Coulometer User Manual

Never work on the machine if you have not been trained to operate the machine.

Coulometric titration

Principle of coulometry according to Karl Fischer

The **coulometric Karl Fischer titration** is a variation of the classic water content determination method according to Karl Fischer. The conventional method works with a methanolic solution of iodine, sulfur dioxide and a base as buffer substance. If an aqueous sample is titrated, then several reactions take place that can be summarized in the following sum equation:

$H_2O + I_2 + [RNH]SO_3CH_3 + 2\ RN \rightleftarrows [RNH]SO_4CH_3 + 2\ [RNH]I$

According to the equation above the I_2 reacts quantitatively with H_2O. This chemical equation serves as a basis for the water content determination.

With the **coulometric Karl Fischer titration**, the necessary iodine is directly and electrochemically generated in the electrolyte containing iodine ("electronic buret"). Between the amount of electric charge and the amount of generated iodine, there is a strictly quantitative relationship, which is used for high-precision dosing of the iodine. Because the coulometric Karl Fischer method is an **absolute determination**, no titer needs to be determined. It must only be ensured that the reaction generating the iodine runs with a 100% current efficiency. All of the reagents available today ensure this.

The endpoint indication is effected voltametrically by modulating an alternating current of constant strength to a double Pt electrode. This results in a voltage differential between the Pt wires. This is drastically reduced as soon as even the slightest amounts of free iodine are present. This circumstance is used for detecting the endpoint of the titration.

Working with water standards

Certified water standards

Commercially available, certified water standards with water content of 1.00 ± 0.003 mg/g and/or 0.10 ± 0.005 mg/g should be used for validating the instrument as a whole, integrated system.

NOTICE

The 1.0 mg/g water standard is easier to handle and is therefore preferred.

Recommended weighing ranges

1.0 mg/g water standard	0.2 - 2.0 g
0.1 mg/g water standard	0.5 - 5.0 g

Practical recommendations

For validation, it is essential to work very accurately. In order to minimize any measurement inaccuracies that could occur, the sample preparation and the sample processing should proceed in accordance with a defined scheme:

1 Put on gloves (always for Karl Fischer titration).

2 Use a clean syringe.

3 Take a new ampoule of water standard and shake it briefly.

4 With a folded paper towel held between thumb and index finger, break open the ampoule at the marking.

5 Draw approx. 1 mL of the water standard into the syringe.

6 Pull the plunger of the syringe up to the end and shake the syringe back and forth somewhat.

The inside of the syringe is rinsed by water standard and freed of water contamination.

7 Dispose of the used water standard in a waste bottle.

8 Draw the rest of the water standard into the syringe, aspirating as little air as possible.

9 Push out any air bubbles that may be present in the syringe.

10 Wipe off the needle with a lint-free paper towel and cover it with the appropriate cap.

11 Place the syringe on the balance and press **[TARA]**.

12 As soon as the drift on the 899 Coulometer is stable, take the syringe in your hand, press **[START]** and inject approx. 1 mL of the water standard through the septum.

There are two possibilities:

▪Version 1:

Inject the water standard without immersing the needle in the reagent liquid. If a little drop remains on the end of the needle, it must be aspirated back before pulling the needle out of the septum.

The water standard should not be sprayed from the syringe onto the electrode nor onto the wall of the titration cell.

▪Version 2:

Inject the water standard directly under the surface of the reagent liquid.

Take care to ensure that you do not aspirate any liquid when you withdraw the syringe from the reagent liquid.

13 Close the syringe with the same cap and place it back on the balance.

14 Read off the value displayed by the balance and enter it on the Coulometer as the sample size.

If you have connected a balance to the Coulometer, you may transmit the sample size directly from the balance.

15 The next determination can be started as soon as the determination has been finished and the titration cell has been conditioned (drift stable) again.

Sample addition

Size of the sample size

The sample weight should be small in order to be able to titrate as many samples as possible in the same electrolyte solution and to keep the titration time short. However, ensure that the sample contains at least 50 µg of H_2O. The following text helps you determine the appropriate sample size.

