An FT-NIR Primer

Industry: **Oils and Gas**
Product: **NR800**

Near Infra-red Spectroscopy

Like other forms of spectroscopy, light from a lamp (usually called the "source") is passed through a sample and measured by a detector. The Beer-Lambert law (also called the Beer-Lambert-Bouguer law or simply Beer's law) is the linear relationship between absorbance and concentration of an absorber of electro-magnetic radiation. The general Beer-Lambert law is usually written as:

$$A = a_{\lambda} \times b \times c$$

Where A is the measured absorbance, a is a wavelength-dependent absorptivity coefficient, b is the path length, and c is the analyte concentration.

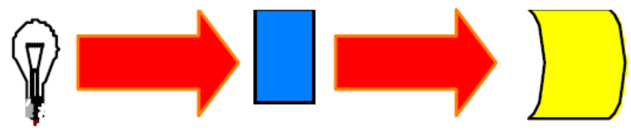
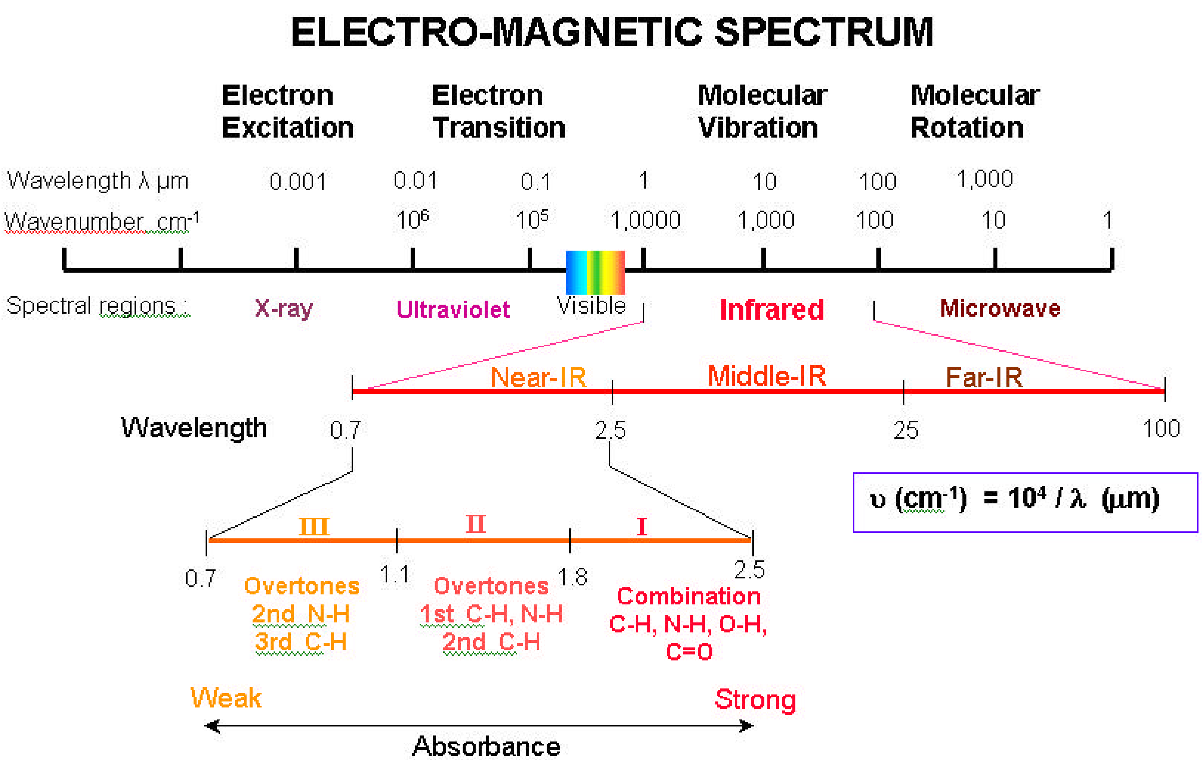


Figure 1: A basic photometer

In Near Infra-red spectroscopy we choose a light source and detector that will operate through most of the NIR range, approximately 700 – 2500 nm (14,000 – 4,000 cm-1).



