917 Coulometer



Tutorial 8.917.8001EN





Metrohm AG CH-9100 Herisau Switzerland Phone +41 71 353 85 85 Fax +41 71 353 89 01 info@metrohm.com www.metrohm.com

917 Coulometer

Tutorial

8.917.8001EN

01.2019

Technical Communication Metrohm AG CH-9100 Herisau techcom@metrohm.com

This documentation is protected by copyright. All rights reserved.

This documentation has been prepared with great care. However, errors can never be entirely ruled out. Please send comments regarding possible errors to the address above.

Table of contents

1	Introductio	n	1
	1.1	Structure of the tutorial	1
	1.2	Additional information	1
	1.3	Symbols and conventions	1
2	Installation		3
	2.1	Setting up and connecting the instrument	3
	2.2 2.2.1 2.2.2 2.2.3 2.2.4 2.2.5 2.2.6 2.2.7	Setting up the titration cell for coulometry Inserting the titration cell Preparing the titration cell Mounting the addition and aspiration tube Titration cell with aspiration equipment (utilization with Dos- ino) Using the titration cell with a Karl Fischer oven Using the titration cell with a sample changer Connecting sensors	7 7 11 . 12 . 13 14 15
	2.3	Setting up the aspiration and reagent bottle	. 20
	2.4	Connecting a pump	. 24
3	Switching t log languag	he instrument on and off and selecting the dia- ge	25
	3.1	Switching the instrument on and off	. 25
	3.2	Selecting the dialog language	. 27
4	Fundament	als of operation	28
	4.1	Touch-sensitive screen	. 28
	4.2	Display elements and controls	. 28
	4.3	Status display	. 30
	4.4	Entering text and numbers	. 31
5	Setup and	configuration	33
	5.1	Preparing instruments and material	. 33
	5.2	Configuring reagent data	. 33
	5.3	Configuring a printer	. 34
6	Carrying ou ple	It the water content determination of a sam-	36
	6.1	Creating a titration method	. 36

	6.2	Carrying out the water content determination	40
7	Performing	titration with supplementary functions	45
	7.1	Saving the determination and the PC/LIMS report	45
	7.2	Modifying the titration parameters	. 47
	7.3	Creating a sample table	. 48
	7.4	Carrying out titrations using the sample table	. 51
8	Setting up	the user administration	52
	8.1	Creating a user list	. 52
	8.2	Automatic login with USB flash drive	. 54
	8.3	Login with password	. 56
	Index		59

Table of figures

Figure 1	Connecting the power supply unit	. 3
Figure 2	USB/RS-232 adapter	. 4
Figure 3	Connecting the USB cable	. 6
Figure 4	Inserting the titration cell	. 7
Figure 5	Filling the adsorber tube	. 8
Figure 6	Equipping the titration cell	. 9
Figure 7	Mounting the addition and aspiration tube	11
Figure 8	Titration cell with aspiration apparatus	13
Figure 9	Remote connection 885 Compact Oven SC – Remote Box – 917 Cou	-
	ometer	15
Figure 10	Unscrewing the protective cap from the generator electrode	16
Figure 11	Screwing the electrode cable to the generator electrode	16
Figure 12	Connecting a generator electrode	17
Figure 13	Unscrewing the protective cap from the indicator electrode	17
Figure 14	Screwing the electrode cable to the indicator electrode	18
Figure 15	Connecting an indicator electrode	18
Figure 16	Connecting the PVC tubings	24

1 Introduction

1.1 Structure of the tutorial

This tutorial describes the installation and the first steps with the 917 Coulometer. You will be guided step-by-step through the most important dialog pages by using a water content determination with a water standard. In addition, you will find information regarding titrations with extended functions and for user administration.

1.2 Additional information

General information regarding coulometric Karl Fischer titration can be found in the monograph "Water Determination by Karl Fischer Titration" at www.metrohm.com (8.026.5011 DE; 8.026.5013 EN).

1.3 Symbols and conventions

(5- 12)	Cross-reference to figure legend				
	The first number refers to the figure number, the sec- ond to the instrument part in the figure.				
1	Instruction step				
_	Carry out these steps in the sequence shown.				
Method	Dialog text, parameter in the software				
File ► New	Menu or menu item				
[Next]	Button or key				
	WARNING				
	This symbol draws attention to a possible life-threat- ening hazard or risk of injury.				
	WARNING				
	This symbol draws attention to a possible hazard due to electrical current.				
	WARNING				
	This symbol draws attention to a possible hazard due to heat or hot instrument parts.				

The following symbols and formatting may appear in this documentation:

	WARNING				
	This symbol draws attention to a possible biological hazard.				
	CAUTION				
	This symbol draws attention to possible damage to instruments or instrument parts.				
•	NOTE				
	This symbol highlights additional information and tips.				

2 Installation

2.1 Setting up and connecting the instrument

Connecting the power supply unit

The 917 Coulometer has an external power supply unit for a 24 V power supply (DC). This is connected to the power socket of the Coulometer.



WARNING

An incorrect supply voltage can damage the instrument.

Operate the instrument only with the supply voltage specified for it. Use the supplied power supply unit exclusively.





Proceed as follows:

1 Connect the plug of the external power supply unit with the power socket of the Coulometer (*see figure 1, page 3*).



NOTICE

The plug of the power supply unit is protected against accidental disconnection of the cable by means of a pull-out protection feature. If you wish to pull out the plug, you first need to pull back the outer plug sleeve marked with arrows.

2 Connect the power cord with the external power supply unit of the Coulometer and with the power grid.



CAUTION

Switch off the Coulometer correctly by pressing the power switch before you disconnect the electricity supply. If this is not done, then there is a danger of data loss.

Connecting a balance

As a rule, balances are equipped with a serial RS-232 interface. To connect a balance, you require a USB/RS-232 adapter (6.2148.050).



Figure 2 USB/RS-232 adapter

The USB/RS-232 adapter can be connected to the 917 Coulometer directly or by means of a USB hub.

Connect the 9-pin plug of the respective balance connecting cable to the RS-232 connector. Consult the manual for the balance to select the correct connecting cable.

