

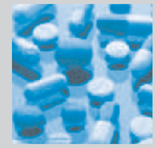

DENVER INSTRUMENT

Volumetric Karl Fischer Applications Introduction

Water analysis is without doubt one of the most frequently performed laboratory analyses in sectors such as the oil, pharmaceutical and food industries. For many products, water content needs to be etermined at every stage of the manufacturing process from raw materials to finished goods. The quality and the preservation of the final product depend on the amount of water present.

Excess levels of moisture will, for example, allow bacterial growth in food, decrease the performance of oils and lubricants, modify the density and the viscosity of paints and inks, disturb the texture of powdered products by forming conglomerates and impede the combustion properties of fuels.

The technique most commonly used for these analyses because of its rapidity, accuracy and ease of use is Karl Fischer titration. The sample is generally injected into the titration cell where it is dissolved by an appropriate solvent. The mixture is then titrated to complete ryness with a solution containing iodine. Volumetric Karl Fischer determination covers the range of 1 to 100 mg of water in the sample taken.

The applications described in this booklet have been chosen to cover a variety of sample types commonly examined for their water content. They should be used as a starting point for your own specific samples and, if needed, they can be optimised in order to suit your analysis requirements.

The Titrant

The determination of water content is based on the oxidation of sulphur dioxide by iodine in the presence of water:

$$I2 + SO_2 + 2H2O \;..\; 2HI + H_2SO_4.$$

This chemical reaction is used in Karl Fischer titration for quantitative water determination. The key components of the titrant, iodine and sulphur dioxide, are dissolved in methanol.

The Solvent

The solvent or working medium must ensure the stoichiometry of the Karl Fischer reaction. It must be able to dissolve the sample and the products of the reaction. It must also enable the detection of the end point. Most liquid samples dissolve in methanol, which is the usual solvent in Karl Fischer determinations. To determine the total amount of water, the sample must dissolve completely.

Other solvents can be added to methanol in specific proportions to liberate the water more efficiently. For example chloroform is a good solvent for fats and formamide improves the solubility of polar substances. Methanol should always be present in the solvent in a proportion not lower than 25%, otherwise unknown stoichiometric ratios will occur for the KF reaction.

The Indicator Electrode

A double platinum electrode is used, which is polarised with alternating square wave current. The detection of the end point of the titration is based on the detection of an excess of iodine. This occurs when water is no longer present in the KF cell. That may be indicated visually by a yellow colouring of the working media.

karl Fischer Titration Workstation

Radiometer Analytical's new volumetric Karl Fischer titrator gives your sample water content associated with its related uncertainty. The TitraLab 55 is a compact workstation combining burette, pump, titration stand and keyboard in one single box. To speed up analysis throughput, the TitraLab 55 keeps your cell ready for immediate use. As water is all around us, TitraLab 55 integrates automatic moisture drift compensation to avoid moisture drift errors that might cause a deviation from measurement trueness.

The Results

The results obtained at the end of a titration can be given directly associated to their corresponding expanded uncertainty. The uncertainties given for your Karl Fischer reagent titre, burette and instruments used to prepare sample (balance, pipette) are programmed based on standard manufacturer specifications. These calculations comply with the following standard: EN 13005 (GUM)(1).

Radiometer Analytical has developed an Uncertainty Plug-in enabling the TIM550 to give results with their expanded uncertainty.

(1) GUM: Guide for the Expression of Uncertainty Measurements