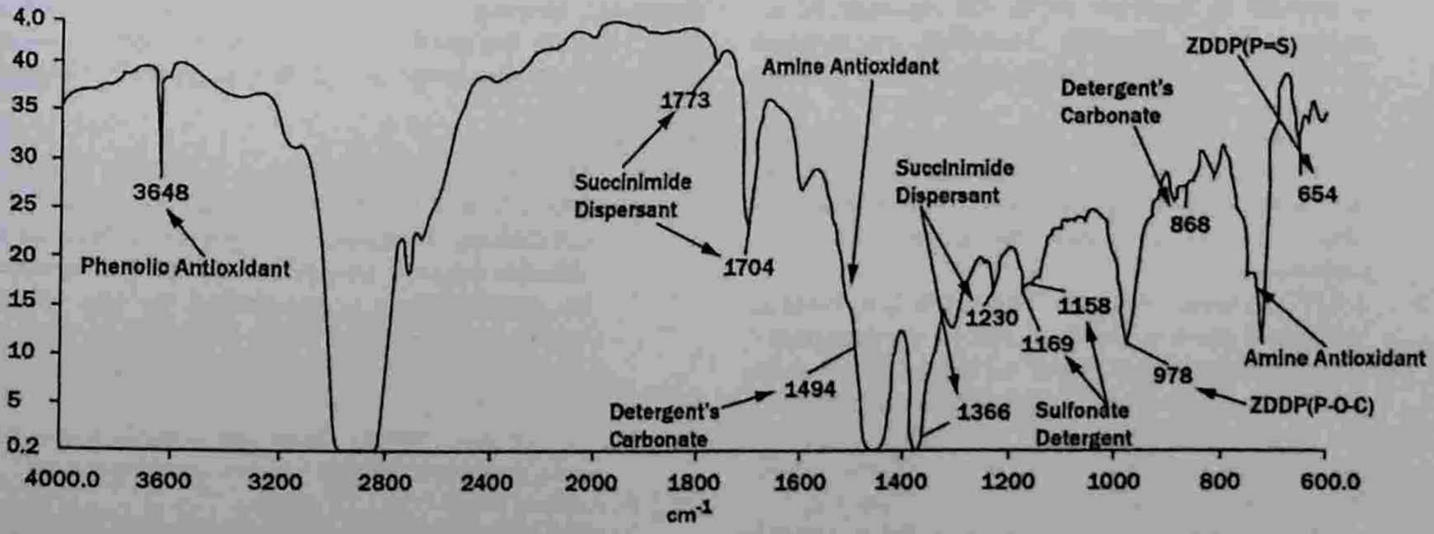


Figure 22.21—FTIR spectrum showing spectral bands of common motor oil additives.

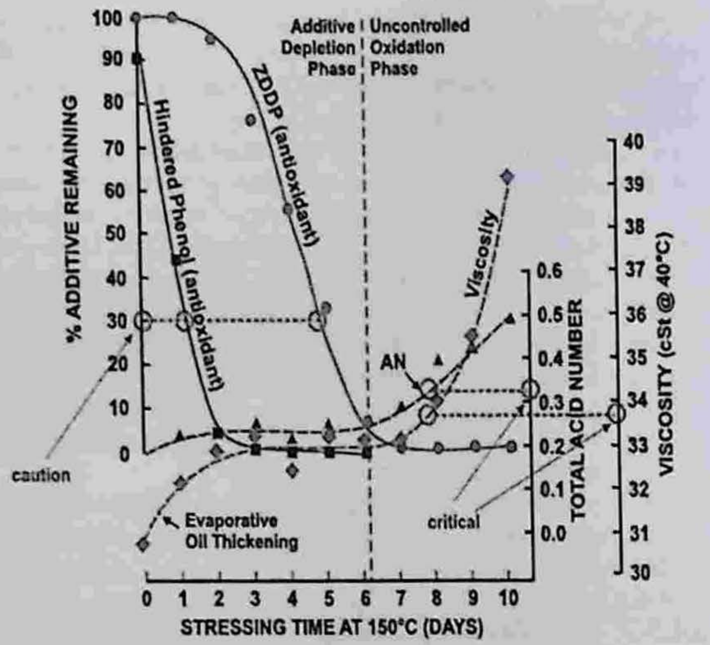


Figure 22.22—Oil analysis properties that characterize pre- and postoxidation physical and chemical oil properties [19].

22.5.8 Glycol Contamination

Glycol (antifreeze) enters lubricating oils from the cooler. Leakage from corrosion, seal failure, cooler core damage, and cavitation are the most common causes of glycol contamination. Glycol is extremely harmful to (1) the lubricant base oil (promotes oxidation and deposit formation), (2) additives (additive reactions forms abrasive oil balls), (3) filtration (plugs pores prematurely), and (4) the engine (corrosion, wear, etc.).