The parameters for the RS-232 interface must match those on the instrument and those on the balance. For this purpose, check the manual for the balance.

The following table offers an overview of the balances that you can use together with the 917 Coulometer and of which cable you will need for connection to the RS-232 interface:

Balance	Cables		
AND ER, FR, FX with RS-232 inter- face (OP-03)	6.2125.020 + 6.2125.010		
Mettler AB, AG, PR (LC-RS9)	In the scope of delivery for the balance		
Mettler AM, PM, PE with interface	6.2146.020 + 6.2125.010		
option 016	Also from Mettler: ME 47473		
or Mettler AJ, PJ with interface option 018	hand switch or ME 46278 foot switch		
Mettler AT	6.2146.020 + 6.2125.010		
	Also from Mettler: ME 42500 hand switch or ME 46278 foot switch		
Mettler AX, MX, UMX, PG, AB-S, PB-S, XP, XS	6.2134.120		
Mettler AE with interface option	6.2125.020 + 6.2125.010		
011 or 012	Also from Mettler: ME 42500 hand switch or ME 46278 foot switch		
Ohaus Voyager, Explorer, Analyti- cal Plus	Cable AS017-09 from Ohaus		
Precisa balances with RS-232-C interface	6.2125.080 + 6.2125.010		
Sartorius MP8, MC, LA, Genius, Cubis	6.2134.060		
Shimadzu BX, BW	6.2125.080 + 6.2125.010		

Connecting the barcode reader

The barcode reader is used as an aid for text and numerical input. You can connect a barcode reader with USB interface.



Figure 3 Connecting the USB cable

A printer can be connected to the USB connector (Type A) (with a 6.2151.020 connecting cable), as can a USB keyboard, a barcode reader or a USB flash drive (for storing and loading methods, etc.).

If several USB devices are to be connected, then it is recommended that a USB hub (available from specialty stores) be used. The USB hub then functions as a distributor to which several USB devices can be connected.

2.2 Setting up the titration cell for coulometry

2.2.1 Inserting the titration cell



Figure 4Inserting the titration cellSet up the titration cell on the magnetic stirrer as follows:

1 Attach the 6.2047.040 titration vessel holder to the magnetic stirrer.

2 Insert the 6.1464.320 titration cell into the titration vessel holder.

2.2.2 Preparing the titration cell

Filling the adsorber tube

Before setting up the titration cell, the 6.1403.030 adsorber tube must be filled with the 6.2811.000 molecular sieve. Proceed as follows:



Figure 5 Filling the adsorber tube

- **1** Insert a small cotton plug into the bottom of the adsorber tube. Do not pack the cotton too tightly.
- 2 Fill the adsorber tube to $\frac{3}{4}$ with molecular sieve granulate.
- **3** Place a small cotton plug on the molecular sieve. Do not pack the cotton too tightly.
- **4** Seal the adsorber tube with the appropriate cover.



NOTICE

Note that the molecular sieve must be replaced at regular intervals. Each time you refill the adsorber tube with the molecular sieve, you can, for example, write the date directly on the adsorber tube.



Figure 6 Equipping the titration cell

Equip the titration cell as follows:

- 1 Place the 6.1903.030 stirring bar in the titration cell.
- 2 Cut the 6.2713.0x0 ground-joint sleeves to the correct length and attach them to the ground joints of the inserts (electrodes, adsorber tube, etc.).

Please take care to ensure that the edges of the ground-joint sleeves are cut to size cleanly and that there are no fringes. The ground-joint sleeves must not protrude at the lower edge of the ground-joint opening.

3 Insert the 6.1403.030 adsorber tube into the generator electrode.

- **4** Insert the 6.0345.100 generator electrode without diaphragm or the 6.0344.100 generator electrode with diaphragm together with the adsorber tube into the large ground-joint opening at the rear.
- **5** Insert the 6.0341.100 indicator electrode into the left ground-joint opening.
- 6 Place the 6.1448.020 septum on the front opening of the titration cell and screw it shut with the 6.2701.040 screw cap.

Tighten the screw cap only enough so that sealing is ensured. The septum must not bend.

Filling the titration cell (generator electrode without diaphragm)

Proceed as follows when using a generator electrode without a diaphragm:

- **1** Fill approximately 100 mL of reagent into the titration cell with the aid of the 6.2738.000 funnel.
- **2** Close the remaining ground-joint opening on the right with the 6.1437.000 ground-joint stopper (with ground-joint sleeve attached).

Filling the titration cell (generator electrode with diaphragm)

Proceed as follows when using a generator electrode with a diaphragm:

- 1 Fill approximately 5 mL of catholyte into the generator electrode.
- **2** Fill approximately 100 mL of anolyte into the titration cell with the aid of the 6.2738.000 funnel. The level of the anolyte should be roughly 1 2 mm above the level of the catholyte.
- **3** Close the remaining ground-joint opening on the right with the 6.1437.000 ground-joint stopper (with ground-joint sleeve attached).

2.2.3 Mounting the addition and aspiration tube



Figure 7 Mounting the addition and aspiration tube

Insert the addition and aspiration tube as follows into the titration cell:

- **1** Attach the 6.2713.000 ground-joint sleeve that has been cut to size to the ground joint of the 6.1437.000 stopper.
- 2 Insert the stopper into the 6.1439.010 addition and aspiration tube.
- **3** Attach the 6.2713.000 ground-joint sleeve that has been cut to size to the ground joint of the addition and aspiration tube.
- **4** Insert this assembly into the ground-joint opening.
- **5** Connect the tubing for the reagent addition at the upper connector of the addition and aspiration tube (5).
- **6** Connect the tubing for the aspiration of the titration cell at the lower connector of the addition and aspiration tube (6).

Details regarding how to connect the addition tubing and the aspiration tubing can be found in the chapter "Setting up the aspiration und reagent bottle" (see chapter 2.3, page 20).

2.2.4 Titration cell with aspiration equipment (utilization with Dosino)

A Dosino allows the automatic replacement of reagents.

