FTIR Measurements of Lubricating Oils Using a Low-Volume, Multiple Reflection ATR

Jenni L. Briggs, Ph.D., PIKE Technologies

Abstract

Using a low-volume, multiple reflection ATR, infrared spectra of new and in-service motor oil were collected. Benefits of this ATR accessory for the measurements of motor oil and other liquids with low-concentration components of interest are discussed. Example spectra are presented.

Introduction

Lubricating oils are often subjected to harsh operating environments, which results in reduced oil performance, depleted additives, and unwanted oxidative byproduct formation. This can lead to excessive wear and catastrophic equipment failure. To screen and monitor lubricating oils, infrared transmission analysis is popular due to its yield of informative data, its simplicity, and its being a relatively low-cost method. Among the parameters IR can be used to evaluate are chemical oxidation, additive depletion, and water and soot contamination. The aim of this application note is to investigate the suitability of a low-volume, multiple reflection ATR for the analysis of lubricating oils.

Experimental Conditions

In-service and commercially-available synthetic blend and conventional motor oils were analyzed using the MIRacle ATR accessory (Figure 1) equipped with a 9-reflection diamond/ZnSe crystal trough plate from PIKE Technologies (Madison, WI). 32 scans were co-added for the background and sample spectrum. The FTIR spectrometer was equipped with a DTGS detector.

Results

In transmission, typical cell pathlengths for oil quality monitoring and life assessment range from 0.2 to 0.5 mm. These transmission pathlengths eliminate single reflection ATR accessories, in most cases, as a viable sampling accessory because ATR is a surface technique. The effective pathlength, which is roughly equivalent to the transmission pathlength, for a single reflection accessory is less than 5 µm. However, a multiple reflection ATR accessory offers the advantage of enhanced sensitivity because the magnitude of the absorbance bands are increased relative to a single reflection ATR accessory. This is important when measuring low-concentration components like additives and oxidative chemicals found in lubricating oils.

Figure 1. MIRacle ATR accessory and 9-reflection diamond/ZnSe crystal from PIKE Technologies.

Another advantage of this particular multiple reflection ATR, the MIRacle with a 9-reflection diamond/ZnSe crystal, is its crystal size. The sampling surface diameter is 5 mm. Considering the shallow depth of penetration of the beam into the sample, less than a few microns, only a small smear of sample across the crystal is sufficient to obtain a quality spectrum. This small crystal surface area offers easy cleaning by using less solvent compared to the cleaning of a transmission cell and for the analysis of mass-limited samples.

The fingerprint region of the FTIR-ATR spectra of motor oil is shown in Figure 2. Comparing spectra from the two unused motor oils, differences in the absorbance bands suggest that the additive formulation in each oil is not the same. In the spectrum of the in-service (3,500 miles) oil, the carbonyl oxidation band at 1,750 cm^{-1} is clearly visible; the magnitude of the absorbance band is nearly 0.08 AU. The band at 1150 cm^{-1} is associated with sulfate oxidation.[1] These spectra support that the MIRacle with a 9-reflection diamond/ZnSe crystal plate has adequate sensitivity to differentiate oil formulations and to gauge oil life. This ATR accessory may find application use in research and development laboratories and oil monitoring programs in the field.

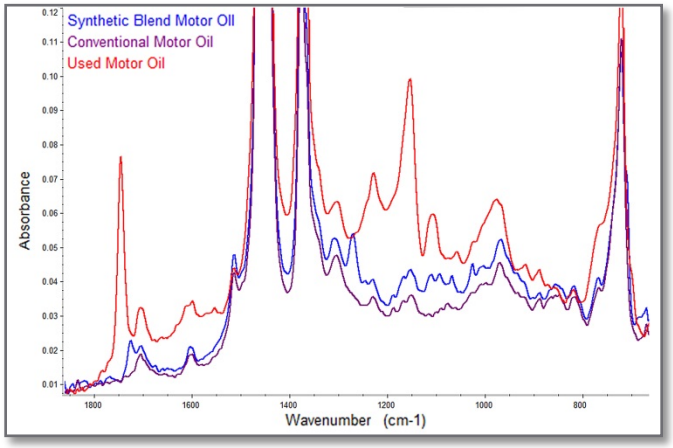

Figure 2. New synthetic blend (blue), new conventional motor oil (purple) and in-service motor oil (red) spectra collected using the MIRacle ATR with a 9-reflection diamond/ZnSe crystal.

Conclusions

For added sensitivity, the PIKE MIRacle 9-reflection diamond/ZnSe ATR is a useful tool to analyze low-concentration components in liquid samples such as motor oil. The small sampling surface area allows for a low sample volume making it ideal for mass-limited samples and also reducing the volume of cleaning solvent required.

Reference

(1) R. Nguele, H.S. Al-Salim, and K Mohannad, *Lubricants* **2**, 206-222 (2014).