IR APPLICATION NOTE 03-03

Analysis of Automotive Fluids Using with FTIR

Introduction

The liquid sample is one of the most common types of samples submitted for analysis and comprises a vast array of pure compounds and solutions. As an example, several liquids are used in an automobile to sustain critical elements such as lubricating the engine or drive train and to supply mundane functions like washing the windows or generating electrical power. This paper will describe the analysis of several solutions used in automobiles.

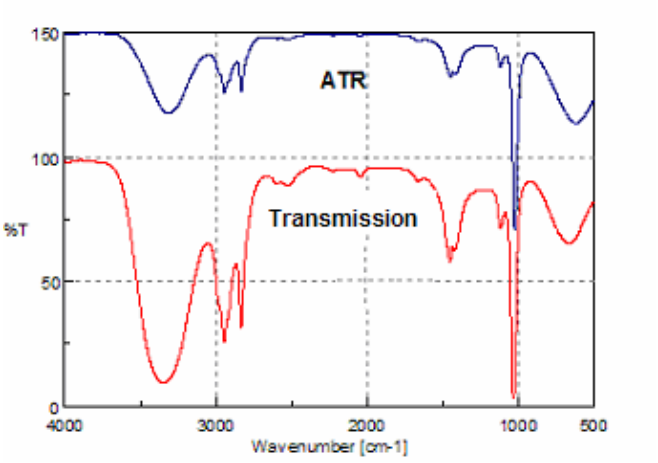

Figure 1. Transmission and ATR spectra of methanol.

The traditional infrared analysis method for fluid samples is to collect an infrared transmission spectrum of a thin film of the liquid contained between the windows of an infrared liquid cell. Not all liquids, however, can be analyzed with infrared spectroscopy in this manner. For samples that are aqueous, viscous or chemically reactive, an infrared liquid cell is cumbersome and labor intensive. Frequently, special windows with a reduced spectral range must be used and the cell can be difficult to keep clean to prevent cross contamination.

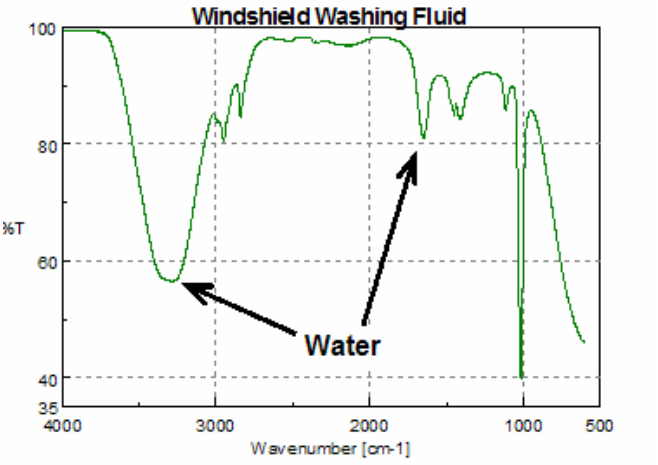

Figure 2. Spectrum of windshield washing solution.

Infrared analysis using Attenuated Total Reflectance (ATR) accessories like the diamond single-reflection micro-ATR require no sample preparation and greatly simplify the collection of FT-IR spectra. The liquid sample is placed onto the ATR crystal and the sample spectrum is collected. A volatiles cover supplied with the accessory can be used to cover the sample to prevent evaporation during analysis. The sample is then cleaned from the crystal surface and the accessory is ready to collect additional spectra. ATR analysis methods are less complicated than using liquid cells, are fast and a very small amount of the sample is needed.

