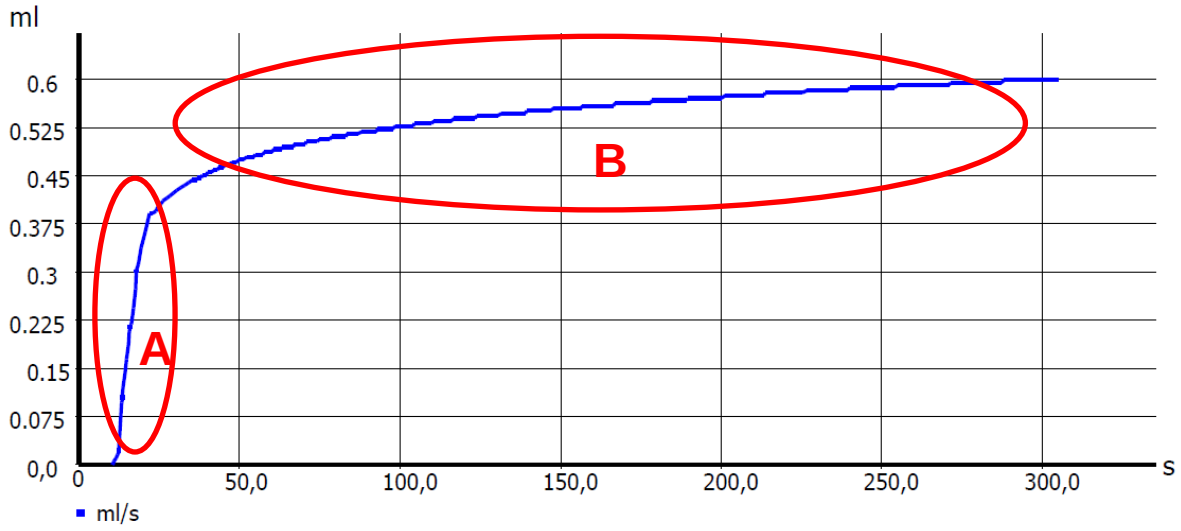
Titration of water in liquid samples

Description

This application can be used for many liquid samples as long as no side reactions occur and the sample dissolves in the KF reagent.
Liquid samples that do not or only partially dissolve in alcohols can often still be titrated in Methanol or similar solvents: The water is extracted from the sample dispersed in the solvent during the titration. The extraction time should be increased.
The solubility of the sample can be improved by adding toluene, chloroform, long-chain alcohols or similar solvents. Various special KF solvents for oils or fats are also available.
In this application, 1-component reagents are used to determine the water content. The use of 2-component reagents is also possible if the corresponding titration parameters are used.

If a sample causes side reactions, can be recognized from the titration curve: After the water has been titrated (the steep rise in the curve at the beginning), the titration curve rises steadily until the max. time has been reached, the µA end criterion is not met. Some side reactions can be prevented by titration with special reagents (ketones), for others it helps to titrate in the cold. Some side reactions cannot be prevented.



Ill.: KF-Titration with side reaction; A: Water + side reaction; B: side reaction

Devices

Titrator	TL 7500 KF or higher
Exchange unit	WA 10
Electrode	KF 1100
Lab accessoires	Magnetic stirrer TM 235 KF
	Karl-Fischer titration vessel TZ 1770
	Glas syringe (or disposable syringe) with needle

Reagents

1	1-component titrant, e.g. Composite 2 or 5 or a similar reagent
2	1-component solvent, e.g. CompoSolver E, Methanol or a similar solvent
3	Molekular sieve, dry
All reagents should be in analytical grade or better.	

Titration procedure

Reagents

Karl Fischer reagents are available as ready-to-use solutions.

The molecular sieve must be replaced or dried regularly, at least every 4 weeks.

Cleaning and handling the KF 1100 electrode

The KF 1100 electrode does not require any special treatment.
The two platinum pins must not touch each other. For cleaning, Isopropanol or other solvents that do not attack glass and platinum are suitable.