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Types of NIR Spectrometers

First we should differentiate between filter photometers and spectrometers. A filter photometer does not produce a spectrum. It uses a filter or system of filters to choose a small portion of the spectrum and measure the light absorbed by the sample. Filter photometers are relatively simple and inexpensive devices that can be used successfully for some measurements. In the case of filter photometers, the absorbance of the sample across the wavelength range selected by the filter is calibrated according to the Beer-Lambert Law. This works quite well for a number of measurements, particularly in binary mixtures and mixtures which can be treated as binary. Using multiple filters in system, it is possible to measure multiple components.

A spectrometer produces a spectrum that is the absorbance at many wavelengths, which allows us to measure very complex mixtures. We correlate changes in concentration with the changes in absorbance at many wavelengths at the same time. In this way, multiple components can be measured simultaneously, and spectral interference can be eliminated.

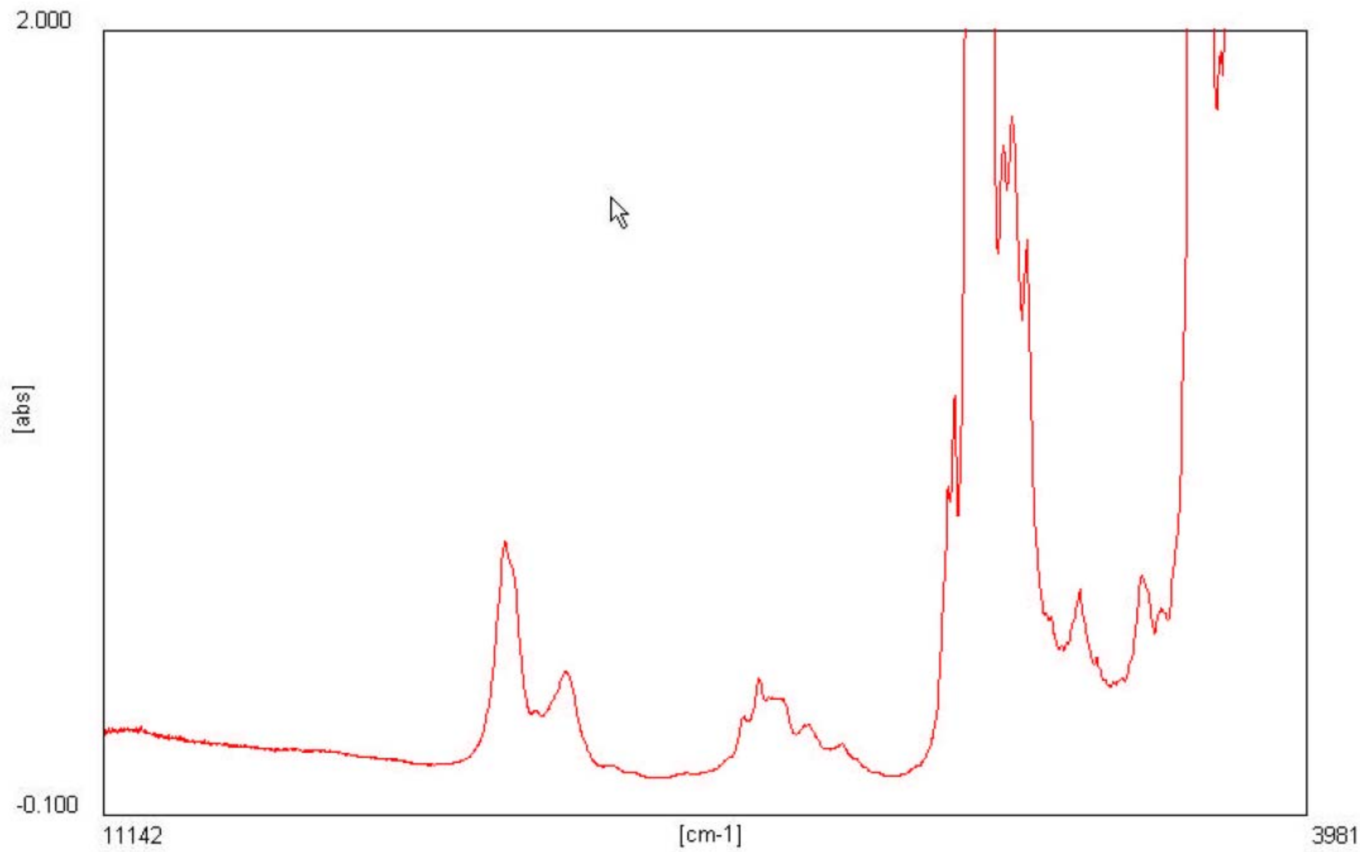

Figure 2: NIR Spectrum of Toluene

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The original infrared instruments were of the **dispersive** type. These instruments employ a prism or grating which works exactly the same as a prism separates visible light into its colors (frequencies). Gratings are a more modern approach, which better separates the frequencies of infrared radiation, offering better resolution.