For aspiration, the 6.5617.000 aspiration equipment is used, along with a complete dosing unit and a 50 mL glass cylinder. For aspirating greasy samples when only the sample and not the whole reagent is to be aspirated, we recommend using a dosing unit with a 20 mL cylinder. For highly viscous samples, a dosing unit with a 10 mL cylinder is more suitable.



Insert the aspiration tip into the titration cell as follows:

1 Screw the 6.2730.030 nipple with stopper and O-ring onto the 6.1446.060 stopper.



3 Slide the 6.1543.070 aspiration tip through the stopper.



Figure 8 Titration cell with aspiration apparatus

4 Place the stopper with the attached aspiration tip into the ground-joint opening with the ground-joint sleeve.

Insert the aspiration tip into the titration cell until it almost touches the vessel base.



Collision with stirring bar

The aspiration tip must not collide with the stirring bar.

2.2.5 Using the titration cell with a Karl Fischer oven

When samples release their water only slowly or only at higher temperatures, the oven method is used. The sample is heated in a KF oven (e.g., *860 KF Thermoprep*) and the water that is released is transferred to the titration cell with a carrier gas.

2.2.5.1 Equipping the titration cell with an oven

The 6.1465.320 titration cell with additional lateral opening is used. The titration cell must be equipped beforehand (*see chapter 2.2.2, page 7*).



1 Gas inlet

2 Seal A.254.0102

- Remove the septum and replace with A.254.0102 seal.
- Insert the gas inlet from the side through the screw cap and the seal and tighten with the screw cap.

2.2.6 Using the titration cell with a sample changer

If a large number of samples have to be processed, the determination of the water content can be automated with the aid of a sample changer with oven module (e.g., *885 Compact Oven SC*).

2.2.6.1 Remote connection

The 885 Compact Oven Sample Changer can be used as a control unit for an automation system with a 917 Coulometer.

The 917 Coulometer must be connected with the 885 Compact Oven Sample Changer by Remote Box (6.2148.010) and by Remote 885 - RM Box (6.2141.340) cable.

The cable transfers start and stop signals of the 885 Compact Oven Sample Changer to the connected 917 Coulometer, and (in the opposite direction) status signals of the coulometer (Cond OK, EOD) to the 885 Compact Oven Sample Changer.

2.2.6.2 "885 Compact Oven SC – 917 Coulometer" sys-

tem

Standard combination for water content determinations with a 917 Coulometer.



Figure 9 Remote connection 885 Compact Oven SC – Remote Box – 917 Coulometer

The 917 Coulometer is operated in KFC mode. The sample series is started on the 885 Compact Oven Sample Changer.

2.2.7 Connecting sensors

The measuring interface contains the following measuring inputs:

- **Gen.** for a generator electrode
- Ind. for a double Pt electrode
- **Temp.** for a temperature sensor of the Pt1000 or NTC type



CAUTION

Under all circumstances, avoid mixing up the electrode cable from the indicator electrode with the one from the generator electrode. Mark the screw heads of the cables accordingly.

2.2.7.1 Connecting generator electrodes

Screwing the electrode cable to the generator electrode

1 Unscrew the protective cap from the generator electrode.



2 Tighten the 6.2104.120 electrode cable to the generator electrode.



Figure 11 Screwing the electrode cable to the generator electrode

Connecting the electrode cable to the coulometer

1 Plug the electrode plug into the **Gen.** socket of the coulometer.







NOTICE

The electrode cable is protected against accidental disconnection of the cable by means of a pull-out protection. If you wish to pull out the plug again, you first need to pull back the outer plug sleeve.

2.2.7.2 Connecting indicator electrodes

Screwing the electrode cable to the indicator electrode





Figure 13 Unscrewing the protective cap from the indicator electrode

2 Tighten the 6.2104.020 electrode cable to the indicator electrode.



Figure 14 Screwing the electrode cable to the indicator electrode

Connecting the electrode cable to the coulometer

1 Plug the electrode plug into the **Ind.** socket of the coulometer.







NOTICE

The electrode cable is protected against accidental disconnection of the cable by means of a pull-out protection. If you wish to pull out the plug again, you first need to pull back the outer plug sleeve.

2.2.7.3 Connecting a temperature sensor

A temperature sensor of the Pt1000 or NTC type can be connected to the **Temp.** connector.

Connect the temperature sensor as follows:

1 Insert the plugs of the temperature sensor into the **Temp.** sockets of the coulometer.



2.3 Setting up the aspiration and reagent bottle

Mode of operation of the built-in pump

With the built-in pump, one can on the one hand add fresh reagent easily at the press of a button, while on the other hand one can also empty the titration cell (coulometry) at the press of a button. This can be accomplished if all of the required tubing connections have been set up correctly and if all of the connectors have been screwed tightly enough to seal.



The following diagram gives an overview of the mode of operation.

- 1. Air will be pumped into the reagent bottle (on the left) for as long as the left-hand key **A** is kept pressed down.
- 2. The overpressure in the reagent bottle pushes fresh reagent into the titration cell.
- 3. Air will be suctioned out of the aspiration bottle (on the right) for as long as the right-hand button **B** is kept pressed down.
- 4. The vacuum in the aspiration bottle suctions the liquid out of the titration cell and into the aspiration bottle.



NOTICE

The catholyte must be replaced manually at regular intervals.

Please observe the manufacturer's specifications.

Setting up reagent bottles

The reagent is pumped out of the supply bottle by overpressure and into the titration cell. All of the bottle cap connectors must be leak-proof for this reason.

Metrohm offers suitable thread adapters for bottles with other threads than GL 45.



To equip the reagent bottle, proceed step-by-step as follows:

- **1** Place a 6.1446.040 threaded stopper in the M6 connector (smallest opening) of a 6.1602.105 bottle cap and screw it tightly.
- **2** Insert the long 6.1819.030 PTFE cannula into the M8 connector (second-smallest opening) of the bottle cap.



NOTICE

The length of the cannula must be adjusted to the size of the bottle beforehand.

3 Insert a 6.1805.200 PTFE tubing into the M8 connector of the bottle cap and screw it tightly.

4 Cut off a piece (approx. 1 m) of the 6.1801.120 PVC tubing and connect to the 6.1609.010 adsorber tube filled with molecular sieve.