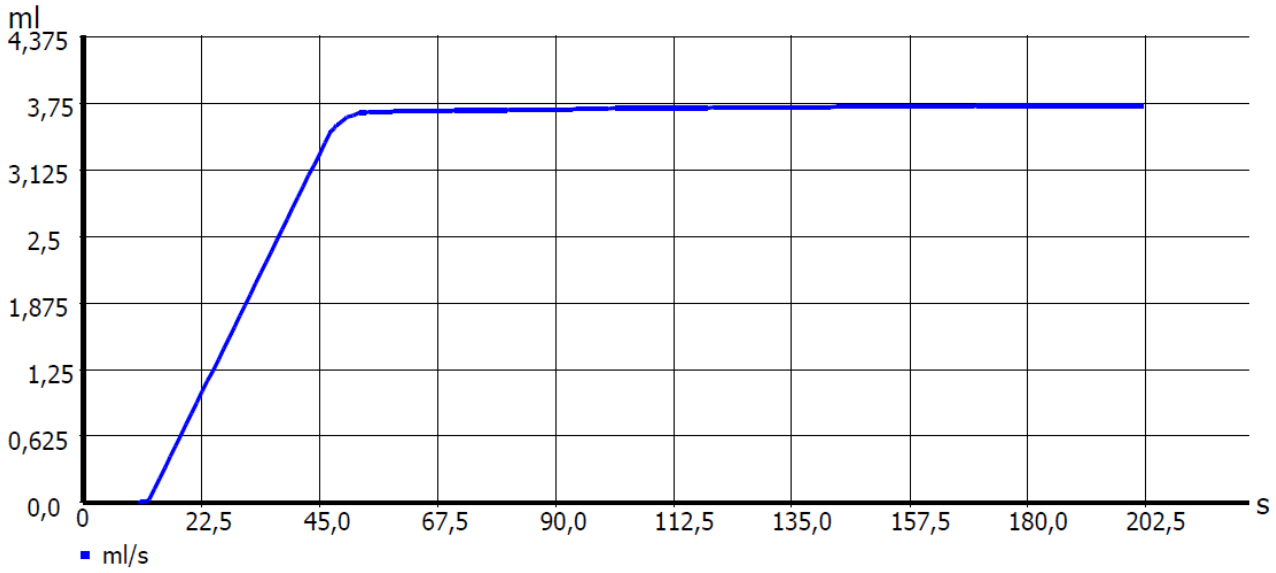
Sample preparation

The titration cell is filled with approx. 30 ml solvent and the conditioning is started. The solvent can be used for several titrations.

The sample is drawn into a syringe with a needle. If possible, the syringe should be rinsed with a small amount of sample before. After conditioning is finished, the sample is injected through the septum into the titration cell and the titration is started. The amount of sample is determined by weighing back.

The required amount of sample can be estimated using this rule of thumb:

$$W(g) = \frac{0{,}5 * Titer\ [\frac{mg}{mL}]}{expected\ \ water\ content\ [\%]}$$



Titration parameter

Suitable method parameters for the reagents used should be selected for the KF titration. Appropriate default methods are stored in the titrator. The parameters used here are well suited for most applications.

The default method "Sample 1-Comp." is well suited for 1-component reagents:

Default method	Sample 1-Comp.
Methoden type	Automatic titration
Mode	KF
Conditioning	On
Extraction time	10 s
Fixed delay time	1 s
Step size	0,005 mL
Pre-titration	aus
Polarization voltage	100 mV
Max. titration time	600 s
Min. titration time	60 s
Max. titration volume	50 mL
Drift	100 µg/min
Endpoint	20.0 µA
Delta Endpoint	3.0 µA
Endpoint delay	10 s
Dosing speed	30%

The default method "Sample 2-Comp." is well suited for 2-component reagents:

Default method	Sample 2-Comp.
Methoden type	Automatic titration
Mode	KF
Conditioning	On
Extraction time	10 s
Fixed delay time	0 s
Step size	0,004 mL
Pre-titration	aus
Polarization voltage	100 mV
Max. titration time	600 s
Min. titration time	60 s
Max. titration volume	50 mL
Drift	50 µg/min
Endpoint	20.0 µA
Delta Endpoint	18.0 µA
Endpoint delay	10 s
Dosing speed	30%

Calculation:

$$Water\ [\%] = \frac{(EP - B) * T * M * F1}{W * F2}$$

B	0	Blank value
EP		Consumption of titrant
T	WA	Exact concentration of the titrant, readed from the Exchange Unit
M	1	Molar mass of
W	man	Sample weight [g]
F1	0.1	Conversion factor 1
F2	1	Conversion factor 2

The calculation is done here as % water. F1 may need to be adjusted for other units.

Any questions? Please contact the application team:

Xylem Analytics Germany Sales GmbH & Co. KG, SI Analytics
Hattenbergstraße 10
D-55122 Mainz, Germany
Telefon: + 49 6131 66 5126
Fax: + 49 6131 66 5101
E-Mail: titration@si-analytics.com