Lab Manager®
April 12, 2017
Run Your Lab Like a Business
LabManager.com

Beyond Karl Fischer: Titration Methods

Analysts are always looking for a handheld, push-button replacement for complicated laboratory processes **by Angelo DePalma**

Almost every industry is concerned about water concentration levels in some way. Food and pharmaceuticals automatically come to mind, but fuel, explosives, agri-chem, and polymer processing businesses (to name a few) all rely on moisture content as a critical quality attribute.

Karl Fischer titration (KFT) is the best-known technique for determining water levels in samples, and it is supported by numerous compendial methods. KFT has two principle forms: volumetric titration for relatively high water content (e.g., honey) and coulometric for low-water samples (plastics, oils).

KFT relies on a redox reaction involving iodine, sulfur dioxide, an alcohol, and water. The volumetric method involves titration of iodine required to react with water in the sample. Coulometric determinations measure the electron loss from iodide to iodine. Familiar vendor names and products dominate this business. Metrohm's KF Titrando and KF Ti-Touch, Mettler-Toledo's C20 and V20 instruments, Hach's TitraLab® AT1000, and YSI's TitroLine KF analyzers are illustrative. KFT reagents and consumables are often purchased separately.

Look, ma, no lab!

Analysts are always looking for a handheld, push-button replacement for complicated laboratory processes. Spectro Scientific (Chelmsford, MA), which specializes in water analysis instruments and software, recently introduced Version 5 of its FluidScan® portable infrared oil analyzer. FluidScan provides a quick answer to whether an industrial or lubricating oil is fit for use.

The instrument measures the infrared signature associated with the presence of water in the oil and translates this into ppm concentrations of water.

Anyone who has run an infrared spectrum can attest to the method's sensitivity to water contamination. FluidScan turns this sometime liability into an asset. The system includes an oil library, recently doubled in size to more than 700 entries.

"You need an oil library because the base infrared

signature of the oil, and how it interacts with moisture, can vary," says Patrick Henning, PhD, chief technology officer. "The library must contain this information to provide an accurate ppm water reading."

The new version of FluidScan illustrates a significant advantage of instrument-based methods over KFT. Where both methods involve sample preparation, for the latter the underlying technology, titration, has not changed that much since KFT was introduced in 1935. Infrared detection hasn't advanced that much either for bench instruments, but advances in miniaturization and signal to noise have greatly improved the accessibility and utility of handheld devices. "We've improved infrared analysis by stabilizing the infrared background reading,"

Henning says, a consequence of broadening the background oil library and upgrading background signal subtraction.

For those who worry about standards and compliance, Spectro claims faithful correlation between FluidScan results and those obtained through ASTM D664 and D4739 titration methods and ASTM D6304 KFT methods.

Getting the wet out of wetness

Eliminating wet chemistry from moisture determinates makes sense since reagent dispensing itself introduces error. Environmental friendliness never hurts either. Arizona Instrument (Chandler, AZ) touts its Vapor Pro XL water measurement system as a chemical-free, or "green," alternative to KFT.

Based on relative humidity (RH) measurements, Vapor Pro uses a cylindrical sample oven, a dry carrier gas, and a moisture sensor. Samples heat up, driving off volatiles, which travel from the sample vial to a polymer capacitor RH sensor. Arizona Instrument also offers the Computrac® Max® 4000, which is based on the more common loss-on-drying measurement, and a Computrac model employing loss-on-ignition technology.

"Karl Fischer methods entail the use of toxic chemicals that require disposal after use," explains Arizona Instrument sales director Rick Ervin. "Our only consumable is the septum users place on the sample vial."

According to the company, applications include "almost any in which Karl Fischer titration is employed," the exceptions being samples containing ammonium hydroxide, ethanol, methanol, and acetone.

As with the FluidScan, Vapor Pro provides equivalence to commonly employed ASTM moisture-detecting methods. Unlike its competitor's system, no "oil database" is required. "Interferences are very minimal," Ervin says. "It does not care about responses in hydrocarbons, and unlike Karl Fischer titration, there is no built-in bias."