Ocean Insight

# Cleaning Up IPA Production with Stage-by-Stage MIR Analysis

Application Note


**KEYWORDS**

- Isopropyl alcohol
- Volume fraction
- Ocean MZ5 MIR spectrometer

**TECHNIQUES**

- Attenuated total reflectance
- MIR spectroscopy

**APPLICATIONS**

- Quality testing
- Process control



2-Propanol (also known as isopropyl alcohol or IPA) is one of the most common solvents in the world, with over 2 million tons produced in 2003 (Sciencing, 2017). The IPA market is expected to reach $6 billion/year or more by 2024 (Isopropyl Alcohol Market, Consumption, Forecast, and Global Analysis: By Regions and Applications, 2018).

A large majority of the IPA produced in the U.S. is made by indirect hydration of propene and sulfuric acid, since this process requires lower quality propene compared to other production methods.

The production of IPA through the indirect hydration process involves the reaction between propylene and sulfuric acid, followed by hydrolysis (NCBI, 2012). IPA forms an 87 wt.% azeotrope with water, and has a boiling point of around 80 °C, which prevents it from being distilled to pure IPA without adding a ternary component, such as diisopropyl ether or cyclohexane.

It is important to spot check volume fractions at various stages in the production process as each stage of the distillation requires certain volume fractions for the system to operate at maximum efficiency. Each step along the process has a unique chemical composition: from equal parts IPA and water, to high concentrations of IPA, high concentrations of cyclohexane, etc. Since MZ5 was successfully able to determine volume fractions in a binary mixture, it was only natural for its next test to be ternary (3-part) mixtures.

## About the MZ5 ATR-MIR Spectrometer

The Ocean MZ5 is both a research tool and a core technology for integration into fully customized solutions for applications such as fuel monitoring and detection of food adulterants.

For the lab, MZ5's small size and ease of use make it an ideal benchtop/fume hood device, which is how we used it for this study. University and institutional researchers investigating biofuels and other energy sources will also appreciate the MZ5's versatility.

For offline applications, MZ5 can anchor a devoted R&D station where process scientists analyze new additives and mixes and build spectral databases. In addition, QC stations within process lines can utilize MZ5 to spot check process fluid streams.

## Experiment Setup

Several mixtures were made using 2-propanol (HPLC grade) from Fisher Scientific, and water (ASTM type 1) and cyclohexane (ReagentPlus, ≥99%) from Millipore Sigma to test several steps along the production process, from crude IPA to 99.9+% anhydrous isopropyl alcohol. **Table 1** shows the volume fractions that were prepared.

**Table 1**

| Φ2 - Propanol | Φ Water | Φ Cyclohexane |
|---|---|---|
| 0.14 | 0.01 | 0.85 |
| 0.45 | 0.05 | 0.50 |
| 0.50 | 0.50 | 0.00 |
| 0.60 | 0.35 | 0.05 |
| 1.00 | 0.00 | 0.00 |

The samples were analyzed using the ATR crystal on the MZ5 spectrometer and the default settings in the Mirror operating software.

## Absorbance Model

**Figure 1** shows the resulting absorbance spectra for the mixtures listed in Table 1, along with pure water, IPA and cyclohexane for reference. Clearly IPA, water and cyclohexane all have distinctly different spectral shapes, which is one of the benefits of MIR spectroscopy. IPA is the dominant influencer of the spectra; however, water is quite absorbent across the entire MIR range measured with MZ5.

