OPERATING MANUAL



TitroLine® 5000

TITRATOR



a xylem brand

Gebrauchsanleitung Se	te	3.	78	3
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Wichtige Hinweise:

Die Gebrauchsanleitung ist Bestandteil des Produktes. Vor der ersten Inbetriebnahme bitte sorgfältig lesen, beachten und anschließend aufbewahren. Aus Sicherheitsgründen darf das Produkt ausschließlich für die beschriebenen Zwecke eingesetzt werden. Bitte beachten Sie auch die Gebrauchsanleitungen für eventuell anzuschließende Geräte.

Alle in dieser Gebrauchsanleitung enthaltenen Angaben sind zum Zeitpunkt der Drucklegung gültige Daten. Es können jedoch vom Hersteller sowohl aus technischen und kaufmännischen Gründen, als auch aus der Notwendigkeit heraus, gesetzliche Bestimmungen der verschiedenen Länder zu berücksichtigen, Ergänzungen am Produkt vorgenommen werden, ohne dass die beschriebenen Eigenschaften beeinflusst werden.

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Important notes:

The operating manual is part of the product. Before initial operation, please carefully read and observe the operating manual and keep it. For safety reasons the product may only be used for the purposes described in these present operating manual. Please also consider the operating manuals for the devices to be connected

All specifications in this operating manual are guidance values which are valid at the time of printing. However, for technical or commercial reasons or in the necessity to comply with the statuary stipulations of various countries, the manufacturer may perform additions to the product without changing the described properties.

Mode d'emploi Page 155 ... 230

Instructions importantes:

Le mode d'emploi fait partie du produit. Prière de lire et d'observer attentivement le mode d'emploi avant la première mise en marche de l'appareil, et de le conserver. Pour des raisons de sécurité, l'appareil ne pourra être utilisé que pour les usages décrits dans ce présent mode d'emploi. Nous vous prions de respecter également les modes d'emploi pour les appareils à connecter.

Toutes les indications comprises dans ce mode d'emploi sont données à titre indicatif au moment de l'impression. Pour des raisons techniques et/ou commerciales ainsi qu'en raison des dispositions légales existantes dans les différents pays, le fabricant se réserve le droit d'effectuer des suppléments concernant l'appareil pour séries de dilution qui n'influencent pas les caractéristiques décrits.

Instrucciones importantes:

El manual de instrucciones forma parte del aparato. Antes de la operación inicial de aparato, lea atentamente y observe las instrucciones de operaciones y guárdelas. Por razones de seguridad, el aparato sólo debe ser empleado para los objetivos descritos en este manual de instrucciones. Por favor, observe las instrucciones de operación para los aparatos a conectar.

Todas las especificaciones en este manual de instrucciones son datos orientativos que son válidos en el momento de la impresión. No obstante, por motivos técnicos o comerciales, o por la necesidad de respetar las normas legales existentes en los diferentes países, el fabricante puede efectuar modificaciones del aparato sin cambiar las características descritas.

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Notes to the Manual

The provided manual will allow you the proper and safe handling of the product. For maximum security, observe the safety and warning instructions in the Instructions.

The pictogram $\mathbf{\Lambda}$ has the following meaning:

- Warning of a general danger to personnel and equipment.
- Non-compliance results (can result) in injury or material damage.

1 Important information for device use.

Refers to another part of the operating manual.

Status at time of printing

Advanced technology and the high quality of our products are guaranteed by a continuous development. This may result in differences between this operating manual and your product. We cannot exclude mistakes. We are sure you understand that no legal claims can be derived from the information, illustrations and descriptions.

A potentially more recent version of this manual is available on our internet website at www.si-analytics.com. The German version is the original version and binding in all specifications.

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1 Technical Specifications of the Titrator TitroLine® 5000

1.1 Summary

The TitroLine® 5000 is a potentiometric titrator and suitable for the following applications:

The possible range of titrations includes pH, and mV with a maximum of 5 memorisable methods.

- The examples of possible use of the TitroLine[®] 5000 include:
- Acid and base determination in aqueous solutions such as p and m value, titration of strong and weak acids and bases
- Redox titrations such as iodometry, manganometry, chromatometry, and COD determinations
- Other mV titrations, e.g. chloride
- Titrations using ion-selective electrodes, e.g. coppe-ISE
- Indices such as iodine and peroxide value.

These methods are mere examples; further applications can be found in food technology, environment, quality control, and process monitoring.

In addition, the TitroLine® 5000 comes with the functionalities of the TITRONIC[®] 300 piston burette:

- Manual titrations with or without calculation of the result
- Dosing

Each method allows for the setting of a variety of dosing and filling rates.

Solutions to be used:

Virtually, any liquids and solutions with a viscosity of $< = 10 \text{ mm}^2 / \text{s}$ such as concentrated sulphuric acid may be used. However, one has to avoid the use of chemicals that may attack glass, PTFE or FEP or that are explosive, such as hydrofluoric acid, sodium azide or bromine! Suspensions containing high solids percentages may clog or even damage the dosing system.

General provisions:

The safety guidelines that are applicable to the handling of chemicals have to be observed under all circumstances. This applies in particular to inflammable and/or etching liquids.

Guarantee

We provide guarantee for the device described for two years from the date of purchase. This guarantee covers manufacturing faults being discovered within the mentioned period of two years. Claim under guarantee covers only the restoration of functionality, not any further claim for damages or financial loss. Improper handling/use or illegitimate opening of the device results in loss of the guarantee rights. The guarantee does not cover wear parts, as lobes, cylinders, valves and pipes including the thread connections and the titration tips. The breach of glass parts is also excluded. To ascertain the guarantee liability, please return the instrument and proof of purchase together with the date of purchase freight paid or prepaid.

1.2 Specifications Titrator TitroLine[®] 5000

(Release: 30. August 2018)

CE Zeichen: C E FC	As per the low voltage directive 2014/35/EU; test basis EN 61 010-1: 2011-07 for laboratory devices As per the EMC directive 2014/30/EU; test basis EN 61 326 Part 1: 2012 As per the EMC directive 2011/65/EU; test basis EN 50 581: 2013-02 FCC part 15B and ICES 003
Country of origin:	Germany, Made in Germany

The following solvents/titration reagents are allowed to be used:

- All common titration solutions.
- As reagent water and all non-aggressive non-organic and organic fluids are allowed. If using combustible fluids fire please adhere to the Guidelines for Explosion Protection and Prevention of the chemical industry.
- For fluids with higher viscosity (≥ 5 mm²/s), lower boiling point or affinity to outgas, the filling and dosage speed can be adjusted.
- Fluids with viscosity over 10mm²/s cannot be dosed.

Measuring input:

pH/mV-input with 12 bit transducer for high-precision readings.

Electrode socket according to DIN 19 262 or additional with BNC socket insert (Z 860) Reference electrode 1 x 4 mm socket

		Measurement range	Display resolution	Measurement accuracy* without	
				sensor probe	$[\Omega]$
рН	рН	- 3.0 17.00	0.01	0,05 ± 1 Digit	> 5 · 10 ¹²
mV	U [mV]	- 1900 1900	1	1,0 ± 1 Digit	> 5 · 10 ¹²

To ensure maximum accuracy of the readings we recommend to allow some reasonable time for the TitroLine® 5000 titrator to "warm up".

Measurement input: Temperature probe - connector for a Pt 1000 resistance thermometer and NTC 30 Connection: 2 x 4 mm - sockets and 1 x 2 mm socket.

	Measurement	Display resolution	Measurement	Measurement
	range		accuracy* without	accuracy* without
			Pt 1000 sensor	NTC 30 sensor
T [°C]	- 30 115	0.1	0.5 K ± 1 Digit	0.5 K ± 1 Digit

* The measurement uncertainty of the sensor probe has to be taken into account as well

Display:	3.5 inches -1/4 VGA TFT display with 320x240 pixels.
Calibration:	Automatically with up to three buffer solutions, sequence during calibration optional, freely definable buffers can be input. Default buffer solutions according to DIN 19 266 and NBS, or technical buffers: pH = 1.00; $pH=4.00$; $pH=4.01$; $pH=6.87$; $pH=7.00$; $pH=9.18$; $pH=10.00$;
Inputs:	
Input pH/mV:	pH/mV-input with electrode socket according DIN 19 262/or BNC
Input Pt 1000/NTC 30:	Temperature sensor probe (Connection sockets: 2 x 4 mm
Power supply:	power supply 100 – 240 V; 50 – 60 Hz, power input: 30 VA Use the Power supply TZ 1853, Type No.: FW 7362M/12 only!

RS-232-C Interface: Data bits: Stop bit: Start bit: Parity: Baud rate: Address: RS-232-1	RS-232-C interface Daisy Chain function available. adjustable, 7 or 8 Bit (default: 8 Bit) adjustable, 1 or 2 Bit (default: 1 Bit) static 1 Bit adjustable: even / odd / none adjustable: 1200, 2400, 4800 , 9600, 19200 (Default 4800 baud) adjustable, (0 to 15, default: 01) for computer, input Daisy Chain
RS-232-2	devices of SI Analytics, titrator TitroLine [®] 5000/6000/7000/7500, TW alpha plus/TW 7400 - Burettes TITRONIC [®] 500/300, TITRONIC [®] 110 <i>plus,</i> TITRONIC [®] <i>universal,</i> - Balances of the types Mettler, Sartorius, Kern, Ohaus (for more, please contact SI Analytics) - Exit Daisy Chain
USB Interface:	1 x USB-type A and 1 x USB-type B (Mini – type)
USB –Typ B ("slave")	for connecting a PC
USB –Typ A ("master")	for connecting: - USB "mouse" - USB keyboard - USB printer - USB data media e.g. USB stick - USB Hub for the connection of more than one USB device
Stirrer connection:	Plug connection with integrated low voltage supply (= 12 V) in the casing bottom of the titrator housing for stirrer TM 50
Housing material: Front keyboard: Housing dimensions: Weight:	Polypropylene Polyester 135 x 310 x 205 mm (W x H x D), height incl. interchangeable unit w.o. stirrer ca. 2 kg
Ambient conditions:	Ambient temperature: + 10 + 40 °C for operation and storage Humidity according to EN 61 010, Part 1: Max. relative humidity 80 % for temperatures up to 31 °C, linear decrease down to 50 % relative humidity at a temperature of 40 °C
Dosing units	
Cylinder:	TitroLine [®] 5000 with 20 ml burette (borosilicate glass 3.3) TitroLine [®] 5000 with 50 ml burette (borosilicate glass 3.3) UV protection coat made out of TROGAMID, blue transparent
Valve: Hoses:	volume neutral cone valve made from fluorocarbon polymers (PTFE), TZ 3000 FEP hose set, blue transparent
Bracket for supply bottle:	suitable for square glass bottle and misc. reagent bottles
Dosing accuracy:	according DIN EN ISO 8655, part 3 Accuracy: 0.15 % Precision: 0.05 %

1.3 Warning and safety information

The TitroLine[®] 5000 corresponds to protection class III. It was manufactured and tested according to DIN EN 61 010, Part 1, Protective Measures for electronic measurement devices and control devices and has left the factory in an impeccable condition as concerns safety technology. In order to maintain this condition and to ensure safe operation, the user should observe the notes and warning information contained in the present operating instructions. Development and production is done within a system which meets the requirements laid down in the DIN EN ISO 9001 standard.

For reasons of safety, the titrator TitroLine[®] 5000 must be opened by authorised persons only; this means, for instance, that work on electrical equipment must only be performed by qualified specialists.

In the case of nonobservance of these provisions the titrator TitroLine[®] 5000 may constitute a danger: electrical accidents of persons or fire hazard. Moreover, in the case of unauthorised intervention in the titrator TitroLine[®] 5000 as well as in the case of negligently or deliberately caused damage, the warranty will become void.