Recommended sample sizes

Water content of the sample	Sample size	Resulting water content
10,000 ppm = 1%	10 mg - 100 mg	100 µg - 1,000 µg
1,000 ppm = 0.1%	100 mg - 1 g	100 µg - 1,000 µg
100 ppm = 0.01%	1 g	100 µg
10 ppm = 0.001%	5 g	50 µg

Working with liquid samples

Liquid samples are added with a syringe. The samples can be injected two different ways:

- One uses a syringe with a long needle, which one immerses in the reagent during the injection.
- One uses a syringe with a short needle and aspirates the last drops back into the needle.

The best way for you to determine the injected sample amount is to reweigh the sample.

Glass syringes should be used for the **determination of traces and validations**. We recommend obtaining these from a specialized syringe manufacturer.

Highly volatile samples and samples of low viscosity should be cooled before sampling. Doing so avoids losses while working. The syringe must, however, not be cooled directly, as condensation could be formed. For the same reason, no air may be aspirated into a syringe into which a cooled sample has been aspirated beforehand.

Samples of high viscosity can be thinned by heating. The syringe must be heated as well. The same target can be reached by diluting with suitable solvents. In this case the water content of the solvent has to be determined and subtracted as a blank value.

Pastes and fats can be added to the titration cell with a syringe without needle. You can use the ground-joint opening for this. If you also wish to aspirate, you can use the opening with the septum stopper. The best way for you to determine the sample amount is to reweigh the sample.

If samples contain only **traces of water**, then the syringe has to be predried well. If possible, the syringe should be rinsed with the sample solution by filling in and discarding solution several times.

Operation

Switching the instrument on and off

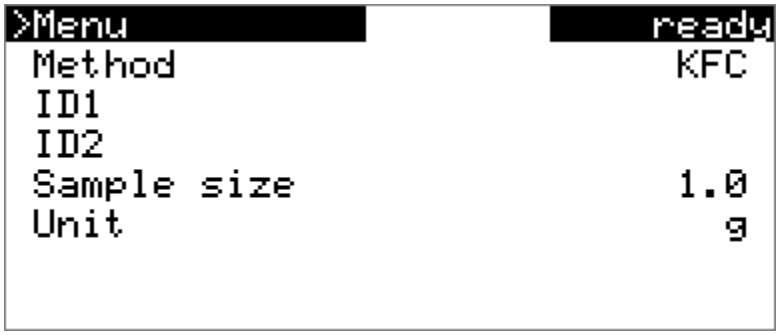
Switching on the instrument

Proceed as follows:

1 ▪ Press the red **[STOP]** key. 

The instrument is initialized and a system test performed. This process takes some time.

The main dialog is displayed:



Switching off the instrument

The instrument is switched off with the **[STOP]** key. The fact that the key needs to be pressed down for an extended time prevents accidental switch off.

Proceed as follows:

1 ▪ Keep the red **[STOP]** key pressed down for at least 3 s.

A progress bar is displayed. If the key is released during this time, then the instrument will not be switched off.

Fundamentals of operation

The keypad

BACK	Apply the input and exit the dialog.
⇧ ⇩	Move the selection bar either up or down by one line at a time.
	Select the character to be entered in the text editor.
⇦ ⇨	Select the character to be entered in the text and number editor.
	Select the individual functions in the function bar.
OK	Confirm the selection.
STOP	Stop an ongoing method run or a manual function.
	Switch the instrument on or off.
START	Start a method run or a manual function.

Carrying out a determination

The sample size can be entered in the following ways when a determination is carried out:

- Enter manually on the instrument.
- Send automatically from a connected balance. For this purpose, check the manual for the balance.

Proceed as follows to carry out a determination:

1 Loading the method

Proceed as follows to load a method template:

a) Opening method templates

- In the main dialog, select **Method** and press **[OK]**.