Additional details can be found in the chapter "Filling the adsorber tube" (*see chapter 2.2.2, page 7*).

- **5** Place the adsorber tube with the SGJ 14 standard ground-joint on the bottle cap.
- **6** Secure the SGJ 14 standard ground-joint of the adsorber tube with the 6.2023.020 SGJ clip.
- **7** Place the completely equipped 6.1602.105 bottle cap on a reagent bottle from your chemicals manufacturer and screw it tightly.

Setting up the aspiration bottle

The aspiration bottle is used as a waste container and must be sealed against leaks.

Metrohm offers suitable thread adapters for bottles with other threads than GL 45.



To equip the aspiration bottle, proceed step-by-step as follows:

- **1** Place a 6.1446.040 threaded stopper in the M6 connector (smallest opening) of a 6.1602.105 bottle cap and screw it tightly.
- **2** Introduce the 6.1623.000 overflow protection into the M8 connector from below (second-smallest opening) of the bottle cap.
- **3** Place the bottle cap on the 6.1608.030 clear glass bottle (or on a different bottle with GL 45 thread) and screw it tightly.
- **4** Place the 6.1808.050 tubing olive in the M8 connector of the bottle cap and screw it tightly.
- **5** Plug the remaining piece of the 6.1801.120 PVC tubing onto the tubing olive.
- 6 Insert the 6.1446.090 stopper into the remaining opening on the bottle cap.

7 Introduce the short 6.1819.050 PTFE cannula into the opening of the stopper.

8 Insert a 6.1805.200 PTFE tubing with M8 connector into the opening of the stopper and screw it tightly.

2.4 Connecting a pump

Connecting tubings to the pump

The pump connectors of the 917 Coulometer are located on the rear of the instrument.



Figure 16 Connecting the PVC tubings

Take care to ensure that you connect the tubings to the pump correctly. Proceed as follows:

- **1** Fasten the 6.1801.120 PVC tubing from the aspiration bottle (clear glass) securely to the **Waste** connector (left-hand connector).
- **2** Fasten the 6.1801.120 PVC tubing from the reagent bottle (amber glass with adsorber tube) securely to the **Solvent** connector (right-hand connector).

3 Switching the instrument on and off and selecting the dialog language

3.1 Switching the instrument on and off

Switching on the instrument



CAUTION

Peripheral devices (e.g., printers) must be connected and switched on before you switch on the 917 Coulometer.



NOTICE

English is set as the default dialog language when the instrument is switched on for the first time.

Information about changing the dialog language can be found in the following section.

Proceed as follows:

 Press the power switch on the left-hand side of the back panel of the 917 Coulometer. The 917 Coulometer is initialized. A system test is performed. This process takes some time.



NOTICE

If a buret unit is connected, then a request appears to carry out the **Prepare** function:

	010-126 Prepare dosing device					
For at least one dosing device, the reques to prepare is switched on.						
	Execute the "Prepare" function. You find it under "Manual control/Dosing".					
	ок					

All tubings and the cylinder are rinsed with the **Prepare** function.

The preparing of the buret unit is described in the *Preparing buret unit* section later in the document.

• Confirm the message with **[OK]**.

The main dialog is displayed:

New method		17:34:58	
User			
Identification 1			
Identification 2			
Sample size	1.0	g	
	_		
System Load method	Control	Edit parameters	Results

Switching off the instrument



CAUTION

) —

The 917 Coulometer must be switched off by pressing the power switch on the rear of the instrument before the electricity supply is interrupted. If this is not done, then there is a danger of data loss. Proceed as follows:

1 Press the power switch on the left-hand side of the back panel of the 917 Coulometer.

The current data is saved and the system is shut down. This process takes just a short time. At the same time, all other instruments connected to the 917 Coulometer via a USB cable are also being switched off.

3.2 Selecting the dialog language

The user interface is available in several languages. English is set as the default dialog language at the time of shipment.

Proceed as follows to select the dialog language:

1 Opening the system settings

- In the main dialog, tap on [System].
- Tap on [System settings].

The System / System settings dialog is displayed.

2 Selecting the dialog language

Tap on the **Dialog language** list box and select the desired language.

3 Saving the settings

■ Tap on the fixed keys [<>>] or [🔐].

The main dialog is displayed in the respective dialog language.

4 Fundamentals of operation

4.1 **Touch-sensitive screen**

The entire 917 Coulometer user interface is touch-sensitive. Simply touch a few of the buttons on the interface to learn how a touch-sensitive screen reacts. You can always return to the main dialog by touching [$\widehat{}$].

In order to activate an element on the 917 Coulometer user interface, just touch the screen with your fingertip, finger nail, the eraser of a pencil or a stylus (special tool for operating instruments with touch-sensitive screens).



CAUTION

Never touch the touch screen with a pointed or sharp object such as a ballpoint pen.

In the default setting, the software is configured in such a way that an acoustic signal will be generated every time an active control is touched. This setting can be deactivated in the system settings.

4.2 Display elements and controls

New method		17:34:58		
Use	r			
Identification				
Identification	2			
Sample siz	1.0	g	\blacksquare	
System Load	Control	Edit	Results	1
	1	Parameters		5
	رالي. ال	۲ ^h m		\triangleright

The following display elements and controls are available:

[Home] always opens the main dialog.
[Back] saves the entry and opens the next-higher dialog page.
[Help] opens the online help for the dialog displayed.
[Print] opens the printing dialog.
[Manual] opens the manual control.
[Stop] cancels the running determination.
[Start] starts a determination.

Table 1Fixed keys which are always available

The file name of the loaded method, the time and the system status are displayed in the main dialog in the **Title bar**.

In the other dialogs, the title bar shows the headings of the next upper level and of the displayed dialog. This is an aid for orientation during navigation through the user dialog.

Table 2Screen elements

System	Buttons open a new dialog when they are tap- ped.			
Sensors				
Delete	Inactive buttons with gray lettering indicate that the respective function is not available at the moment.			
1.0	Input fields open an input dialog when tapped.			
\mathbf{V}	Tapping on the selection symbol opens a selection list.			
	A check box can also be activated or deactiva- ted by tapping on it.			