Prior to switching the device on it has to be ensured that the operating voltage of the titrator TitroLine[®] 5000 matches the mains voltage. The operating voltage is indicated on the specification plate. Nonobservance of this provision may result in damage to the titrator TitroLine[®] 5000 or in personal injury or damage to property.

If it has to be assumed that safe operation is impossible, the titrator TitroLine[®] 5000 has to be put out of operation and secured against inadvertent putting to operation. In this case please switch the titrator TitroLine[®] 5000 off, pull plug of the mains cable out of the mains socket, and remove the titrator TitroLine[®] 5000 from the place of work.

Examples for the assumption that a safe operation is no longer possible,

- the package is damaged,
- the titrator TitroLine® 5000 shows visible damages,
- titrator TitroLine® 5000 does not function properly,
- liquid has penetrated into the casing.
- If the titrator TitroLine® 5000 has been altered technologically or if unauthorized personnel tried or succeeded to open the instrument as attempt to repair it.

In case that the user operates such a device, all thereof resulting risks are on the user.

The titrator TitroLine[®] 5000 must not be stored or operated in humid rooms.

For reasons of safety, the titrator TitroLine[®] 5000 must only be used for the range of application described in the present operating instructions.

In the case of deviations from the intended proper use of the device, it is up to the user to evaluate the occurring risks.

The relevant regulations regarding the handling of the substances used have to be observed: The Decree on Hazardous Matters, the Chemicals Act, and the rules and information of the chemicals trade. It has to be ensured on the side of the user that the persons entrusted with the use of the titrator TitroLine[®] 5000 are experts in the handling of substances used in the environment and in titrator TitroLine[®] 5000 or that they are supervised by specialised persons, respectively.

During all work with titration solutions: **Please wear protective glasses!**

The titrator TitroLine[®] 5000 is equipped with integrated circuits (EPROMs). X rays or other high energy radiation may penetrate through the device's casing and delete the program.

For working with liquids, not beeing common titration solvents, especially the chemical resistance of the construction materials of the titrator TitroLine[®] 5000 have to be considered (please also refer to chapter 1.1).

For the use of liquids with high vapour pressure or (mixture of) substances not being mentioned in chapter 1.1 as allowed substances, the safe and proper operation of the titrator TitroLine[®] 5000 has to be guaranteed by the user.

When the piston moves upwards within the cylinder, a microfilm of dosing liquid or titration solution will always remain adhered to the inner wall of the cylinder, but this has no influence on the dosing accuracy. This small residue of liquid, however, may evaporate and thus penetrate into the zone underneath the piston, and if non-admitted liquids are being used, the materials of the titrator TitroLine[®] 5000 may be dissolved or corroded (please refer also to chapter 8 "Maintenance and Care of the titrator TitroLine[®] 5000").

2 Unpacking and First Operation

2.1 Unpacking and First Operation of the titrator

The titrator itself as well as all related accessory and peripheral parts have been carefully checked at the factory to ensure their correct function and size.

Please ensure that the small accessories are also removed in full from the packaging.

For the scope of delivery, please refer to the enclosed parts list.

The titrator TitroLine® 5000 may be placed on any flat surface.

2.2 Connection and installing of Piston Burette and magnetic stirrer TM 50

The low voltage cable of the power supply TZ 1853 has to be plugged in to the 12 V socket "in"(see Fig. 3 back panel, chapter. 2.3), on the back panel of the Piston Burette. Then plug the power supply into the plug socket.



Fig. 1a)

Place the power supply easily accessable in order to be able to remove the Piston Burette anytime easily from the power circuit.

Insert the stirrer at the lower right side and fasten it by pushing backwards (Fig. 1). This automatically connects the power supply to the Stirrer TM 50



The tripod rod TZ 1748 is screwed into the thread and the titration clip Z 305 can now be mounted on the tripod rod (Fig. 2). Instead of the magnetic stirrer TM 50, you can also install the titration stand without stirring function TZ3866.

2.3 Connecting the Titrator - Combination with Accessories and Additional Devices

2.3.1 Back panel of the titrator TitroLine® 5000



Fig. 3

2.3.2 Connection ports of the TitroLine® 5000 . Connection of Electrodes

The TitroLine[®] 5000 has following connections:

- 1) pH/mV measurement input (DIN or BNC through adapter) for the connection of pH, redox and other measurement or combination electrodes.
- 2) Input for reference electrodes (Ref.)
- 3) Temperature measurement input for connecting Pt 1000/NTC 30 electrodes Two RS232 ports, 4-channel (Mini-DIN):
- 4) RS2 for connection of a weighing balance and other devices from SI Analytics (burettes etc.)
- 5) RS1 for connection to the PC
- 6) USB-A ("Master") interfaces for connecting USB devices such as a keyboard, printer, manual control unit, USB memory device
- 7) USB-B interface (mini type) for connection to a PC
- 8) Connection of the external power pack/supply TZ 1853
- 9) On/Off switch

2.3.3 Connecting a printer

Printers with a USB interface are to be connected one the USB-A interface. These printers **have to** feature HP PCL emulation (3, 3 enhanced, 5, 5e no GUI or GDI printers!). Alternatively the thermocompact printer Seiko S445 can be connected.

2.3.4 Connecting a USB device (manual controller, keyboard, memory device, hub)

The following USB devices can be connected to the USB-A interfaces:

- TZ 3880 manual controller (in the following: "mouse")
- PC-keyboard
- Printer
- USB storage devices, e.g. USB sticks
- USB hub
- USB barcode scanners

2.3.5 Connection of analytical balances

Analytical balances are to be connected to the RS232-2 using an appropriate cable.

2.4 Setting the Language of the Country

The ex-factory default language setting is English. When the piston burette is switched on, the main menu will appear once the boot sequence is completed:



Fig.4

Using <MODE> followed by <System settings> you navigate to the system settings. The very first menu is to be used for setting the language of the country:

Select met Methode 01		
System setti	ngs	
Balance data		
Rinsing		
Output		
	OK	ESC
20 ml	OK	09/01/14 15:55

Fig. 5

The first menu are the language settings.

System settings	
Language settings	
Calibration settings	
Reagents / dosing unit	
Global memory	
RS232 Settings	
Printer	PDF
Stirrer control	On▼
A V OK	ESC
20 ml	09/01/14 15:55

Fig. 6

Select the language using the $<\uparrow\downarrow>$ arrow keys, confirm it with <ENTER>/<OK>.

System se Language sett		
English	-	
Deutsch		
Français		
Español		
Polski		
Russian/Pyc	ский	
^ V	OK	ESC
20 ml		07/17/14 13:48

The selected language will appear immediately. Pressing the <ESC> key twice will return the user to the main menu.

Fig. 7

2.5 Dosing unit and Accessories



- TZ 2003 drying tube
 TZ 3282 dosing hose without dosing tip and holding bracket
- 3) TZ 3802 threaded cap with borehole GL 45, incl. adapter with 2 openings for drying tube and suction hose
- 4) TZ 1748 stand rod
- 5) Z 305 titration clamp
- 6) TZ 3620 dosing hose with dosing tip and holding bracket: bracket = TZ 3875
- 7) TZ 3803 1 litre reagent bottle, brown
- 8) TZ 3656 titration tip unit, blue
- 9) TZ 3801 valve cover lid and TZ 3000 3/2-way valve
- 10) TZ 3130 20 ml dosing unit or TZ 3160 50 ml dosing unit
- 11) TZ 3283 connection hose
- 12) TZ 3281 suction hose

2.6 Installing the burette tip

The burette tip consists of the elements shaft with threaded clamping joint, hose and slip-on tip.



Burette tip - Sequence of assembly:

- 1. Cut of hose end evenly.
- 2. Slip parts of the threaded clamping joint on to the hose.
- 3. Guide hose through shaft.
- 4. Press the free hose end over the ring marking until it reaches the stop of the tip.
- 5. Push the tip with pressed in hose onto the shaft.
- 6. Hold tip firmly, and screw threaded clamping joint to the shaft.

2.7 Initial Filling and Rinsing

The dosing unit and the tubes are already mounted and ready-to-use. After the reagent bottle is connected, the initial filling of the dosing unit can be performed. While performing the initial filling or washing program, an adequately sized waste bin has to be placed below the titration tip.

On the main menu (fig. 9)

Main menu	
0.0	000 ml
Methode 01 Method parameter Select method / system ^{20 ml}	START EDIT MODE 07/20/14 16:24

Fig. 9

Press <MODE> key and select Rinsing:

Select method ,	/ system	
HCl titrtion Methode 01 System settings Balance data		man dos
Rinsing Output		
20 ml	ок	ESC 07/20/14 16:28

Fig. 10

Confirm the selection by pressing <OK>.

At this point you can select the number of rinsing cycles (Fig. 11). Initial filling requires a minimum of two rinsing cycles. You can stop the rinsing operation (Fig. 12 and 13) at any time by pressing <STOP> and then resume rinsing with <START>.

Rinsing		
Rinse 1 x		
Rinse 2 x		
Continueous	s test	
∧ ∨ 20 ml	ОК	ESC 07/20/14 16:31

Fig. 11

The device fills first before the rinsing procedure starts:



Fig. 15

The rinsing program (Fig.12-15) can be canceled at all time with <STOP> and the continued with <START>. When the rinsing is finished, you can get back to the start menu by pushing 2 x<ESC>.

3 Working with the Titrator TitroLine[®] 5000

3.1 Front Keyboard



Fig. 16

Apart from alphanumeric input (a-z, A-Z, 0-9) and a few other functions, almost all functions can be performed using the front keyboard.

<mode>:</mode>	Methods selection, rinsing, system settings
<edit>:</edit>	Changing the current method, new method, copy and delete method
<esc>:</esc>	<esc< b="">> will take you back to the previous menu level.</esc<>
<start stop="">:</start>	Start and Stop of a current method
<cal></cal>	CAL> starts the pH calibration
<fill>:</fill>	Filling the unit
< ↑>	Arrow up: Selection of individual menus and numeric values
< ↓ >	Arrow down: Selection of individual menus and numeric values
<→>	Arrow right: change position in entry menu

The individual functions are described in detail in Chapter 3.4, External PC Keyboard.

3.2 Display

The display consists of a graphical LCD display with a resolution of 320 x 240 pixels. It also offers the possibility to display graphics, e.g. the measuring curve while or after the titration is/was running.



3.3 Manual controller "mouse"

The "mouse" (Fig. 18) is needed for manual titration. It can also be used for starting dosage or automatic titration methods.



Fig. 18

Mode	Black key	Grey Key
Manual titration	Start of titration, single-step and continuous titration (please refer to chapter 3.6.1, manual titration)	Filling Stop of titration including evaluation
Dosage through Dosage method	Start dosage	Filling
Automatic titration	Start of the method	Stop of the method including evaluation

3.4 External PC Keyboard

Keys	Function
<esc></esc>	<esc> will take the user to the previous level on the</esc>
	menu.
<f1>/<start></start></f1>	Start of a selected method
<f2>/<stop></stop></f2>	Stop of the current method
<f3>/<edit></edit></f3>	Change of the current method, new method, copy method
<f4>/<fill></fill></f4>	Fill the interchangeable unit
<f5>/</f5>	Display and modification of the balance data. With <shift +="" f5=""> display and modification of the global memories</shift>
<f6>/<mode></mode></f6>	Selection of method, rinsing, system settings
<f7>/<sys></sys></f7>	System settings (language selection, time/date)
<f8 <cal=""></f8>	Start calibration menu
<f9>/+ / -</f9>	Change of sign
<f10>/<dos></dos></f10>	Start dosing menu
Num/ Scroll	Without function
Lock/ Lock	
Prt Sc	Without function
Sys Rq	
$<\uparrow><\downarrow><\leftrightarrow><\rightarrow>$	Selection of individual menus and numeric values
09	Input of numeric values
<enter></enter>	Confirmation of input parameters
< ←Backspace >	Deletion of one input digit / an input character to the left of the blinking cursor
Letters, ASCII-symbols	Alphanumeric input possible. Uppercase and lowercase possible.
All other keys	Do not have any function

3.5 Menu Structure

There are 5 selection menus:

- Start or main menu
- Method parameters
- Method selection
- CAL menu
- System settings

After power-up, the main menu is always the first menu to appear. The method displayed will always be the last method that was used (Fig. 19).