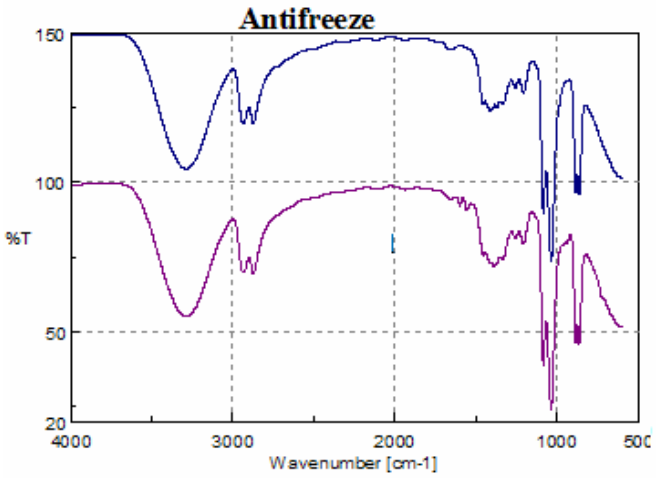

Figure 3. Spectra of two samples of anti-freeze, normal-blue and the extended lifetime-purple.

The resulting data can be searched against a digital database of ATR spectra for positive identification. Despite changes in the relative peak intensity of the absorption bands, due to the internal reflection mechanism of ATR accessories[1], spectra can also be compared to transmission data. As an example, Figure 1 is a plot of the transmission and ATR spectra of methanol.

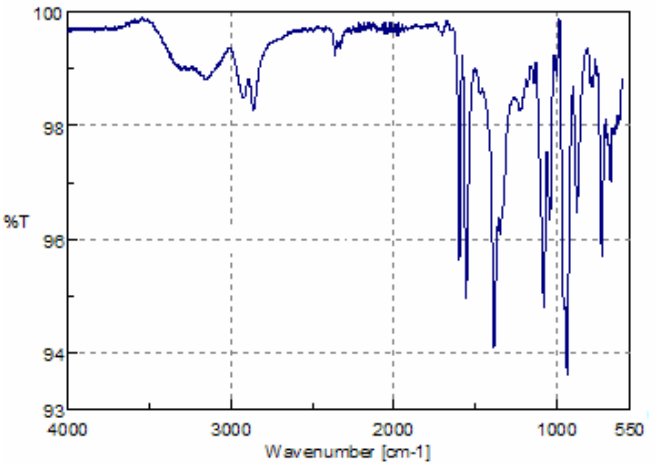

Figure. 4. Extended mileage antifreeze minus the normal.

Experimental Results

Spectra were collected using a Jasco FTIR-460 Fourier transform infrared spectrometer equipped with a Golden Gate™ micro-ATR accessory. Sample volumes of 20 microliters were pipetted onto the ATR surface and the spectra collected. No sample preparation was necessary to obtain the various spectra and the liquid sample is simply wiped from the crystal surface after data collection. If necessary, a solvent that will remove the sample is used to clean the diamond crystal surface.

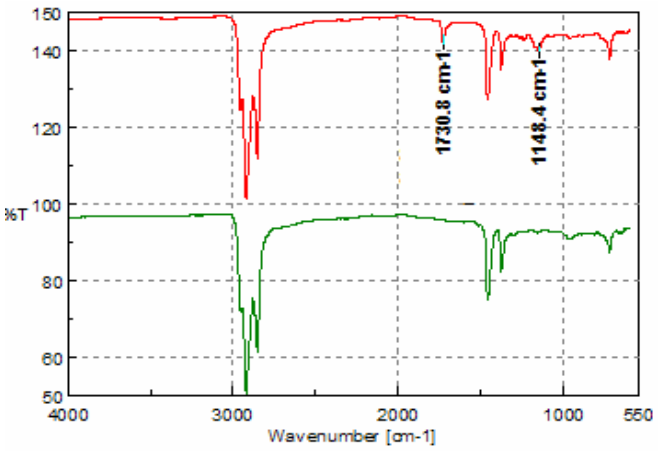

Figure 5. Analysis of 10W30 engine oil (green), power steering fluid and two transmission fluids (red)

FT-IR spectra of 64 scans at 4 cm^{-1} resolution were coadded and averaged to obtain the single-beam background and sample spectra. Figure 2 is a spectrum of windshield washing solution and it can be readily observed that methanol (Figure 1) is the major component. The water solvent increases the intensity and band broadening for the O-H stretch and bending modes.

