

Fourier Transform Infrared Analysis Applied to Maintenance  
Management -  
Making the Most of this Powerful Tool

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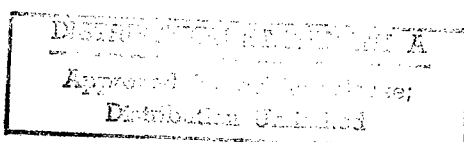
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**Abstract:** The utility of infrared analysis for industrial applications has increased due to the availability of Fourier transform infrared (FT-IR) spectrometers. This increase has come about because of the advantages that FT-IR offers over other types of infrared equipment. These advantages include speed of analysis, good wavelength stability, high-energy throughput, and high sensitivity. The above factors coupled with PC control, easy-to-use software; small footprint and relatively low purchase cost have made modern FT-IR systems a more powerful and cost-effective analytical tool. Application of this tool to maintenance management is reaching new heights because of the many facets of maintenance quality assurance to which infrared analysis can be applied. It is through a better understanding of the capabilities of FT-IR analysis to solve maintenance management problems that its use will continue to grow.

**Key Words:** Condition monitoring; Fourier transform infrared spectroscopy; FT-IR; incoming material identification; lubricant analysis; power generation; quality control.

**Introduction:** In modern proactive maintenance management, the goal is to provide maximum uptime and proper operation of mechanical devices. This is needed to assure that the work done by a piece of mechanical equipment is done with the greatest possible safety and reliability. In support of maintenance management efforts, several diagnostic techniques are applied to provide information that can be used to assess the performance of equipment. These techniques include such things as vibration analysis and lubricant analysis.



The use of these diagnostic tools is justified by the predictive capabilities they provide. The information from diagnostic tests can be used in the prevention of catastrophic failures and scheduling of maintenance activities. However, many times the economic justification for completing analysis associated with equipment maintenance is difficult to assess. The value of a particular technique used in maintenance management is measured not only by the cost of purchasing the equipment, but also by the cost and quality of information associated with using the tool. Also important is the ease with which useful information is obtained. One diagnostic tool that has been finding increased use in maintenance management is Fourier transform infrared (FT-IR) spectroscopy.

The increase in use of FT-IR instrumentation has evolved for several reasons related to the technological advancement of the technique. The advancement in the utility of FT-IR spectroscopy for routine quality control and process analysis has occurred because of the advantages of FT-IR compared to other infrared techniques. These advantages include high sensitivity, high-energy throughput, high resolution, and good wavelength stability. However, the expansion of the technique into routine analysis areas has only occurred within the last fifteen years. This expansion has occurred due to four basic reasons: the rapid advancement in high powered PC computers, decrease in instrument size, lowering of prices, and the development of easy to use software.

Two application areas in maintenance management where FT-IR spectroscopy can be used include verification of incoming material and lubricant condition monitoring. The utility of FT-IR instrumentation in completing both of these functions provides greater value and justification for the purchase of the equipment. It also provides a method that can be applied in a total quality management for fluids and non-metallic solid materials.

Many problems occur as a result of incorrect materials being applied in maintenance practices. FT-IR is an excellent tool for identification of non-metallic incoming materials of all types, including gaskets, o-rings, greases, oils and other fluids. In the nuclear power industry, gaskets and o-rings must be certified before they are stocked for use in maintenance. FT-IR is quick, easy, and most times non-destructive, and it provides positive identification of many non-metallic materials. Because of these facts, FT-IR is one of the best tools available for a variety of identification and quality verification purposes. This is because of the availability of a variety of sampling devices that allow the FT-IR to be used in ways that provide molecular structure information quickly and easily. Application to the identification and verification of lubricants and other fluids is a sound practice in a well-controlled lubricant condition-monitoring program.

