

Trace Water-in-Methanol Monitoring

Application Note AN 71007-A

Introduction

Organic solvents are used as reaction media or for extracting products in the pharmaceutical, fine chemical and fragrance industries in large quantities. Solvents can represent a significant part of the costs associated with any particular product and often need to be recycled. They are generally recovered by either filtration or centrifuging, and are often contaminated by water. Before it can be re-used, a purification step is performed to ensure that the water content is below the acceptable limit. Currently, the most widely accepted and used measurement technique is Karl Fischer titration.

A Continuous On-Line Monitoring Solution

With an OXS-2205 Optical Trace Water Analyzer, on-line and real-time monitoring of water content in solvent can be achieved. The sampling system is that of a flow-through type, suitable for continuous monitoring using a slip stream sampling configuration. No reagents are required. The analyzer can be configured for low ppm level trace analysis as well as percent level analysis.

Using a unique and proprietary Tunable Filter Spectroscopy (TFS) engine, high-throughput near infrared (NIR) absorption spectroscopy is performed, resulting in high sensitivity measurement. Wavelength scanning coupled with advanced spectra analysis enables drifts compensation, resulting in high long-term stability measurement and robust interference resolution.

Measurement

Methanol is one of the most commonly used solvents. Measurement of trace levels of water in methanol was conducted using a closed loop sampling system with a continuous flow driven by an externally mounted miniature pump (LTC series, Hargraves, NC). The measurement started with a high-purity methanol ("Super Methanol", American Bioanalytical, MA) with a maximum water content of 500 ppm. This original solution represented the "zero" reference.

A series of water was injected into the sampling system to generate a "staircase" profile with the following theoretical concentration values:

- Step 1: 277 ppm
- Step 2: 554 ppm
- Step 3: 831 ppm
- Step 4: 1107 ppm
- Step 5: 1382 ppm
- Step 6: 1657 ppm
- Step 7: 1932 ppm

[All concentration values are volumetric (v/v)]

Results and Discussions

With the measurement updated every ten seconds, continuous staircase profile was obtained and is shown in Figure 1. Figure 2 shows the measured concentration versus the theoretical values.

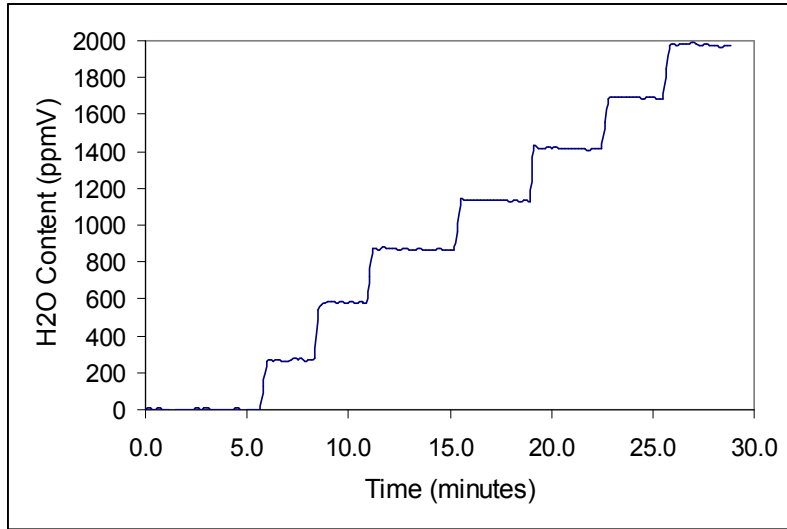


Figure 1. Measured concentration vs. time

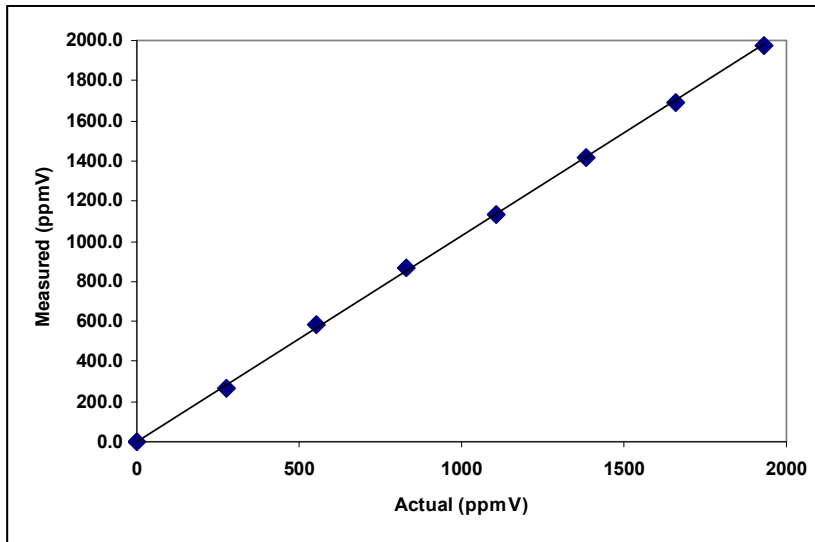


Figure 2. Measured concentration vs. theoretical values

The table below shows the calculated root-mean-squared (rms) value of the noise, which can be used to calculate the lower detection limit (LDL).

Averaging time	rms noise	LDL [5X rms]
10 seconds	3.4 ppm	17 ppm
1 minute	1.5 ppm	7.5 ppm

Table 1. Zero rms noise values and lower detection limits