

Comparison of FT-NIR and NMR Spectral Analysis

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Overview

FT-NIR and NMR spectroscopy techniques are popular analytical tools for many chemists because of its versatility in elucidating molecular structure, optimizing reaction dynamics, measuring reaction kinetics, monitoring reaction content and controlling product purity. These two technologies are both nondestructive and complimentary to each other; and provide a wealth of information on physical, chemical and structural aspects of molecular systems. This poster presents spectral data of common solvent and liquid mixture analysis using a Thermo Scientific™ Antaris™ FT-NIR analyzer and a Thermo Scientific™ picoSpin™ portable NMR spectrometer.

Thermo Scientific Antaris FT-NIR and picoSpin NMR Instrumentation

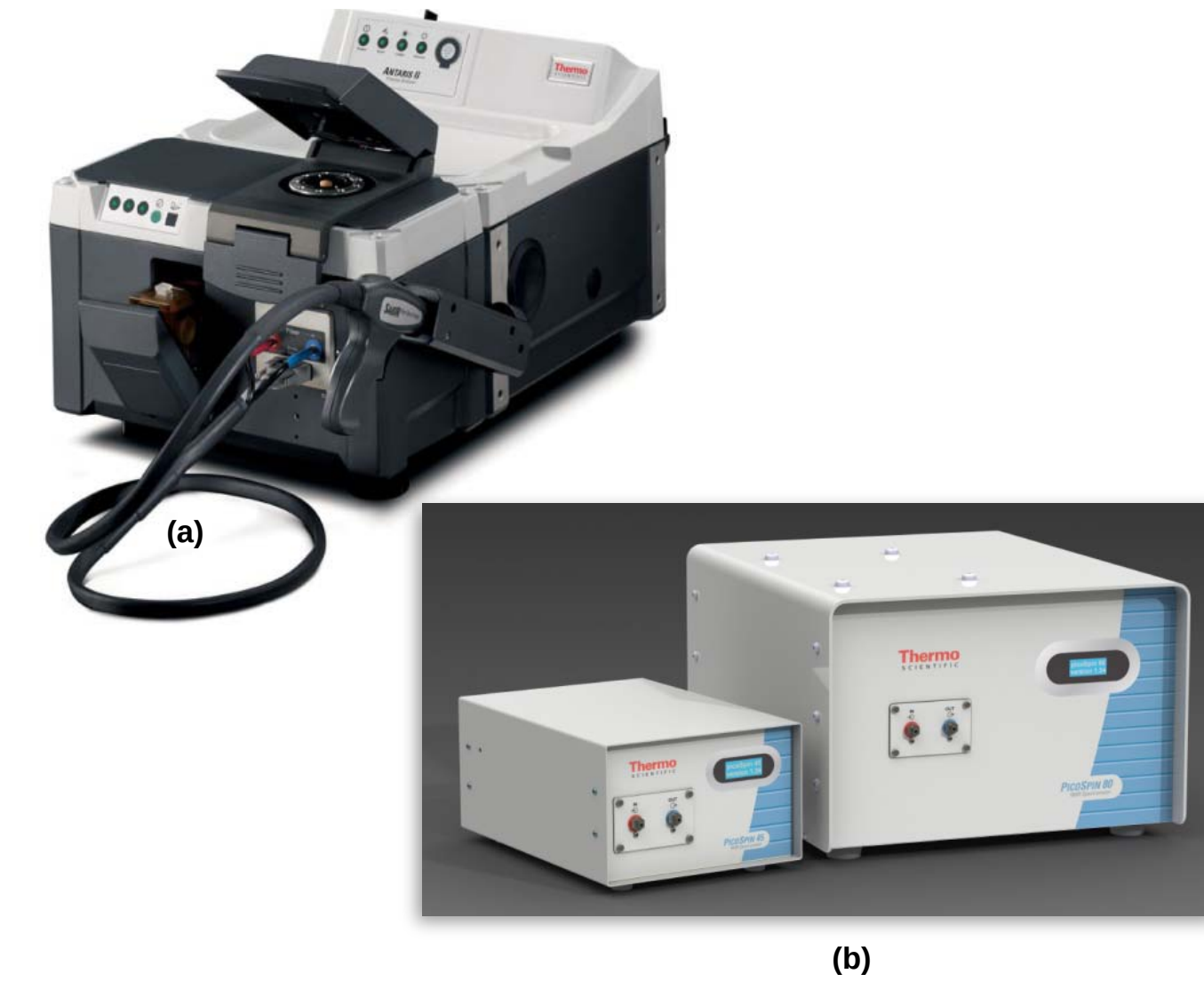
Antaris FT-NIR Analyzer

Thermo Scientific Antaris is the dedicated FT-NIR analyzer built specifically for use in the industrial environments of the pharmaceutical, chemical, and polymer industries. Uses include identification of incoming material at load docks and checking product quality on factory floors. Antaris is the first fully integrated system with various sampling solutions for different applications that meets regulatory requirements.. It offers everything you need to run any sample, without reconfiguring the analyzer or changing accessories.

picoSpin NMR Spectrometer

The Thermo Scientific™ picoSpin™ 80 spectrometer is a true breakthrough in chemical instrumentation. It provides the power of a standard Nuclear Magnetic Resonance (NMR) spectroscopy instrument in a compact and affordable form. The superior resolution of the 2 Tesla magnet reveals chemical information not available using weaker magnetic fields. The easy of use and affordable NMR spectrometer offers: Practical performance – small molecule sensitivity designed for everyday use Personal convenience – portable NMR without waiting for core facility schedules Low cost operation – no cryogenics, no deuterated solvents, mains energy supply Unique technology – capillary cartridge system ideal for monitoring chemistry

FIGURE 1. (a) Antaris FT-NIR Analyzer; (b) picoSpin 45 and picoSpin 80 NMR Spectrometers