Fig. 19

Pressing <START> will result in the immediate execution of the method shown. <EDIT>/F3 will take you to the method parameters (Fig. 20).

Method parameter HCI	
Edit method	
New method	
Default method	
Recalculation	
Copy method	
Delete method	\bullet
A V OK	ESC
20 ml	09/30/14 18:15

Fig. 20

At this point you can

- modify the current method
- create a new method
- call and memorise standard methods
- copy or delete an existing method
- print an existing method (only titration methods)

Use the < \downarrow > und < \uparrow > keys to select the submenus, confirm your selection with <OK>/<ENTER>. <ESC> will take you back to the main menu.

<MODE>/F6 leads you to the "select method" menu (Fig. 21).

Select method / system	n
Alkalinity (p+m)	
Dosing method	dos
HCI	
Methode 01	
System settings	
Balance data	
Rinsing	▼
A V OK	ESC
20 ml	09/30/14 18:17

Fig. 21

Existing methods (maximum 5) can be selected by pressing the $<\downarrow>$ und $<\uparrow>$ keys and confirming the selection with <OK>/<ENTER>. Once the selection made, you will return to the main menu with the newly selected method. If no method is selected, <ESC> will also take you back to the main menu.

To navigate directly to the system settings (Fig. 22 and Fig. 23) you can use the <SYS>/F7 key; you can also navigate there through the method selection menu.

System settings		
Language settings		
Calibration settings		
Reagents / dosing unit		
Global memory		
RS232 Settings		
Printer	PDF	
stirrer	On▼	
A V OK	ESC	
20 ml	10/14/14 14:49	

Fig. 22

System set	ttings	
stirrer		On 🛦
Date/time		
Reset		
Device infor	mations	
System tone	!	
Data exchan	ge	
Software Up	date	
~ V	ок	ESC
20 ml		10/14/14 14:49

Fig. 23

3.6 Main Menu

After power-up, the main menu is always the first menu to appear. The method displayed will always be the last method that was used (Fig. 24).



Fig. 24

3.6.1 Automatic Titration

Г

The method being displayed can now be carried out immediately with <START>. Depending on the method settings, you will be prompted for the sample identification (Fig. 25) and the sample weighed (Fig. 26). You can use an external PC keyboard (optional) for entering a 20-digit alphanumeric sample ID.

	Sample ID Alkalinity (p+m)	
	sample abc 123	
Fig. 25	 OK ESC 20 ml 09/30/14 18:22 	
	Edit weight	
	003.372 0 0g	
5- 00		

Fig. 26

The balance data can be entered using the front keyboard or an external keyboard. The input is to be confirmed with <OK>/<ENTER>.

In the case of an automatic acceptance of the balance data, the weighed-in quantities will be read in from a memory. If the memory does not contain any balance data, a message will appear to indicate that no balance data are present:



Fig. 27

Pressing the Print key will transfer the balance data, too. Titration will then begin directly after the transfer of the balance data without any further confirmation being necessary. The display will show the measured value (pH or mV) and the current consumption. The measured value is displayed in a slightly larger font. The top of the display will show the "Titration is running" status indication and the method being used, i.e. "HCI":



Fig. 28

Pressing the <Mode>/<F6> will cause the titration curve to be displayed (Fig. 34).



Fig. 29

The consumption in ml will be displayed on the X axis, the Y axis will show the measurement reading. Scaling of the chart will be done automatically. The result will be displayed at the end of the titration (Fig. 30).

Device is filling HCI	
EQ Result Start pH/temp End pH/temp	4.669 ml / pH 6.495 0.508 % pH 2.148 / 25.0 ℃ pH 9.326 / 25.0 ℃
MODE no USB Stick	ESC 09/30/14 18:32

Fig. 30

<MODE>/<F6> can be used to view the titration curve or further resuts. pH und mV titration curves will show the measurement curve (blue) and the 1st derivation (red). The values and the location of the equivalence point are identified directly in the curve itself.



Fig. 31

If a printer is connected, the results will either be printed according to the settings made for the method, or else they will be memorised in the form of a PDF- and CSV-file file on a connected USB stick. If no printer or USB stick is connected, the bottom left corner of the display will show the message "no printer" or "no USB stick".<ESC> will take you back to the main menu where you can start the next titration immediately.

3.6.2 Calibration (CAL menu)

If you are on the main menu (Fig. 32), calibration is started by pressing the <CAL> key on the titrator or the <F8/CAL> key from an external keyboard.



The titrator will ask you to rinse the electrode and immerse it successively into 2 or 3 buffers.

pH calibrat	tion	
Rinse electroc into Buffer	de and 1 (TEC_	4.000) dip
START 20 ml	ESC	MODE 10/01/14 11:12

Fig. 33

Fig.

Fig.

Fig.

The 1st buffer is started with <Start>. The 2nd and 3rd buffers (optional) are to be started with <Enter/OK>. During calibration, you can view the current mV and temperature values of the buffer:

	pH calibration Calibration of buffer 1 in operation		
		172.3 mV	
		25.2 °C (a)	
34	20 ml	ESC 10/01/14 11:14	
	pH calibratio Calibration active		
	Rinse electrode into Buffer	and 2 (TEC_7.000) dip	
35	OK 20 ml	ESC 10/01/14 11:14	
	pH calibratio Calibration of but	on ffer 2 in operation	
		-3.0 mV	
		24.6 °C (a)	
36	20 ml	ESC 10/01/14 11:15	

Once calibration completed, the display will show the slope and the zero point of the electrode.

pH calibration Calibration ready	
Slope Zero point Temperature	98.7% / -58.4 mV/pH pH 6.95 / -3.0 mV 24.9 ℃ (a)
no USB Stick	ESC

Fig. 37

The calibration values will be automatically printed or stored as a PDF file. <ESC> will take you back to the main menu. The current calibration values can be viewed at any time by pressing the <CAL> keys:

pH calibration		
Rinse electrode and into Buffer	1 (TEC_4.000) dip	
START ES	SC MODE 10/01/14 11:12	

Fig. 38

followed by <Mode>:

pH calibratio Current values	n
Slope	98.7% / -58.4 mV/pH
Zero point	pH 6.95 / -3.0 mV
Temperature	24.9 °C
Date	01.10.14 - 11:15
	ESC
20 ml	10/01/14 11:18

Fig. 39

3.6.3 Manual Titration

Manual titration is always performed using the "mouse". Manual titration is impossible without the "mouse". The mV or pH reading will be displayed. The value can be selected in the "Titration parameter" menu item. In the present case this is the pH value.



Fig. 40

Using **<START>/<F1>** or pressing the black key on the "mouse" will start the manual titration method. Following the input of the sample description and/or the weight/volume (optional - please compare also the explanations are regarding automatic titration in **Chapter 3.6.1**), the following display will appear:



Fig. 41

You can control the metering rate with the black key of the "mouse". A single depression of the key will cause a step up to the first level. Depending on the size of the dosing unit it is 0.0025 ml (20 ml dosing unit) or 0.00625 ml (50 ml dosing unit). However, only three decimal places will be shown. Therefore you will only see the dosing volume in the display starting with the 4th (20 ml dosing attachment) or the 2nd titration step (50 ml dosing attachment).

If one keeps the black key depressed on the first level, titration will be continued at a low rate. If you press the black key fully down (2^{nd} level) titration will proceed at a higher rate. The rate of the second level can be set in five stages using the < $\downarrow\uparrow$ > arrow keys. These stages can also be changed during manual titration.



Fig. 42

Stage 5 corresponds to maximum titration speed. Speed is reduced by 50% each time.

Example: 20 dosing unit:

Stage 5	100 % (ca. 40 ml/min)
Stage 4	50% (ca. 20 ml/min)
Stage 3	25% (ca. 10 ml/min)
Stage 2	12.5 % (ca. 5 ml/min)
Stage 1	6.8 % (ca. 2.5 ml/min)

As soon as the titration is completed, press the <STOP/F2> key or approx. for 1 sec. the grey key of the "mouse". The titration result will be calculated and displayed.

End of titration man Titration TA	
Consumption	3.288 ml
ТА	2.47 g/l
Start pH/temp	pH 4.208 / 25.0 ℃
End pH/temp	pH 7.023 / 25.0 ℃
Back	ESC
Printing!	10/01/14 11:37

Fig. 43

The result can also be printed or stored in PDF- and CSV-format. <ESC> will take you back to the start menu way to start the next titration immediately. Filling of the dosing unit occurs automatically.

3.6.4 Dosage

To start a dosage method, please use the <START>/<F1> or the black key of the "mouse".



The dosed volume will be briefly displayed before the display returns to the main menu (Fig. 44)

The next dosage operation can be started immediately. Filling of the unit will occur automatically. This option can be switched off. Then the cylinder will be filled when the maximum cylinder volume is reached. The unit can be filled at any time using <FILL>.

A dosing operation can also be performed without any dosing method with the <DOS>/<F10> key of the external keyboard:



Fig. 47

This is the point to input the volume which will be dosed following the confirmation with <OK> or <ENTER>:



Fig. 48

Pressing the <ENTER>/<OK> key will cause the next dosing operation to be performed immediately.

In this case further dosages can be performed using<OK>. or <ENTER>. Filling of the unit following dosage will not occur automatically here, unless the maximum cylinder volume has been reached. The unit can be filled at any time using <FILL>. <ESC> will take you back to the main menu.

4 Method Parameters

From the main menu (Fig. 40), <EDIT> will take you to the method parameters:

Method parameter Alkalinity (p+m)	
Edit method	
New method	
Default method	
Recalculation	
Copy method	
Delete method	•
A V OK	ESC
20 ml	10/01/14 13:28

Fig. 49

4.1 Method editing and new method

If you select <edit method> or <new method> you will be taken to the modification or new creation of a method. Selecting <new method> will always lead to the prompt for the input of a method name (Fig. 50). This prompt will not appear in the case of the modification of an already created method.

New method Method name	I	
Methode 01		
< > 20 ml	ок	ESC 10/01/14 13:29

Fig. 50

The method name can contain up to 21 characters. Special characters are also possible. If no keyboard is connected, the method name being displayed has to be adopted (in the present case "Method 01"). Numbering of methods will occur automatically. Press <OK>/<ENTER> to confirm the input. The method name can be changed at any time. Please continue at this point with **Chapter 4.6**.

4.2 Default methods

The <Default methods> item of the TitroLine® 5000 contains a series of ready-made standard methods which can be conveniently selected (Fig. 51).

Default method	
Alkalinity (p+m)	
Chloride in %	
Chloride in mg_l	
COD Blank	
COD Sample	
COD Titer	
Dosing method	dos 🔻
A V OK	ESC
20 ml	10/01/14 13:32

Fig. 51

Once the selection made, you are directly prompted for the input of the method name.

New method Method name	od	
<u>p</u> H strong ac	sid	
< > 20 ml	ОК	ESC 10/01/14 13:32

Fig. 52

The standard name may be adopted or modified. Subsequently, you will be taken to the <Change method parameters> item. Please continue at this point with **Chapter 4.6**.

