Metrohm

## Application Bulletin 407/1 e

# Automated volumetric Karl Fischer titration with MATi 10

### 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 10; water content; branch 1; branch 4; branch 7; branch 11; branch 12



### Summary

This Application Bulletin provides information on the MATi 10 system (MATi = **M**etrohm **A**utomated **Ti**tration). MATi 10 is a completely configured system used for the automated volumetric Karl Fischer titration and the water content determination in liquid or solid samples. Up to 24 samples are directly analyzed in 75 mL titration beakers. The samples are weighed in the titration beakers and covered with Al-foil and foil holders. In this way the samples do not loose or absorb water.


### Instruments

The MATi 10 system consists of:

- Sample Changer
- Titrator with KFT mode
- 10 mL buret for KF Titrant
- 50 mL buret for KF Solvent
- Magnetic Stirrer
- Required accessories for the KF titration

### Electrodes

| | |
|---|---|
| Double Pt Elektrode | 6.0340.000 |

### 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 | Tap water Herisau |
| 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 depends on the type of sample and its water content. Information on sample preparation for different types of samples can be found in the Metrohm KF Monograph.

### Analysis

#### *System preparation*

To prepare the system, a blank value determination is run and the result of the determination discarded.

#### *Blank determination*

Insert a magnetic stirring bar, 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 Dosing unit and the water content of the solvent is determined. The mean value of at least three blank determinations is saved.

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, insert a magnetic stirring bar, 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.

| | 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 |
| Water | * | * | 0.02 … 0.04 |

* We do not recommend using pure water as standard for low titrant concentrations.

Table 1: The table shows the approximate amount of standard [g] which leads to a titrant consumption between 10 to 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.

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 the sample or an aliquot of it in a titration beaker. Add a magnetic stirring bar, 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 dissolved and the water content determined.

## Calculation

### *Blank*

$$\text{Blank} = V_{EP} \quad (1)$$

$V_{EP}$: Titrant consumption up to the end point in mL
Blank: Titrant consumption for the pure solvent in mL

### *Titer*

$$\text{Titer} = \frac{m_{standard} \times w(standard)}{V_{EP} - \text{Blank}} \quad (2)$$

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

### *Sample*

$$w(H_2O) = \frac{(V_{EP} - \text{Blank}) \times \text{Titer} \times 0.1}{m_{sample}} \quad (3)$$

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

## Example

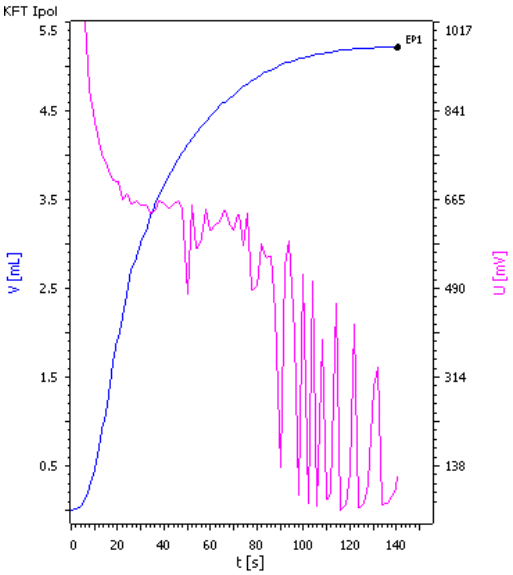



Fig. 1: Titration curve of a titer determination with water standard 10 mg/g (blue = added volume of titrant in mL, pink = measured value in mV)

## 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 (2) and (3)).

- The used solvent should be as dry as possible to obtain small blank values (< 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 is immersed properly. We recommend using a volume of 20 mL.
- 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 the 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.
- When weighing in solid samples, make sure that no sample is sticking to the wall of the titration beaker. This leads to wrong results because not the complete amount of sample is dissolved.
- The sample has to dissolve quickly in the solvent. If the dissolution is slow, an extraction time can be used to avoid the titration to finish too early. Another possibility is the addition of solubilizer. In case the sample does not dissolve at all, the water can be extracted using a suitable solvent and an aliquot of the solvent used as sample. The water content of the solvent used for the extraction needs to be determined separately and subtracted from the result of the sample determination (see KF Monograph for further information).
- 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.
- A picture of the complete MATi 10 system can be found in the appendix.

