

Application Note AN-K-068

Water content determination and acid-base titration in parallel

Reliable and reproducible water content determination in parallel to an aqueous potentiometric titration

The water content determination by volumetric Karl Fischer titration is one of the most important analyses worldwide. Using an OMNIS system consisting of an OMNIS Titrator and an OMNIS Sample Robot, the fully automatic analysis of water content is possible in various products and matrices. The OMNIS Sample Robot is capable of running several different titrations in parallel.

In this Application Note, we present the results of a volumetric Karl Fischer titration run in parallel to an aqueous acid-base titration on the same system. The water content is not influenced by the parallel running aqueous titration, allowing the combination of potentiometric titrations and Karl Fischer titrations on the same automated system.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on certified sodium tartrate dihydrate with a water content of 15.71% ± 0.07%. No sample preparation is required.

EXPERIMENTAL

The determinations are carried out on an automated system consisting of an OMNIS Sample Robot S with Dis-Cover functionality, OMNIS Dosing Module, and an OMNIS Professional Titrator (equipped with a double Pt-wire electrode for automated systems for KFT and a dEcotrode plus for acid-base titration).
The sample is weighed into the sample beaker, which is then covered with the DIS-cover lid and placed on the rack. The OMNIS Sample Robot automatically brings the beaker to the workstation and uncovers it just prior to the analysis. A solvent mixture consisting of methanol, imidazole, and sulfur dioxide is added automatically and the sample is titrated with a single-component titrant to the endpoint.
In parallel, an aqueous acid-base titration was performed on a second workstation on the same OMNIS Sample Robot.



Figure 1. Automated OMNIS System for the parallel volumetric Karl Fischer titration and aqueous acid-base titration consisting of an OMNIS Sample Robot, OMNIS Dosing Module, and OMNIS Titrator Professional equipped with a Pt-wire electrode for automated systems and a dEcotrode plus.

RESULTS

As the focus is on the reproducibility of the KF results while performing acid-base titration in parallel, we only present the KFT results here. Reproducible results for the water content are obtained. For the tested sodium tartrate dihydrate a water content of 15.67% (n = 6, SD(rel) = 0.3%) is found, which is well within the given certified water content.

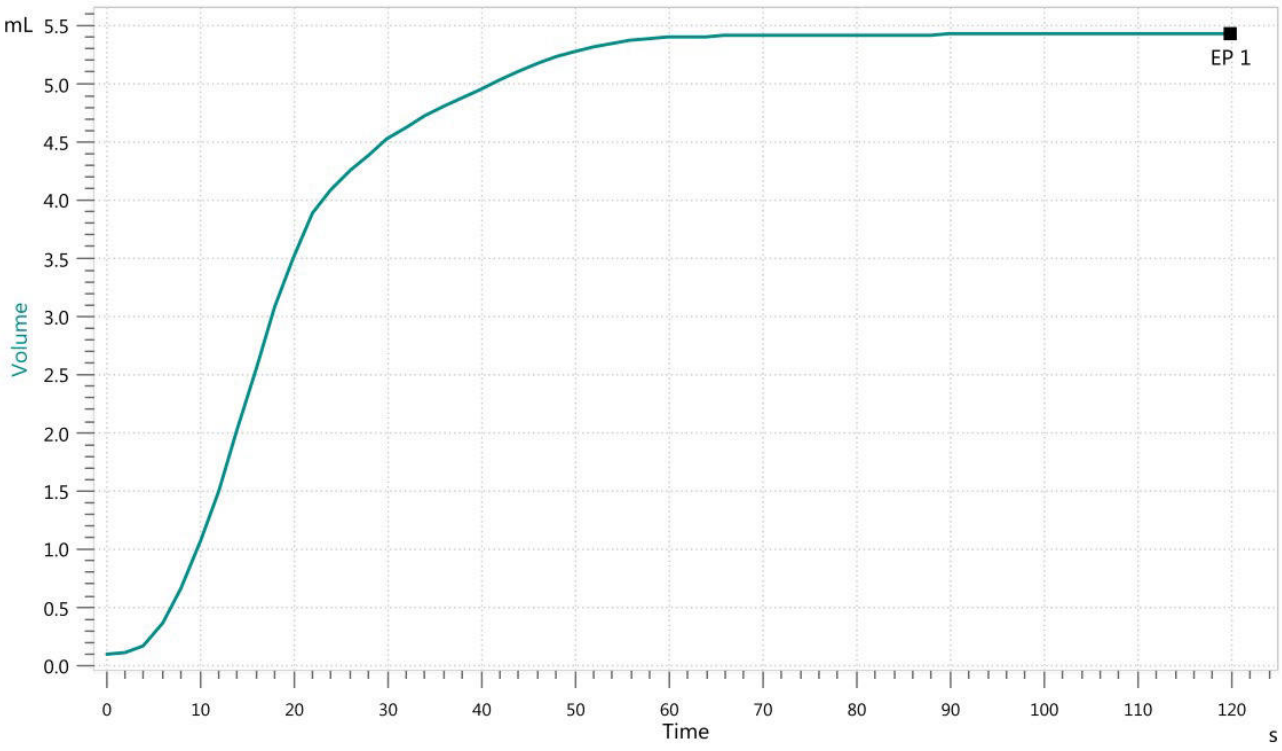


Figure 2. Titration curve of the determination of the water content in sodium tartrate dihydrate.