4.3 Status display

The current status of the system is displayed in the upper right-hand corner of the title bar.



4.4 Entering text and numbers

In the editing dialog for text or numerical input, enter the individual characters by tapping in the input field. The following functions are available:

Text editor

٢	New method 17:35:32						35:32		
				User					
	А	В	С	D	E	F	G		×
	Н	Ι	J	К	L	М	Ν	Delete entry	
	0	Р	Q	R	S	Т	U	A	
	V	W	Х	Y	Z	ш		V	
ĺ	Cancel a - z				0 - 9	e ch	Special aracters		ок

Table 4Editing functions

[OK]	The modification is applied and the editing dialog is exited.
[Cancel]	The editing dialog is exited without applying the modification.
[Delete entry]	The content of the input field is deleted com- pletely.
[図]	The character in front of the cursor is deleted.
[⇔]	The cursor within the input field is shifted to the left by one character.
[⇔]	The cursor within the input field is shifted to the right by one character.
[az]	The lower-case letters are displayed. The label changes to [AZ] . The upper-case letters are displayed again by tapping.
[09]	Numbers and mathematical characters are displayed.
[Special charac- ters]	Special characters are displayed. You can use the button [More] to navigate through all available characters.

Number editor

Edit command / Stop conditions							
	Stop time			off	s		
Input: 1 999999		7	8	9	off	Ŧ	
Default value: off		4	5	6	R1		
		1	2	3			
		0	+/-				
Cancel	Delete entry				Oł	<	

Table 5 Editing functions

[OK]	The modification is applied and the editing dialog is exited.
[Cancel]	The editing dialog is exited without applying the modification.
[Delete entry]	The content of the input field is deleted com- pletely.
[off]	If not only numbers but also special values (e.g., off) can be entered, then the corresponding but- tons will be shown to the right of the numerical keypad.
[R1]	For many parameters, a result previously defined in the method can also be entered in place of a number. You can select the result variable by touching [R1] .



NOTICE

A commercially available USB keyboard can be connected to make text and numerical input easier.

5 Setup and configuration

5.1 **Preparing instruments and material**

What you will need:

- A 917 Coulometer, including accompanying accessories
- An analytical balance
- A water standard with a water content, e.g., of 1 mg/g
- A USB printer with connecting cable, in case you wish to print out reports.

5.2 Configuring reagent data

The coulometric reagents used can be administered and monitored. The following conditions for the monitoring of a reagent can be defined:

- Number of determinations
- Working life
- Reagent capacity
- Drift

If a monitored parameter reaches the set limit, then the reagent replacement can be carried out manually or automatically.

The reagents are configured under **System ► Reagents**.

Proceed as follows to configure the reagents:

Configuring reagents

1 Open the list of reagents.

- In the main dialog, select the **[System]** button.
- Tap on [Reagents].
- Select **[New]** and enter a name for the reagent.
- Tap on [Reagent mon.] and set the limit of the monitored parameter.
- Select the [Reagent replacement] button and specify [manual] or [auto].

i

NOTICE

A suitable method (aspirating used reagent, adding fresh reagent) must be available for automatic reagent replacement. This will be started automatically when the set limit has been reached.



NOTICE

Note that the catholyte solution must be replaced manually when a generator electrode with diaphragm is used for automatic reagent replacement.

2 Use the **[Home]** button to return to the main dialog.



NOTICE

In the method used for the water content determination for the sample, the reagent monitoring of the reagent used must be activated under **[Cell]**.

5.3 Configuring a printer

If you wish to print out results and titration curves, then you must configure the printer in the device manager.

Proceed as follows:

- **1** Opening the printer dialog
 - In the main dialog, tap on **[System]**.
 - Tap on [Device manager].

2 Entering the printer data

• Select **Printer** and tap on **[Edit]**.

Device manager / Edit		
Device type: Printer		
Device name	Printer	
Comment		
Printer	off	
Connector	USB	
	PDF Networ settings printer	More options

- Tap on the selection symbol next to the input field **Printer** and select a printer type.
- Once you have connected a USB printer, tap on the selection symbol next to the input field **Connector** and select **USB**.
- If you would like to use a printer in your company network, tap on the selection symbol next to the input field **Connector** and select **Ethernet**.

3 Return to the main dialog with the $[\widehat{\omega}]$ fixed key.



NOTICE

You can also compile reports as PDF files and store them on a USB flash drive or on a shared data directory in your company network.

6 Carrying out the water content determination of a sample

6.1 Creating a titration method

Below you will find a description of how to create your own method for water content determination. The 917 Coulometer contains method templates which are already configured except for a few parameters.

The water content of a sample should be determined by means of a triple determination. If you have a printer connected, then a report with the result and the curve should be printed out automatically at the end of each determination.

Proceed as follows:

Loading a method template

- 1 In the main dialog, tap on **[Load method]**.
 - Tap on [New method].
 The method table with the stored templates is opened.
 - Select the **Coulometric KF titration** template.
 - Tap on [Load template].
 - Confirm the following message with **[Yes]**.

Lo	Load method / New method						
			Metrohm met	hod tem	plates		
	Coulometric KF titration						
			020-121 Met	hod mo	dified		
	The modifications of the current method have not been saved.					ave	
		Do yo	ou want to load	the met	hod anyway?		
	Yes No						
					Empty method	Load template	

Adjusting method parameters

1 In the main dialog, tap on [Edit parameters].

The command sequence of the loaded method is displayed.

Par	Parameters / Sequence							
Current method: New method								
01	WAIT	Wait						
02	REQUEST	Data request						
03	KFC	Karl Fischer coulometry						
04	CALC	Calculation						
05	REPORT	Report						
06								
	Save Method Insert Delete Edit method options command command							

2 Selecting a Karl Fischer command

Sequence / Edit command	i 😑
01 KFC	Karl Fischer coulometry
Control parameter	rs Sensor
Titration paramete	rs Stirrer
Stop conditions	
Conditioning	
Cell	

- Highlight the line **KFC**.
- Tap on [Edit command].

