KF-Titration with Oven TO 7280

Description

Indirect coulometric Karl Fischer titration with the TO7280 headspace oven is suitable for many samples. The water content of solid samples or samples that cannot be titrated directly due to side reactions can also be determined in this way.
The general procedure: The sample is placed in a vial and the vial is closed. The water is heated out and transferred through a carrier gas into the titration cell where it is titrated.
The temperature at which the water is heated out depends on the sample: The temperature must be chosen so high that the water is completely released from the sample. However, the temperature must not be too high: some samples decompose at high temperatures.
For the carrier gas, you have the choice between air (via the pump built into the TM 235 TO) or, for sensitive samples, an inert gas (e.g. nitrogen).
The background drift of the complete system is determined with the 0-vial.
The blank value, i.e. the water content of an empty vial, must be determined for accurate results. Depending on the humidity, the blank value of the 5ml - vials is approx. 40µg to over 100µg!
For samples that have a small volume with a high water content, the volume of the sample can be neglected. For samples with very low water content (a few ppm), such as insulating oils, the volume of the sample must be considered and the blank value corrected.

Devices

Titrator	TL 7500 KF trace M6
Oven	TO 7280
Generator elektrode	TZ 1752 (without diaphragm)
Elektrode	KF 1150
Vials	TZ 3988
Titration cell	TZ 1754

Reagents

1	Reagent for generator without diaphragm, suitable for use with oven
2	Molecular sieve, dried

Titration procedure

Reagents

Karl Fischer reagents are available as ready-to-use solutions.
The molecular sieve must be replaced 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.

Cleaning and handling the generator electrode

The TZ 1752 electrode does not require any special treatment.
The platinum sieve must not be mechanically stressed or bent. For cleaning, Isopropanol or other solvents that do not attack glass and platinum are suitable.

Preparation of the titration cell

The titration cell is filled with approx. 140 ml KF reagent and the titrator is switched on. A method with headspace oven is selected or the oven control is activated. The cell is automatically titrated dry and is ready when the drift is constantly below 15µg/min.

Preparation of the vials

The vials and crimp caps should be in equilibrium with the laboratory air. Especially different temperatures between vial and laboratory air lead to wrong or strongly fluctuating blank values. Previous drying in a drying oven or breathing air in the vial also lead to strongly fluctuating blank values! It is best to place vials and crimp caps a few hours before starting work where the sample will be weighed into the vial (i.e. next to the balance).
The vials for the blank value must be treated exactly in the same way as the vials for the sample and also closed at the same time if possible.
Another empty vial is closed and used as a 0-vial.

The correct temperature

The correct oven temperature depends strongly on the sample. The temperature must be high enough to quickly release all the water from the sample. However, it must not be too high that the sample decomposes. Usually, the suitable temperature is found in the range of 100 - 160°C.

Background drift

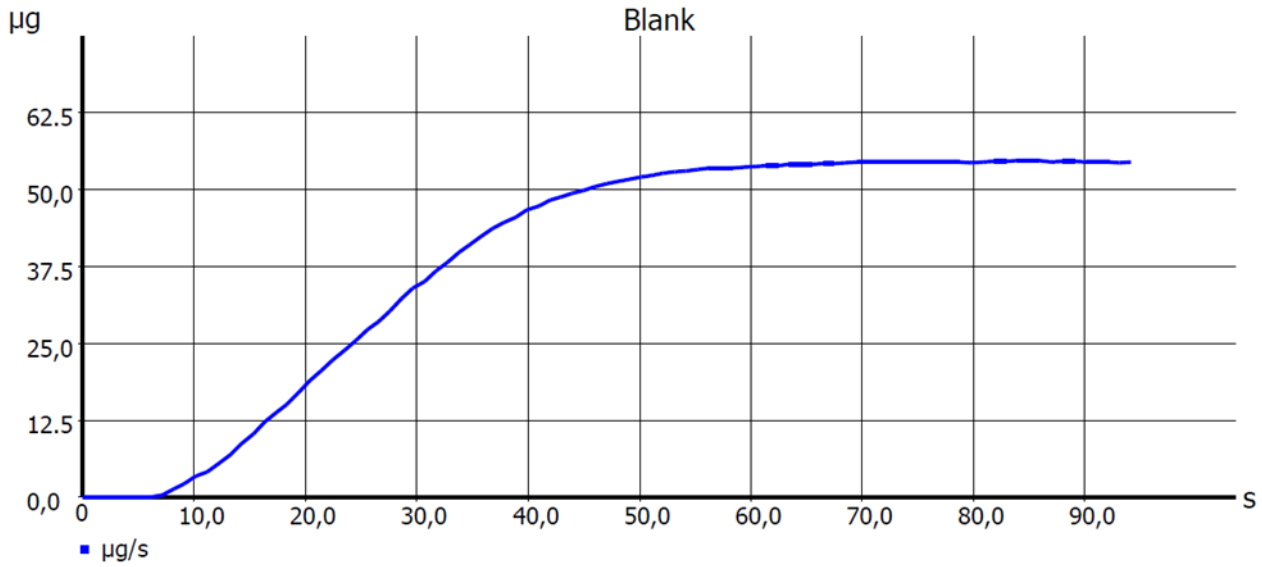
The 0-vial is placed in the oven and the method is started. The gas supply is set to approx. 0.1 L/min on the flowmeter. The background drift is now determined. The sample method can be started as soon as all start criteria (temperature, start drift, Δ) are fulfilled.
It is sufficient to determine the background drift at the beginning of a measurement series using the same method.