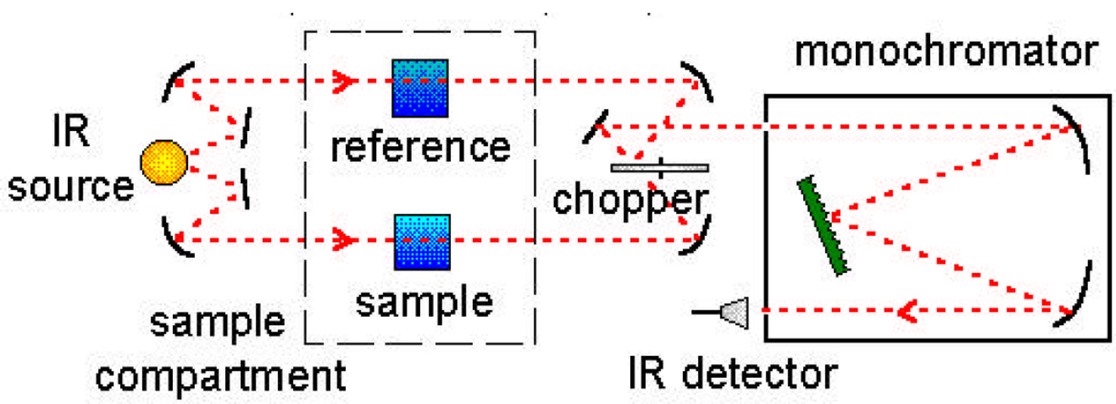
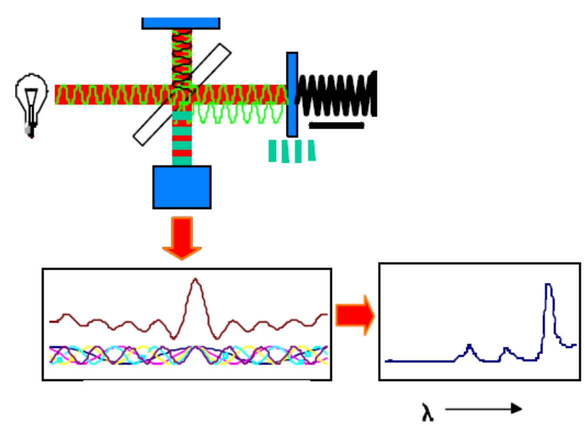


Figure 3: Dispersive IR System

Dispersive systems can only measure a small part of the spectrum at a time, so they usually scan the spectrum by moving the grating, the mirrors or the detector. These early systems worked reasonably well in laboratories, where temperature variations and vibration could be controlled. In process environments dispersive systems are plagued by variations in wavelength calibration, baseline stability and mechanical reliability. The small movements in the optical components caused by changes in temperature or vibration severely affect the measurement of absorbance at each wavelength, which in turn affects the multivariate calibration models. In some instruments, the IR detector consists of a diode array, in order to measure more of the spectrum at once and minimize movement of the optical components, but these instruments tend to be limited in resolution by the number of elements in the diode array.

FT-NIR Advantages

Most Fourier Transform Near Infra-red (FT-NIR) spectrometers are based on the Michelson interferometer, where the light from an NIR source lamp is split and reflected off two mirrors, then recombined. One of the mirrors is fixed, while the other moved. The various wavelengths of light interfere with each other, causing a change in the intensity of the light at the detector. The resulting measurement at the detector is light intensity vs. time called an **interferogram**. This signal is converted using the **Fourier Transform** from the time to the frequency domain to produce a spectrum.



FT-NIR spectrometers have three very well known advantages over dispersive NIR systems:

The **Fellgett Advantage** refers to the fact that all frequencies are measured simultaneously. This gives FT-NIR systems a great advantage in the speed. The spectrum can be acquired in a fraction of a second, instead of several minutes. Also, there is less likelihood of changes in the spectrum due to changes in mechanical alignment during the scan, so spectra are more reproducible.