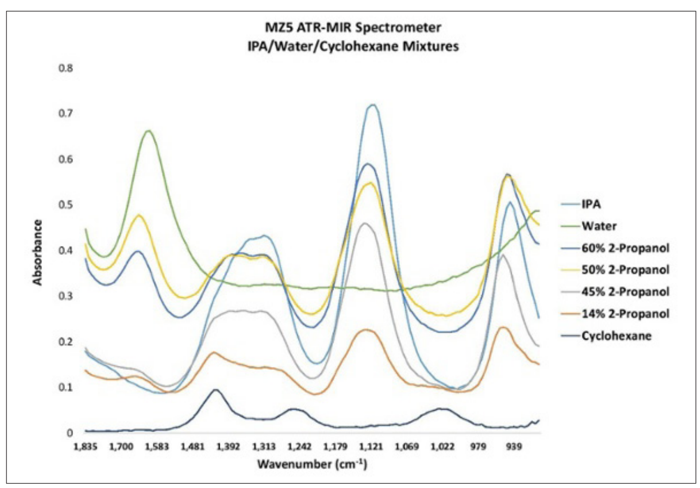


*Figure 1. Absorbance of IPA/Water/Cyclohexane Mixtures*

**Figure 2** shows a plot of volume fraction versus absorbance. The strong peak around 1121 $cm^{-1}$ corresponds to the strong C-O stretching of IPA, a secondary alcohol. This strong peak allows for determination of the volume fraction of the ternary mixtures.

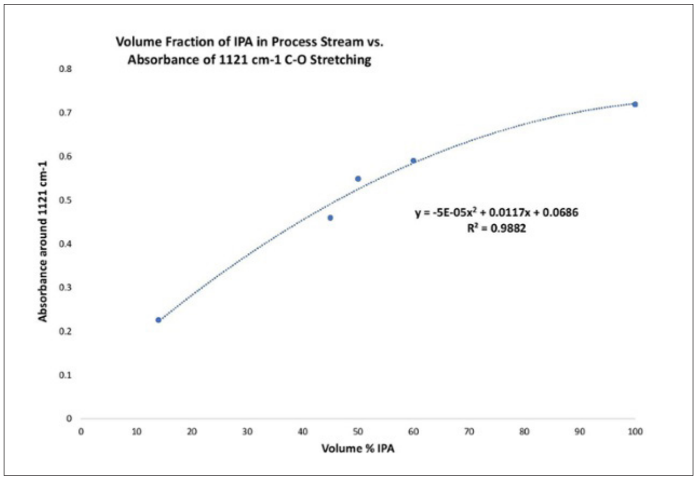


*Figure 2. Absorbance trend of C-O Stretching Band*

**Figure 3** shows the corrected MZ5 absorbance spectra for the mixtures listed in Table 1. Each spectrum was normalized with respect to pure IPA to more clearly illustrate the differences in the spectra.

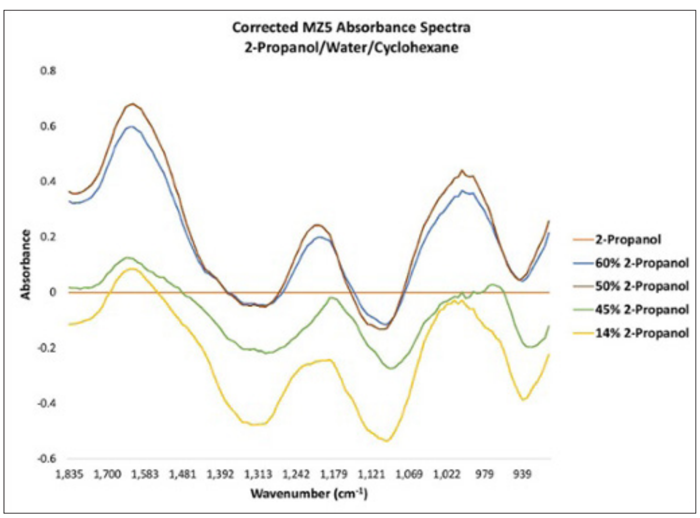


*Figure 3. Corrected Absorbance of IPA/Water/Cyclohexane Mixtures*

**Figure 4** shows a plot of volume fraction versus corrected absorbance. Again, there is a very strong correlation that allows for accurate volume fraction determinations across a wide range of IPA concentrations.

