Fourier Transform Infrared (FT-IR) Technique in the QC/QA Laboratory and on the Manufacturing Floor

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FT-IR, QA, QC, quality control, quality assurance, production monitoring

INTRODUCTION

The growth in popularity and acceptance of Fourier transform infrared (FT-IR) spectrometers for use in quality assurance laboratories and on manufacturing floors is one of the major developments affecting industrial environments in the last five years. FT-IR spectroscopy offers almost unlimited analytical opportunities in many areas of production and quality control. It covers a wide range of chemical applications especially in the analysis of organic compounds. In addition to its more classical role in qualitative analysis, its use in quantitative determinations has grown due to the high signal-to-noise of the technique coupled with the development of advanced statistical curve analysis algorithms. Thanks to compact design and ruggedness, the instrumentation can be located in the analytical laboratory near the production line and, in a growing number of cases, directly on-line. Low cost, speed and ease of analysis make FT-IR a method of choice for many industrial applications.

FT-IR spectrometers overcome many limitations affecting older, dispersive IR systems. The most import advantages include a drastic reduction of the time needed for data acquisition as well as improvements in energy throughput and sensitivity. Other benefits include the elimination of stray light interference which removes the need for external wavelength calibration. Modern FT-IR instrumentation features another advantage – a powerful computer as an integral part of the system. Thanks to this fact, in addition to data collection, storage, and manipulation, a typical FT-IR spectrometer can automatically identify samples by searching a wide range of commercially available digital spectral libraries. Several new sophisticated programs for the quantitative determination of multiple components in a single sample are also available. Many FT-IR spectrometers can be interfaced with host computers for automatic data transfer and remote operation.

Further aids to the growth of acceptance of FT-IR systems for routine quality control analysis are the lower cost and simplification of the user interface of many modern compact instruments. These attributes overcome some of the shortcomings FT-IR spectrometers that limit their use in this environment.

Recent developments in FT-IR instrumentation are paralleled by equally dramatic changes in sample handling techniques. An extensive range of new accessories simplify and, in many cases, eliminate tedious sample preparation. Several new sampling devices feature constant optical pathlength regardless of the sample volume or thickness, making reproducible quantitative analysis simple and elegant.

Nicolet Instrument Corporation, which manufactures a wide range of FT-IR spectrometers, offers products addressing the needs of the quality and process control markets. These dedicated chemical analyzers are tailored for the production plant laboratory environment. These product lines offer the full advantages of the latest FT-IR technology combined with a simplicity of push button operation. Rugged construction and a well-isolated optical compartment allow uninterrupted use of these instruments in many industrial laboratory environments. Depending on the sampling interface, the products can be used for gas, liquid or solid sample evaluations.

The analyzers are designed to handle specific methods developed for individual applications as an integral part of the system. This makes them ideal as dedicated, easy-to-use QA/QC and process control laboratory analyzers. The systems can be programmed to include custom methods which allow full flexibility of data collection, analysis, and interpretation of results with a single keystroke. This configuration permits operation by non-technical personnel. It

Figure 1: Protégé 460

also ensures consistent, high quality analytical results with a great degree of accuracy and precision. One of Nicolet's dedicated analyzers is shown in Figure 1.

Several application examples that are currently run on dedicated analyzers are presented herein:

Analysis of oxygenated extenders in gasoline

The use of organic extenders in gasoline for octane rating improvement and emission control is increasing. Owing to their unique spectral features, oxygenated extenders are easily detected and quantified in gasoline. Refer to Figure 2.

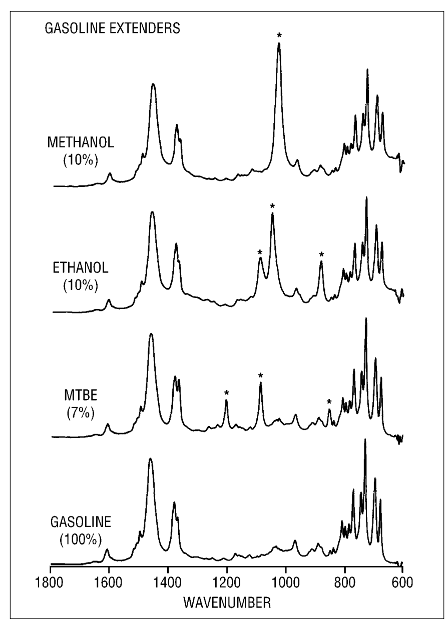

Figure 2: Methanol (10%), Ethanol (10%), and MTBE (7%) in gasoline spectra. Major extender peaks marked with (*)

Monitoring of toluene diisocyanate in pre-polymer mixtures.

Toluene diisocyanate (TDI) is used in various resin blends in the manufacture of various polymeric foams. The TDI concentration in a pre-polymer mixture affects the quality of the final product. Quantitative determination of TDI in resin blends is possible through the use of a horizontal attenuated reflectance accessory (HATR) mounted in the spectrometer. Once the calibration method is developed, the analysis can be performed with a single keystroke after placing the sample on the accessory. Refer to Figure 3.

