Metrohm

Application Bulletin 417/1 e

Automated volumetric Karl Fischer titration using MATi 11

Branch

General analytical chemistry; pharmaceutical industry; food, stimulants, beverages, flavors; fertilizers, base materials, explosives; detergents, surfactants, cosmetics

Keywords

Titration; Karl Fischer titration; volumetric; automation; MATi 11; water content; homogenization; sample preparation; Polytron PT 1300 D; branch 1; branch 4; branch 7; branch 11; branch 12

Summary

This Application Bulletin provides information on the MATi 11 system (MATi = **M**etrohm **A**utomated **Ti**tration). MATi 11 is a completely configured system used for the automated volumetric Karl Fischer titration for the water content determination in liquid or solid samples. The system includes a Polytron PT 1300 D for the homogenization of samples. Up to 53 samples are directly analyzed in 120 mL titration beakers. The sample is weighed in the titration beaker and covered with aluminum foil and a foil holder. In this way the samples do not lose or absorb water.

Instruments

The MATi 11 system consists of:

- Sample Processor
- Titrator with KFT mode
- Polytron PT 1300 D
- Pump for removal of waste
- Dosino (2)
- 10 mL Dosing unit for KF Titrant
- 50 mL Dosing unit for KF Solvent
- Required accessories for the KF titration

Electrodes

Double Pt Elektrode	6.9903.048

Reagents

One- or two-component reagents can be used:

- Titrant for volumetric KF titration
- Solvent for volumetric KF titration
- Depending on the application auxiliary solutions (solubilizers) might be needed. More information on solubilizers is available in the Metrohm KF Monograph.

Standard

Water standard 10 mg/g	Commercially available standard with an approximate water content of 10 mg/g should be used.
Sodium tartrate dihydrate	

Sample preparation

The sample preparation (homogenization) is carried out directly in the titration beaker using a Polytron (picture 1). Because the titration takes place in the same beaker, there is no risk of changing the water content during sample preparation and obtaining false results.

The homogenization time strongly depends on the type of sample. The time should be optimized for each sample. It is also recommended to vary the speed during the homogenization. More information on the use and handling of the Polytron can be found in AB 418.

Figure 1: Polytron PT 1300 D (left) and an aggregate (right)

Analysis

System preparation

To prepare the system, a blank value determination is run and the result of the determination discarded. After the titration to dryness, the titrant consumption during 300 s is recorded and the drift (mL/s) is stored as common variable. This value is used for the drift correction of the endpoints of the blank and the titer determinations as well as the sample measurements.

Blank determination

Cover the titration beaker with Al-foil and foil holder and place it on the rack. An appropriate, predefined amount of solvent is automatically added by a Dosino and the water content of the solvent is determined. The mean value of at least three blank determinations is saved as common variable.

Please note that the solvent volume for all subsequent determinations must be identical to the volume used for the blank determinations.

Titer determination

Weigh an appropriate amount of standard (see table 1) into a titration beaker, cover the titration beaker with Al-foil and foil holder and place it on the rack. The same amount of solvent as for the blank determination is automatically added. The mean value of at least three titer determinations is saved as Titer on the Dosing unit.

Table 1 shows the approximate amount of standard in grams which leads to a titrant consumption between 10 and 90% of the volume of a 10 mL buret. The additional consumption of titrant for the blank value of the solvent is not included and could lead to a consumption of more than 90% of the buret volume.

Table 1: Recommended amount of standard [g]

	Titrant 1	Titrant 2	Titrant 5
Water standard 10 mg/g	0.1 … 0.9	0.2 … 1.8	0.5 … 4.5
Sodium tartrate dihydrate	0.02 … 0.05	0.02 … 0.11	0.03 … 0.28

Titrant 1: 1 mL of titrant reacts with approximately 1 mg H_2O

Titrant 2: 1 mL of titrant reacts with approximately 2 mg H_2O

Titrant 5: 1 mL of titrant reacts with approximately 5 mg H_2O

Sample determination

Place an aliquot of the sample in a titration beaker. Cover the beaker with Al-foil and foil holder and place the beaker on the rack. The same amount of solvent as for the blank determination is automatically added, the sample homogenized and the water content determined.