Blank value

The blank value should be carried out at least as a 3-fold determination. For the blank value, the same titration parameters must be used as for the sample titration.

The empty vials are closed with the crimp cap. The vials for the blank value must be handled in the same way as the vials for the sample and must also be closed at the same time if possible. It is best to close vials with sample and vials for the blank value alternately.

Titration parameter Blank value



Default method	With Headspace Blank
Method typ	Automatic Titration
Mode	Coulometrie
Start Drift	15
Start Drift Tolerance	0,3 µg/min
Start Drift Tolerance Intervall	20 s
Stop Drift Delta	5
Stop Drift Tolerance	0,02
Min. titration time	90 s
Max. titration time	1200 s
Endpoint delay	5 s
Working point	300 mV
Control faktor	4
Oven	
Oven temperature	150°C*
Oven temperature Delta	0,5 °C
Gas supply	Pump
Automatic fan control	on
0-Vial Interval**	Never
0-Vial on Method change**	yes

*or another suitable temperature **only with TitriSoft

Calculation:

$$B\ [\mu g] = \mu g \quad \rightarrow \text{M01}$$

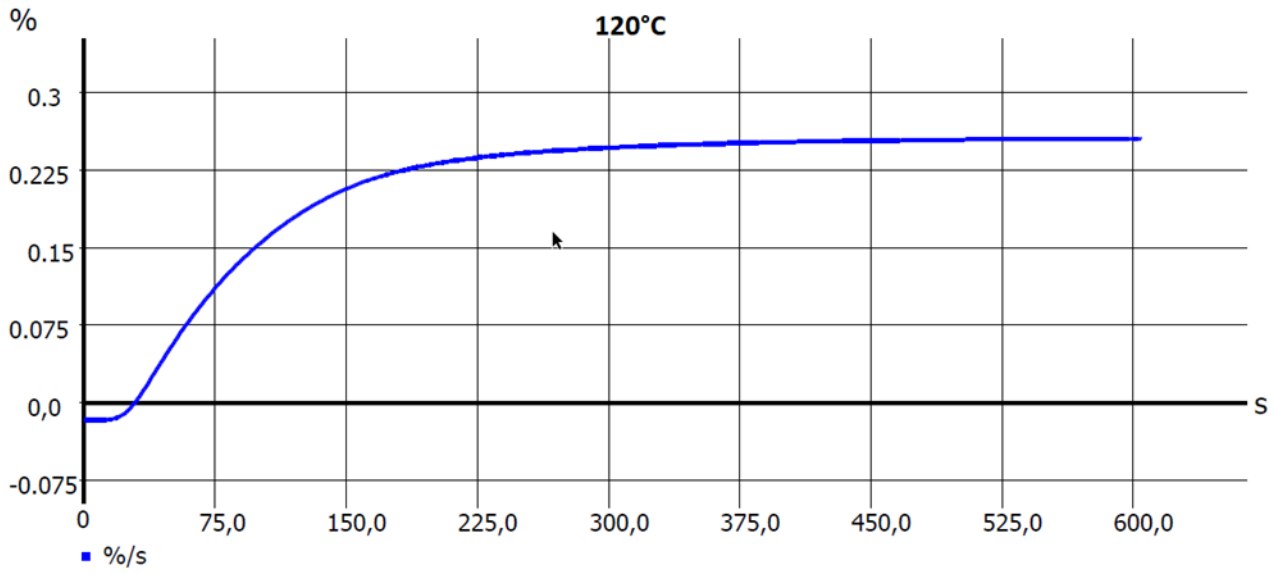
µg		Absolute Water content in µg

The determination of the blank value should be carried out at least as a 3-fold determination and the mean value calculated. The blank value is stored in a global memory.

Sample preparation

The sample is weighted into a vial and the vial is closed with the crimp cap.
The sample amount should be chosen so that there is no more than approx. 10 mg of water in it - otherwise the titration time will be unnecessarily long.
For samples with very low water contents, the sample amount should be chosen use as large as possible, but the volume should not exceed approx. 4.5 mL. For liquid samples, the sample volume should be large enough to immerse the needle in the sample.

Titration parameter



For KF titration, method parameters suitable for the reagents used should be selected. The gas supply should be approx. 0.1 L/min.

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Calculation

Berechnung:

$$Wasser\ [ppm] = \frac{(\mu g - B) * M * F1}{W * F2}$$

B	M01	Blank value in µg, stored in global memory M01
µg		Water content in µg
M	1	Molekular weight
W	man	Sample amount [g]
F1	1	Conversion factor 1
F2	1	Conversion factor 2

The result is calculated as ppm water. If the result should be calculated in other units, F1 must be adjusted.

Hint:
The oven system should be checked regularly by titration of a suitable standard. Suitable standards are e.g. lactose standards with approx. 5% water used at 150 - 160°C. The recovery should be 100 ± 5 % and the standard deviation (RSD) <1.5 %.

Any questions? Please contact the application team:

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