FT-NIR Experiment and Spectral Data

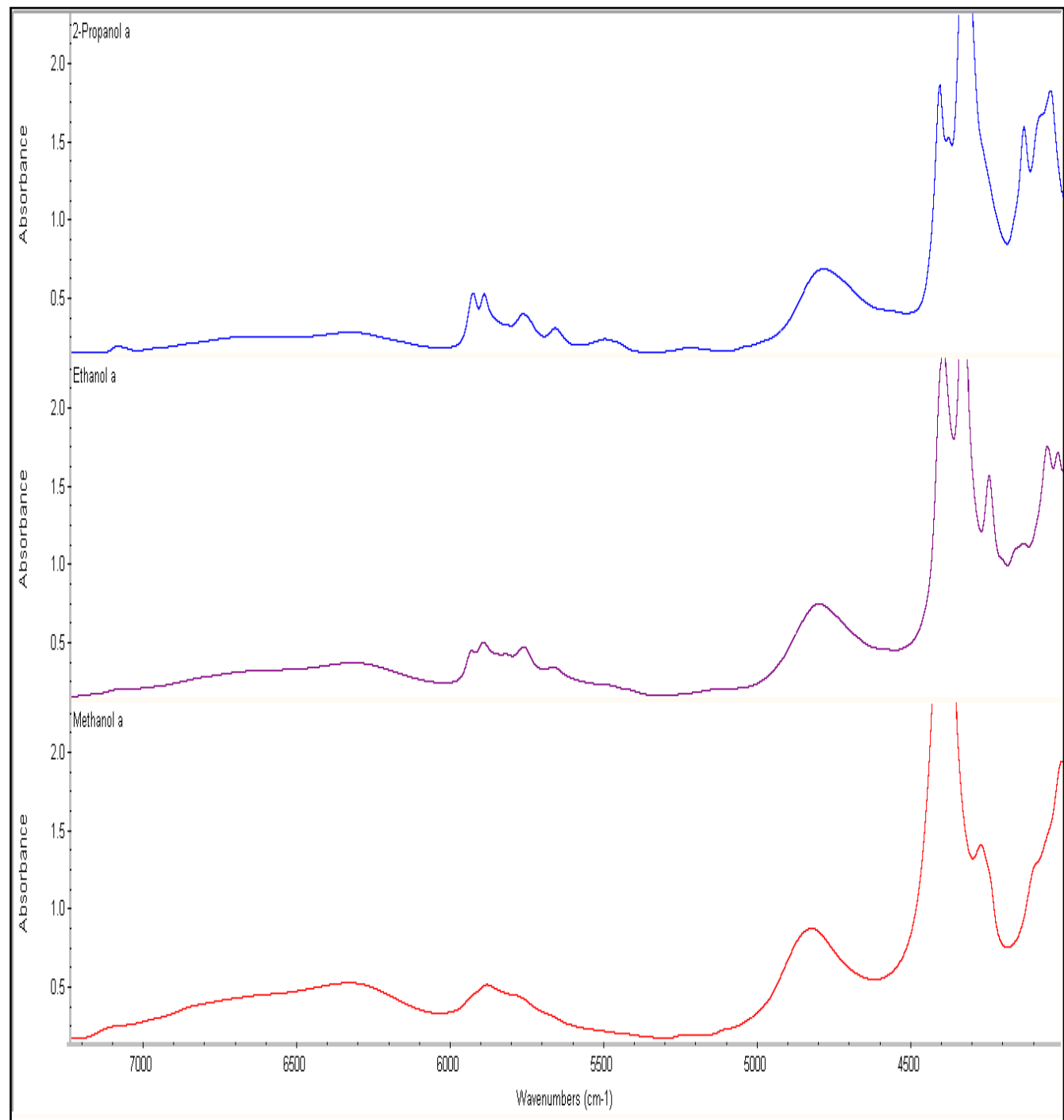
Liquid Transmission Sampling Technique

Solvent Samples were transferred to a 1 mm path length transmission cuvette, and scanned in the transmission sampling module of Antaris FT-NIR MDS analyzer. The transmission sampling arrangement involves a three position sample shuttle with a dual temperature controlled (heated) cell-block (see Figure 2). The sample holder can accept the disposable vials or cuvettes and keep them at the computer selectable temperature. Collection of background spectrum is done automatically, allowing both the sample and background to be measured in the exact same conditions. The scanning parameters were 32 scans for spectral averaging, 8 cm⁻¹ resolution, and the wavelength range was from 4000 cm⁻¹ to 10,000 cm⁻¹.

FIGURE 2. Antaris II FT-NIR Liquid Sampling Technique



FIGURE 3. FT-NIR Spectral Plots of Methanol, Ethanol, and 2-Propanol Solvents



picoSpin 80 NMR Spectral Data

Sample spectra were acquired using a picoSpin 80 NMR spectrometer. The spectrometer is an 82 MHz pulsed FT 1H NMR permanent magnet instrument. It uses capillary sample technique for the spectral measurement. Solvent samples were manually injected into the 1/16" sampling port with a 1 ml slip-tip polypropylene syringe and 22-gauge blunt-tip needle. The fluid path is 400 micro meter ID Teflon/Quartz capillary tubing with a total flow path volume of 40 micro liters, and an active volume within the RF coil of 70 nL. Each sample was scanned 16 times and averaged, and the total sampling time was about 1.5 minutes. The sample FID data was processed and plotted with mNova™ NMR software.