- *Primary tests:*
  - *Elemental spectroscopy:* Most commercial antifreeze formulations include corrosion inhibitors that contain sodium, boron, or potassium, or a combination of these, among other elements. The inhibitors transfer into the oil with coolant (glycol) contamination. As such, they can be detected elementally by trending boron, sodium, or potassium, or a combination of these. The presence of these elements effectively serves as "markers" in detecting and quantifying glycol contamination.
- *Secondary tests:*
  - *Viscosity:* When a significant amount of glycol is present in used oil there will typically be an increase in oil viscosity. Therefore, an increase in oil viscosity should be investigated as possible antifreeze contamination.
  - *Water testers:* While not all coolant leaks result in the prolonged presence of water in the crankcase oil (water boils off quickly at normal engine running temperatures) some amount of water is not uncommon.
  - *Schiff's reagent method:* This is a colorimetric procedure used in the field and in the laboratory. If there is antifreeze in the oil there will be a distinctive color produced by the chemical change of the glycol-contaminated oil when mixed with the Schiff's reagent. Often glycol transforms rapidly into other chemicals after it contacts the oil. This condition frequently results in a false negative from this test.
  - *Blotter spot test:* By placing a couple drops of oil on common blotter paper or card stock a qualitative assessment of glycol contamination can be obtained.

22.5.9 Fuel Dilution

Fuel typically gets into the crankcase as an incomplete combustion byproduct (blowby) or by leakage. Either way, the influence of the fuel on lubrication can be substantial. For instance, just 10 % fuel dilution can be enough to reduce an SAE 30 viscosity to an SAE 20. The reduced oil viscosity can alter critical oil film thickness in engine components. Additionally, the fuel dilutes additive concentrations. If excessive fuel levels are measured (>3 %); the source of the fuel needs to be determined and remedied.

- *Primary tests:*
  - *Flash point testing:* When a lubricant is properly baselined, a sudden drop in flash point is a positive indication of fuel dilution.
  - *FTIR spectroscopy:* There are specific search areas in the infrared spectra that can be used to assess the presence of gasoline and diesel in lubricants.
  - *Fuel dilution meter:* This instrument uses a headspace sampler to measure fuel vapor. This correlates well with fuel dilution using Henry's law.
- *Secondary tests:*
  - *Viscosity:* Fuel contamination of crankcase oils sharply influences the blended viscosity. Fuel that enters the crankcase through the combustion chamber (blowby) may only consist of the heavier molecules (i.e., reducing the resultant viscosity effect). However, raw fuel from leakage will sharply lower viscosity and oil film thickness.

22.5.10 Soot and Lost Dispersancy

Soot enters crankcase oil from combustion blowby. Excessive amounts occur when oil drains are overextended, air cleaners are plugged, rings/liners are worn, or overfueling conditions occur, or any combination of these. As soot builds in the oil, the performance of the oil can degrade to eventually impair lubrication and result in the formation of sludge and deposits. Soot load and the quality of soot dispersancy (influenced by additives) affect the timing of a condition-based oil change for crankcase lubricants. Dispersancy is considered impaired when soot particles coagulate, forming carbon suspensions larger than 1 μm. Depending on the grade of oil and engine type, soot concentrations in the range of 2–5 % are typically flagged as abnormal.

- *Primary tests:*
  - *FTIR spectroscopy:* Infrared provides a reliable, time-efficient test for soot load. However, it does not evaluate the quality of dispersancy. Because soot absorbs infrared energy across the full spectrum, its concentration is quantified by the resulting baseline shift (spectral shift). It is typically presented as percentage soot or percentage transmittance (for infrared).
  - *Optical soot meters:* These can provide a quick and reliable determination of soot concentration.
- Secondary tests:
  - *Viscosity:* Viscosity will generally increase somewhat with increasing concentrations of soot. There is

even a sharper effect on viscosity when dispersancy is lost.

- *Blotter spot test:* In addition to soot load, the appearance of well-defined annular structure (rings, halos, dark center spot, etc.) points to loss of dispersancy.

22.5.11 Alkalinity Reserve

Monitoring and controlling the reserve alkalinity of crankcase oils is key to any extended, condition-based oil drain strategy. Certain fuels (e.g., high in sulfur) and operating conditions (cold climates, short trips, etc.) contribute to loss of alkalinity reserve. High blowby from worn engines can also lead to corrosive conditions.

- *Primary test:*
  - *BN:* This particular test has been performed for many years to assess alkalinity reserve by oil laboratories. The preferred procedure is ASTM D4739 or ASTM D5984-96. Typically, crankcase oils are scheduled for change when BN drops to 50 % of the original, new-oil BN. BN results less than 2 are considered critical (corrosive).

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