The application of infrared analysis to used lubricant condition monitoring has grown tremendously in the last ten years. The speed of analysis makes FT-IR an attractive option for the rapid screening of used lubricants for oxidation breakdown

products and contaminants, such as water, fuel, antifreeze, fuel, and soot. This can shorten laboratory turnaround times and decrease the number of time-consuming traditional oil quality tests that need to be completed. The growth in the use of infrared analysis has been fostered by a general increase in the importance of used lubricant condition monitoring for large industrial and transportation equipment. It provides a way to assure safe operation, prevent catastrophic failures, as well as increase lubricant drain intervals.

This presentation will provide an overview of the approaches currently in use for applying FT-IR analysis to maintenance management. Included will be a discussion of various methods of sample analysis and data treatment schemes, as well as how the FT-IR can be set up to allow non-chemist operators to use it successfully. Particular focus will be on its application in the nuclear power industry. Case studies with cost savings data will be provided.

**Principles of Infrared Spectroscopy:** Infrared spectroscopy in the mid-infrared region (2.5 to 25 micrometers or 4000 to 400 wavenumber) has been traditionally used to obtain information about the structural composition of molecules. It is particularly useful for identifying structural features and functional groups in organic molecules, but can also be used for the analysis of inorganic molecules, particularly those containing oxygen or nitrogen. The technique is sensitive to atomic combinations where there is a change in dipole moment. This means that hydrocarbons and molecules with combinations of dissimilar atoms are detected. Thus, gases such as carbon monoxide (CO) or nitrous oxide (NO) are readily detected using infrared measurement. Infrared spectra are not obtained from diatomic molecules that are symmetrical, such as nitrogen (N<sub>2</sub>) or oxygen (O<sub>2</sub>).

Metal ions are not detected by infrared so that infrared is not useful for wear metal analysis. However, because of the different information obtained, atomic absorption (AA) and atomic emission (AE) spectroscopy are complementary techniques to infrared. For example, zinc in the commonly used additive zinc dithiodiphosphate can be measured directly using AA. The phosphorous-oxygen bond in the phosphate group to which the zinc is bound is detected using infrared. However, tricresol phosphate, another antiwear additive, can only be measured using infrared since it contains no zinc.

Infrared spectroscopy can not only be used to identify materials or components in mixtures; it also can be used to quantify the levels of components in a sample. This fact has led to an expanded role for the technique, especially aided by the introduction and evolution of FT-IR spectrometers.

**Modern FT-IR Spectrometers:** The advances in FT-IR spectrometers have led to their widespread use for quality control and process monitoring applications. The high sensitivity of FT-IR is brought about by the combination of high-energy throughput and high resolution, which are due to the way the instrument operates. Another feature these instruments have is good wavelength stability because a reference laser is used. These features alone did not lead to a full migration of the technique to quality control and process monitoring applications. The evolution of smaller size, lower cost instruments coupled with high speed, low cost PC computers contributed strongly to the widespread use of the technique that is seen today. Also contributing to the more widespread use has been the development of easy to use software and convenient sampling accessories.

With the advancement of graphical user interfaces, all software on PC computers has become more intuitive to use. FT-IR systems, following this trend, now have software that utilizes these graphical interfaces to allow operators to obtain useful data quickly without having to spend long periods of time learning how to use the software. To further simplify operation, customizable operator interfaces allow a lab manager to set the system up so that only the features needed are seen. Another major advancement that some manufacturers have made is communication between the spectrometer and the computer so that on-board diagnostics are available. These diagnostics allow the operator to quickly determine if problems exist with the system that would prevent them from getting usable data. In addition to these hardware diagnostics, programmable system checks can verify proper operation of the sampling configuration.

**Sampling Accessories:** The high-energy throughput feature of FT-IR spectrometers is another major reason that has allowed the technique to gain widespread use. It is because of the high-energy throughput and resulting high signal to noise, that a variety of sampling accessories has been developed. These accessories have created more sampling options, which allow simpler and more optimized system operation. A recently introduced feature in some sampling accessories is the ability to place them into the FT-IR instrument and have them recognized by the spectrometer. The operational parameters are automatically set up and a test scan is run to verify that it is working properly. This means that the operator does not have to remember the parameters, since the system sets them automatically.