FIGURE 4. picoSpin NMR Spectrometer: Capillary cartridge and magnet

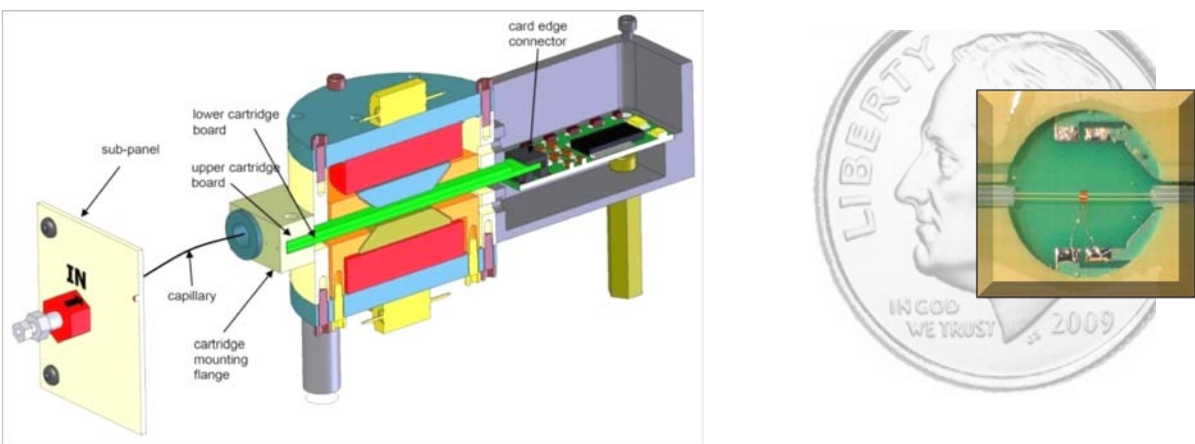
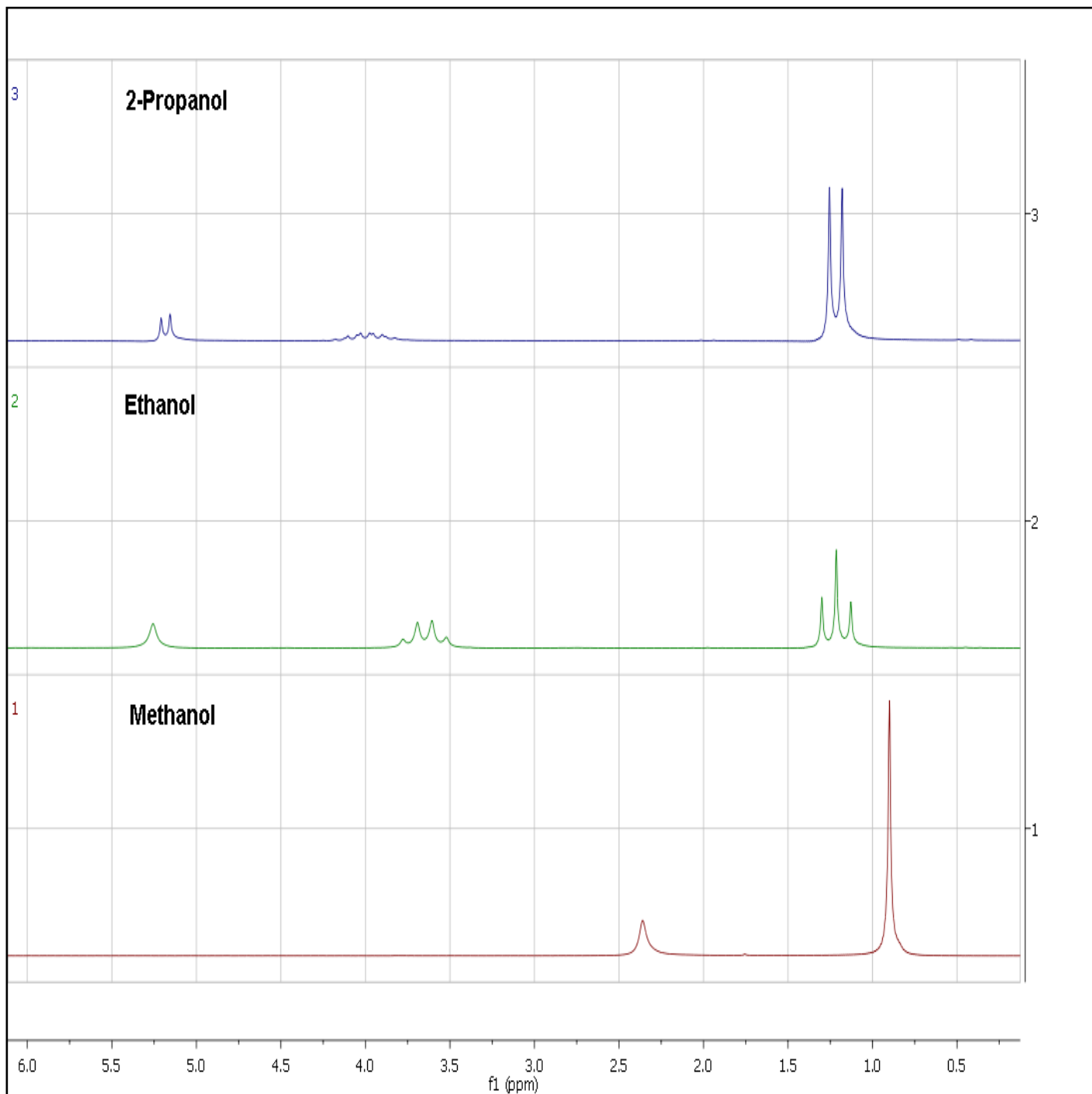


FIGURE 5. NMR Spectral Plots of Methanol, Ethanol, and 2-Propanol Solvents



FT-NIR and NMR for Mixture Analysis

As a demonstration of mixture analysis, various amount of methanol was added to ethanol to generate mixture solutions, and the solution samples were then scanned by both the Antaris FT-NIR analyzer and picoSpin 80 NMR spectrometer. The FT-NIR data was processed with Thermo Scientific™ TQ Analyst™ Chemomatrix software and the NMR data was processed with mNova™ software.

