Temperature ramp KF Oven

Description

In indirect Karl Fischer titration using a headspace oven, the temperature has a decisive influence on the result.
If the temperature is too low, the water is only released slowly or not completely. If the temperature is too high, the sample may decompose, which can lead to too high results.
At the correct temperature, the water is released quickly and completely without decomposition of the sample.
To determine this temperature, a temperature ramp (heat-out curve) is recorded. A sample is placed in the oven and the temperature is slowly increased. Meanwhile, the drift is recorded. The temperature ramp shows how much water is released at which temperature.
The Titrisoft 3.5.4 software or higher is required for a temperature ramp.

Instruments

Titrator	TL 7500 KF trace M6 with Oven TO 7280
Electrode	KF 1150 and TZ 1752
Stirrer	Magnetic stirrer TM 235 TO
Vials	TZ 3988
Lab accesory	Titration cell TZ 1754
	Magnetic stirrer bar 30 mm

Reagents

1	Reagent (Anolyte), suitable for use with oven, e.g. Hydranal Coulomat AG-Oven
2	Molekular sieve, dry
3	Methanol, dry
4	Water Standard 1 mg/g or 0,1%
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

Regent (Anolyte)

Suitable reagents are available ready for use.
As methanol and other volatile components are carried out of the titration cell by the carrier gas, there is a loss of volume, and the reagent becomes noticeably more viscous. The reagent must then be replaced. Sometimes it can be refreshed by adding dry methanol.
The condition of the reagent should be checked regularly by titration of a standard (1 mg/g or 0.1%).

Molekular sieve

The molecular sieve must be replaced or regenerated regularly, every 1-2 weeks depending on use. To regenerate, the molecular sieve is dried at 250 - 300°C for 1-2 hours.

Vials

The vials and crimp caps are best stored open, but protected from dust, next to the balance. This keeps the vials and caps in equilibrium with the humidity, resulting in stable blank values.

Sample preparation

The sample is weighed into a vial and the vial is sealed with a crimp cap.

The sample quantity is chosen so that there is approx. 1 - 3 mg of water in the sample. The maximum volume should not exceed approx. 4 mL.

Expected Water content	Sample amount [g]
1 – 10 ppm	5 – 10*
10 – 100 ppm	2 – 5*
0.01 – 0.1 %	0.5 – 2
0.1 – 1 %	0.1 – 0.5
1 – 5 %	0.05 – 0.1
5 – 15 %	< 0.05**

* or max. 4 ml!

** for sample amount <50mg a balance with 5 digits is recommended.

Titration parameter

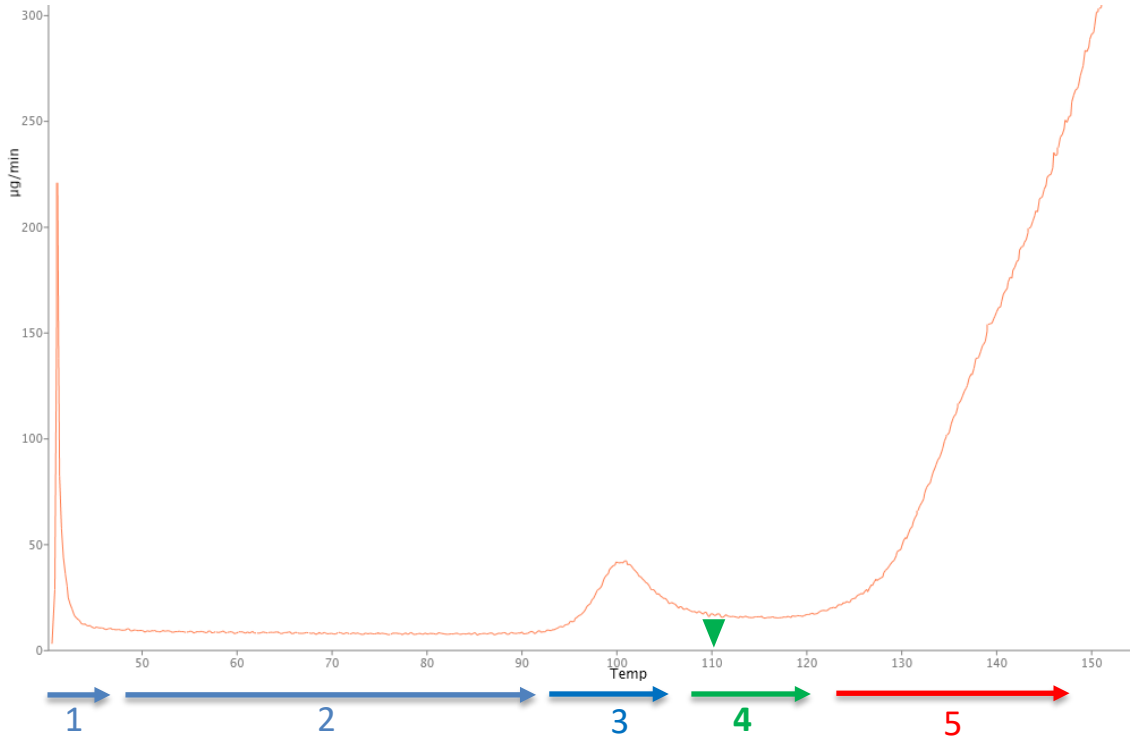
Default method	---
Start drift	15.0 µg/min
Interval	20 s
Stability:	0.3 µg/min
Working point:	300 mV
Control factor	4
Oven	
Gas source	Gas
Start Temperature	40°C
End Temperature	280
Temperature Delta	0,5°C
Temperature ramp	1°C
Fan automatic	On
Gas flow (set manually at Flowmeter)	100 – 150ml/min

* This range covers the entire temperature range of the oven. The start and end temperature can be adjusted depending on the sample.

Evaluation:

No water content is calculated!
Instead, the temperature ramp is analyzed visually. A minimum is sought that is at a higher temperature than the water release of the sample.

For example, Potassium hydrogen carbonate provides the following curve:



1. Water from blank or gas phase of the vial.
2. The sample does not yet release any or only a little water, temperature too low.
3. Water from sample is released.
4. Ideal temperature range - no further water is released, but no decomposition.
5. Decomposition, temperature too high.

A high drift can be recognized between 40 - 45°C, which is caused by easily released water (e.g. from the gas phase of the vial, the blank value). The drift then remains very low up to approx. 95°C, where no water is released. Between 95 - 110°C the drift is increased again. In this range, the water contained in the sample is released. From 110 - 120°C the drift is again quite low, the water of the sample is already released, decomposition does not yet take place. Decomposition of the sample begins above 120°C.
For sample titration, a temperature is chosen at which the water is safely released, but which still has a clear distance to decomposition. In the example of potassium hydrogen carbonate, 110°C is suitable.
The carrier gas can have a decisive influence here: If air is used as the carrier gas, the usable temperature window can become significantly smaller due to oxidation of the sample or decomposition can take place before the water is completely released. An inert gas (e.g. nitrogen) should be used for sensitive substances. Some samples decompose before the water is released completely – such substances cannot be titrated with the oven method.

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Let's Solve Water