Calculation

Drift for drift correction

$$\text{Drift} = \frac{V_{300s}}{300} \quad (1)$$

Drift Drift for drift correction in mL/s
V_{300s} Volume of titrant consumed after 300 s in mL
300 Determination time in s

Blank

$$\text{Blank} = V_{EP} - t_{Det} \times \text{Drift} \quad (2)$$

Blank: Titrant consumption for the pure solvent in mL
V_{EP}: Titrant consumption up to the end point in mL
t_{Det}: Determination time in s
Drift: Drift for drift correction in mL/s

Titer

$$\text{Titer} = \frac{m_{standard} \times w(\text{standard})}{V_{EP} - \text{Blank} - t_{Det} \times \text{Drift}} \quad (3)$$

Titer: Titer of the selected titrant in mg/mL
$m_{standard}$: Mass of standard in g
w(standard): Certified water content of standard in mg/g (for pure water = 1000)
V_{EP}: Titrant consumption up to the end point in mL
Blank: Titrant consumption for the pure solvent in mL
t_{Det}: Determination time in s
Drift: Drift for drift correction in mL/s

Sample

$$w(H_2O) = \frac{(V_{EP} - \text{Blank} - t_{Det} \times \text{Drift}) \times \text{Titer} \times 0.1}{m_{sample}} \quad (4)$$

$w(H_2O)$: Water content in %
V_{EP}: Titrant consumption up to the end point in mL
Blank: Titrant consumption for the pure solvent in mL
t_{Det}: Determination time in s
Drift: Drift for drift correction in mL/s
Titer: Titer of the selected titrant in mg/mL
0.1: Conversion factor for %
m_{sample}: Mass of sample in g

Example

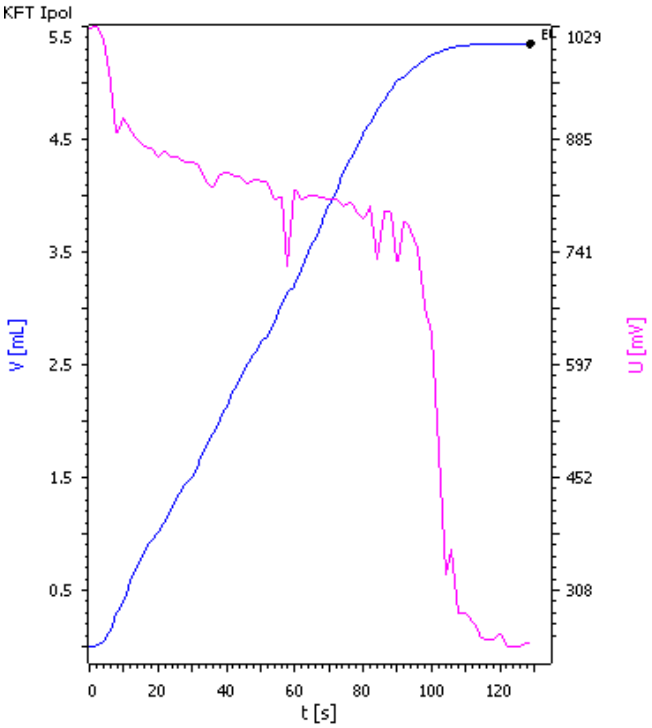

Figure 2: Titration curve of a titer determination with water standard 10 mg/g (blue = added volume of titrant in mL, pink = measured value in mV)

Comments

- The titration head includes an o-ring gasket which seals the titration beaker during determination. When defining the work position for the titration head, make sure the o-ring gasket is slightly pressed against the foil holder. A leak can lead to instable drift values and to irreproducible results.
- The substance to be titrated is weighed in the titration beaker before the solvent is added. Therefore, conditioning of the solvent is impossible and the water content of the used solvent or solvent mixture must be determined separately. The blank value is saved and the volume subtracted from the titrant consumption for the titer and sample determination (see equation (3) and (4)).
- The used solvent should be as dry as possible to obtain small blank values (preferably < 1 mL). To ensure small blank values the solvent can be dried, e.g., with molecular sieve.
- If the solvent is changed (addition of solubilizer, different batch …) the blank determination must be repeated and a new blank value determined.
- The amount of solvent added must be large enough to make sure that the electrode and the Polytron are immersed properly. We recommend using a volume of 50 mL. Never run the Polytron dry, means without immersing the connected aggregate in a liquid.
- Once the blank value is determined, all subsequent measurements must be carried out in the same volume of solvent.
- The titer determination can be done using different types of standards (see table 1). We recommend using a liquid water standard, as there are no problems regarding solubility. Additionally, the sample sizes can be large enough to avoid influences caused by a weighing error.
- To weigh standards and samples a balance with a resolution of ± 0.1 mg should be used.
- Close the titration beaker immediately after weighing to avoid changes of the water content.
- In case of electrostatic problems use an ionizer during the weighing procedure.
- The stirring speed should be high enough to ensure a proper mixing. Too high stirring speeds can have a negative effect on reproducibility, due to air bubbles disturbing the measurement.
- Except for the calculation, the method and the parameters used for the system preparation, blank, titer and sample determination have to be identical. Usually the standard parameters can be used. Depending on the sample it might be necessary to adapt the parameters.
- Make sure that the Dosing unit used for the solvent addition is air bubble free. Because the complete cylinder is dosed for a determination, air bubbles can have an influence on the blank value and subsequently on the results for the samples as well. To avoid air bubbles aspiration speed can be reduced.
  Another option is the dosing of two times 25 mL instead of 50 mL.
- A picture of the complete MATi 11 system can be found in the appendix.