A selection of parameters (e.g. start conditions) and instruments (e.g. dosing devices, see next step) appears, which can be selected and adjusted individually.

3 Adjusting the calculation

- Select CALC.
- Tap on [Edit command].
- Tap on [New].
 - A selection of templates appears.
- Select e.g., the template **KFC content (ppm)**.
- Tap on [Load template].
- Confirm the subsequent note with **[Next]**.
- Under [Result options], ensure that the selection SMN1 is set for the parameter Variable for mean value.

This ensures that the mean value of the results is calculated.

i

NOTICE

The calculation is carried out continuously, i.e. the calculation is updated after every determination that is carried out until the defined number of determinations has been reached.

Under [More options], define parameters, e.g., Save result in result table.

See the detailed manual for precise information.

 Return with the [] fixed key to the command sequence of the method run.

4 Adjusting the report options

In the default settings, **Result report** and **Curve** are defined in the report options.

Adjust these parameters as needed and supplement them with additional reports.

See the detailed manual for precise information.



NOTICE

If you have not connected a printer, delete the command **REPORT**. If this is not done, then an error message will appear at the start of the determination.

5 Adjusting additional method parameters

If necessary, adjust parameters in the **KFC** command such as the pause or the extraction time.

See the detailed manual for precise information.

6 Activating the statistics

• In the main dialog, tap on [Edit parameters].

• Tap on [Method options].

Sequence / M	equence / Method options						
Current method: New method							
	🖌 Statistic	s					
	Number of	determinatio	ns 5				
Save automat.	Properties	Note	Start/Stop options	Sample data			

Here you can define the statistics function for this method. You can specify the number of determinations for which the respective statistical calculations are to be carried out.

These settings apply for all determinations which are carried out with this method.

- Tap on the check box **Statistics**.
- Enter a value under Number of determinations.



NOTICE

Under **[Save automat.]**, you can also save the determination as a determination file (MDTM file) and as a PC/LIMS report (TXT or UTF8 file) *(see chapter 7.1, page 45)*.

Tap on the fixed key [<>].

7	Sa	ving a me	thod			
	•	Tap on [Sa	ive me	thod].		
		Sequence / S	ave metho	d		
			Memory	Internal memory		
			Group	Main group		
		F	ile name	New method		
		Cancel			Sa	ve

You can save the method in the instrument's internal memory, on a USB storage medium or in a shared data directory in a network. In addition, you can also create different groups and/or folders for the methods.

- Tap on the input field **File name**.
- Enter a name for the method.
- Confirm the entry with **[OK]**.
- Tap on [Save].
 The method is stored in the folder selected in the [Group] input field. Main group is selected by default.

6.2 Carrying out the water content determination

Conditioning the titration cell

1 Starting conditioning

Tap on the start key [\triangleright].

The following dialog continues to be displayed until the status **Con-ditioning OK** is achieved, i.e. until the titration cell has been titrated dry.

KFC		►
03 KFC	Karl Fischer coulometry	
	Conditio	ning
	Drift	1094 µg/min
Hold	Cancel Sample command data	Edit parameters Results

KFC			V
03 KFC	Karl-Fischer-Coulome	strie	
	Cond	itioning OK	
	Drift	1.9 µg/	/min
Hold	Cancel	ample Edit	Results

The water seeping in is titrated off continuously (basic drift).

Entering sample data in the main dialog

1 Entering sample identification and sample size

- Tap on the input field **Identification 1**.
- Enter a designation for the sample (e.g., type of sample or an analysis number).
- Tap on the input field **Identification 2**.
- Enter a further designation for the sample (e.g., batch number or sampling date).

Adding sample

1 Prepare the sample for adding into the titration cell.

- **2** Tare the balance with the prepared sample and then remove the sample from the balance once again.
- **3** Tap on the start key [\triangleright].

Conditioning is stopped. The request for adding the sample will be displayed for 8 s.

KFC				- >			
01 WAIT	Wait						
	A	dd samp	le				
8 s							
Hold	Cancel command	Sample data	Edit parameters	Results			

4 Introduce the sample into the titration cell during the specified time.

Starting the titration

- **1** Reweigh the sample in order to determine the sample size.
- 2 Enter the sample size and confirm with [Continue].

KFC		
02 REQUEST	Data request	
	Sample size 1.06210 g	
	Continue	

i

NOTICE

The sample size can also be adopted directly from a connected balance.

 The titration curve is shown live. After the completion of the titration, the results dialog is displayed. If a printer is connected, then the report defined in the method will be printed out.

Conditioning is restarted automatically in the background.

3 Carry out the two remaining water content determinations in the same way.

If the statistics function is activated, you can have the statistics data displayed after the titration. Proceed as follows:

1 Opening the statistics page

- In the main dialog, tap on [Results].
- Tap on [Statistics].

The mean value of the result is displayed in the statistical overview.

2 Displaying statistics data

Tap on **[Details]**.

Stati	stics / Details			
Re Me sa	esult name: KFC content (p ean value abs rel	pm) 1000 2.5 0.25	ppm ppm %	SMN1 n=03
No.	Sample size		Result	
1	0.9496 g		1003 ppm	I
2	1.7160 g		998 ppm	
3	1.5813 g		1000 ppm	I
Sa	ample data		Determ. on/off	Result on/off

In the upper part of the display you will see the mean value of the result as well as the absolute and the relative standard deviation. The single results of the titrations are listed in a table.

If you would like to exclude a titration from the statistical calculation, you can select it and then tap on **[Result on/off]** or **[Determ. on/off]**. The recalculation of the statistics data will be performed immediately.

If you would like to add other titrations to the statistics, you can do this in the statistical overview.

7 Performing titration with supplementary functions

7.1 Saving the determination and the PC/LIMS report

You can have the determination data (measuring point list, results etc.) of a titration which has been carried out saved automatically. This makes it possible to reprocess or print out this data later. You can save the determinations to a USB storage medium or to a drive in a computer network.

If you would like to manage the determination data in a database on a PC, then you can save it in a report called PC/LIMS report or transfer it directly to a PC through a serial connection.

PC/LIMS reports can be saved either as TXT file (in accordance with ISO 8859-1) or UTF8 file.