4.3 Copy Methods

Methods can be copied or stored with a new name. If you select this function, the current method will be copied and you can include a new name

New method Method name	
HCI[1]	
< > OK	ESC 10/01/14 13:35

Fig. 53

A new name with the suffix [1] is assigned automatically in order to avoid the existence of two methods having the same name. Subsequently, you will be taken to the <Change method parameters> item. Then you proceed with **Chapter 4.6**.

4.4 Delete Methods

If this function is selected, you will be prompted to know whether the current method is actually to be deleted. You have to reply **<Yes>** in explicit terms and also confirm this reply with <OK>/<ENTER>.

Delete me HCI[1]	Delete method HCl[1]	
Yes		
No		
^ V	ок	ESC

Fig. 54

4.5 Print method

The currently selected method can be printed on a connected printer or stored on a USB drive as PDF file.

Method par HCl	rameter	
New method		
Default meth	Default method	
Recalculation	1 I	
Copy method	d l	
Delete metho	bc	
Print method		
^ V	ок	ESC
20 ml		10/01/14 13:40

Fig. 55

4.6 Change Method Parameters

The input or modification of the method name was already described in Chapters 4.1 and 4.3.

Edit method parameter HCI	
Method name	
Method type	auto
Mode	Dynamic
Result	
Titration parameter	
Dosing parameter	▼
A V OK	ESC
20 ml	10/01/14 13:41

Fig. 56

4.6.1 Method type

On the <Method type> you can select whether you wish to perform a manual or automatic titration or a dosage:

Method ty HCl	pe	
Automatic titration Manuel titration Dosing mode		
_∧ ∨ 20 ml	ок	ESC 10/01/14 13:43

Fig. 57

The selection of the Method type will have an influence on the further parameterisation of the method. For instance, if you select the dosing mode, neither a selection of a formula nor a change of the Titration mode (dynamic or linear, etc.) will be available.

4.6.2 Titration mode

In the case of an automatic titration, you can make a selection between the following modes:

- Linear titration (pH and mV)
- Dynamic titration (pH and mV)
- End-point titration (pH, mV)

4.6.2.1 Linear titration

In the case of linear titration, the step size remains identical over the entire titration cycle. Linear titration is often used for complicated or unknown samples. Complicated examples include, for instance, chloride in the trace range (-> very flat curve shape) or titrations in non-aqueous media. If one would use a dynamic titration control in these cases, this would not yield any benefit. Depending on the parameters, the step sizes used in excessively flat curves would either be too small or too large. Below an example of a flat and rather unsteady curve shape:



Fig. 58

Titration was performed as a linear titration with a step size of 0.05 ml. In this case, dynamic titration control with a step size adapted to the curve slope would generate an even more unsteady course of the curve.

Linear Titration is possible for mV und pH titrations.

4.6.2.2 Dynamic titration

in the case of dynamic titration, the titration steps are adapted to the change of the measurement readings/ml (slope, curve slope). Small slope values mean a large step sizes, and large slope values indicate small step sizes. Within that section, this leads to the inclusion of most of the measurement points which are later on of importance with regard to the evaluation of the equivalence point (EQ).

Dynamic titration begins with three identical small step sizes, for instance 0.02 ml, and this value is then doubled until the maximum step width is reached, for instance 0.5 or 1 ml. Should the slope values now increase during the titration, the step sizes will decrease down to minimum step size, for instance 0.02 ml. In the example below (Fig. 59) titration was performed between 100 and 300 mV with the smallest step sizes (in the present case 0.02 ml). With linear titration control involving step sizes of 0.05 or even 0.1 ml, only 1-2 measurement points would be recorded between 100 and 300 mV. This would result in an inaccurate calculation of the equivalence point.



Fig. 59

Dynamic titration is possible for mV and pH titrations.

4.6.2.3 End-point titration

The goal of end-point titration consists in titrating as precisely as possible to one or two end points given in terms of pH or mV. Consumption in the end points will be used as a result. The classical examples of pH end-point titration include total acidity in wine or beverages and the p+m value (alkalinity).

The first stage of end-point titration consists in the continuous dosing up to a delta value away from the set end point. The dosing speed can be adjusted. Subsequently, titration is performed in a drift-controlled manner with linear step sizes between the delta value and the end point.

Example: Determination of the alkalinity (m value)

pH in the point:	4.30
delta pH value:	1.00
linear step width:	0.02
dosing speed:	12 %
end-point delay:	10 s
drift:	medium (20 mV/min)

Up to a pH value of 5.30, titration is performed with the set dosing speed. Subsequently, the method will change to a linear step size of 0.02 ml, until the end point of pH 4.30 is either reached or fallen short of. Should this value raise again to above pH 4.30 within 10 seconds, another titration step of 0.02 ml will be added. Consumption will be determined precisely at pH 4.30.



Fig. 60

4.6.3 Result

At first, the calculation options are specified (dynamic and linear titration only):

Result Chloride in %		
Calculation of Formula	options	1 EQ
_∧ ∨	ок	ESC 10/01/14 14:2

Fig. 61

One inflection point (1 EQ) can be analyzed in the TitroLine[®] 5000:

Calculation Chloride in %	ı options	
only total co	nsumption	
Evaluate 1 E	Q	
20 ml	ОК	ESC 10/01/14 14:23

Fig. 62

With "**only total consumption**" the consumption at the last measured pH/mV value will be used.

With ",1 $\ensuremath{\text{EQ}}\xspace$ respectively the calculated equivalence points of the titration curve will be used.

"Formula" offers the following settings:

Result Chloride in %		
Result text		
Formula Unit Decimal plac Global mem		% 2
^ V 20 ml	ОК	ESC 10/01/14 14:25

Fig. 63

The Result text may contain up to 21 alphanumeric characters including special characters.

Result text Chloride in %	1	
Chloride 123	45678% Aa	
< > 20 ml	ОК	ESC 10/01/14 14:26

Fig. 64

Please confirm your input with <OK</ENTER>. If there are two results - such as in the case of titration for two pH end points - you can enter two result texts.

4.6.3.1 Calculation Formula

The appropriate calculation formula is selected on the **Formula selection** submenu:

Formula selection Chloride in %	
EQ1	
(EQ1-B)*T*M*F1/(W	*F2)
(B-EQ1)*T*M*F1/(W	*F2)
(B*F3-EQ1*F1)*T*M	/(W*F2)
(W*F2)/((EQ1-B)*M*	۴ 1)
EQ1*T*M*F1/(W*F2)
A V OK	ESC
20 ml	10/01/14 14:27

Fig. 65
The following calculation formulae are available for EQ and EP:

Formula for linear and	Formula for titrations to end-	Additional information
dynamic titration to EQ1	point (EP 1 and EP2)	
No formula		No result will be determined
(EQ1-B)*T*M*F1/(W*F2)	(EP1-B)*T*M*F1/(W*F2)	Formula for calculating the concentration of a sample taking into account a blank value in terms of ml. Direct titration to one EQ or EP1 (ex.:: chloride, p or m value)
(B–EQ1)*T*M*F1/(W*F2)	(B-EP1)*T*M*F1/(W*F2)	Formula for calculating the concentration of a sample taking into account a blank value in terms of ml. Reverse titration (examples. CSB, saponification number)
(B*F3–EQ1*F1)*T*M/(W*F2)	(B*F3–EP1*F1)*T*M/(W*F2)	Formula for calculating the concentration of a sample taking into account a blank value, including a multiplicative factor. Back titration.
(W*F2)/(EQ1-B)*M*F1)	(W*F2)/(EP1-B)*M*F1)	Formula for calculating a titer (T) of a titration solution.
(W*F2)/(EQ1-B)*M*T*F1)	(W*F2)/(EP1-B)*M*T*F1)	Formula for calculating the concentration of a sample taking into account a blank value in ml. Direct titration to one EQ or EP1.
(W*F2)/(B-EQ1)*M*T*F1)	(W*F2)/(B-EP1)*M*T*F1)	Formula for calculating the concentration of a sample taking into account a blank value in ml. Back titration (NCO-value, Epoxy-number).
EQ1	EP1	Calculation of the consumption in the equivalence or end point.
	EP2*T*M*F1/(W*F2)	Formula for the calculation of concentration of a sample. Direct titration to 2 EP. Here EP2 (p and m value)
	(EP2-EP1)*T*M*F1/(W*F2)	Formula for the calculation of the concentration of a sample. Direct titration to 2 EP. Here calculation of the difference between EP2-EP1.
	(F3*EP2-EP1)*T*M*F1/(W*F2)	Formula for the calculation of the concentration of a sample. Direct titration to 2 EP. Here: calculation of the difference between EP2-EP1, taking into account a multiplicative factor for EP2.
	(F1/W) * EP1 *F2	Calculation of the des TAC (Total Anorganic Carbonat reserve)
	((F1/W)*(EP2-EP1) * F3-F4)*F5	Calculation of the FOS (<u>V</u> olatile <u>O</u> rganic <u>A</u> cids) FOS/TAC-value

The abbreviations used here have the following meaning:

- ml: Total consumption, e.g. for pH Stat
- EQ: Consumption at the equivalence point 1 in ml
- EP: Consumption at the end point 1 and 2 in ml
- B: Blank value in ml. Mostly determined by way of titration
- T: Titer of the titration solution (e.g. 0.09986)
- M: Mol; mol- or equivalence weight of the sample (e.g. NaCl 58.44)
- F1-F5 Factor 1-5. conversion factors
- W "Weight", weighed-in quantity in g or volume in ml."

Formula parameter (B-EQ1)*T*M*F1/(W*F2)	
B (Blank value)	0.0000 ml
T (Titre)	0.10000000
M (Mol)	35.45000
F1 (Factor 1)	0.1000
W (Amount)	man
F2 (Factor 2)	1.0000
A V OK	ESC
20 ml	10/01/14 14:30

After selecting a formula, please confirm your selection with <OK>/<ENTER>:

Fig. 66

The values for the blank value, the titers and factors F1-F5 can be entered or read from a global memory. The values from the global memory were defined in advance by a titration or were manually entered:

ок

ESC

10/01/14 14:45

	Formula parameter B (Blank value) fix value Global memory			
67	^ V 20 ml	ОК	ESC 10/01/14 14:42	
	Blank value Global memory M01 M02	blank value M02	*0.0220 * 1.0000	

^ V

20 ml

Fig. 67

Formula parameter (EQ1-B)*T*M*F1/(W*F2)	
B (Blank value)	M01
T (Titre)	0.10000000
M (Mol)	35.45000
F1 (Factor 1)	0.1000
W (Amount)	man
F2 (Factor 2)	1.0000
A V OK	ESC
20 ml	10/01/14 14:47

The global memory used is displayed. Here, in this example, it is M01:

Fig. 69

Storing results in global memories is described in Chapter 4.6.3.5.

The values of the individual parameters of the selected calculation formula can now be input one by one.







4.6.3.2 Sample weight and volume (sample quantity)

Fig. 72

The Sample Quantity (W) item is used to select whether one is wishing to use a sample weight or a sample volume for titration or solution preparation.

You have the following options:

- Manual sample weight: The sample weight is enquired by a prompt at the start of the method • and manually input.
- Automatic sample weight: The sample weight is automatically transferred by a connected • balance.
- Fixed sample weight: A fixed sample weight is input in g. This weight will then automatically • be used for each start of the method.
- Manual sample volume: The sample volume in ml is prompted at the start of the method and ٠ manually input.
- Fixed sample volume: A fixed sample volume is input in ml. This volume will then automatically be used for each test of the method.

4.6.3.3 Formula unit

The formula unit can be selected in the **Unit** submenu.

Unit 1 Chloride in %		
None		
ml		
96		
ppm		
g		
mg		•
^ V	OK	ESC
20 ml		10/01/14 14:57

Fig. 73

Once the selection made (e.g. %), the unit will also be displayed as piece of information on the display.