Reference

- AB 418 – Use of the Polytron PT 1300 D

Author

Competence Center Titration

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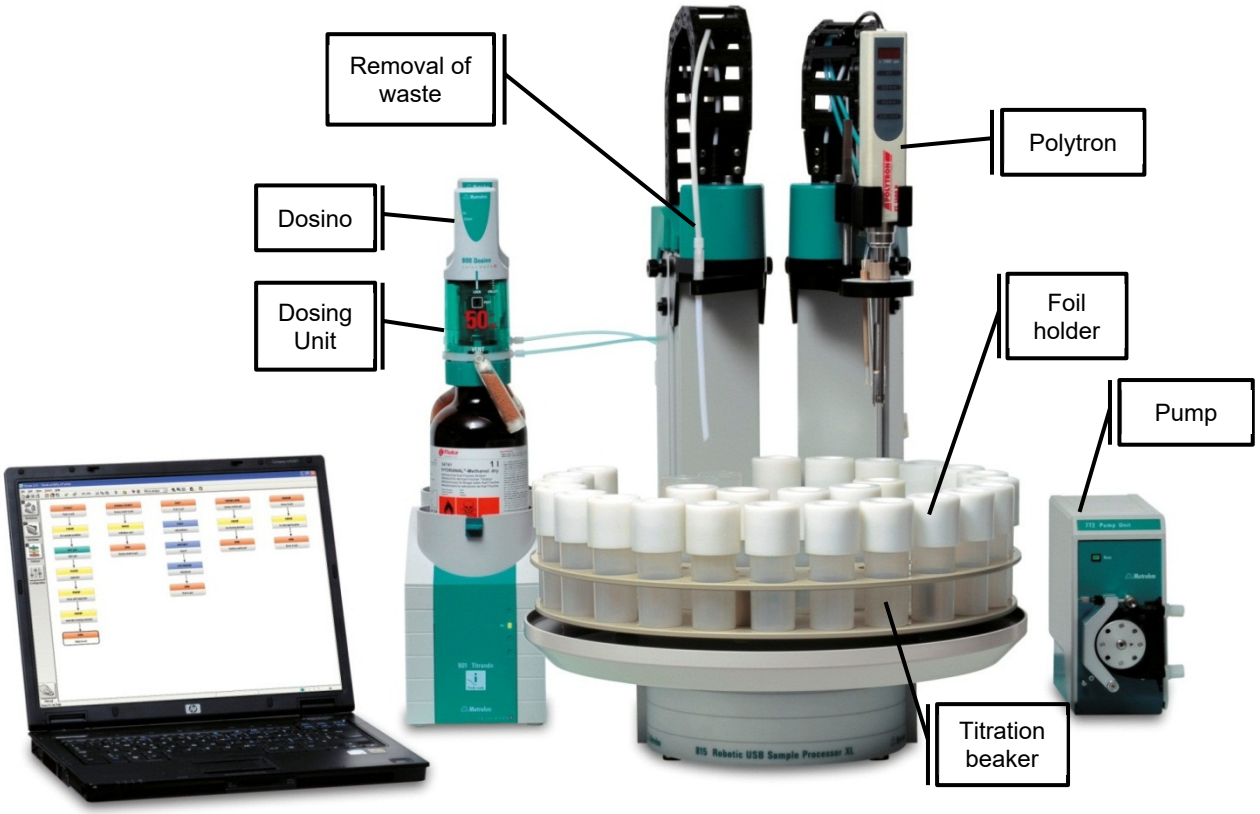
Appendix


Removal of waste
Polytron
Dosino
Dosing Unit
Foil holder
Pump
Titration beaker