FT-NIR instruments employ a HeNe laser, which is aligned with the IR source and passes through the interferometer along with the IR radiation as an internal wavelength calibration. The **Connes Advantage** is that FT instruments are wavelength calibrated on each scan, and so wavelength accuracy is unaffected by changes in temperature or vibration. Since all the light that passes through the sample is measured at the same time, the light throughput to the detector of FT instruments is much greater, resulting in much higher sensitivity and lower noise levels. This is the **Jacquinot Advantage**.

There are a number of different interferometer designs used by different manufacturers. All have the three advantages listed above, and all benefit from being mechanically less complex than dispersive systems and therefore, more reliable. Most interferometer designs were originally developed for laboratory spectrometers and employ motors, slides, or other friction parts for moving the mirror. Until recently, there has not been an FT-NIR system designed from the beginning as a process instrument, intended for rugged industrial applications, with a virtually frictionless interferometer mechanism.

Yokogawa FT-NIR

Yokogawa has developed a rugged, high resolution, high signal-to-noise ratio interferometer for process FT-NIR which employs a set of four spring steel plates supporting the moving mirror. The driving force comes from a voice coil that has no friction parts. A second damping voice coil is mounted at the top of the carriage. The spring steel plates are made of the same material, so changes with temperature are minimized. The spring steel plates allow the mirror to move up to 2.5 mm, giving the analyzer a 4 cm^{-1} resolution. The spring steel plates resist movement from side to side making it resistant to vibration. To further dampen vibration, the interferometer is installed, along with the IR source and laser, in a cast aluminum enclosure mounted on soft rubber cushions.