Г		
	Result HCI	
	Result text	
	Formula	
	Unit	%
	Decimal places	3
	Statistics	None
	Global memory	
	A V OK	ESC
	20 ml	10/08/14 11:37

Fig. 74

4.6.3.4 Decimal places

To conclude, it is possible to determine the number of decimal places from 1-6. The standard setting is 2.

4.6.3.5 Global Memories

If a titration result is to be used again later, such as the factor or titer of a solution or a blind value, this can be saved automatically. The creation of a global memory is only possible if an external keypad is used. The creation of a global memory is possible in the system settings or by pressing Shift+F5 on the external keypad. This will take you to the <Global Memories>:



Using F3 it is possible to add a global memory:



Fig. 77

The titrator proposes a memory name, such as **M01** (M01- M10). The name of the memory can be changed in reference to the application. Here in this example of "**M01**" for "**Blank value Chloride**".

Add memo M01	ry	
Blank value (Chloride_	
< > 20 ml	ОК	ESC 10/01/14 15:26

Fig. 78

This simplifies later the allocation of the global memory in another method.

Formula pa B (Blank value)			
fix value			
Global memo	ory		
V 20 ml	ОК	ESC 10/14/14 14:59	

The blind value which was possibly titrated in advance, is always taken into consideration automatically.

Blank value Global memory	-		
M01 Blank	M01 Blank value Chloride *0.0350		
20 ml	ок	ESC 10/14/14 15:00	

Fig. 80

Example: The blank value of a chloride titration is defined with the support of an extra method. The result in ml is thereby automatically written into global memory M01 by using the name "Blanc value Chloride". The blank value is then automatically deducted from the titrant consumption within the chloride method. Here in our example it is 0.035 ml:

System set Global memory		
->M01 Blank	value Chlor	*0.0350
20 ml	OK	ESC 10/01/14 15:39

Fig. 81

4.6.3.6 Statistics

The mean value and relative standard deviation can be automatically calculated and documented by using the statistics.

Result Chloride in %	
Result text	
Formula	
Unit	%
Decimal places	2
Statistics	None
Global memory	
A V OK	ESC
20 ml	10/08/14 11:39

The calculation of the mean value is already possible from 2 individual values, the calculation of the relative standard deviation is only possible from 3 single values. the maximum quantity is 10.



Fig. 83

The mean value and relative standard deviation (RSD) are shown directly on the display.

End of titration 1 of 3 Chloride in % 3 of 3		
EQ Chloride Mean value RSD	3.614 ml / -259.7 mV 4.50 % 4.49 % 2.47 %	
MODE 20 ml	ESC 10/08/14 12:32	

Fig. 84

4.6.4 Titration parameters

The <Titration parameter> submenu is used to determine the actual parameters of the method:

Edit titration parameter HCI	-
Measured value	pН
Measuring speed / drift	Normal
Initial waiting time	0 s
Dynamic	Steep
Titration direction	Increase
Pretitration	Off ▼
A V OK	ESC
20 ml	10/14/14 15:02

Edit titration paramete	r
Measuring speed / drift	Normal 🔺
Initial waiting time	0 s
Dynamic	Steep
Titration direction	Increase
Pretitration	Off
End of titration	
A V OK	ESC
20 ml	10/14/14 15:03

Generally applicable titration parameters

Depending on the titration mode (dynamic, linear and end-point titration), it is possible to enter a variety of parameters. The following parameters are valid for all automatic titration modes:

- Measured value (pH, mV)
- Measurement speed
- Initial waiting time
- Pre-titration
- Titration end

But please note that the measurement speed and the titration end differ again as a function of the respective titration mode.

<Measured value> is the first selection to be made. In the present example, the selection is "pH".

Measured y HCl	value	
mV		
рH		
^ V	OK	ESC

Fig. 87

The selected measured value is displayed for information.

Edit titration paramete HCI	r
Measured value	pН
Measuring speed / drift	Normal
Initial waiting time	0 s
Dynamic	Steep
Titration direction	Increase
Pretitration	Off ▼
AV OK	ESC
20 ml	10/14/14 15:02

<Measuring speed> or drift will determine the span of time after which the measured value will be accepted following a titration step.

Measuring s HCl	peed / drift	
Normal		
Fast Fixed delay tir User-defined	ne	5 s
20 ml	ок	ESC 10/02/14 10:58

Fig. 89

Drift-controlled acceptance of the measured value in terms of mV/min is set by selecting "**norma**l", "fast or "**user-defined**". The drift values at predefined in terms of in mV/min for normal and fast drift:

Normal drift	20 mV/min
Fast drift	50 mV/min

Small drift value = slow and precise Large drift value = fast and "less precise" The following parameter selection can be made for user-defined drift setting:

Minimum holding time [s]:	01 - 99
Maximum holding time [s]:	01 - 99
Measuring time t: [s]	01 - 99
Drift [mv/min]	01 - 99

15s
_
2s
20mV/min

Fig. 90

If normal or fast drift was selected before, the values will be defaulted for user-defined drift. In the present case, for instance, 20 mV for normal drift:



Drift-controlled acceptance of the measured value is used in most applications. However, there are applications in which the setting of a fixed holding time for measured value acceptance following the titration step is recommendable. Examples hereof include titrations in non-aqueous media. In the case of dead-stop titration no holding time other than the fixed one can be selected. The fixed delay time can be set between 0 and 999 seconds:



Fig. 92

After the start of titration, it makes frequently sense to have the sample stirred over a defined period of time, for instance, to allow for the sample to be dissolved. The waiting time to be observed prior to the first addition of titration solution can be set using the **<Initial waiting time>** item. The initial waiting time can be set between 0 and 999 seconds:



Dynamic control

If dynamic control was selected, one has a selection of 3 different stages: steep, medium and flat:

dynamic d HCl	rive	
Steep Average Flat		
V 20 ml	ок	ESC 10/02/14 11:10

Dynamic parameters	Min./max. step size	Applications
Steep	0.02/1.0	Strong acids and alkali (HCI, NaOH, HNO3 etc.), redox titrations such as iron (permanganometric or cerimetric), halogenides high concentrations
Average	0.02/1.0	lodometric titrations, halogenides, medium-strength acids and alkali
Flat	0.05/0.5	Weak acids and alkali, titrations involving Ca- or Cu-ISE

If linear titration control was selected, you have to define the step size.

r
pН
5 s
Off
0 s
0.100 ml
Increase 🔻
ESC
10/02/14 11:13

Fig. 95

Linear step size can be set from 0.001 to 5.000 ml.

Step size HCI	
00.	.0 5 0 ml
< > 20 ml	OK ESC 10/02/14 11:13

Fig. 96

Linear step width can also be set for end-point titration (pH and mV). In this type of titration, linear step width is used after the first continuous titration stage.

Titration direction

The titration direction can be set to "**increase**" or "**decrease**". For instance, if you wish to perform a total acidity titration to a pH value of 8.1 using NaOH, you have to select "**increase**". When titrating for the alkalinity ("m value") to a pH value of 4.5 using HCl, you have to select "**decrease**".

Titration d HCI	irection	
auto		
Decrease		
Increase		
_∧ ∨ 20 ml	ОК	ESC 10/02/14 11:14



Pretitration

If the titration agent consumption is roughly known, you can set a pretitration volume on the **<Pretitration>** menu. In this process, a defined volume is dosed (= pretitrated) following the initial waiting time. After the addition of the pretitration volume, another defined span of time is observed as the waiting time before the next titration step is added. The pretitration volume is automatically added to the titration agent consumption. The pretitration volume can be set from 0.000 and 99.999 ml, the possible range for setting the waiting time following pretitration is between 0 and 999 seconds.

Pretitration HCI Off	I	
Volume [ml]		12.000ml
Delay time		15s
_	ОК	ESC 10/02/14 11:16

Fig. 98

Titration end

The end of a titration is reached, and the result will be calculated as soon as, or if, respectively:

- The defined End value pH and mV value has been reached
- The criteria (steep, flat, **slope value**) have been met for one turning point (**EQ1**) in the case of a linear or dynamic titration.
- The predefined value ml has been reached (Maximum titration volume)
- or if titration was terminated manually by operating the <Stop> key.

End of titr HCI	ation		
End value		12.000 pH	
EQ		On	
Slope value		Steep	
Max. titratio	on volume	50.00 ml	
^ V 20 ml	ОК	ESC 10/02/14 11:21	

Fig. 99

It is also possible to switch off the criteria for the end value for pH and mV.

End value HCI Off		
On		12.000 pH
^ V 20 ml	ОК	ESC 10/02/14 11:22

The possible pH end value input ranges from 0.000 to 14.000.

The possible mV end value ranges from -2000 to +2000.

The range of the μ A input can be selected between 0.0 and 100.0.

Automatic detection of the equivalence point (EQ) can be switched on and off for linear or dynamic titration.

EQ HCI Off On		
_ ∧ ∨ 20 ml	ок	ESC 10/02/14 11:23

Fig. 101

If automatic EQ detection is off, titration will continue to the predefined end value in mV or pH or to the maximum mI value, respectively. Nevertheless, it is possible to calculate the EQ subsequently on the basis of the recorded measurement data.

If EQ detection is activated, you can define the slope value for the EQ:

Slope valu e HCI	e	
Steep		
Flat Value		700
_∧ ∨20 ml	ок	ESC 10/02/14 11:24

Fig. 102

The determination of the equivalence point (EQ) is done on the basis of the maximum of the first derivation (red curve) of the measurement data.

Setting of the **maximum titration volume** should always make sense. It also serves as a safety criteria to prevent excessive titration, i.e. a possible overflow of the titration vessel. The maximum titration volume can be set between 1.000 und 999.999 ml:



4.6.5 'End-point titration' Titration parameters

When working with end-point titration, there are some differences in context with linear and dynamic equivalence-point titration.

As was already described in **Chapter 4.5.2.3**, end-point titration, in a first stage, proceeds by continuously dosing until a specific Delta value ("**Delta end-point**") at a distance from the set end value is reached. The dosing speed of this first stage can be set in terms of % on the "**Dosing parameters**" menu. Subsequently, titration continues in a drift-controlled manner or with a fixed holding time with a linear step width between the Delta value and the end value. As soon as the end value has been reached, a defined waiting time is observed. If the end value is fallen short of, one or more than one additional titration step(s) is/are added until the end value has become stable. The waiting time at the end is referred to as **End-point delay**.

In the case of an end-point titration for two endpoints, it is possible to set both of the endpoints with different Delta values and end-point delays:



4.6.6 Dosing parameter

Edit method parameter Alkalinity (p+m)	
Method name	
Method type	auto
Mode	End pt.
Result	
Titration parameter	
Dosing parameter	•
A V OK	ESC
20 ml	10/02/14 11:31

Fig. 106

The dosing parameters (dosing speed, filling speed and max. dosing/titration volume) are determined for each method. This applies to all types of methods such as manual and automatic titration, dosing and Solution Preparation.

Edit dosing paramete Alkalinity (p+m)	er
Dosing speed	15.00 %
Dosing speed	6.00 ml/min
Filling speed	30 s
Max. titration volume	50.000 ml
∧ ∨ OK	ESC
20 ml	10/02/14 11:33

Fig. 107

The dosing speed can be set in % from 1 to 100 %. 100 % is the maximum dosing speed.

Dosing unit	Max. dosing speed [ml/min]
20 ml	40
50 ml	100

The filling speed can be set in terms of seconds from 20 to 999. The standard setting of this value is 30 seconds. For diluted aqueous solutions the filling speed can be six to 20 seconds. For non-aqueous solutions the filling speed should be set to the 30 seconds. In the case of highly viscous solutions such as concentrated sulphuric acid the filling speed should be further reduced down to 40 - 60 seconds.

