KF-Titration with Oven TO 7280 Troubleshooting

Description

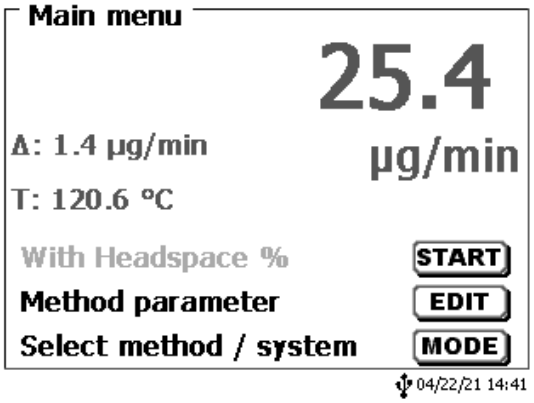
The coulometric Karl Fischer titration with the headspace oven TO7280 is quite a convenient procedure for the user: The sample is placed in a vial and sealed. The water is heated and transferred through a carrier gas into the titration cell. After the end of the titration, the system is immediately ready for the next sample. In normal operation, the user has no direct contact with the chemicals - except when changing reagents.
But what if things don't work as they should? In this guide, we describe the most common errors that can occur in the TO 7280 headspace oven and how to fix them. In this guide, we describe the most common errors that can occur with headspace oven TO 7280 and how to fix them. For errors that affect the titration itself or the titration cell, we recommend our application "Coulometric KF Titration Troubleshooting".

Basics

The titration cell must be sufficiently filled with the reagent so that the contacts of the generator and indicator electrodes are completely covered by liquid.
A reagent suitable for oven operation must be used. These reagents are less volatile than normal reagents.
A temperature must be selected that is suitable for the standard or sample being used. When starting up the oven, it may take some time for the starting drift to become low enough - the complete system must be dried first.
If problems occur, a liquid water standard (a standard with a concentration of 1 mg/g is recommended) should be titrated first to rule out the possibility that there is a problem with the titration cell or reagent. Used up reagents often lead to very low recoveries.
An oven standard is then titrated. Oven standards for different temperature ranges are available. A standard should be used that is suitable for the temperature range in which the sample is titrated.
The blank value of the empty vials should always be determined. Depending on the humidity, the blank value of the 5 ml vials is approx. 40 µg to over 100 µg!

Drift too high, start drift is not reached

Problem: The drift (with oven) no longer reaches the start drift (usually 15 µg/min), it remains permanently too high. Without an oven, the drift is low. The base drift is the water that enters the titration cell and is "titrated away" there.



Various causes can be responsible for excessive drift - it is often used desiccant, a leaky titration cell, side reactions or even just a used reagent.

Used desiccant

A frequent cause of excessive base drift is used desiccant. As a result, moisture from the carrier gas enters the titration cell and the drift increases.

If the base drift is high, the desiccant in the two desiccant bottles on the TM 235 TO and in the desiccant tube on the generator electrode should first be replaced and the tubing connections checked for leaks.

Too high temperature, unsuitable septa

If unsuitable septa are used at very high temperatures, the septa may release water contained in them or even decompose and thus lead to excessive drift.

In this case, it helps to reduce the oven temperature or to use septa suitable for high temperatures. Most samples release their water at temperatures well below 160°C. The septa of Xylem Analytics tolerate these oven temperatures without any problems if used correctly.

Too high or too low results when oven is used

If it is suspected that too much or too little water is detected with the KF oven, the titrator with titration cell without oven is first checked with a water standard. Standards with a water content of 0.1% or 1mg/g are well suited. Solid standards are not suitable due to their poorer solubility. Pure water is not suitable due to the very small amount required and the associated high inaccuracy in weighing. The water content of the standard should be recovered within the specified imprecision of the standard, but at least the recovery should be 100 ± 2 %. The relative standard deviation should not exceed 0.5%. If the recovery rate with the liquid standard is within this range, it can be assumed that the titrator and titration cell are in good condition. If problems should already occur here, you can proceed according to the application note "Coulometric KF Titration Troubleshooting".

If the titration cell is OK, a solid water standard suitable for the desired temperature range and the blank value of the empty vial are titrated with oven. At least a 3-fold determination should be carried out for blank value and standard. It is important that the temperature range specified for the standard is maintained. If the temperature is too low, not all the water will be released; if the temperature is too

high, pyrolysis of the standard may occur. The water content of the standard should be recovered within the specified imprecision of the standard, but at least the recovery should be 100 ± 5 %. The relative standard deviation should not exceed 1.5%.

