

Karl Fischer titration and near-infrared spectroscopy in perfect synergy



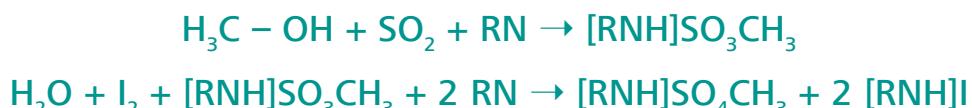
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Metrohm has shaped moisture analysis for more than half a century. Find out about new developments in water analysis and learn how near-infrared spectroscopy in combination with Karl Fischer titration can increase your sample throughput and boost your productivity.

Metrohm White Paper

Karl Fischer titration

In addition to the determination of the pH value, weighing, and acid-base titration, the determination of the water content is one of the most common analyses in laboratories worldwide. Karl Fischer (KF) titration for water determination was introduced in the 1930s. It is a fast and highly selective method. Water and only water is determined. KF titration is



based on the following two redox reactions. In the first reaction, methanol and sulfur dioxide react to form the respective ester. Upon addition of iodine, the ester is oxidized to the sulfate species in a water-consuming reaction. The reaction finishes when there is no water left.

Sample preparation & measurement

Depending on the water concentration and the solubility of the sample substance in KF reagents, the sample can be added to the titration vessel directly or has to be dissolved in a suitable solvent before addition. Suitable solvents are solvents that do not react with the KF reagents. This excludes the use of ketones and aldehydes. A blank determination has to be performed if the sample is dissolved or the water is extracted. For the measurement, the sample is injected directly into the titration vessel using a syringe and needle. The equivalence point is detected by a polarized double Pt pin electrode.

Results

The determined water content can directly be used for the method development and subsequent routine analysis with NIRS technology.

Choice of the right method

KF titration is suitable for water content determinations in all sample forms: solids, slurries, liquids, and even gases. For low water contents between 0.001% and 1%, coulometric KF titration is recommended, whereas samples with a water content from 0.1% up to 100% are determined volumetrically.



Use KF for NIRS validation

The concordance of these two methods has to be tested now and then. For this purpose, a sample with a known amount of water is tested with KF titration as well as with NIRS and the values are compared.

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Near-infrared spectroscopy

Near-infrared spectroscopy (NIRS) shines light on molecular overtone and combination vibrations. This information can be used for identification, qualification, and quantification of substances. For quantitative determinations, a calibration model is needed to link NIR spectra to results from primary methods such as KF titration. Such models are developed with the help of a software. After being verified, they can be used to analyze samples and predict the water content indepen-

dently of the primary method. NIRS yields results that are similar in accuracy to the primary method results, yet without any sample preparation and in a shorter time. In many situations, NIRS can also be used to determine other parameters in addition to moisture in the same measurement. This whitepaper outlines the steps for developing a NIRS model for moisture quantification from KF data and using it in synergy with KF for routine quality control to achieve higher sample throughput.

Spectral data acquisition

Samples in the form of powders, liquids, and creams can be analyzed with NIRS without any sample preparation. Solids are most conveniently measured in high-quality disposable glass vials, while liquids should be measured in high-quality disposable glass cuvettes. For creams, the «NIRS Slurry Cup» is recommended as it can be cleaned with only one swipe. Place the sample vessel in the instrument and start the data acquisition.



Convenient NIRS routine analysis

The use of NIRS for routine analysis is straightforward. Only two clicks are required in the software to select and start the method. The instrument automatically runs an internal wavelength standardization measurement, followed by the actual sample measurement, all in less than 60 seconds. The Vision Air 2.0 software automatically displays the result and, if required, indicates if the water content is within the user defined limits.

Method development

Use the water content values determined using KF titration and the acquired spectra to develop a model. The software contains well-established standard analysis algorithms such as Partial Least Squares Regression (PLS) and helps you create your multivariate calibration model. After validating the model to determine the standard error and demonstrate its robustness, it is ready for routine analysis.

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Summary

NIRS provides convenience in analysis, while KF titration gives you the confidence in your results. We know: These two analysis methods form the most powerful duo when it comes to water determination.

Metrohm laboratory analyzers can be operated in QC, R&D, and plant laboratories alike. The KF and NIRS systems are highly versatile and allow analysis of nearly all sample types.

Benefit from more flexibility, superior analytical performance, seamless method transfer, shortened implementation time, as well as robust and reliable results.



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