Depending on the method type, the (maximum) the living volume or titration volume can be set to 999.999 or even 9999.999.

The following filling options can be set for the dosing mode

Automatic Methode 01	filling	
Off intelligent be intelligent af always		
 20 ml	ок	ESC 10/02/14 11:39

Fig. 108

If"off"is selected for filling, filling it will not occur automatically after each dosing step.

If "intelligent before" is selected for filling, a verification will be performed each time prior to the next dosing step in order to determine whether the dosing step can still be made without a filling operation. Should this prove to be impossible, the first thing to occur is filling, followed by the dosing step.

If "intelligent after" is selected for filling, a verification will be performed after the next dosing step to find out whether the next dosing step can still be made without filling.

If "always" is selected for filling, filling will occur automatically after each dosing step.

4.6.7 Sample identification

In the manual titration and in the preparation of solutions it is possible to input a sample identification. The possible input includes manual, automatic or no sample description at all.

Sample ID HCI	,				
Without sam	nple ID				
	Automatic sample ID				
Manual sam	ple ID				
_ ∧ ∨ _20 ml	ОК	ESC 10/02/14 11:40			

Fig. 109

For a sample description of the 'manual', a prompt for the sample description will always be displayed at the start of the method (Cp. also chapter 3.6, Main menu). For an 'automatic' sample description there will be selected a master description (in the current case this is water, cp. Fig. 110), which will then automatically be numbered starting on 01.

Sample ID HCI		
water		
< > 20 ml	ОК	ESC 10/02/14 11:41

Fig. 110

After a new power-up, numbering will resume with 01.

4.6.8 Documentation

Edit method paramet HCI	er
Mode	Dynamic 🔺
Result	
Titration parameter	
Dosing parameter	
Sample ID	man
Documentation	GLP
A V OK	ESC
20 ml	10/02/14 12:17

Fig. 111

Three different format settings are available for documentation on a printer or USB device: "short", "standard (with curve)" and "GLP":

Documenta HCl	ition	
Short		
Standard (wi	th curve)	
GLP		
Only Display		
V 20 ml	ок	ESC 10/02/14 11:42

Method type	Short documentation	Standard documentation	GLP-Documentation
Automatic titration	Method name, date, time, duration of titration, sample description, weight/volume, starting and end measurement values (pH/ mV Temp), slope and zero point of the pH electrode, results and calculation formula	Same as 'Short documentation' + titration curve	Same as 'Standard documentation' + method content
Manual titration	Method name, date, time, sample description, sample weight/sample volume, results and calculation formula	N/A	Same as 'Short documentation' + plus method content
Dosing	Only method printout possible: method name, date, time and dose parameter	N/A	N/A

5 System settings



Fig. 113

From the main menu (Fig. 113), using the front keys <MODE> and then <System settings> will get you to the system settings:

System settings	
Language settings	
Calibration settings	
Reagents / dosing unit	
Global memory	
RS232 Settings	
Printer	PDF
Stirrer control	Off ▼
AV OK	ESC
20 ml	10/02/14 12:44

Fig. 114

Setting the national language was already described in Chapter 2.5.

5.1 Calibration settings

The Calibration settings item is used to select the buffers for the calibration of the pH electrode as well as to set the temperature of the buffer solution. The temperature has only to be set if neither a resistance thermometer (Pt 1000, NTC 30), nor a pH electrode with an integrated temperature measurement probe is connected.

System se Calibration set	=	
Temperature	e	25.0 °C
pH buffer se Type of calil		2
_∧ ∨20 ml	ок	ESC 10/02/14 12:44



The temperature can be set from 0.0 to 100.0 °C in increments of 0.1 °:

System settings Temperature		
025.0°C		
<u>∧ v < > OK ESC</u> 20 ml 10/02/14 12:47		

Fig. 116

The type of calibration items is used to define whether a 2-point or a 3-point calibration is to be performed:

Type of cal 2-point calib 3-point calib	ration	
^ V 20 ml	ОК	ESC 10/02/14 12:47

Fig. 117

The pH buffers for the buffers 1 - 3 can be determined individually.

System set pH buffer	tings	
pH buffer 1		TEC_4.000
pH buffer 2		TEC_7.000
pH buffer 3		TEC_10.000
Accept value	S	
∧ ∨ 20 ml	ОК	ESC

Fig. 118

A list of technical and so-called DIN/NIST buffers will appear:

System settings Selection pH buffer	
TEC_4.000	
DIN_4.010	
DIN_6.865	
TEC_7.000	
DIN_9.184	
TEC_10.000	
A V OK	ESC
20 ml	10/02/14 12:49

After having determined the buffers for buffers 1 - 3, the selection is to be confirmed with <Accept values>. If the distance between 2 buffer values is too small (for instance, buffer 1 "6.87" and buffer 2 "7.00"), an error message will appear:

System pH buffer	settings	
	Error!	
Buffers do not match		
	At least 2 buffer values are to close to each other or equal.	
20 ml	ESC	14 12:50

Fig. 120

5.2 Reagents – Dosing unit

You can set up the attachment size in the menu (20 or 50 ml), perform an attachment change and enter reagent data, which are put out into the GLP documentation during manual titration.

System settings Reagent	
Unit size	20 ml
Reagent	
Concentration	1.00000
Conc. determined at	
Expire date	
Opened/compounded	🔻
A V OK	ESC
20 ml	10/02/14 12:52

Fig. 121

5.2.1 Replacing the dosing unit

As a rule, the need for replacing the dosing unit occurs only rarely. The dosing unit has to be replaced, if such a replacement becomes necessary as a result of a defect, or of an inspection of the titration unit.

The dosing unit is equipped with lateral ribs around its circumference, with one of these ribs being in double design. This double rib serves as a mark for the correct placement of the dosing unit (fig 125.).

With <OK/ENTER> confirm the <Unit size>

System settings Unit size	
Unit size Unit size Dosing unit exchange Rinsing	20 ml 50 ml
OK	ESC 10/02/14 12:56

Select <Dosing unit exchange>:

Caution: the exchange procedure starts directly with any additional warning. Please take care that the titration tip is placed in a beaker or in the reagent bottle.

The piston is raised to about 85%:

System settings Dosing unit exchange	
Dosing unit moves up	
ESC 20 ml 10/02/14 12:59	

Then you will be prompted to unlock the attachment:

System s Dosing unit		
Please	unlock de	osing unit
OK 20 ml		ESC 10/02/14 13:00

Fig. 124

Now unlock the dosing attachment as shown in Figure 125:



Fig. 125

Confirm with <OK> after the attachment has been unlocked. Now, the attachment will be raised all the way up:

	i settings iit exchange	
Dos	sing unit mov	es up
20 ml	ESC	10/02/14 13:02

Fig. 126

The following message will be displayed:

		settings it exchange	
		e remove dosi I attach a new	
Fig. 127	20 ml	ОК	10/02/14 13:02

Pull the attachment off toward the top and attach the new dosing attachment in the same manner. The two struts of the UV protection must match up with the marking on the housing (Fig. 128, right).



Confirm with <OK>/<ENTER>. If you changed the attachment size, you can select the size here now:

System set Neue Unit size	ttings		
Unit size		20 ml	
Unit size		50 ml	
_ ▲ ♥ 20 ml	ОК	ESC 10/02/14 13:06	

Fig. 129

If you want to change reagents, you can reset the data completely:

System set Reset reagent (
Yes			
No			
^ V	OK	ESC	
20 ml		10/02/14 13:07	

Fig. 130

Then, the attachment will lower back down. Now please lock the attachment.





Reagents:

- Unit size 20 or 50 ml (selectable)
- Reagent name (default: empty)
- Conzentration (default: 1.000000)
- Concentration determined on (default: empty)
- Expire date (default: empty)
- Opened/Produced on: (default: empty
- Test according to ISO 8655: (default: empty)
- Batch ID: (default: empty)
- Last modification (default: current Datum)

System settings Reagent	
Unit size	20 ml
Reagent	
Concentration	1.00000
Conc. determined at	
Expire date	
Opened/compounded	🔻
A V OK	ESC
20 ml	10/02/14 13:14

Abb. 133

System set	ttinas		
Reagent	2-		
Conc. deterr	nined at	🔺	
Expire date	Expire date		
Opened/com			
Inspection a	ccording ISC)	
Batch ID			
Last modific	ation	10/02/14	
^ V	ок	ESC	
20 ml		10/02/14 13:18	

5.2.2 Replacing the titration solution

If titration solutions are to be changed, since differing analysis methods are used, one should first consider whether the time required for frequent changes is not more expensive than the acquisition of another dosing unit.

As a principle and in the case of all piston/cylinder- systems, a substitution of the titration solution by another one involves mixing and carry-over processes. The reason for this is the dead volume above the piston in the cylinder and in the hoses. The disturbances to be anticipated are the greater, the more the new solution differs from the previous type and concentration. In the case of highly different solutions, the first substitution liquid (rinsing) should be distilled water, and the new titration solution should be filled in only subsequently.

The possible disturbances are very much different in the individual cases and cannot be predicted without knowledge of the specific case. Therefore the replacement of titration solutions must always be performed under the supervision of experts who ensure the correctness of the future analyses.

If the decision to change the titration solution has been made, the first thing to do is to remove the dosing unit as it is described in chapter 5.2.1. If possible, the residue of the titration solution should be removed by hand by carefully pushing the projecting piston rod towards the hoses. When doing so, more liquid will leak out of the titration tip, and the residual volume is furthermore reduced. Removing the old titration solution can be accelerated by moving the piston rod of the dosing unit positioned top down. The suction hose is then immersed in the new solution or in water as intermediate liquid. By moving the piston several times in both directions (pumping) the previous liquid is gradually replaced by new liquid. Subsequently, the dosing unit is set on again according to the description in chapter 5.2.1.

5.3 Globale Memory

The handling with the global memories were already described in the chapter 4.5.2.5.

5.4 RS232 Settings

The <RS232 settings> item can be used to determine the device address of the TitroLine® 5000 and set the parameters of the two RS232 interfaces independent from each other:

RS232 Setting:		
Device addre	255	01
RS232-1 (Pr	inter/PC)	
RS232-2 (Ba	alance)	
Reset RS set	tings	
	OK I	ESC
^ V	0	

Fig. 135

The device address can be set from 0 - 15. Address 1 is the default setting:

System se Device addres		
Off		
1		
2		
3		
4		
5		▼
V 20 ml	ОК	ESC 10/02/14 13:47

RS232-1 Settings	
Connection	RS
Baud rate	4800
Parity	No
Data bit	8
Stop bits	1
۸V OK	ESC
20 ml	10/02/14 13:55



The baud rate is present to 4800. It may be set to 1200 – 19200:

Systemein Baudrate	stellungen	
1200		
2400		
4800 (Stand	lard)	
9600		
19200		
^ V	ок	ESC
20 ml		02.10.14 13:45

The parity can be selected amongst <No>, <Even> and <Odd>. <No> is the default setting.

Systemein: Parität	-	
No (Standar Even Odd	d)	
20 ml	ок	ESC 02.10.14 13:46

Fig. 139

Fig. 138

You may select between 7 and 8 data bits. 8 bits is the default setting.

	System settings Data bit 7 Data bit 8 Data bit (Default)
Fig. 140	∧ ∨ OK ESC 20 ml 10/02/14 13:52

You can set data bits at 1, 1.5 and 2. 1 bits is the default setting.

System se Stop bits	ettings	
1 Stop bit (1,5 Stop bit 2 Stop bits	-	
F		
∧ ∨ 20 ml	ок	ESC 10/02/14 13:53

The connection via RS can be changed to USB (USB-PC).