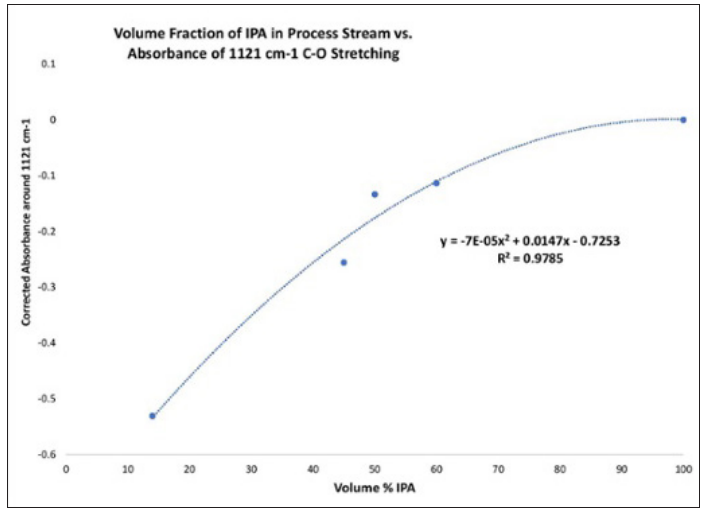


*Figure 4. Corrected Absorbance trend of C-O Stretching Band*

# Principal Component Analysis (PCA)

Real-world measurements are often disturbed by some unwanted influence such as an impurity or fluctuating environmental parameter. When these disturbances translate to distortions and shifts in the spectral output there is need for more advanced correction and broadband characterization of the incoming signal. As computing power grows in both strength and general accessibility, the ability to use numerical methods such as principal component analysis on massive data sets becomes increasingly valuable and mainstream.

PCA is quite popular in many disciplines, and the world of spectroscopy is no stranger to it, either. PCA offers a quick way of classifying the incoming spectra without relying on background knowledge of the physiochemical and optochemical interactions of the system. The plot in Figure 5 shows the tight PCA distributions of the raw spectra from the spectrometer.

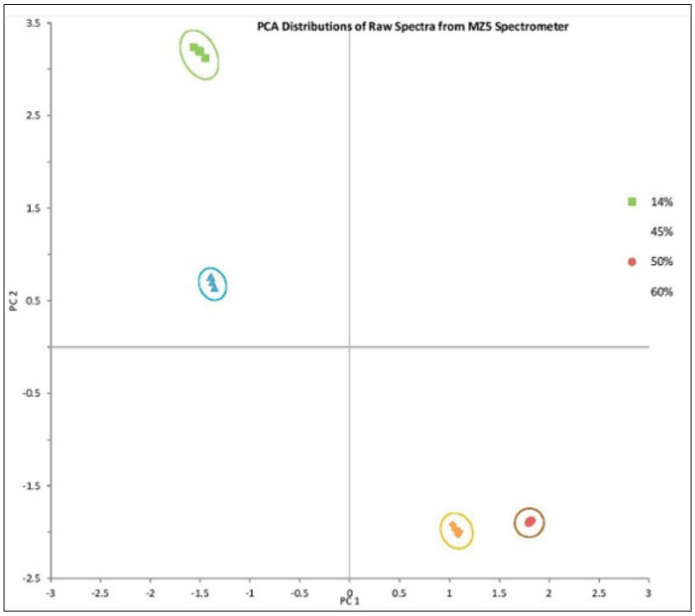


*Figure 5. PCA is a useful tool for deriving information from large data sets.*

# Conclusion

This study truly illustrates the power of MZ5, and the MIR engine inside of it. Determining volume fraction of a single component in a ternary system can often be difficult or inaccurate. Yet with MZ5, the process is quick, simple and accurate.

Also, Ocean Insight can provide users with algorithm development and machine learning tools that derive additional insight from the spectral data the MZ5 collects. Tools can be developed and refined to handle complex fuel mixtures and to account for issues associated with optical interference.

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