Sampling techniques for the FT-IR spectrometer that are of most interest in maintenance management include transmission analysis and horizontal attenuated total reflectance (HATR) analysis. With these approaches, most types of samples that will be encountered can be successfully analyzed.

Transmission analysis can be used for most fluids from very low viscosity to relatively high viscosity. For fluids to analyzed in traditional transmission cells they must be pushed or pulled through a thin space between two windows. The

magnitude of the space is around 100 micrometers, which is approximately the thickness of a human hair. Because of this thin film, the practical limit for the use of flow-through transmission cells is between 300 and 400 centistokes (@40°C).

An alternative method for transmission analysis is by the use of a membrane made of porous polyethylene. In this case, a small measured amount of fluid material is pressed into the surface of the polyethylene and it is analyzed once it has been absorbed into the pores. While this method is not as reproducible as the flow-through transmission cell, it is typically adequate for routine analysis. Membranes that have an effective pathlength of around 100 micrometers need to be used to obtain comparable sensitivity to the 100-micrometer transmission cell. The advantage of the porous membrane over the flow-through cell is that it does not require solvents to clean since the membrane is disposable. This is an important safety advantage.

In practice, flow-through transmission cells or porous polyethylene membrane cells with nominal pathlength of 100 micrometers are used to analyze unused and used oils. The transmission cell technique provides good sensitivity for low level additive and contaminant measurement. For new lubricants or other fluids, a comparison can be made of the lubricant or other fluid to determine if it is similar to previously obtained batches. They can also be checked for the presence of contaminants such as water.

For used lubricant analysis, methods are available for condition monitoring which assess the status of the lubricant and mechanical system. The methods apply either new oil reference subtraction followed by assessment of spectral regions, or direct analysis of spectral regions to obtain information from the sample. Spectral features are used to determine relative levels of lubricant breakdown products, such as carbonyl oxidation, nitro-oxidation, sulfur oxidation, and other base oil breakdown products in the case of certain synthetic lubricants. Contaminants that are assessed include water, antifreeze, fuel, or soot. Additive loss can be determined for certain additives, such as phosphate antiwear/antioxidant additives and phenolic antioxidants.

One problem with diesel engine oils is the high levels of soot that are contained in the oil. If the level exceeds 3-4% by weight of soot, the transmission cell technique using a 100-micrometer pathlength becomes no longer useful. The light infrared light can no longer transmit through the sample, and no useful data can be obtained. In this case, an HATR cell is a reasonable alternative to both trend the soot level and check for breakdown products or contaminants. It must be kept in mind however; that the HATR method has lower sensitivity than the transmission cell and increased scanning times should be used as discussed below.

For grease sample analysis, which is becoming of greater concern in maintenance management, neither the flow-through cell nor the membrane can be used. Greases can not be pumped through cells. When placed on a porous membrane they

separate into oil and soaps constituents, which leads to a significant change in the spectral features. The only way to analyze these by transmission is by the use of thin films between removable windows. This approach is time consuming and some type internal standard approach must be used to account for pathlength variations. A much simpler way to handle grease sample analysis is by using an HATR accessory.

HATR is a sampling method that allows a thin film of sample to be placed on the upper surface of an infrared transparent crystal. The infrared light is transmitted through the crystal and bounces off the sample to obtain a spectrum. However, because the total number of bounces through an HATR element is limited, sensitivity of the measurements with HATR is lower than that of a transmission cell. To overcome this situation, a crystal with the highest number of bounces should be used. In practice, either a 45° ZnSe (10-12 bounce) or a 40° ZnSe element can be used. The number of scans collected should be at least two to four times that of the transmission cell. The HATR cell can be used for very thick or very dark samples that can not be done using the transmission cell. Because of optical effects, spectra from HATR and transmission cells can not be directly compared as regards the peak intensities. Corrections can be made to make the peak intensities more comparable. However, peak locations are the same for HATR and transmission cells.

