

Specac

Application Note

**Rapid determination of alcohol
content using the Arrow™ consumable
ATR slide**



**Specac Quest and
Arrow ATR**

INSIDE: Find out how Arrow™ can help you quantify food and drink content in seconds with zero cleaning between samples.

Introduction

Accurate quantification of the composition of food and drink is essential for compliance with local regulations on labelling of goods for general sale and for correct application of customs duties when imported. Food and drink labelling should provide correct information about calorific and nutritional content to help people stay within recommended limits.

Chromatographic techniques provide accurate determination but take time and skill to operate effectively. FTIR offers an attractive low-cost alternative that provide results in seconds and be carried out by personnel with limited training. Here we set out how the Arrow™ can be used to dramatically increase sample throughput with no cleaning required between samples.

Silicon ATR consumable technology helps you zero in on your analysis. Designed for the Quest™ ATR accessory, Arrow™ allows rapid assessment of a range of liquid analytes.

- Long duration ATR experiments.
- Batch sample preparation.
- Archive samples.
- Prevent sample cross contamination.
- Remote sample preparation.

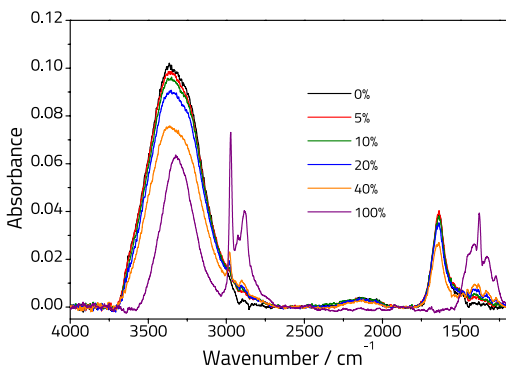


Figure 1: Spectra showing various concentrations of Ethanol/Water solutions recorded on an Arrow™ ATR Slide.

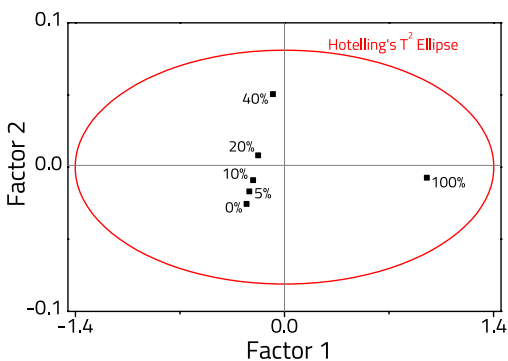


Figure 2: PLS Scores plot showing the sample distribution in 2 -factor space.

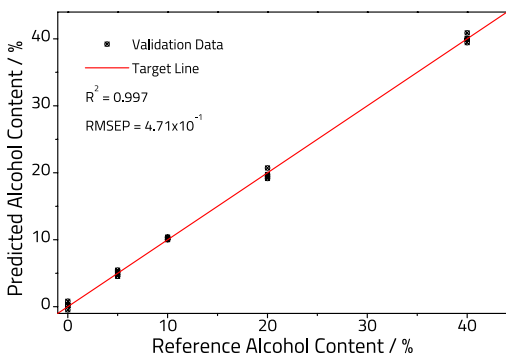


Figure 3: Calibration plot comparing known and PLS predicted alcohol content.

Quantification Techniques

Basic FTIR quantification methods utilize the Beer-Lambert law to produce a simple straight-line absorbance vs concentration calibration plot. This works well for simple solutions at low concentration. At higher concentrations deviations from a straight line are usually observed due to solute interactions. Where a simple calibration plot is inappropriate, chemometric techniques can be used to tease out hidden information within the dataset. One such technique is Partial Least Squares (PLS) analysis. This correlates two matrices – in this case FTIR spectra and composition data – aiming to extract the variance in the former to explain the variance in the latter. This technique can be used to quantify samples over a broad range of concentrations.

Experimental

Solutions of water/ethanol at the desired concentration were prepared using volumetric dilution. Samples were transferred to an Arrow™ slide *ex-situ* to the spectrometer. One Arrow™ slide was retained for use as a background, then samples were recorded on an alternate slide. To prevent evaporation the Arrow™ sample cap was fitted immediately after loading the sample. Spectra were recorded on a commercially available spectrometer. By batch preparing the samples prior to the start of the experiment 26 samples were measured in under 10 minutes. This speed helped minimize atmospheric changes between the background and sample which could result in the appearance of water vapor and CO₂ bands in the spectra. A simple PLS model was constructed using 6 samples, and then a further 20 samples were used to test it.

Results and Discussion

Figure 1 shows selected spectra for different concentrations of Ethanol solution. The pure water spectrum (black line) shows characteristic peaks at *ca.* 3350 cm⁻¹ (assigned to two overlapped peaks: symmetric, ν_1 , and antisymmetric, ν_3 stretching vibrations) and *ca.* 1640 cm⁻¹ (assigned to the ν_2 bending vibration). The pure spectrum of ethanol (purple line) displays peaks due to an OH stretching vibration at *ca.* 3330 cm⁻¹, CH stretching vibrations typical of an alkyl group between 3000-2800 cm⁻¹ and further peaks in the fingerprint region below 1500 cm⁻¹. The spectra of the mixture samples

are a combination of the two pure samples, however due to interactions between the two some small changes are observed. For instance, the ν_2 peak position of water shifts with increasing alcohol content due to disruption of hydrogen bonding; This peak is shifted by ca. 6 cm^{-1} in the 40% ethanol content spectrum, relative to the pure water spectrum. Small changes such as these can be analyzed via chemometrics to provide insight into chemical species even when that species is IR silent. [1]

A 1-factor PLS model was found to explain 93.6% of the variance within the training dataset. However, a closer inspection of [Figure 2](#) shows that whilst it does a good job at separating the pure ethanol spectrum from the others it does a very poor job at separating out water and the water/ethanol mixture samples from one another. Adding in a second factor explains 99.9% of the variance within the dataset, and this second factor separates out the remaining spectra from one another. Therefore, in order to fully explain the variation within our data whilst preventing overfitting of our dataset we chose to use a 2-factor model.

[Figure 3](#) shows the validation calibration plot of the model comparing the known quantification values of samples to the PLS predicted values from their spectra. The high R^2 value and low root mean squared error of prediction (RMSEP) demonstrates a high predictive power for the model.

Conclusion

Arrow™ has been used to successfully quantify alcohol concentrations in a simple model system using PLS. Spectra were obtained very rapidly, with clear implications for the productivity of your lab.

References

[1] Specac Application Note AN19-05: NIR Process Monitoring of Continuous Industrial Acetic Acid Production Using the ProCell™ Vortex.



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