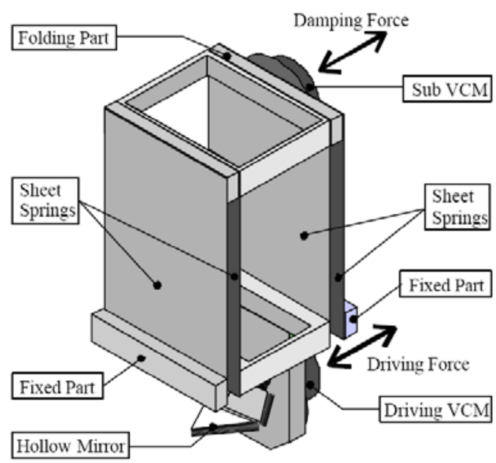


Figure 4: Yokogawa NR800 FT-NIR Interferometer Actuator

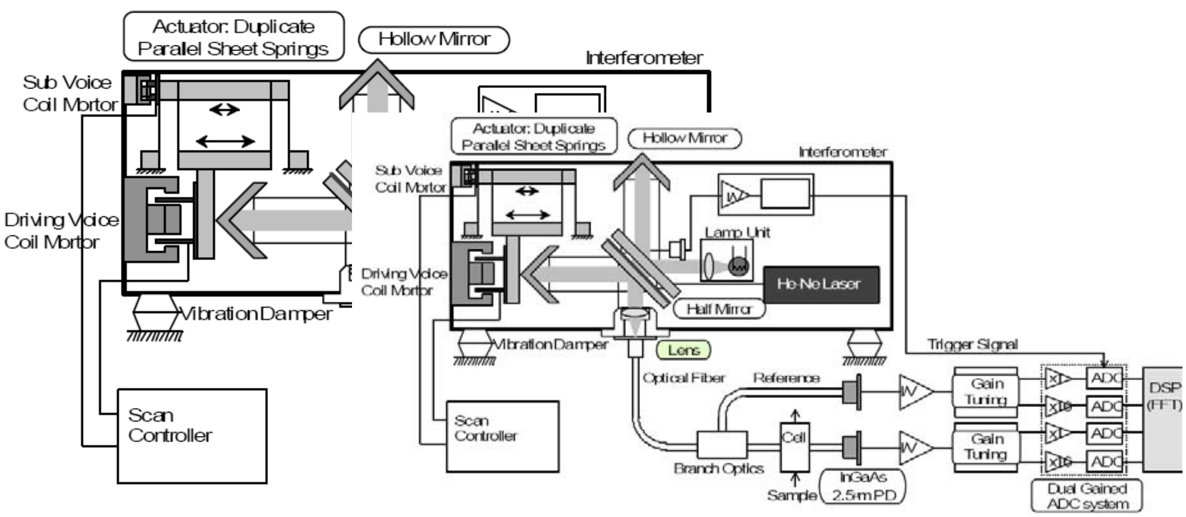


Figure 5: Yokogawa InfraSpec NR800 block diagram

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Outside the interferometer, the InfraSpec NR800 optical path is fiber optic. The NR800 is designed as a process instrument and many of the processes being monitored will be flammable, toxic or both. For safety and cost reasons it is undesirable to bring flammable sample streams into the room or enclosure where the analyzer is mounted where small leaks and routine sample conditioning system maintenance can lead to accumulation of vapors or expose people to flammable or toxic liquids. Up to 4 sample cells or probes can be mounted up to 300m from the analyzer. All four cells can be monitored simultaneously and the onboard computer supports calibration models for up to 12 simultaneous measurements per channel. Infrared radiation leaves the interferometer enclosure through a collimating lens and through infrared optical fiber to a splitter inside the analyzer cabinet. The light is split into sample and reference beams with separate optical fibers bundled into a single cable. Both the sample and reference beams are carried to the flow cell or probe and back to analyzer cabinet to separate detectors. Since the refractive index of fiber optic cable changes with temperature, the dual-beam optical configuration is designed to compensate for changes in the fiber optic cable by comparing the sample and reference beams. Thus for a 4-channel system, the analyzer will have 8 detectors, 4 sample detectors and 4 reference detectors. The Yokogawa InfraSpec NR800 uses a newly developed InGaAs (Indium Gallium Arsenide) detector having a spectral range of 900 - 2500 nm (11,000 – 4000 cm^{-1}). The spectrometer covers a ll the NIR spectral zones, including the important combination zone. Yokogawa has developed dual gain auto-ranging Analog-to-Digital Converters (ADC), which produces a very high signal-to-noise ratio (less than 50 µAbs units RMS noise at 4800 – 5800 cm^{-1}. A typical configuration for 2 channels is shown in Figure 6.

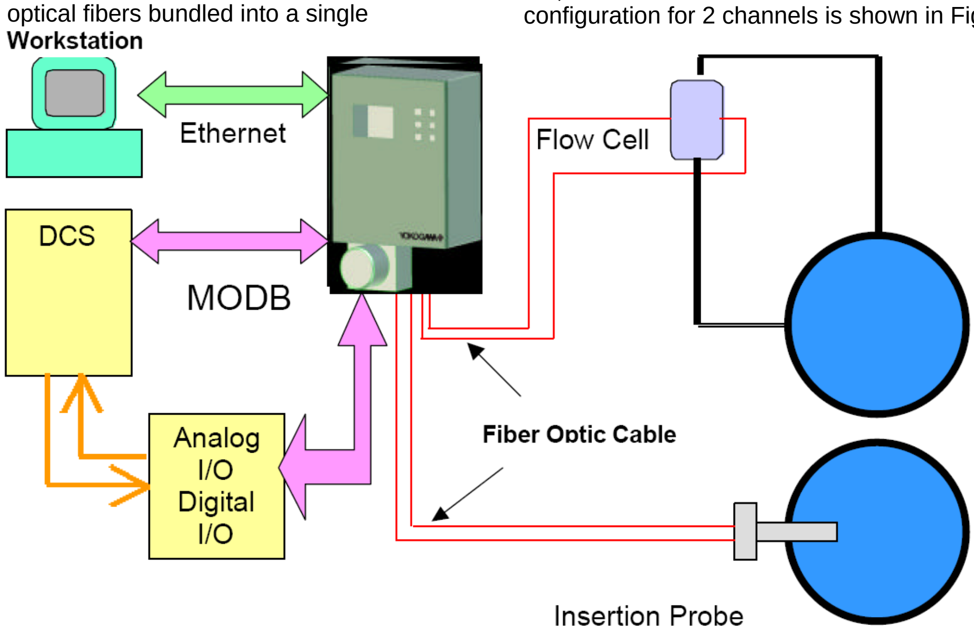

Figure 6: Typical configuration for the InfraSpec NR800 for two channels

Conclusion

The advantages of FT-NIR over dispersive NIR systems are well known in laboratories. The purpose of developing a new Michelson-type interferometer, new wide-range InGaAs detectors and new low-noise circuitry was to provide the spectrometric performance required for fast, simultaneous multi-component and multi-channel NIR analysis in the process environment. Ultimately, the success of an analyzer application is dependent on how well a spectrometer produces the spectra. Stability, reliability, wavelength accuracy, resistance to vibration and temperature change are all factors that determine the performance of the analyzer and the application. The Yokogawa Infra-Spec NR800 is designed to bring a new level of performance to a process field analyzer.