Too low results

If, in spite of a functioning titrator and titration cell, there are false low readings, the cause should be sought in the area of the oven. First of all, it should be checked whether the correct temperature range has been maintained and whether the titration cell is working properly.

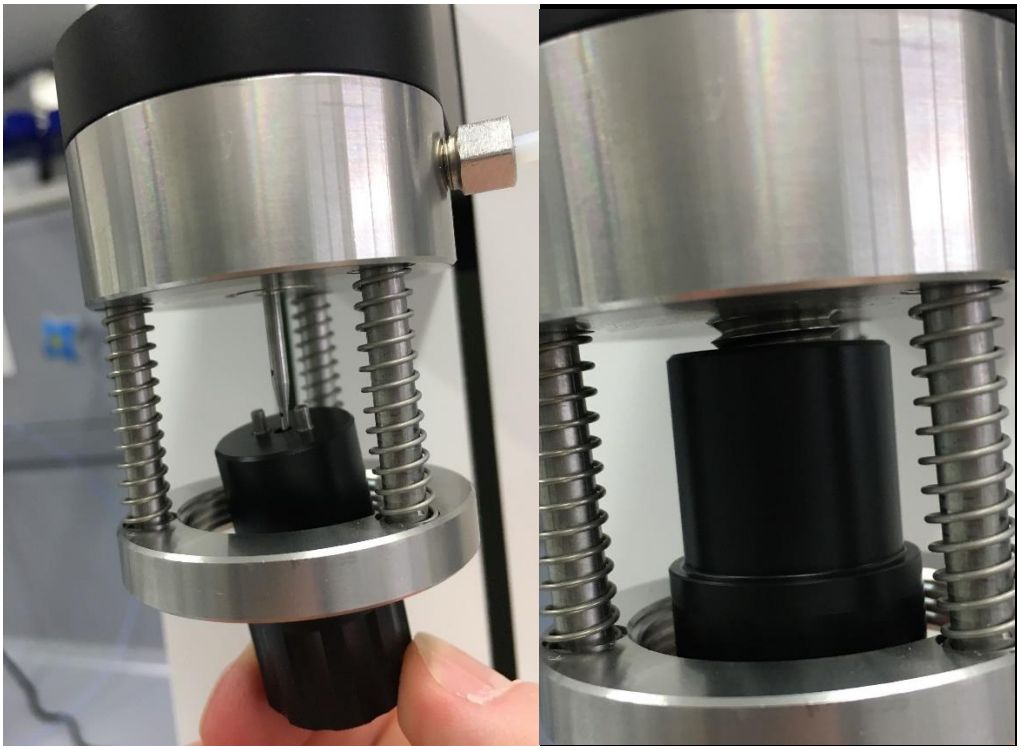
If the temperature is correct, too low results are usually due to a leak between the vial and the titration cell. To be checked are:

- Gas flow correctly adjusted (100 mL/min)
- tight fit of tubes and screw connections
- tight fit of the needle
- correct fit of the seals
- condition of the septa (elastic, must not tear when pierced with the needle)

