Application Notes AN M156

Quantification of low-concentrated liquid contents via FTIR

Introduction

When applied correctly, FTIR spectroscopy allows to quantify contents in liquids reliably and with great spectral quality. This is needed, when impurities in solvents, contents in extracts or liquid mixtures are investigated. While ATR measurement may be disadvantageous when it comes to liquid quantifications, it is very well possible to reliably detect and evaluate even very low concentrations below 0.1% in transmission.

One obstacle however is sample preparation as well as the choosing of the optimal path length needed for the experiment. Preparing numerous samples under specific conditions and multiple path lengths often takes a lot of time, not to mention the cleaning process.

The Accessory

In this application note we like to introduce an innovative, Bruker exclusive FTIR accessory that brings ease to an otherwise tedious task:

The Select-a-Path liquid transmission cell.

Made of stainless steel and ZnSe, the unique design of this cell enables simplified measurement of IR transmission spectra of liquids at three different path lengths (50, 100, and 200 µm). Sampling itself is fast and easy.

Keywords	Instrumentation and Software
Quantification	ALPHA II FTIR Spectrometer
Quality Control	INVENIO FTIR Spectrometer
Oil Analysis	Select-a-Path Transmission Cell
Liquid Analysis	OPUS/Quant

Add a few drops of liquid into the cell, close it and simply rotate the cell in its holder to change the pathlength and select the appropriate sample well (Figure 1). Afterwards, the cell can be readily disassembled for cleaning and refilling.

Figure 1: Select-a-Path cell in holder (left) and during sampling (right).

Unlike other liquid cells, the Select-a-Path facilitates analyses of multicomponent solutions of varied component concentrations. Optimized pathlengths are simply selected for each component and a wide dynamic range of concentration levels.

The cell is retained in a holder that conveniently fits onto the spectrometer's standard sample slide plate holder. This holder includes a built-in aperture to prevent overfilling the sample wells with infrared radiation.

Experimental Benchmark

Quantitative analysis in optical spectroscopy is achieved by measuring the absorbance of light by a chemical compound. Absorbance is proportional to the sample concentration and to the pathlength of the transmission cell. Longer pathlengths are needed for low concentrations, while shorter pathlengths are suitable for the accurate measurement at high concentrations.

Quick and reproducible switching of three different pathlengths in the Select-a-Path cell allows fast optimization for quantitative analysis. As mentioned before, quantifications are needed throughout research and industry applications.

Therefore, we benchmarked the new cell in two real-life application examples:

Example #1: Residual water in ethanol

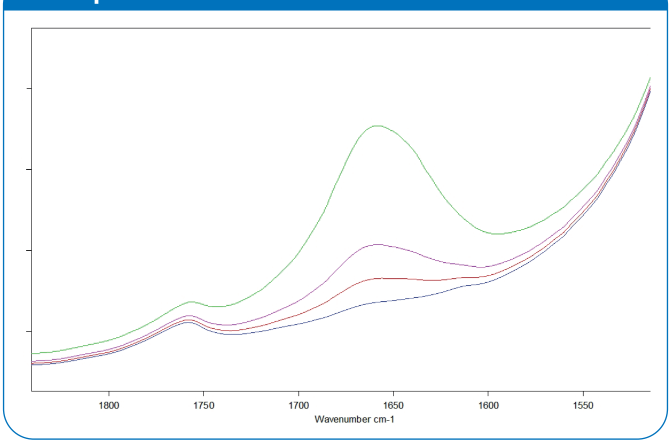

Figure 2: Spectra of 0.5% (black), 1% (red), 2% (pink) and 5% (green) water in ethanol measured in Select-a-Path cell at 200 µm pathlength.

Ethanol that is used in the production of gasoline must be pure and may only feature minimum amounts of water. If the ethanol contains to much water, the gasoline may damage engines or reduce the efficiency of the combustion process. The tightly sealed Select-a-Path cell is first filled with ethanol and then closed to prevent ambient water to contaminate the sample. Figure 2 shows the analysis of different amount of waters. Calibrations are easily created and the amount of water can be unambigously identified.

Example #2: Heavy water determination

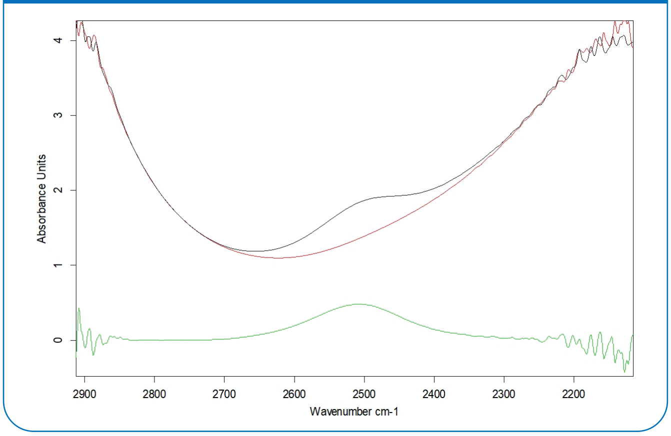

Figure 3: Spectra of 0.027% D_2O in H_2O (black), pure H_2O (red) and difference spectra (green) at 200 µm pathlength

Heavy water is used in nuclear reactors, since the amount of D_2O in H_2O is important for the analysis of coolants in nuclear power plants. D_2O is also important in medical and biological research. For example, the measurement of deuterium enrichment in saliva or blood allows estimating total body water to determine body composition. The concentration of heavy water in these applications varies from 0.01% to 5%. FTIR spectroscopy using transmission cells is traditionally employed for both applications.

Care must be taken to avoid evaporation during sample analysis because the vapor contains less deuterium than the liquid water. Subsequently, evaporative losses would result in an increase in deuterium content. This issue is eliminated with the tightly sealed Select-a-Path cell. Figure 3 shows an example analysis of a small amount of D_2O in water.

Conclusion

The Select-a-Path cell allows fast and reproducible switching of three defined pathlengths. It is a very convenient accessory for the quantitative analysis of solutions with a wide range of component concentrations, including volatile and viscous liquids.

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