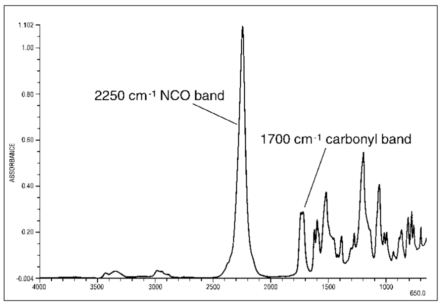

Figure 3: Toluene diisocyanate pre-polymer spectrum obtained on a horizontal ATR accessory. Bands used for quantitative determination are indicated

Monitoring of fluorination level of polyethylene materials

Chemically reinforced polyethylene finds many uses in industrial applications. Fluorination of the polyethylene surface is one of the processes for improving its performance. The fluorination level can be conveniently monitored using an FT-IR spectrometer. The infrared technique offers price and performance advantages over currently used neutron activation analysis (NAA) and electron spectroscopy (ESCA). Refer to Figure 4.

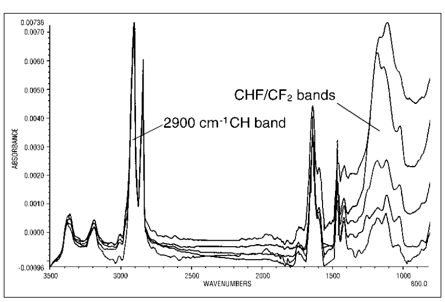

Figure 4: Series of calibration spectra obtained for fluorinated polyethylene samples. Bands used for determination of fluorination levels are indicated

Monitoring corn syrup manufacturing

Rapid measurement of dextrose equivalent (DE) and dry substance (DS) at intermediate steps of corn syrup processing allows for better control of syrup production. The parameters are calibrated against the reference method using Partial Least Squares quantitative analysis software. Refer to Figure 5.

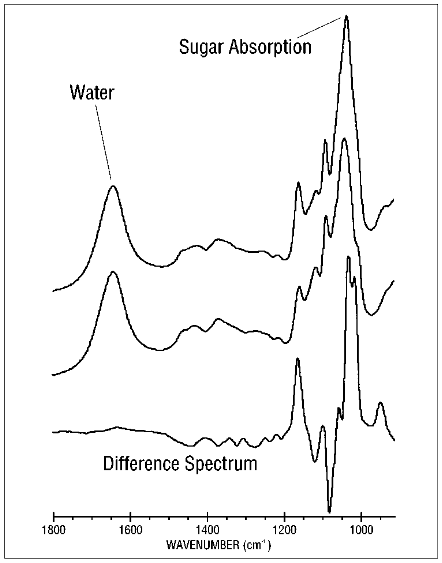

Figure 5: Spectra of corn syrup production lot samples having different DE and DS levels with the spectral subtraction result below

Lubricating oil blending quality control

Lubricating oils are blended from different components including base oil, additive packages, pour point depressants, and viscosity index improvers. FT-IR can be used to measure the levels of these components in the finished product. Refer to Figure 6.

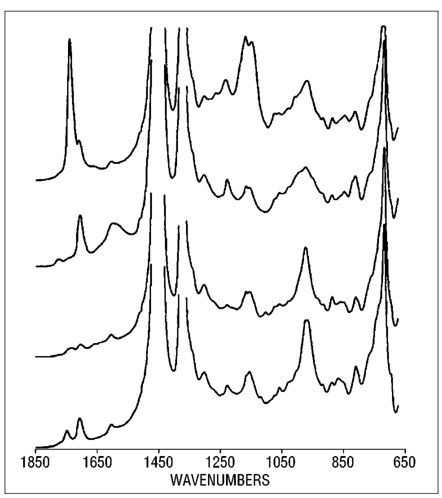

Figure 6: Overlay spectra of commercial lubricating oils showing differences in base oils and additives

Lubricating oil condition monitoring

FT-IR analysis of used lubricating fluids followed by subtraction of the appropriate new oil reference is an effective tool for monitoring changes in the lubricant during its use. The changes that occur are the result of oxidation processes or contamination from other parts of the mechanical system. Refer to Figure 7.

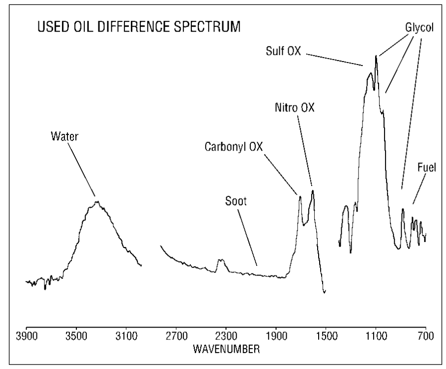

Figure 7: Typical used engine oil difference spectrum with components of interest in condition monitoring labeled

Hydroxyl number determination in polypropylene glycols

Knowledge of the hydroxyl group content of polypropylene glycol is important for predicting the functional characteristics that will be imparted on the products for which they are used. Assessment of this value can be quickly and easily done using FT-IR. Refer to Figure 8.

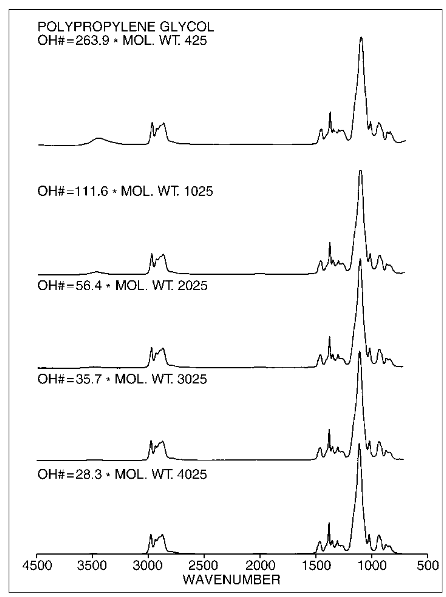

Figure 8: Spectra of polypropylene glycol samples with different hydroxyl values

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