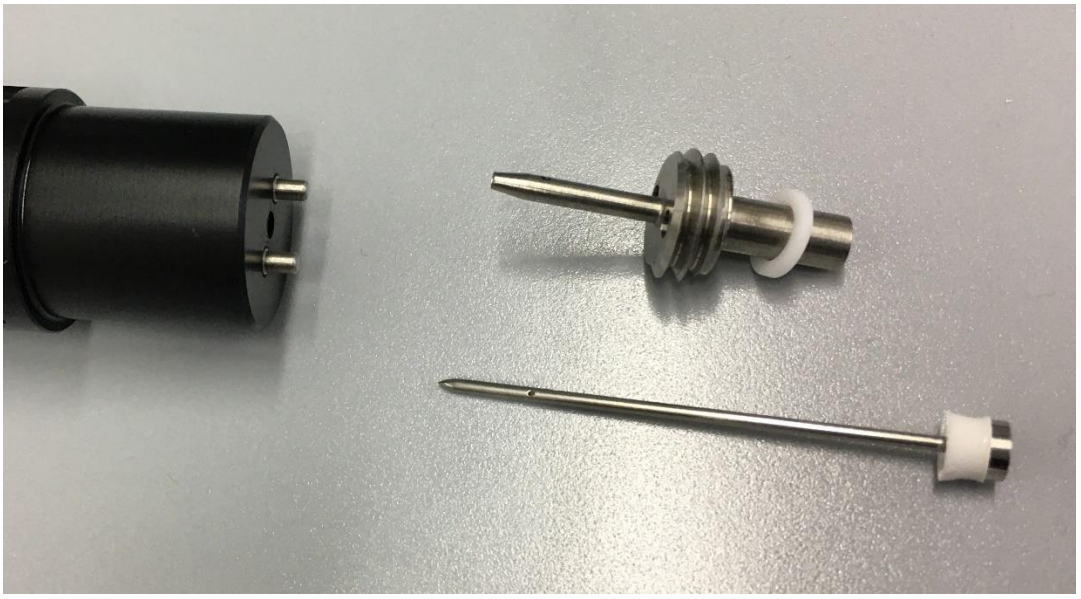
The sealing surfaces of the tube fittings on the needle head in particular must be absolutely clean: here, for example, even the fiber of a cellulose cloth can lead to a significant loss of gas (and thus to significantly lower results).

To check the needle, it must be removed. To do this, unscrew the lower centering disk from the needle head (clockwise).

The needle can then be unscrewed with the needle wrench (special tool, included with the TO 7280):



Now the needle can be removed, checked and cleaned if necessary. The inner needle (below) is only inserted through the outer needle.



Reassembly is performed in reverse order.

An incorrect (too high) blank value also leads to too low results. The vials for the blank value should always be closed at the same time under the same conditions as the sample vials.

Too high results

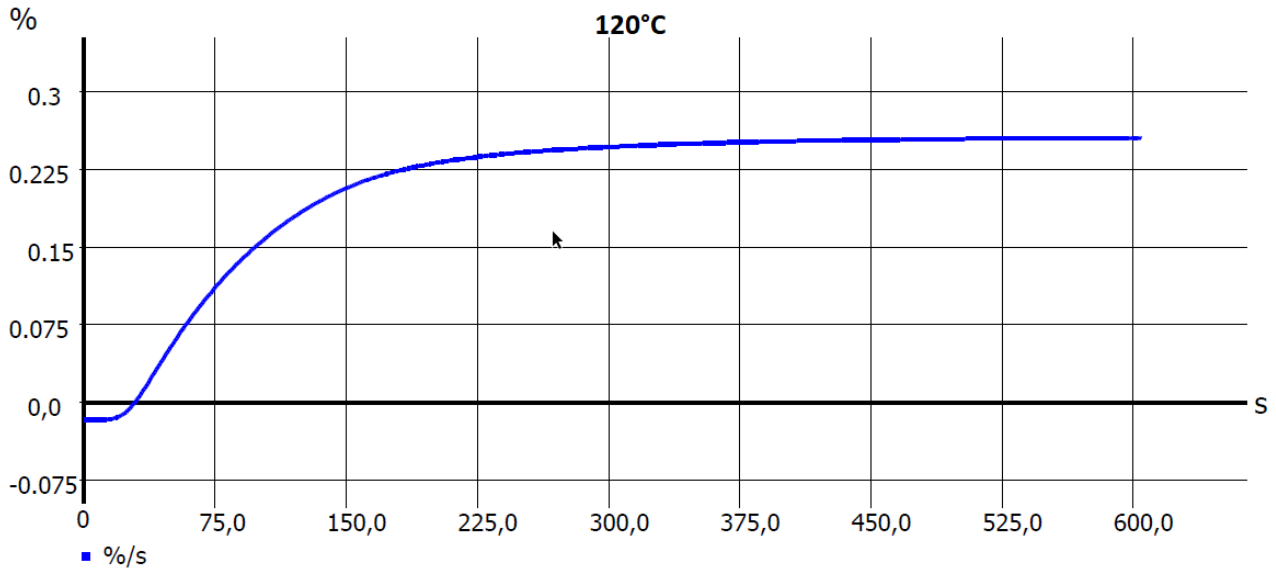
Too high results during the titration of a standard are very rare if the temperature is correct. If there are excess results, the supply lines for the carrier gas and the drying flasks should be checked for leaks and the molecular sieve should be replaced. False high readings in samples can also be caused by decomposition reactions.