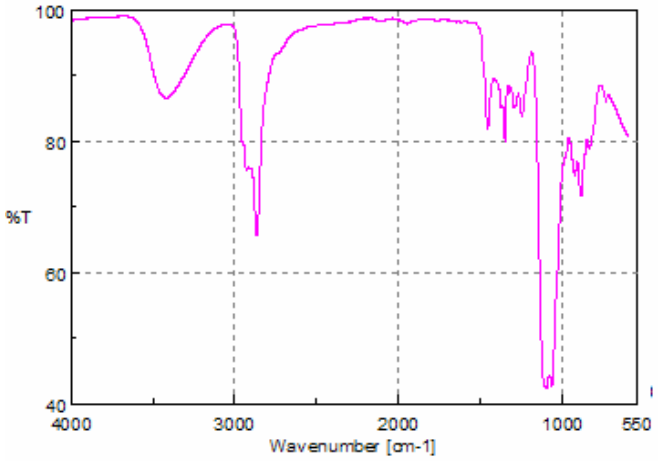

Figure 6. Spectrum of brake fluid.

Figure 3 illustrates the spectra of two samples of anti-freeze. While the spectra for the "normal" anti-freeze and the extended lifetime solution appeared identical, a subtraction of the standard ethylene glycol solution from the extended mileage formulation reveals a spectrum of the component(s) that presumably extends the anti-freeze lifetime.

Analysis of 10W 30 engine oil, power steering fluid and two automatic transmission fluids (ATF 3 and ATF 4) all yielded spectra representative of long-chain aliphatics as illustrated in Figure 5. The only discernible difference is the additional peaks at 1730 and 1151 cm^{-1}, which highlight a similar additive in the ATF and power steering samples. Figure 6 is a spectrum of brake fluid and was interpreted as a complex alcohol or possibly a diol, providing the requisite lubrication and compression characteristics.

The most challenging sample was battery acid. Mainly sulfuric acid, battery acid is extremely corrosive and will etch or react with almost every standard infrared window or ATR element. By contrast, the diamond element of the ATR is not damaged during analysis. The spectrum of the acid solution is displayed as Figure 7.

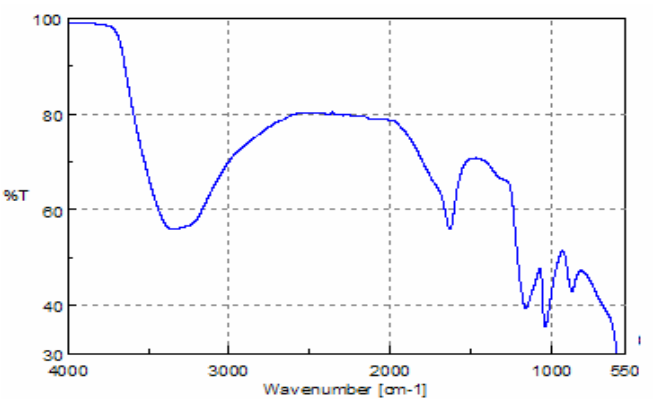

Figure 7. Battery Acid.

Conclusions

The Golden Gate™ single reflection micro-ATR is a simple, easy-to-use accessory for the analysis of liquids and solutions. The ATR technique is simple, rapid and very reliable for sample characterization. The analysis method is non-destructive and can be used to collect data from a minimal amount of sample.

Acknowledgements:

Pine Belt Automotive, Lakewood, NJ. and **Specac Inc, 500 Technology Court, Smyrna, GA 30082 Tel: 800 447 2558 Fax: 770 319 2488 www.specac.com, for the ATR.**

Contact:
Jasco, Inc.
8649 Commerce Dr. Easton, MD 21601
800-333-5272 Fax: 410-822-7526
www.jascoinc.com