CONCLUSION

Karl Fischer Titration is a precise and reliable method for the determination of the water content of a sample. This study shows that a parallel determination of the water content besides potentiometric aqueous acid-base titration on an automated OMNIS system is possible. The potentiometric determination does not interfere with the Karl Fischer titration.

A reliable water content determination is possible while running aqueous titrations in parallel with Karl Fischer titration on the same OMNIS system.

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CONFIGURATION



Main module Pick and Place S

Main module for setting up an OMNIS Sample Robot Pick&Place in size S. This module is comprised of module and rack plates. It is already equipped with the main lift and one gripper. In addition to sample racks and gripper fingers, workstations such as Pick&Place module or a pump module are required for using it to set up a functioning Sample Robot. The selection of these components proceeds in accordance with the application.



Pick and Place module

Module for installation in the module plate of the Pick&Place of the OMNIS Sample Robot. This workstation takes up the sample beaker for the analysis. Used sensors are cleaned or parked in the storage beaker of the Pick&Place module between analyses. If stirring is required during the analysis, then a separate rod stirrer must be used for this workstation.



Peristaltic (2-channel) pump module

Module for installation in the module plate of the Pick&Place of the OMNIS Sample Robot. This workstation is equipped with both a rinsing pump and an aspiration pump. They are used to clean the sensors in a Pick&Place module and to empty the sample beaker after the analysis before it is returned to the rack once again.



OMNIS Rod Stirrer Sample Robot

Rod stirrer with permanently mounted cable (2.5 m) for direct connection to the OMNIS Sample Robot.



OMNIS Professional Titrator without stirrer

Innovative, modular potentiometric OMNIS Titrator for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is safer than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a stirrer added as needed. Including "Professional" function license for parallel titration with additional titration or dosing modules.

- Actuation via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Can be supplemented with magnetic stirrer and/or rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Safe handling of chemicals, automatic transfer of the original reagent data from the manufacturer

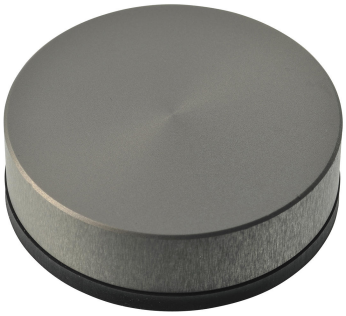
Measuring modes and software options:

- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with 5-way parallel titration: "Professional" function license



OMNIS Dosing Module without stirrer

Dosing module for connection to an OMNIS Titrator for extending the system to include an additional buret for titration/dosing. Can be supplemented with one magnetic stirrer or rod stirrer for use as separate titration stand. Freely selectable cylinder unit with 5, 10, 20 or 50 mL.



KF Dis-Cover lid for OMNIS 120 mL sample beaker, 16 pieces

Lid with seals for volumetric Karl Fischer Titration in 6.01400.200, 6.01400.300 and 6.01400.303 sample beakers in the OMNIS Sample Robot Pick&Place system.



OMNIS sample rack, 16 x 120 mL, (PP)

OMNIS sample rack for OMNIS Sample Robot Pick&Place, suitable for 16 sample beakers. The following sample beakers can be used: 6.01400.200, 6.01400.300, 6.01400.303.
Plastic: Polypropylene (PP)



Cylinder unit OMNIS 20 mL

Intelligent 20 mL cylinder unit for an OMNIS Titrator, Titration Module or Dosing Module. Includes dosing tubing and antidiffusion tip.

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS Stand-Alone license

Enables stand-alone operation of the OMNIS software on a WindowsTM computer.

Features:

- The license already includes one OMNIS instrument license.
- Must be activated via the Metrohm licensing portal.

OMNIS
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OMNIS Stand-Alone: 1 instrument license

1 instrument license for operating one additional OMNIS instrument in OMNIS Stand-Alone.

The following instruments are supported:

- OMNIS instruments
- Metrohm USB devices
- RS-232 instruments (e.g., balance)

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Function license KFT with conditioning

Function license for volumetric Karl Fischer titration with conditioning for an OMNIS titration system.



Double Pt-wire electrode for sample changer (fixed cable)

Indicator electrode (diameter = 5.3 mm, fixed cable 2 m) used for automated Karl Fischer titration.



dUnitrode with Pt1000

Digital, combined pH electrode for OMNIS with integrated Pt1000 temperature sensor. This electrode is particularly suitable for :

- pH measurements and titrations in difficult, viscous, or alkaline samples
- at elevated temperatures
- long-term measurements

The fixed ground-joint diaphragm is insensitive to contamination.

Reference electrolyte: c(KCl) = 3 mol/L, storage in storage solution.

Alternatively: Reference electrolyte for measurements at T>80°C: Idrolyte, storage in Idrolyte.

dTrodes can be used on OMNIS Titrators.