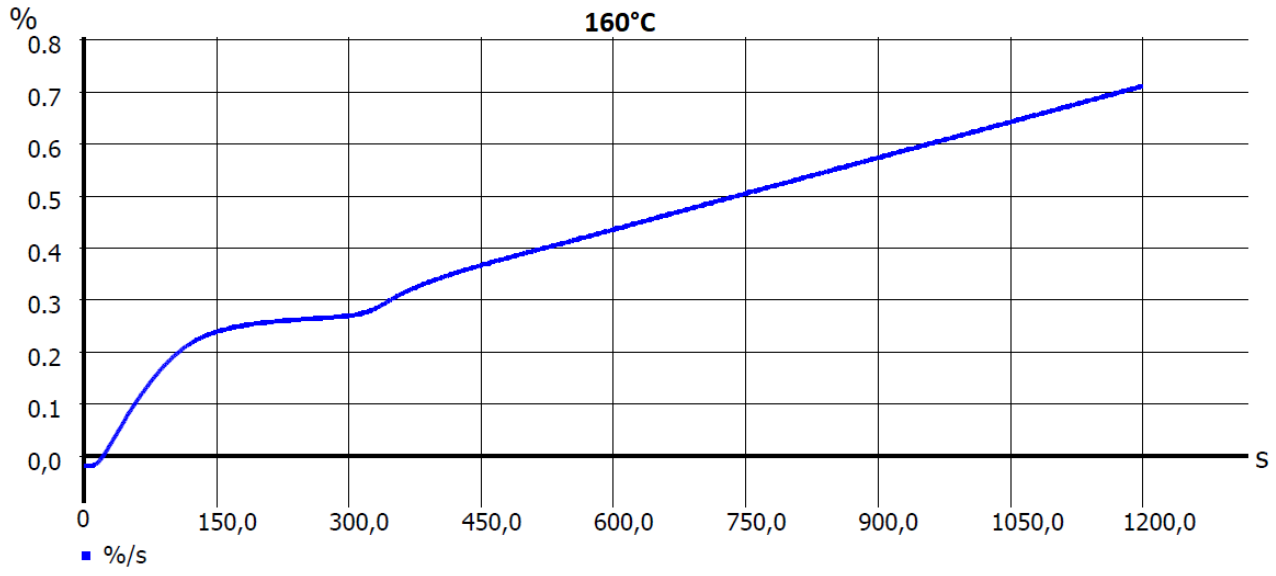
An incorrect blank value (too low) also leads to false high readings. The vials for the blank value should always be sealed at the same time under the same conditions as the sample vials.

Decomposition reactions

If the temperature is too high or if the wrong carrier gas is used (air in the case of oxidation-sensitive samples), the sample may decompose and split off water. The titration then finds no end, the water content found is much too high.

Here is a polyethylene oxide that can be titrated without problems at 120°C, but slowly decomposes at 160°C:





In such a case, it only helps to reduce the temperature so that decomposition no longer occurs. If air is used as the carrier gas, decomposition can be avoided in the case of easily oxidizable substances by using nitrogen or argon.

Gas flow too low

The gas flow should always be set with vial so that the gas is directed through the vial into the titration cell. Without vial, the gas flow is somewhat higher due to the lack of flow resistance in the needle. The gas flow should be about 400 - 500 mL/min when the valve on the flowmeter is fully open. For the titration the gas flow is adjusted to 100mL/min.

If the gas flow is significantly lower, test first without the vial in the oven. Without the vial, the gas flow is >500mL/min. If this is not the case, either a tube is clogged or a screw connection is leaking.

To be checked:

- All tubes and screw connections between TM 235 TO and needle head, especially the tubes on the drying flasks.
- The screw fittings on the drying bottles.
- The seals of the tube screw connections in the needle head.
- The needle (clean if necessary)

If the gas flow without vial is about 500mL/min, the cause is probably between the needle head and the titration cell.

To be checked:

- The seals of the tubing screw connections in the needle head.
- The seals of the tube fittings in the needle head.
- The needle, especially the bores in the outer needle (clean if necessary).
- All connections between the TM 235 TO and the needle head (a leak here can also lead to an insufficient flow with vial).

Any questions? Please contact the application team:

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