The **tiBase** database software from Metrohm is available to you for administering the determination data on the PC.

Defining the automatic saving

Proceed as follows:

1 Opening the method options

- In the main dialog, tap on [Edit parameters].
- Tap on [Method options].
- Tap on [Save automat.].

Method options / Save automatically				
The file name is extend with date and time.	led automatically			
Save determinatio	n automatically			
Memory	External memory 1			
Group				
File name	Identification 1			
Write protection				
Create PC/LIMS n	eport			

2 Activating Save determination and defining the memory location

- Activate the Save determination automatically check box.
- Select the memory location under Memory. You can choose between External memory 1 (e.g., a USB flash drive) or Shared memory (memory location in a computer network).
 A USB flash drive (external memory 1) must be plugged in on the 917 Coulometer before a determination is carried out. This memory location must already be defined in the device manager at the time of the selection of a shared memory location in a computer network. Before a determination is carried out, the 917 Coulometer must be connected to the network with an Ethernet cable.
- Tap on the selection symbol next to the input field Group and select an existing group or create a new group. The determination data can be stored on the storage medium in various groups (= folders on the storage medium).
- Define the designation for the file name.
 Two sample identifications or the method name are available for selection. If you tap on the input field File name, then you can enter a designation of your own for the file name.
- Activate or deactivate write protection. You can protect the original determination data against overwriting.

3 Activating the PC/LIMS report and defining the memory location

- Activate the Create PC/LIMS report check box. Define the memory location for the PC/LIMS report in the device manager.
- Return to the main dialog with the [$\widehat{\mathbf{m}}$] fixed key.



NOTICE

These settings are method-specific. The determination data is saved to the defined memory location for all determinations which are carried out with this method. You can define different memory locations for your methods.



NOTICE

The settings for the PC/LIMS report (memory, RS-232, coding) must be undertaken in the "Edit instrument/PC/LIMS report" dialog.

7.2 Modifying the titration parameters

You can optimize a titration run by modifying individual titration parameters according to the needs of your analysis. For the following titration, the stirring rate, the extraction time and the start drift are being modified.

Setting the stirring rate

1 Opening the stirrer settings

- In the main dialog, tap on [Edit parameters].
- Select the KFC command and tap on [Edit command].
- Tap on [Stirrer].

2 Modifying the stirring rate

- Tap on [-] or [+] to modify the stirring rate.
 You can use the check box Switch off automatically to set whether or not the stirrer is switched off at the end of the titration.
- Use the [] fixed key to return to the **KFC** command.



NOTICE

Thorough mixing is important with coulometric titration.

We recommend a Stage 8 stirring rate or Stage 15 when used in combination with an oven.

Modifying the settings for the titration

1 Setting an extraction time

NOTICE

The input of an extraction time is recommended, e.g., with samples that release water slowly or if a Karl Fischer oven (e.g., *885 Compact Oven SC*) is used. The extraction time corresponds to the minimum duration of the titration.

- Tap on [Titration parameters].
- Under Extraction time, enter the desired time.
 Use the [] fixed key to return to the KFC command.

2 Modifying the start drift

- Tap on [Conditioning].
- Under **Start drift**, enter the desired value.
- Define additional stop parameters under [Cond. options].
 Conditioning will be canceled if one of the stop parameters set here is achieved before the start drift is reached.

7.3 Creating a sample table

If you would like to prepare a sample series in advance, you can use the sample table. Proceed as follows:

Filling the sample table with sample data

1 Activating the sample table

• In the main dialog, tap on [Control].

Control				•
	Statistics			
	Sample t	able		
	Autostart			
	Number	of autostarts	Table	
	Sa	mple number	0	
	Favorites	Delete table	Reset autostart	Delete statistics

- Activate the **Sample table** check box.
- Return to the main dialog with the [] fixed key.
 A new button is now visible in the main dialog.

New method		17:41:35	•
User		D. Müller	
Sample table	Line 0 of 0		
	S	ample table	
System Load	Control	Edit	Results

2 Entering the sample data

• Tap on [Sample table].

		•			
Sam	ple table				
No.		Identificatio	n 1	Sam	ple size
1					
L	_oad/ Save	Properties	Insert line	Delete	Edit

The sample table is still empty. The first line is highlighted.

Tap on [Edit].

Sample table / Edit	
Line number	- 1 +
Method	\checkmark
	New sample
Identification 1	
Identification 2	
Sample size	1.0 g 🖤

- Tap on the selection symbol next to the input field **Method**.
- Select a saved method.
 A particular method can be selected for each sample which is to be processed. If no method is specified, then the currently loaded method is executed.

 Fill out the fields for sample identification and the sample size. The line number is automatically increased by one after the sample size has been entered. The next sample size can be entered directly.

The criterion, after which the line number should be increased by one, can be modified in the properties dialog of the sample table under **[Editing options]**.

Properties / Editing options		0
End line with	Sample size	
Copy automatically into next line:	Identification 1	
Method	Identification 2	
Identification 1	manual ▼ Sample size	
)		

- Use the [<>] fixed key to return to the sample table.

7.4 Carrying out titrations using the sample table

Now carry out the titrations with the samples for which you have entered sample data.

Each time that you press the [\triangleright] fixed key, the sample data will be loaded from the top line of the sample table and applied for the current titration. This line will be deleted at the end of the titration. The sample data of the next line down will be used for the next titration.

A result report will be printed out and a PC/LIMS report will be saved after each titration if you have defined or activated these options.



NOTICE

The sample table can also be used with automated determinations.

8 Setting up the user administration

If different persons are working on the instrument, we recommend that the user administration of the 917 Coulometer be used. This means that everyone can use their user identification to log on to the instrument. The reports will then automatically contain the respective user name.

In addition, a dialog level can be set up for each user. In addition to the expert dialog, with access to all functions and settings, a routine dialog can be selected with limited access rights. The available functions and dialog areas can be configured for the routine dialog.

Administrator rights can be assigned to users who are permitted to administer methods and to make configuration settings.