The method table with the stored methods opens:

b) In the function bar, select **New** and press **[OK]**.

- The list with method templates opens:

c) Loading the method template

- Select the desired method template **KFC** and press **[OK]**.

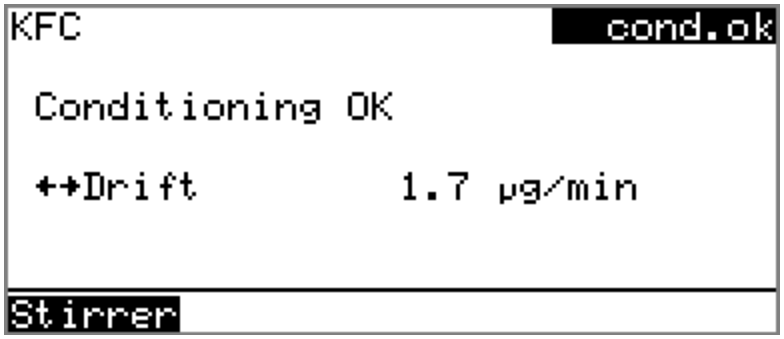
The method is now loaded and is displayed in the main dialog under **Method**.

2 Starting conditioning

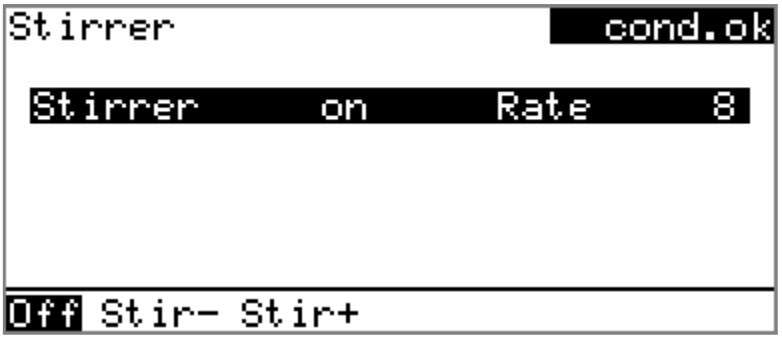
- Press **[START]**.

Conditioning starts. **Conditioning not OK** is displayed until the endpoint is reached. The working medium is titrated to the endpoint.

This is indicated by **Conditioning OK**. The status is kept stable.



The stirring rate can be modified with the **Stirrer** function. The following dialog is opened by pressing **[OK]**:



The stirring rate can be reduced with **Stir-** and increased with **Stir+**. **Off** switches the stirrer off. **On** is now displayed instead. This can be used to switch the stirrer back on. This dialog is exited with **[BACK]**.

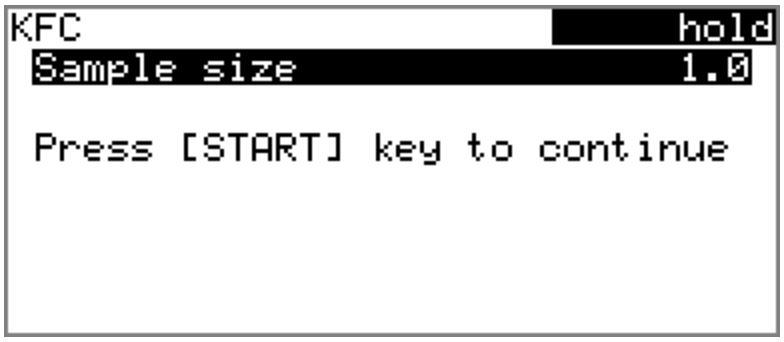
3 Adding sample

▪ If Conditioning OK is displayed, press **[START]**.
Conditioning is stopped. The request for adding the sample will be displayed for 8 s.

The sample must be added during this time.

▪Add the sample.

