Recommended

AQUASTAR®

Water Standards for Karl Fischer Titration

Introduction

Karl Fischer (KF) titration is an instrumental analytical technique used in Quality Assurance/Quality Control for quantifying water content. Instrument validation and control is a key component of quality systems based on ISO, GLP, GMP, and FDA guidelines.

Use of Water Standards in Volumetry

Volumetric KF requires both titer determination/standardization and periodic performance checks to monitor instrument accuracy and precision using standards appropriate to each task. While 100% water may theoretically be used for both of these procedures, there are a number of practical advantages to using a certified Water Standard instead.

The main disadvantage to using 100% water is that the required sample size is sufficiently small (10-20mg) to require highly skilled handling in order to avoid errors related to sample weighing and transfer. Furthermore, laboratory water may not necessarily be free of potentially interfering contaminants. Finally, a number of regulatory bodies have recently begun to emphasize the need to have standards used in the laboratory be traceable to an appropriate national standard reference material.

The use of solid standards necessitates the opening of the titration cell to atmosphere, which risks contamination by atmospheric moisture. Additionally, finely ground solids tend to stick to greased sample ports and adhere to the walls of the titration cell due to static generated during their transfer. The use of Sodium Tartrate Dihydrate, in particular, is further complicated by its limited solubility in methanol, the solvent most frequently used in volumetric KF. This poor solubility necessitates limiting the sample size, which, again, can lead to errors related to sample weighing and transfer.

For these reasons we recommend the use of a specially formulated liquid standard containing 1% water for both titer determination/standardization and periodic instrument performance checks in volumetric KF. The **AQUASTAR® Water Standard 1.0% NIST** (Cat. No. 1.88052.0010) is a volumetric standard that is directly traceable to SRM 2890 from NIST (U.S. National Institute of Standards and Technology). Each package of 10 sealed single-use ampoules is shipped with the exact water content, accuracy, method traceability to the NIST batch, and minimum shelf life listed on the enclosed Certificate of Analysis.

Use of Water Standards in Coulometry

Though coulometric KF is considered an absolute method, periodic performance checks to monitor instrument accuracy and precision are still recommended, since such factors as ambient temperature and

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humidity conditions, quality of titration cell seals and septa, freshness of desiccant in drying tubes, chemistry of the samples, and others may introduce error into the analysis. While 100% water may, again, theoretically be used for this purpose, the sample size and handling issues relative to its use are even more challenging than in volumetry.

Because coulometric KF is a semi-micro method, the required sample size for100% water is extremely small (100-500µg), and weighing and sample transfer errors even more difficult to avoid. As previously stated above, some laboratory water may not necessarily be free of potentially interfering contaminants, and electrochemical techniques, like coulometric KF, are typically more sensitive to impurities that can change the conductivity of the system.

Therefore, we recommend the use of a specially formulated liquid standard containing ca. 1000ppm water for periodic performance checks in coulometric KF. The **AQUASTAR® Water Standard 0.1% NIST** (Cat. No. 1.88051.0010) is a coulometric standard that is directly traceable to SRM 2890 from NIST. Each package of 10 sealed single-use ampoules is shipped with the exact water content, accuracy, method traceability to the NIST batch, and minimum shelf life listed on the enclosed Certificate of Analysis. In those cases where analyses are carried out in the low ppm range, we recommend using the **AQUASTAR® Water Standard 0.01% NIST** (Cat. No. 1.88050.0010), a similar ampoulized standard with the certified water content of ca. 100ppm.

Special care must be exercised while using low-water liquid standards so as to prevent their contamination by atmospheric moisture. Please refer to the application note entitled "Handling Aquastar Water Standards", available on www.emdchemicals.com/analytics under Karl Fischer Analysis Tech Notes.

Use of Standards with the Karl Fischer Oven

Certain insoluble samples, such as plastics, as well as samples which react with the KF reagent/iodine, such as ascorbic acid, are typically analyzed by means of a Karl Fischer Oven in conjunction with either a volumetric or coulometric titrator. In such a configuration, the sample is heated by the oven, and only the vaporized water is transferred to the titration cell by means of a dry inert carrier gas. In order to monitor the performance of the KF Oven, a suitable standard must be used.

For this purpose we recommend the **AQUASTAR® Water Standard Oven 1%** (Cat. No. 1.88054.0005). This product is an entirely new solid standard for the KF Oven method. In contrast to other solid standards like lactose, citrate, or tartrate, it has a water content of only 1%, making it applicable to both coulometry and volumetry, and it can be used over a wide temperature range from 140-400°C.

Use of Standards with the Oil Evaporator

Certain highly formulated petrochemical products, such as lubrication oils, may contain as additives substances that interfere with the Karl Fischer titration, making direct analysis impossible. In such cases, an auxiliary piece of equipment, known as an Oil Evaporator, is used in conjunction with the titrator, such as in e.g., ASTM D6304 (Method B).

The **AQUASTAR® Water Standard Oil 15-30 ppm** (Cat. No. 1.88055.0010) is a unique reference material developed specifically for use in verifying the accuracy of coulometric KF equipment used in direct analysis of oils, as well as Oil Evaporators used in indirect analysis, as described above. This transformer oil-based 15-30 ppm ampoulized liquid standard is directly traceable to NIST SRM 2890.

For additional **AQUASTAR®** product Technical and Applications Support call: **1-800-222-0342**