One other important aspect to maintenance management that has not been discussed thus far is the analysis of non-metallic parts used in mechanical systems. This refers to gaskets, o-rings, and other polymeric parts that must be replaced during maintenance activity. This is of particular interest in the nuclear power industry, where these parts must be certified prior to their use. This is regulated in the nuclear power industry to assure that the specified materials are used during maintenance. Even though it is not regulated in other industrial operations, the practice of obtaining and verifying materials from other sources can provide significant cost savings. The FT-IR spectrometer is one of the best methods for analyzing these materials.

In this case, the materials need to be pressed against the surface of a single-bounce HATR cell and scanned to obtain a spectrum. This spectrum is compared to spectra of previously analyzed certified materials to obtain a positive identification. The HATR method is, in most cases, non-destructive. Several different types of single-bounce HATR accessories depending on how hard of materials must be analyzed. The harder the material to be analyzed, the more pressure is required to obtain a useful spectrum. The hardness of the material to be analyzed must be considered when determining which particular accessory is needed.

**Case Studies:** To help obtain a better feel for the value of the FT-IR technique, several case histories have been compiled. They include examples from the nuclear power industry for incoming lubricant verification, used lubricant condition monitoring, and material analysis.

Incoming lubricant case history: A nuclear plant was in an outage and decided to check the incoming lubricant using the FT-IR while the tanker was still on the premises. The analyst stopped the tanker at the gate, pulled a sample, and took it to the FT-IR. Five minutes later (after running the sample through the FT-IR) the analyst realized the lubricant was contaminated by a substance the tanker had previously hauled. The analyst forced the supplier to buy back the lubricant. The cost savings from the lubricant alone: \$5,148.00 (1800 gallons at 2.36 per gallon). This cost saving does not take into account the damage or downtime that could have resulted if the lubricant had been added to the turbine it was headed for.

Incoming lubricant case history: A nuclear plant received a shipment of turbine oil that was added to the make up lube reservoir. The plant did not test the lubricant for two days since the plant was powering up from an outage. An operator decided to test the lubricant. When he analyzed the lubricant using the FT-IR, he confirmed that it was contaminated. The supplier came to the plant and paid to clean up the reservoir, but the plant lost two days of running time and had to purchase power from an alternate source in order to supply its customers. Total dollars lost: approximately 1 million dollars.

Lubricant condition monitoring case history: A nuclear plant was using FT-IR for condition monitoring of a condensate pump. The plant noticed the antioxidant additive was depleting at an accelerated rate. Working with the lubricant supplier, the plant was able to "sweeten" the lubricant by adding an additive package concentrate. The plant then monitored the pump closely to prevent having to complete work on the pump prior to the scheduled outage. This close monitoring took place for three months before the outage began. If the pump had needed to be shut down prior to the scheduled outage, the loss of production and scheduling issues could have cost approximately 1 million dollars per day.

Material certification case history: Based on the regulation in the nuclear power industry, the plant must provide "reasonable assurance" that the material used meets the specification for the application. If the plant does not do this internally, they must obtain "Nuclear Grade" certification from outside sources. This certification can cost five times the cost of the parts from a standard commercial source that they certify internally. Therefore, it is very cost effective to buy materials on the open market, and have them certified in-house. Examples of the cost savings are as follows:

<b>Part Description</b>	<b>One time savings (\$)</b>	<b>Total savings (\$) life of plant*</b>
Heat Removal Seals	22,629	107,163
Gasket	291	2,334
O-Ring	159	4,308
Filter	875	7,634

\* - Life of Plant typically is 20 to 30 years, depending on the type and years in service.

**Conclusions:** FT-IR spectroscopy is a technique that continues to grow in popularity as more applications are found for its use. The technological advances in spectrometers, computers, software and sampling along with lower purchase costs have and will continue to feed this growth process. For maintenance management, the simplification of FT-IR systems, their software and sampling accessories have made them easier to use to obtain useful information. Their use will continue to grow based on the above factors coupled with more standardized methods for interpretation of the data they generate.