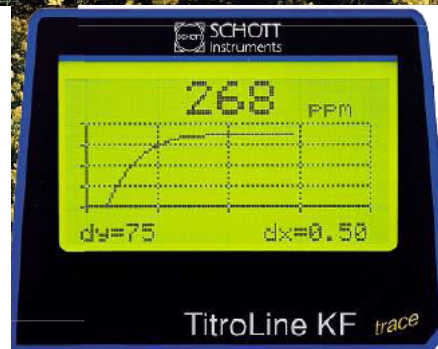


BASIC EQUIPMENT -Trace amounts of amounts

STEFAN KAUS

The Karl Fischer titration is considered one of the most important methods in determining the water content due to its high selectivity. In almost all branches of industry, e.g., chemical, petrochemical, pharmaceutical or food industry, the method is used routinely. Numerous ASTM, ISO and DIN standards, as well as specifications from European and US medical books (USP) indicate use of this method.



TitroLine KF trace until 2012

The water content can be displayed as a curve.

Karl Fischer titration (KF titration) is an established method for the analytical determination of water. There is a difference between volumetric and coulometric KF titration.

Both methods are differentiated primarily in the type of dosing with the titration reagent iodine, which under certain conditions react stoichiometrically with water. In volumetric titration, iodine dissolved in alcohol is supplied via a highly precise piston burette. In coulometry, the iodine is generated electrochemically via a generator electrode in an iodine containing solution. Figure 2 shows the schematic of this type of system.

Functional principle

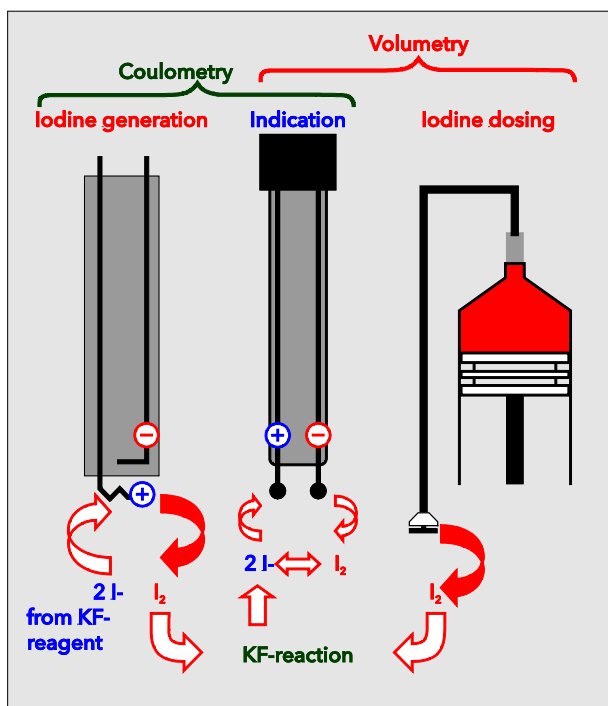
The electrochemical generation is based on the Faraday Law where 1 Mol corresponds to generated iodine 96 485 AS (charging amount, coulomb; thus the designation coulometry). The current measured in AS corresponds exactly to the water content of the sample provided.

The detection of the titration end is identical in both methods. With coulometric KF titration, there is an absolute method in which the titration reagent checking can be omitted. It is not necessary to determine the titer, but it would also not be possible based on the equipment setup.

Another advantage to coulometry is the low verification limit. Absolute values of 10 micrograms or concentrations of 1 ppm water are still verifiable. By using low specimen quantities, you can work up to one week with the same reagent before it has to be replaced.

Because materials cannot generally be added directly, their water is extracted externally or released via a heating oven and supplier automatically into the titration vessel. For plastic samples such as polyethylene or polypropylene as well as for oil samples with additives, the use of a heating oven is specified.

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2 Schematic representation of the volumetric and coulometric Karl-Fischer titration.

Volumetry is more flexible in use of solution media and can cover a broader range from 0.01 percent to 100 percent water. If water content is more than five percent, coulometry is costly by comparison. Very small sample quantities are needed so that the titration times are not too long. The maximum absolute values of 100 or 200 milligrams cited by many manufacturers is more of a theoretical nature. At a measuring speed of approx. 2 milligrams water per minute, the titrator would need up to 100 minutes.

The volumetric KF titration is a more commonly used universal method, however, coulometry has gained considerably in recent years. This is primarily due to its simple handling and operation.

Simple operation

Schott Instruments has manufactured piston burettes and titrators for almost 30 years. From the beginning, simple operation was a focal point of development, even when it sometimes involved very complex devices. With the new Titroline KF trace, there is now a Karl-Fischer titrator available that is designed for simple handling. The goal of Schott Instruments was to develop a titrator with which the user can complete coulometric KF titrations with practically no advance knowledge. After switching on the device, you only need to press the start button for each new sample. There is no time consuming programming method.

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```

*           Method 01           *
  Start                <Start>
  Selection:           <Mode>
  Edit Parameter      <F2>

Water                 0 ppm
Mw.                   0 ppm
Drift                 µg/min
    
```

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*           Method 01           *

Model/net weight
Result
Documentation
Titration parameters
Selection < >
Enter<F1>           ESC<F4>
    
```

```

*           Method 01           *
*           Documentation       *
short
Standard(with curve)
GLP
Display only
Selection < >
Enter<F1>           ESC<F4>
    
```


Conducting a test

The measurement is markedly simple: After the titrator is set up, the special KF reagents are filled into the titration vessel; if there is a generator electrode with diaphragm, the generator electrodes as well. The device begins to work immediately. The existing humidity of the KF reagent and the titration vessel are eliminated immediately via the automatic conditioning. If the drift drops below 10 micrograms after a few minutes, the first sample can be titrated. After the liquid sample is injected through the septum, the water content can be tracked dependent on time as a curve on the display (see Fig. 1). The result appears after approx. two minutes. In addition, it can be output to an attached printer. "Standard with curve" and "GLP" are available for printing out the "short" options. For the GLP output, all of the titration parameters including the calculation formula are output along with the titration curve, result, date, time, sample designation as well as sample weight or sample volume (see Fig. 3).

In most cases, a change of the predetermined titration parameters of the ten methods, e.g., the start or stop drift, is not necessary. If it should become necessary, entry takes just a few seconds. The clearly structured operating surface allows for quick and simple navigation. If needed, the method parameterization can be password protected (see Fig. 4).

To communicate with a PC, the Titroline KF trace uses a serial or USB interface. In addition to a printer or PC, an analysis scale can be connected for automatic takeover of weighing and a PC keyboard for entering alphanumeric sample designations. An optional titer stand with a pump is also available, which simplifies the supply and suction extraction of the KF reagents.

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