**Literature**

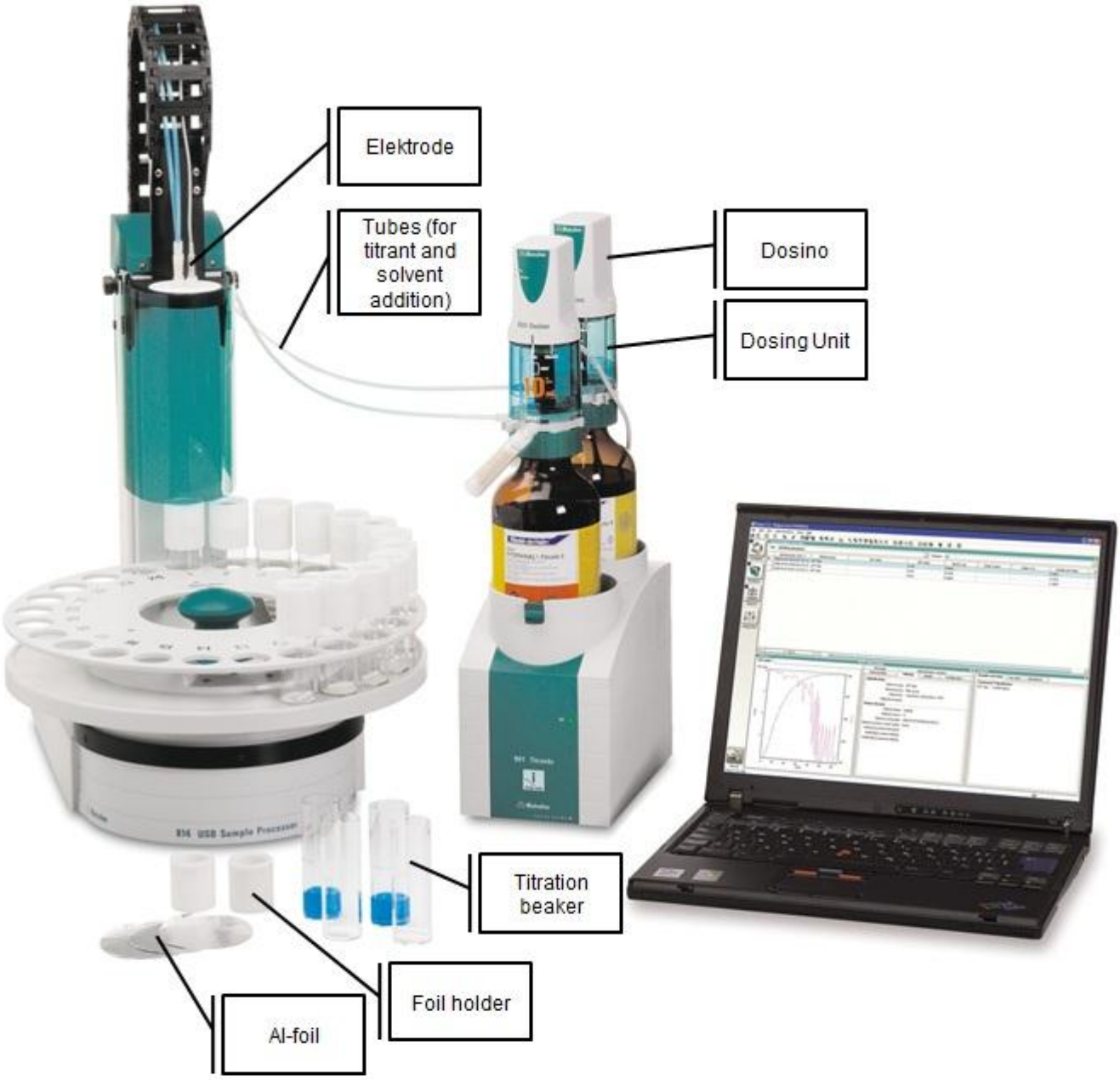
- Monograph: Water Determination by Karl Fischer Titration

**Author**

Competence Center Titration

Metrohm International Headquarters

# Appendix


Elektrode
Tubes (for titrant and solvent addition)
Dosino
Dosing Unit
Titration beaker
Foil holder
Al-foil