

APPLICATION NOTE

No. 1.03 CHEMICAL

WATER DETECTION

- 0 – 100 ±0.05 % water detection
- Trace (±10 ppm) water detection
- Real time continuous measurement
- Many industrial applications
- Replaces Karl Fischer titration

Water can be accurately monitored in a wide range of samples from trace quantities to 100% using a Kemtrak photometric process analyzer. A Kemtrak water analyzer is a recommended replacement for inconvenient and expensive Karl Fischer titration analyzers. Benefits include no maintenance, no reagents and continuous results in real time.

The objective of this application note is to help understand the correct choice and configuration of a Kemtrak industrial analyzer for the measurement of water.

APPLICATION

Water analysis is influenced by the following factors:

1. Concentration range and required precision
2. Background sample absorption characteristics
3. Temperature variation
4. Solubility

Range and Resolution

Dissolved water is measured in the NIR (near-infrared) using a Kemtrak DCP007-NIR photometer. Typical samples include alcohols, glycols, caustics, organic solvents, acids and trace water in hydrocarbons and fuels. In the NIR there are two spectral regions where water has a significant absorption which is related to the hydroxyl or "OH" content of the water molecule.

The 1400nm region represents the first overtone of the O-H stretch in both water and ethanol as shown in *Figure 1*. The 1400nm NIR region is recommended for a water content greater than *ca.* 1% concentration with a full scale resolution of ± 0.05 % *e.g.* 0 – 100 ±0.05 % (±500 ppm) and 0 – 10 ±0.01 % (±100 ppm) water.

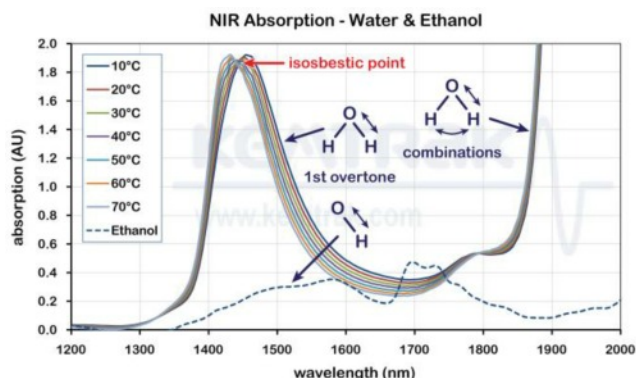


Figure 1: NIR absorption spectra of water & ethanol

The 1900nm region represents a combination of O-H stretch and H-O-H bending which is specific for the water molecule and has approximately five times greater absorption than the 1400nm region. The 1900nm region is generally used for water less than *ca.* 5% concentration and for trace water detection with a resolution of *ca.* ±10 ppm in alcohol.

Background Absorption

Absorption from the non-water component(s) of a sample is referred to as the background absorption.

For water and ethanol as shown in *Figure 1*, the measurement of water is the difference in absorption between the water and alcohol background at a specific measurement wavelength and optical path-length (OPL) and this determines the range and resolution. In the example shown the difference at 1440nm is *ca.* 1.8 AU and at 1930nm is *ca.* 9 AU using an OPL of 1.5mm.

The background absorption determines the maximum OPL that is possible and is a significant factor when making trace water analysis where long OPLs are necessary. For ideal instrument performance, the maximum absorption signal from the water should be below 2 AU, while background absorption should not exceed 1 AU. Instrument resolution is 0.001 AU.

There is considerable variation of absorption in the 1900nm region with some organic liquids having a very low absorption allowing a long OPL *e.g.* CCl₄ has negligible absorption at 1900nm so a 20 cm OPL can be used providing a resolution < 1ppm.

A reference wavelength is also used to accurately measure and compensate for variations in the clarity of the sample due to non-dissolved substances, particulates and window fouling. The reference wavelength should be placed where absorption is minimal *e.g.* < 1300nm in *Figure 1*.

Temperature variation

The absorption of many liquids in the NIR will change as a function of temperature and this will influence the reported concentration. In spectroscopy, molecules have a phenomenon where the absorption at a specific wavelength will be the same at different temperatures (*see Figure 1*) – this point is referred to as the isosbestic point. Where possible, a Kemtrak photometer will be factory configured to measure at the isosbestic point to minimize the effect of temperature.

For highest performance and measurement resolution, it is recommended to measure samples with constant temperature. If this is not possible and when the sample temperature varies more than *ca.* 10°C it is possible to measure the sample temperature and bring the signal back to the Kemtrak 007 where it is used to compensate the absorption.

Solubility

Water has a low solubility in many organic liquids, *for example* hexane (100ppm) and kerosene (5ppm). If water is present at concentration greater than the solubility point then it will become suspended as droplets forming a turbid emulsion which can interfere with an NIR absorption measurement.

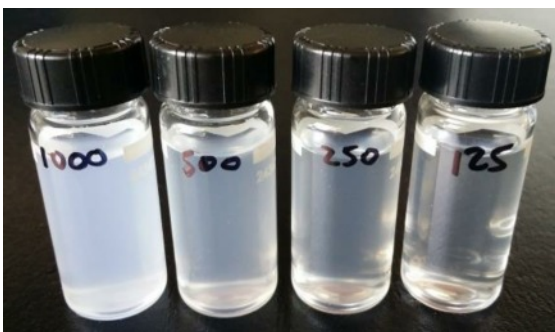


Figure 2: Water dispersed in Kerosene at 1000, 500, 250 & 125 ppm concentration.

Turbid emulsions are accurately measured using the reference wavelength of a Kemtrak DCP007 NIR photometer, allowing both dissolved and suspended water to be monitored. Alternatively, a Kemtrak TC007 turbidimeter is suitable for monitoring water at concentrations above the solubility point. The instruments are calibrated in units of ppm water, providing an accurate measurement of the water content of the sample.



Figure 3: Kemtrak DCP007 NIR photometer with DIN DN50 measurement cell.

INSTALLATION

Kemtrak manufactures two NIR photometers that differ in measurement range and detector technology.

- Kemtrak DCP007-N 800 – 1500 nm
- Kemtrak DCP007-L 800 – 2100 nm

Measurement at longer wavelengths (>1500nm) require advanced NIR LED light source and InGaAs detector technology. Both light source and detector incorporates multiple stage solid-state Peltier temperature regulation to eliminate LED light source and sensor drift to ensure low noise and high measurement resolution.

The measurement wavelength and the optical path-length of the measurement cell is factory configured. A Kemtrak representative will assist with application development to ensure the instrument is optimally configured to meet application requirements and deliver outstanding performance.