System set Connection RS (Default)	-	
USB		
_∧ ∨ 20 ml	ОК	ESC 10/02/14 13:55

Fig. 142

Fig. 143

After switching from RS232 to USB and vice versa, a restart is always necessary.

System RS232-19	s ettings Settings	
	connection was sw start the device	itched to USB.
20 ml	ESC	10/02/14 13:55

The USB driver can be downloaded from our homepage or it may be available on an included USB stick. The RS232 parameters can be set to the factory settings.

5.5 Connection of Printers

The results, calibration data and methods can be printed on the following media:

- HP PCL compatible printer (A4), color and monochrome (e.g. laser printer)
- Seiko DPU S445 (Thermo paper 112 mm width)
- On the USB stick in PDF- and CSV-format

To connect the printers to the burette please use the USB socket. When printing, please check whether the correct printer is connected. It is not possible to print "HP" printer layouts on another thermal printer or vice versa. The printer settings should always be checked and adjusted after changing the printer.

System set Printer	tings	
HP-PCL A4 (-	
HP-PCL A4 (monochrome)
DPU S445		
Print PDF		
20 ml	ОК	ESC 10/02/14 14:04

Only one printer should be connected for one Titrator because an automatic printer recognition is not activated. Print PDF is the default setting. If you select "Print PDF", please make sure that a USB stick is connected to the device.

5.6 Stirrer

Stirrer <ON> means that the magnetic stirrer TM 50 can also be used for stirring if now method has been executed. This is the standard setting:

System se t Stirrer control	ttings	
Off		
On		
^ V	ок	ESC
20 ml		10/02/14 14:40

Fig. 145

If the stirrer is set to <OFF>, it is only started if a method is executed.

5.7 Date and Time

The factory time setting is Central European Time. This setting may be changed, where necessary:

System set Date and time	ttings	
Date		10/02/14
Time		14:42:31
∧ ∨ 20 ml	ОК	ESC 10/02/14 14:42

5.8 RESET

RESET will reset all settings to the factory setting.

Please note: All methods will also be deleted. So please print the methods or export/copy them to a connected USB storage medium (cp. chapter 5.11).

The RESE	T has to he	confirmed	senarately	once again:
THE RESE	1 11/05 10 06	commed	separately	/ Unce again.

System set Reset to factor		
Yes		
No		
ΛV	ок	ESC

Fig. 147

5.9 Device Information

<Device Information> contains information about

- the serial number of the device
- the current software version
- printer driver and update version
- Export version
- device address (Hardware version)

Device informations System settings	
Serial number Software version Printer driver version Update version Export version	BETA 1440 1.14.9.4 2.14.8.8 2.13.2.14
Hardware version ESC 20 ml	O1 10/02/14 14:46

Fig. 148

Your display can deviate to Fig. 148.

5.10 System Tone

The system tone (sound) can be set on or off.

System set System tone	ttings	
Sound on		
Sound off		
ΛV	ОК	ESC
20 ml		10/02/14 14:49

Fig. 149

5.11 Data exchange

All methods with all parameter settings and global memories can be stored and restored on a connected USB-memory. It is also possible to transfer the settings from one titrator to another one. The backup will be started with **Settings backup**:

System se Data exchang		
Settings bac	:kup	
Restore sett	ings	
^ V	OK	ESC
20 ml		10/02/14 14:55

Fig. 150

Backup settings is displayed during the backup in blue:

System set	tings	
Printer		PDF 🔺
Stirrer contro	ol	Off
Date/time		
Reset		
Device inform	nations	
System tone		
Data exchanç	je	T
^ V	ок	ESC
Backup settings		10/02/14 15:16

Fig. 151

After a Reset or a maintenance case it is possible to restore the backup with **Restores settings**:

The backup folder on the USB-memory Stick starts with the backup date. Here it is 141002_151627 Settingsb...That means the backup is from 02th October 2014 15.16 hour:

CAL <dir> CSV <dir> Dyn_Debug <dir> method <dir> result <dir> 141002_151627_Settingsb ^ V OK ESC 20 ml 10/02/14 15:24</dir></dir></dir></dir></dir>	System settings Select backup	
Dyn_Debug <dir> method <dir> result <dir> 141002_151627_Settingsb ^ v OK ESC 20 ml 10/02/14 15:24 System settings Data exchange Settings backup</dir></dir></dir>	CAL	<dir></dir>
52 method <dir> result <dir> 141002_151627_Settingsb ^ v OK ESC 20 ml 10/02/14 15:24 System settings Data exchange Settings backup</dir></dir>	CSV	<dir></dir>
52 result <dir> 141002_151627_Settingsb A V OK ESC 20 ml 10/02/14 15:24 System settings Data exchange <u>Settings backup</u></dir>	Dyn_Debug	<dir></dir>
52 141002_151627_Settingsb N V OK ESC 20 ml 10/02/14 15:24 System settings Data exchange Settings backup	method	<dir></dir>
152 System settings Data exchange Settings backup	result	<dir></dir>
152 20 ml 10/02/14 15:24 System settings Data exchange Settings backup	141002_151627_Set	tingsb
152 System settings Data exchange Settings backup	A V OK	ESC
System settings Data exchange Settings backup	 20 ml	10/02/14 15:24
Settings backup		
	-	
Restore settings		
	Restore settings	

^ V

Settings are being restored

ESC

10/02/14 15:27

OK |

Fi

5.12 Software Update

	System settings	
	Stirrer control	Off ▲
	Date/time	
	Reset	
	Device informations	
	System tone	
	Data exchange	
	Software Update	
	A V OK	ESC
Fig. 154	20 ml	10/02/14 15:28

An update of the device software requires a USB stick containing a new version. For this operation, the two files that are needed have to be located in the root directory of the USB device:

Computer > Wechseldatenträger (F:)		 ✓ ✓	hseldatenträger (F:) durci	<u>×</u> م
Datei Bearbeiten Ansicht Extras ?				
Organisieren 🔻 🛛 Freigeben für 💌 Brennen 🔊	Neuer Ordner			0
 ☆ Favoriten ■ Desktop Bownloads ∑uletzt besucht 	• •	TLXXXX_Application_14_29.bin TLXXXX_Update_14_29.def]	

Plug the USB device into a free USB-A port, wait for some seconds, and then select the Software Update function. The valid software updates will be shown on the display. In the present case this is Version "14_38" which means week 38 from 2014.

Fig. 155

After starting the update using <OK/ENTER>, next thing to appear is the following graphic:



which will change after a few seconds to the following display:

	TitroLine [®] 5000
	Waiting for system readiness
Fig. 157	Vers.2.14.8.8.20
	TitroLine [®] 5000
	System is updating. Please wait
Fig. 158	Vers.2.14.8.8.20

Fig. 158

Upon completion of the update (approx. 2-3 minutes), the device will shut down the software completely and proceed to a new start.

Important: In the course of an update, the methods will not be deleted! You can continue to use them. If no valid update file is stored on the USB stick, the following message will appear:

Software Update Software version: BETA 1440
No update found
ESC A V OK ESC 20 ml 10/02/14 16:01

6 Connection of Analytical Balances and Printers

6.1 Connection of Analytical Balances

As it often happens that the sample is weighed in on an analytical balance, it makes sense to connect this balance to the TitroLine[®] 5000. To connect the balance to the TitroLine[®] 5000, the balance must have a RS-232-C-interface and the connection cable must be configured accordingly. For the following types of balances there are already assembled connection cables:

Balance	TZ-Number
Sartorius (all types), partially Kern,	TZ 3092
Mettler, AB-S, AG, PG	TZ 3099
New Sartorius with USB-interfacee via RS-Adapter	TZ 3099
Precisa XT-Series	TZ 3183
Kern with 9-pole RS232	TZ 3180

For all other types of balances it is possible to obtain an already assembled connection cable (on demand). For this we need detailed information about the RS-232-C-interface of the balance used.

The connection cable is to be connected to the RS-232-C-interface 2 of the TitroLine[®] 5000. This side of the connection cables always consists of a 4-pole mini-plug. The other side of the cable can, depending on the type of balance, be a 25-pole plug (Sartorius), or a 9-pole plug (Mettler AB-S) etc.

In order to allow the balance data to be sent to the TitroLine[®] 5000, the data transmission parameters of the titrator and the balance must correspond to each other. Additionally, it is necessary to carry out some more standard settings on the side of the balances:

- > The balance is to send the balance data via RS-232-C only by means of a print command.
- > The balance is to send the balance data only after the display standstill.
- > The balance should never be set to 'automatic sending' and/or 'send continuously'.
- > 'Handshake' on the balance must be set to 'off', or even 'Software Handshake' or 'Pause'.
- No special characters such as S or St are allowed to be used as prefix in the balance data of the balance data string. In such a case it might be possible that the TitroLine® 5000 cannot process the balance data correctly.

After you have connected the balance with the appropriate cable to the TitroLine[®] 5000 and have adjusted all settings in the balance software, and possibly in the TitroLine[®] 5000, you can now test the data transfer of the balance very easily. Start the one method. Confirm the sample designation. Then, the display asks you:

- a) To press the print-button at the balance \rightarrow Parameters to 'weighted sample automatically'
- b) To enter the weighted sample \rightarrow then the parameters are still set to 'weighted sample manually'

Put an object onto the balance and press the print button. After the standstill of the balance display there will be beep at the TitroLine[®] 5000 and the transmitted balance data appear:

- a) After approx. 5 sec. in the display and the display changes automatically into the measuring display.
- b) The weighted sample must again be confirmed with <Enter> or <F1>.

6.2 Balance data editor

Pressing the die **<F5/balance symbol >** function key on the optional external keyboard will invoke the so-called balance data editor.

A list with the existing balance data will appear:

List of 3 Weigh		nce data	
003	м	1.65470	g 16:13:55
004	м	0.53600	g 16:14:01
005	М	29.76000	g 16:14:08
005	Ivi	29.76000	g 10:14:08
^ v	1	OK	ESC
20 ml			10/02/14 16:1

The balance data can be edited one by one. Following a change, a cross will appear opposite the weighed-in quantity:

Fig. 161

Weights may be deleted or added individually. It is also possible to delete all weights at one stroke.

Balance data 004 *M 0.55600		
Edit weight Delete weight Add weight Delete all?		
20 ml	ок	ESC 10/02/14 16:15

Fig. 162

If no balance data is available, the "No balance data found" message will appear:

List of balance data No balance data found		
_ ▲ ♥ 20 ml	ОК	ESC 10/02/14 16:15

7 Data Communication via RS-232- and USB-B interface

7.1 General Information

The burette TitroLine® 5000 has two serial RS-232-C interfaces to communicate data with other devices. By means of these two interfaces it is possible to operate several devices on one computer (PC) interface.

In addition to that, the TitroLine® 5000 also has an <u>alternatively</u> USB-B interface, which can only be used to connect a PC.

RS-232-C-1 establishes the connection to a connected computer or to the previous device of the "Daisy Chain". At the RS-232-C-2 it is possible to connect additional devices (Daisy Chain Concept).

PIN assignment of the RS-232-C interfaces:

PIN-No. Meaning / Description

- 1 T x D Data output
- 2 R x D Data input
- 3 Digital mass

7.2 Chaining multiple devices — "Daisy Chain Concept"

In order to activate several devices in a chain individually, each device must have an own device address. For this it is at first necessary to establish a connection from the computer to the RS-232-C interface 1 of the first devise in the chain by means of a RS-232-C data cable, e.g. Type No. TZ 3097. With the additional RS-232-C data cable, Type No. TZ 3094, the RS-232-C- interface 2 of the first device is connected with the RS-232-C-interface 1 of the second device. At interface 2 of the second device it is possible to connect an additional device.

