

Titer determination in Karl-Fischer-Titration

Description

This application report describes the general procedure for titer determination in Karl Fischer titration with one- or two-component systems.

Three methods are described: the use of a liquid standard, a solid standard (di-sodium tartrate dihydrate) and pure water.

It is recommended to use a liquid standard, because it is the easiest and most accurate way to inject the standard into the titration vessel with a syringe.

It is not advisable to use pure water as a standard: The very small quantities which are needed are difficult to weigh accurately and bring into the titration vessel. Even small weighing errors lead to very large errors in the range of several %.

The titer is generally a dimensionless number of about 1 for correcting the indicated concentration. Deviating from this, the term "titer" in the Karl Fischer titration describes the exact concentration of the titrant in mg(H₂O)/ml(titrant) and not the dimensionless factor. The most commonly used titrants are 2 and 5 mg / ml.

Instruments

Titration vessel	Karl Fischer titration vessel TZ 1770
Stirrer	Titration stand TM 235 KF
Lab accessoires	Syringe 5 or 10 ml with needle 0.7 x 70 mm
	Microliter Syringe 20 or 50 µl
	Weighing boats
Titration vessel	Karl Fischer titration vessel TZ 1770
Exchangeable Unit	WA 10
Electrode	KF 1100
Titration vessel	Karl Fischer titration vessel TZ 1770
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Reagents

1	KF Titrant, e.g. Hydranal Composite (1-component) or Hydranal Titrant (2-component)
2	Solvent for Karl-Fischer-Titration, 1-component system, e.g. Hydranal Methanol rapid or similar
3	Solvent for Karl-Fischer-Titration, 2-component system, e.g. Hydranal Solvent or similar
4	Liquid standard in Ampoules, 10 mg/g (1%)
5	Di-sodium tartrate dihydrate reference material
6	Distilled Water
All reagents should be in analytical grade or better.	

Titration procedure

Reagents

Water standard 10 mg/g or 1% in ampoules is used as supplied.

Di-sodium tartrate dihydrate reference material is dried as described in the corresponding analysis certificate.

Basic procedure

Choose default method Titer...



Titration vessel is conditioned.
Wait for the message "Conditioning finished"



Add standard.



Enter sample name



Enter sample mass or transfer from balance.



Titration is running.

After the end of the titration, the titer is automatically stored in the exchangeable unit.

A multiple determination is recommended. The default setting for the titer methods is a 3-fold determination.

Sample preparation liquid standard 10 mg/g or 1%

The method titer 1-comp (liq. st.) or titer 2-comp (liq. st.) is selected. In the calculation formula, the water content of the standard (according to the certificate, in mg/g) is entered as factor F2 (default is 10.00).

The big advantage of the liquid standard 10 mg/g is the simple addition with a syringe, the good solubility in the KF reagent and the easy-to-handle weight of the standard. In addition, virtually no water from the air gets into the titration vessel as it does not have to be opened to add the standard.

The amount of liquid standard depends on the size of the burette and the concentration of the KF reagent. The amount should be chosen so that about half of the burette volume is consumed. The most common is the 10 ml burette. The required amount of standard 10mg/g can be estimated according to this rule of thumb:

$$W [g] = 0.5 * concentration[mg/ml]$$

The Liquid Water Standard is injected with a syringe through a septum into the titration vessel. The ampoule is opened and the syringe is rinsed with about 1-2 ml of the standard. The rest of the standard from the ampoule is drawn into the syringe. The syringe is placed on a balance and tared. After conditioning is ready, the needed amount of standard is injected through the septum into the titration vessel and the syringe is weighed back. Several titrations can be carried out with one ampoule of standard.

Sample preparation solid standard

The method titer 1-comp (solid st.) or titer 2-comp (solid st.) is selected. In the calculation formula, the water content of the standard (according to the certificate, in mg/g) is entered as factor F2 (default is 156.6).

Di-sodium tartrate dihydrate is only sparingly soluble in methanol. When working with a solid standard, care must be taken that the standard dissolves completely in the KF reagent. Since the crystals of the standard are colorless, it is difficult to see if everything is solved. If necessary, a higher extraction time must be set.

The amount of solid standard di-sodium tartrate dihydrate depends on the size of the burette and the concentration of the KF reagent. The amount should be chosen so that about half of the burette volume is consumed. The most common is the 10 ml burette. The required amount of di-sodium tartrate dihydrate can be estimated by this rule of thumb:

$$W [g] = 0.05 * concentration[mg/ml]$$

The standard is weighed into a weighing boat and then the balance is tared with weighing boat and standard. After conditioning is complete, the standard is added to the titration vessel (this requires removal of the septum) and the weighing boat is weighed back.

Sample preparation pure water as standard

The method titer 1-comp (water st.) or titer 2-comp (water st.) is selected.

The big disadvantage of this method is the small amount of water that is needed. Even small weighing errors lead to very large errors. The titer determination with pure water should be carried out only by very experienced staff!

The amount of pure water depends on the size of the burette and the concentration of the KF reagent. The amount should be chosen so that about half of the burette volume is consumed. The most common is the 10 ml burette. The required amount of water can be estimated according to this rule of thumb:

$$W [g] = 0.005 * concentration[mg/ml]$$

The water is drawn up in a microliter syringe. The syringe is placed on a balance and tared. Subsequently, the titration is started. After conditioning is ready, the standard is injected through the septum into the titration vessel and the syringe is weighed back.

The dosage of the water standard can also be done directly with a calibrated microliter syringe by volume.

Titration parameter

The TitroLine 7500 KF, TitroLine 7750 and TitroLine 7800 titrators contain 6 different titration methods for titer determination in KF titration.

Method	Suitable for
Titer 1-comp (liq. st.)	1-Component-system, liquid standard 10 mg/g
Titer 1-comp (solid st.)	1-Component-system, solid standard
Titer 1-comp (water st.)	1-Component-system, pure Water standard
Titer 2-comp (liq. st.)	2-Component-system, liquid standard 10 mg/g
Titer 2-comp (solid st.)	2-Component-system, solid standard
Titer 2-comp (water st.)	2-Component-system, pure Water standard

These methods are optimized for their application and should not be changed. The result of the titer determination is stored in mg/ml directly in the exchangeable unit.

Any questions? Please contact the application team:

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