Afterward, the request for the sample size appears:



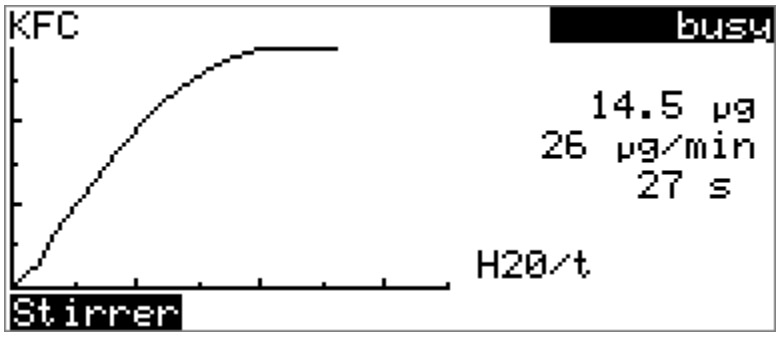
4 Entering the sample size

▪ Press **[OK].**
The editing dialog opens.

▪ Enter the sample size and apply with **Accept** or **[BACK]**.

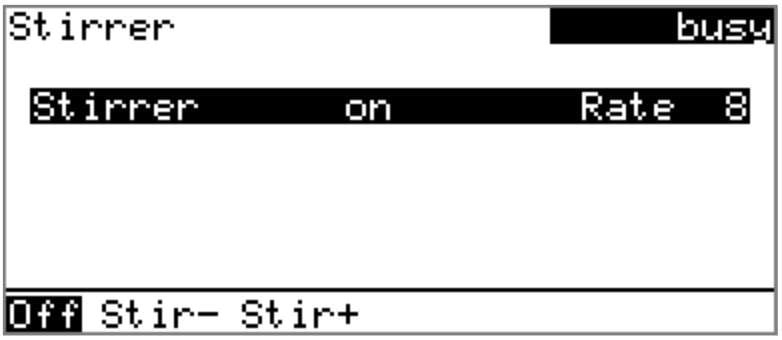
5 Starting the titration

▪ Press **[START]**.
The titration starts and the curve is displayed:



The axes are scaled automatically.

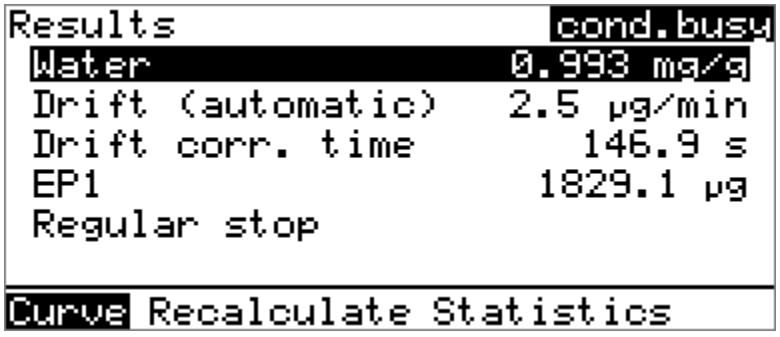
The stirring rate can be modified during titration with the **Stirrer** function. The following dialog is opened by pressing **[OK]**:



The stirring rate can be reduced with **Stir-** and increased with **Stir+**. **Off** switches the stirrer off. **On** is now displayed instead. This can be used to switch the stirrer back on. This dialog is exited with **[BACK]**.

6 Results

After the completion of the titration, the results dialog is displayed:



Conditioning is restarted automatically in the background. You can see the current status of the conditioning in the status display at the upper right in the dialog window (**cond.busy** or **cond.ok**).

7 Returning to the conditioning dialog

▪ Press **[BACK]**.
The main dialog with the sample data of the previously ended titration is displayed.

▪Select **Menu** and press **[OK]**.

▪Select the menu item **Live dialog** and press **[OK]**.

The current status of the conditioning is displayed (see instruction step *2*).
If you wish to start the next titration, repeat the actions starting with instruction step *3*.

Canceling a determination manually

A determination can be canceled at any time with the **[STOP]** key.