FIGURE 6. FT-NIR Absorbance Spectra of Methanol in Ethanol Samples

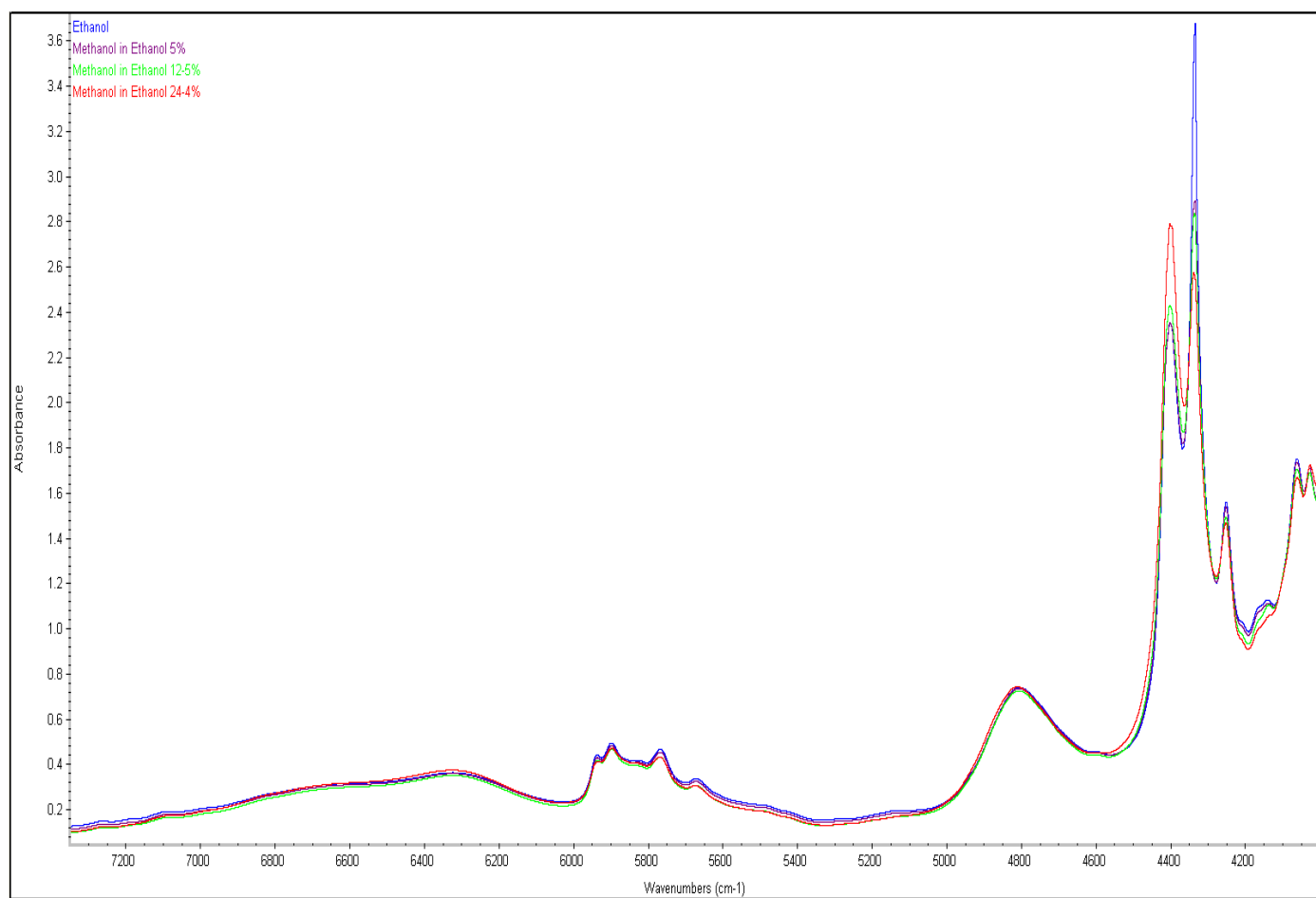


FIGURE 7. FT-NIR 2nd Derivative Spectra of Methanol in Ethanol Samples

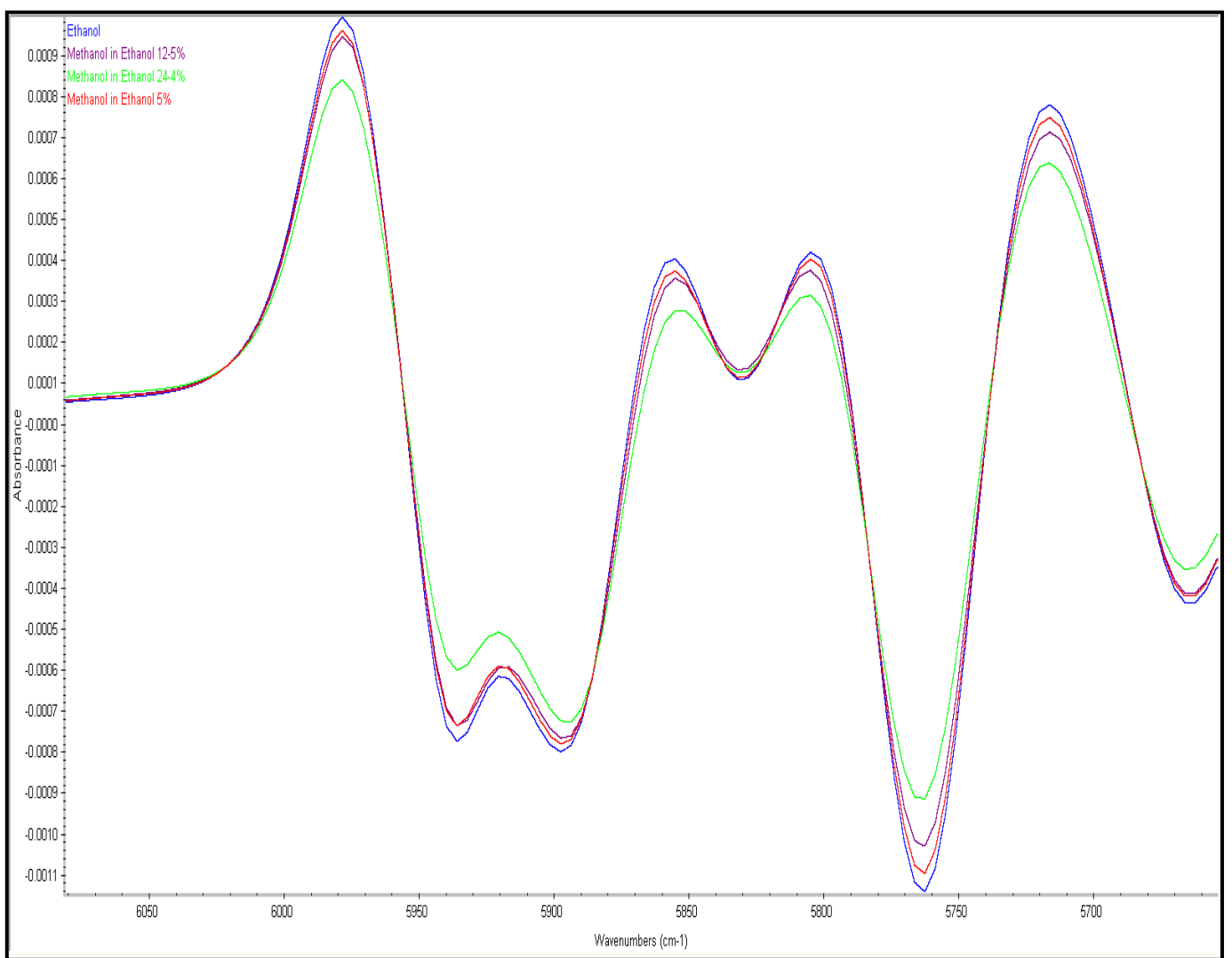
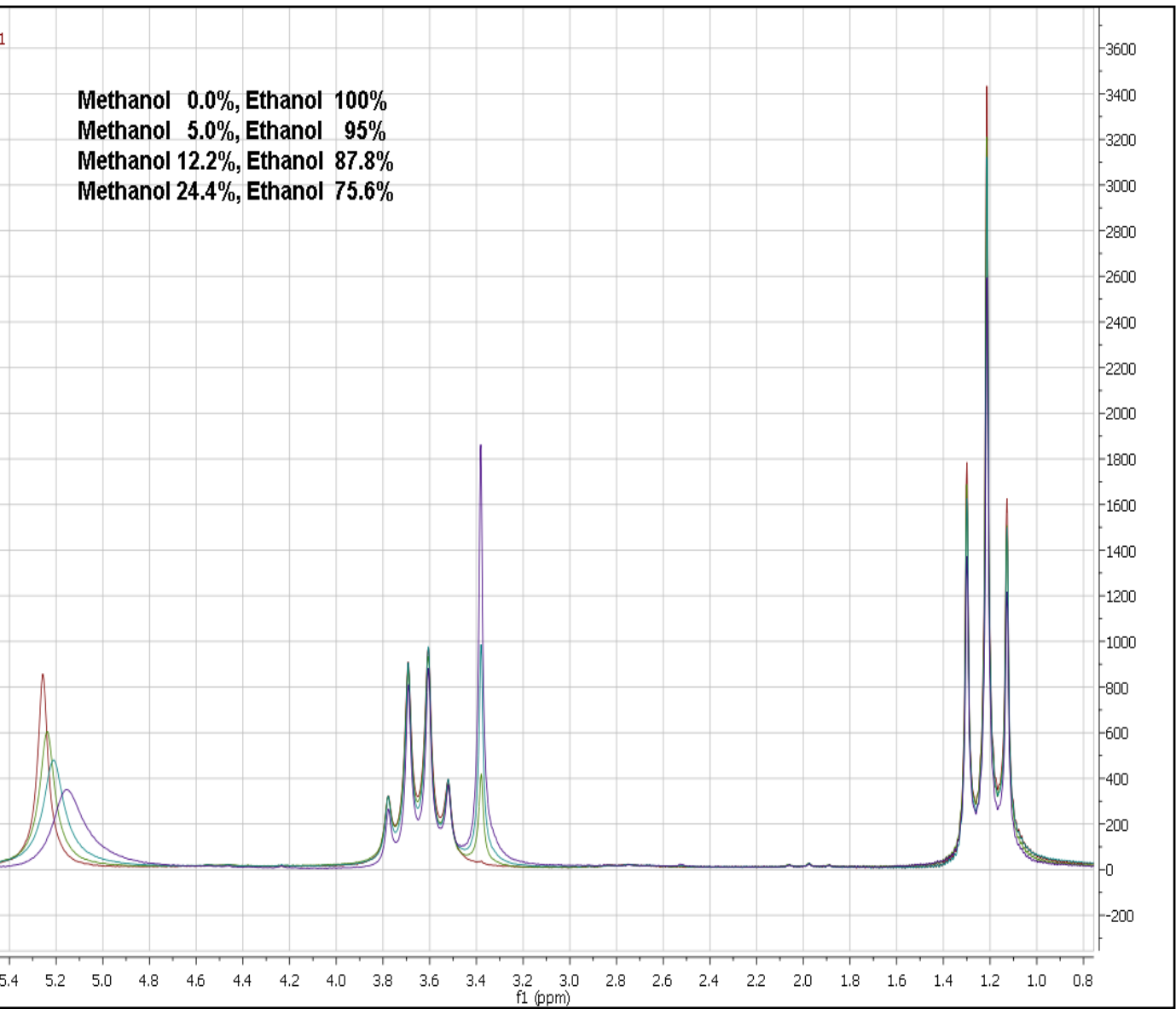


FIGURE 8. NMR Spectral Plots of Methanol in Ethanol



Conclusions

With the advance of new electronic and science technologies, more analytical tools to obtain a deep scientific and engineering understanding of products are available to the pharmaceutical, food, and chemical industries. This post has illustrated that both the Thermo Scientific Antaris FT-NIR analyzer and benchtop picoSpin 80 NMR spectrometer provide chemical structure selectivity and sensitivity for the material classification and composition analysis.

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