If you create a user list, then you can use it in different ways. You can apply different combinations of login options. Three possibilities are listed in the following:

- User name can be selected in the main dialog without login
- Automatic login with USB flash drive
- Login with password

8.1 Creating a user list

The creation of a user list is required for all of the possibilities of the user login.

Defining users

Proceed as follows:

1 Opening the user administration

- In the main dialog, tap on [System].
- Tap on [System settings].
- Tap on [User admin.].

System settings / User administration			
User	Dialog	Status	
Login options ID profile	New Delete	Edit	

2 Creating a new user

Tap on [New].

User administration / Edit u	user		
User			
Full name			
Dialog	Expe	ert dialog	
Status	a	active	
Admin. rights			
Cancel	Favorites	Signature method	Signature determ.

- Tap on the input field **User** and enter an unambiguous user identification (e.g., abbreviation). Close the input dialog with **[OK]**.
- Tap on the input field **Full name** and enter the name of the user. Close the input dialog with **[OK]**.
- Tap on the selection list **Dialog** and select either **Expert dialog** or **Routine dialog**. Remember that the system settings cannot be changed except in the expert dialog. The setting is effective only when working with login.
- Activate or deactivate the administrator rights. The user administration can be used only with administrator rights. At least one person must be in possession of administrator rights.
- Return to the user administration with [<>].
- Define additional users.
- Return to the main dialog with [$\widehat{\mathbf{m}}$].

If you would like to work without a login, it is sufficient to create a user list. Each user can select his or her entry from the user list in the main dialog. Then the user name will be printed out with reports and/or saved along with a determination.

8.2 Automatic login with USB flash drive

It is possible to have automatic user recognition without requiring that a password be entered. If each user uses a USB flash drive of his or her own with his or her user profile, then the instrument can recognize whose USB flash drive it is at the time of the switching on. User login then takes place automatically.

The USB flash drive can be used for saving determination data, methods, PC/LIMS reports or for backing up the entire system.

Creating user profiles

You now need to create an ID profile for each user on a separate USB flash drive. The USB flash drive must be formatted.

Proceed as follows:

1 Plugging in the USB flash drive

- Return to the main dialog with [$\widehat{\mathbf{m}}$].
- Plug in a USB flash drive into the rear of the 917 Coulometer.
- Wait until a message appears which confirms that the USB flash drive has been recognized.

2 Saving the user profile

- Switch over to the user administration with [System], [System settings] and [User admin.].
- Select a user name.
- Tap on [Create ID profile].

A message will appear which confirms the creation of the ID profile.

If you now set the login options, you will automatically be logged in at once with this ID profile.

Setting the login options



NOTICE

The following steps are possible only if the user has administrator rights.

Proceed as follows:

1 Opening the login options

• In the user administration, tap on [Login options].

User adm	inistration / Login options	
	Login via user name Login via identification profile Password required Logout automatically after 10 min Login only for the same user	
Audit Trail	Reasons Modific. Passwo	ord 1s

2 Activating login with ID profile

- Activate Login via identification profile.
- Deactivate all other settings.
- Return to the user administration with [<>].

You can confirm the request following the creation of ID profiles with **[Yes]** if you have created an ID profile beforehand. Make sure that the USB flash drive is plugged in.



NOTICE

If you have not yet created any ID profiles, then you must confirm the request with **[No]**. Then you can deactivate the **Login via identification profile** and create an ID profile in the user administration.

Confirm any messages which may appear with [Yes].

An automatic login will now take place.

3 Logging in

- Tap on **[OK]** after the request that you plug in the USB flash drive with your ID profile.
- In order to log in another user with ID profile, tap in the main dialog on [Control/ Logout] and afterwards on [Logout].
 The request appears once again to plug in a USB flash drive.

8.3 Login with password

If you wish to make it imperative that each user log in on the instrument with a password, then you can activate this in the login options.



NOTICE

Please note that you will no longer be able to delete the entries created in the user administration once you have activated the password request. The only option available afterwards is to deactivate users.

Setting the login options

Configure the following settings:

1 Opening the login options

- Use System ➤ System settings ➤ User admin. to switch over to user administration.
- Tap on [Login options].



2 Adjusting the settings

• Activate Login via user name.

• Activate **Password required**.

Many settings are possible in the login options. Consult the manual of the 917 Coulometer to look these up if necessary.



NOTICE

The login dialog is called up as soon as you exit the login options dialog with [\triangleleft] or [\bigtriangleup]. You must then log in for the first time. You must define and enter a password to accomplish this.

If the login dialog is not called up, switch the instrument off and then back on again.

First login

You must define a password at the time of the first login on the instrument. Proceed as follows:

Login	•
User	user1
Password	
	Change password OK

1 Entering the user name

- Tap on the input field User, enter the user name and confirm with [OK].
- Tap on [Change password].

Change password		
User	user1	
New password	******	
Confirm password		
Cancel		ок

2 Defining a password

- Tap on the input field **New password**.
- Enter a password. It may not be more than 10 characters long.



NOTICE

Note the password.

- Confirm the entry with **[OK]**.
- Enter the password once again under **Confirm password**.
- Tap on **[OK]**.

3 Logging in

• Enter the new password under **Password** and tap on **[OK]** in the login dialog.

Index

Α

A	
Addition and aspiration tube	
Mount	11
Adsorber tube	
Fill	. 7
Aspiration bottle	
Set up	22
Aspiration equipment	12

В

Balance	
Connect	4
D	
Dialog language	27
E	
Electrode	
Connect	15
-	

G

-	
ienerator electrode	15
Fill	10

I

Indicator electrode 17

Instrument	
Switch off	26
Switch on	25

Κ

Karl Fischer oven	
Use	13

М

	_
Molecular sieve	
Replace	8

Ν

Numerical inpu	t 3	1

0

-	
Operation	
General	28
Oven module	14

Ρ

Power supply unit	
Connect	3

R

Reagent bottle	
Set up	21

S

Sample changer	
With oven module	14
Sensor	
Connect	15
Switch off	26
Switch on	25

т

Temperature sensor	19
Connect	15
Text input	31
Titration cell	
Equip	9
Fill	10
Insert	7
Touch screen	28
U	

0	
USB devices	
Connect	6