The TitroLine® 5000 can also be connected via USB cable TZ 3840 (type A (M) – type B (M), 1.8m). It is also possible to connect the TitroLine® 5000 via USB cable TZ 3887 (type A (M) --- USB type B (Mini), 2.0 m) to a USB interface of a PC. To accomplish this connection, a USB driver has to be installed on the PC. Then the USB-B interface takes over the function of the RS232-1 interface. The USB driver can be downloaded from our website.

The address always consists of two characters: e.g. address 1 of the two ASCII- characters <0> and <1>. The addresses can be set from **00** to **15**, i.e. 16 possibilities. It must be ensured that the devices in a chain have different addresses. If a device is addressed with its address, this device will process this command without sending it to another device. The reply to the computer has also an own address. The addresses are allocated as described in \square Chapter 5.3.

The burette TitroLine® 5000 receives commands from a PC at the interface **1** (USB- B) if the computer knows the address. It also sends the answer via this interface. If the address of the incoming command does not match the device address, the complete command will be forwarded to interface **2**. Interface 2 is connected to interface 1 of another device. This device checks the address as well and reacts to the command as the first TitroLine® 5000 did before.

All information (data strings) which arrive at interface 2 of the burette TitroLine® 5000 will immediately be send to the computer via interface 1 (or USB-B interface). Thus, the computer receives the data of all devices. In practice it is possible to connect up to 16 devices to one computer- (PC-) interface.

7.3 Instruction Set for RS-Communication

The commands consist of three parts:	Address	two-digit aa,	e.g.: 01
	Command	-	e.g.: DA
	Variable, if	necessary	e.g.: 14
	and end of	command	<ČR> <lf></lf>

Every command must be completed with the ASCII - sign <CR> and <LF> (Carriage Return and Line Feed). Only if the respective action has ended the answers will be returned to the computer.

Example: The command to dose 12.5 ml shall be sent to the burette TitroLine® 5000 with the address 2.

The command consists of the characters: 02DA12.5<CR LF> In detail:

- 02 = Device address
- = Dosage command with filling and zero points of the display DA
- 12.5 = Volume in ml to be dosed

<CR LF> = Control character as command end

Command	Description	Reply
aaAA	automatic allocation of device address	aaY
aaMC1XX	choosing a method	aaY
aaBF	"filling burette". Aufsatz wird gefüllt.	aaY
aaBV	output of dosed volume in ml	aa0.200
aaDA	dose volume without filling, with adding the volume	aaY
aaDB	dose volume without filling, reset of the volume	aaY
aaDO	dose volume with filling, without adding the volume	aaY
aaGF	filling time in seconds (min is 20, default 30)	aaY
aaEX	"exit" function.back to main menu	aay
aaFP	pH measurement function	aay
aaFT	temperature measurement function	aay
aaFV	mV measurement function	aay
aaGDM	dosing speed in ml/min (0.01 – 100 ml/min)	aaY
aaGF	filling time in sec (adjustable 20 – 999 seconds)	aaY
aaGS	output serial no. Of device	aaGS08154711
aaLC	output of the CAL parameters	
aaLD	output of the measurement data	aaY
aaLR	output report (short report)	aaY
aaM	output of the preset measurement value (pH/mV/ug)	aaM7.000
aaLl	output method content	
aaRH	request of identification	aaldent:TL500
aaRC	send last command	aa"last command"
aaRS	report status	aaStatus:"text
	possible answers are:	
	"STATUS:READY" for ready	
	"STATUS:dosing" dosing	
	"STATUS:filling" filling	
	"ERROR:busy" if no interchangeable unit has been attached	
aaSM	start selected method	aaY
aaSEEPROM	EEPROM reset to factory defaults	aaY
aaSR	stop the actual function	aaY
aaSS	titration start with the transfer of the pH end value	aaY
aaVE	Version number of the software	aaVersion

8 Maintenance and Care of the TitroLine[®] 5000

The preservation of the proper functioning of the piston burette requires testing and maintenance work to be performed on a regular basis.

Regular inspections are essential prerequisites for the correctness of the volume and the proper functioning of the piston burette.

The accuracy of the volume is determined by all chemicals-carrying components (piston, cylinder, valve, titration tip and hoses). These parts are subject to wear and tear, i.e. zthey are or wearing parts, respectively. The piston and cylinder are subject to particular strain, hence they require special attention.

Heavy strain:

Use of e.g. concentrated solutions, reagents and chemicals (> 0.5 mol/L); chemicals attacking glass, such as fluorides, phosphates, alkali solutions; solutions with a tendency to crystallising out; Fe (III) chloride solutions; oxidising and corroding solutions such as iodine, potassium permanganate, Cer (III), Karl-Fischer titration agent, HCl; solutions with a viscosity of > 5 mm²/s; frequent, or even daily use.

Normal strain:

Use of solutions, reagents and chemicals (up to 0.5 mol/l) which do not attack glass, crystalize out or corrode.

Interrupted use :

If the dosing system is not in use for more than two weeks, we recommend emptying and cleaning the dosing unit [6]. This applies in particular under the operating conditions referred to in the "Heavy strain" section. If this recommendation is not adhered to, the piston of the valve may become leaking, this may result in damage to the piston burette.

If the liquid is left within the system, you will also have to reckon with corrosion and an alteration of the solutions used over time, which includes e.g. crystallisation. Considering that as of the state of the art there are no plastic hoses available for the use in titration equipment which would be perfectly free of diffusion phenomena, particular attention is to be paid to the range of the hose lines.

We recommend the following inspection and maintenance work		Heavy strain	Normal strain	
Simple cleaning:		Whenever required in	Whenever required in	
	Wiping off splashed chemicals from the outer surface. [1]	operation	operation	
Cight ob a	alu	Weekly, when putting	Monthly, when nutting	
Sight che		Weekly, when putting back into operation	Monthly, when putting back into operation	
	Check for leakage in the area of the dosing system. [2]	back into operation	back into operation	
	Is the piston tight? [3]			
	Is the valve tight? [4]			
	Titration to clear? [5]			
Basic cle	aning of the dosing system:	Every three months	Whenever necessary	
	All parts of the dosing system to be cleaned separately. [6]			
Technica	l inspection:	Semi-annually when	Semi-annually when	
	Check for air bubbles in the dosing system. [7]	putting back into	putting back into	
	Visual inspection	operation	operation	
	Check of the electrical connections. [8]			
Verification	on of the volume according to ISO 8655:	Semi-annually	Annually	
	Perform basic cleaning			
	Inspection according to ISO 8655 Part 6 or Part 7. [9]			

Please note: Depending on the respective application, there may be different specifications for the entirety of the inspection and maintenance work to be performed. The individual intervals may be extended if no complaints occur, but they will have to be shortened again as soon as any problem has arisen.

The inspection of the metrological reliability including maintenance work is offered as a service (including a manufacturer's certificate, if so ordered). In this case the titration device is to be sent in. Please contact the service (see backside of this manual).

Detailed description of the inspection and maintenance work:

- [1] Wipe off using a soft cloth (and some water with a normal household detergent).
- [2] Leaking connections can be identified by moisture or crystals at the threaded connections of the hoses, at the sealing lips of the piston inside the dosing cylinder or at the valve.
- [3] If any liquid becomes visible below the first sealing lip, it has to be checked at short timely intervals whether any liquid will build up under the second sealing lip, too. In this case both the piston and the glass cylinder have to be replaced immediately. It is easily possible that in operation small liquid droplets build up under the first sealing lip, but they may also disappear again. This phenomenon alone is no reason for replacement.
- [4] The valve has to be removed from its housing for inspection. In this process, the hoses remain connected to the valve. Please check for moisture underneath the valve. When reinserting the valve, please make sure that the small cam at the rotating axis is fitted into the corresponding groove again.
- [5] The titration tip must be free of sedimentation or crystals which might obstruct the dosing process or falsify the results.
- [6] Remove the cylinder, take the valve out of the valve housing, unscrew the hoses and then rinse all parts carefully with distilled water. For the assembly of the cylinder, hoses and other parts of the interchangeable unit, please refer to the operating instructions.
- [7] Dose one burette volume, then refill. Air bubbles will gather at the tip of the cylinder and in the titration hose where they can be detected easily. If bubbles become visible, please re-tighten all connections finger tight, and then repeat dosing. If air bubbles still remain within the system, [6] please check the valve and replace the hose connections. The air bubbles may also occur at the interface between the sealing lip of the piston and the cylinder. If a reduction of the filling speed will not do, the dosing unit has to be replaced.
- [8] Check the electrical plug contacts for corrosion and mechanical damage. Defective parts have to be repaired or replaced by new parts.
- [9] Please refer to the application "Burette inspection according to ISO 8655 Part 6".

9 Storage and transportation

If the titrator TitroLine® 5000 or the interchangeable units have to be stored over some time, or to be dislocated, the use of the original packing will be the best protection of the devices. However, in many cases this packing will not be available anymore, so that one will have to compose an equivalent packaging system. Sealing the lower section in a foil is hereby recommended.

The devices should be stored in a room with a temperature between +10 and +40°C, and the (relative) humidity of the air should not exceed 70 %.

If the interchangeable have to be stored over some time, or to be dislocated, the fluids inside the system, especially aggressive solution have to be removed (please refer also to chapter 8. "Maintenance and Care of the burette").

10 Recycling and Disposal



Please observe the applicable local or national regulations concerning the disposal of "waste electrical and electronic equipment".

The present titrator and his packaging are manufactured as far as possible from materials which can be disposed of environmental-friendly and recycled in a technically appropriate manner. If you have any question regarding disposal, please contact the service (see backside of this manual).

The main printed board carries a lithium battery. Batteries should not to be disposed of with the normal domestic waste. They will be taken back and recycled or disposed of properly by the manufacturer at no cost.

SI Analytics®

EG - KONFORMITÄTSERKLÄRUNG EC - DECLARATION OF CONFORMITY CE - DÉCLARATION DE CONFORMITÉ CEE - DECLARATIÓN DE CONFORMIDAD

Wir erklären in alleiniger Verantwortung, dass das folgende Produkt	We declare under our sole responsibility that the following product	Nous déclarons sous notre seule responsabilité que le produit ci-dessous	Declaramos bajo nuestra única responsabilidad, que el producto listado a continuación
Titrator	Titration unit	Titrateur	Titulador
	TitroLir	ne® 5000	
auf das sich diese Erklärung bezieht, übereinstimmt mit den folgenden EG Richtlinien.	to which this declaration relates are in conformity with the following EC directives.	auxquels se réfère cette déclaration est conforme directives CE soul vantes	todo lo relativo a esta declaración está en conformidad con las directivas CEE siguientes
EMV	EMC	CEM	CEM
EG-Richtlinie 2014/30/EU	EC-Directive 2014/30/EU	CE-Directive 2014/30/EU	CEE siguientes 2014/30/EU
Sicherheit	Safety	Sécurité	Seguridad
EG Richtlinie 2014/35/EU	EC-Directive 2014/35/EU	CE-Directive 2014/35/EU	CEE siguientes 2014/35/EU
RoHS	RoHS	RoHS	RoHS
EG Richtlinie 2011/65/EU	EC-Directive 2011/65/EU	CE-Directive 2011/65/EU	CEE siguientes 2011/65/EU
Harmonisierte Normen oder normative Dokumente	Harmonized standards or normative documents	Normes harmonisées ou documents normatifs	Estándares armonizados o documentos normativos
EMV	EMC	CEM	CEM
EN 61326-1:2013	EN 61326-1:2013	EN 61326-1:2013	EN 61326-1:2013
Sicherheit	Safety	Sécurité	Seguridad
EN 61010-1 :2010	EN 61010-1 :2010	EN 61010-1 :2010	EN 61010-1 :2010
RoHS	RoHS	RoHS	RoHS
EN 50581: 2012	EN 50581: 2012	EN 50581: 2012	EN 50581: 2012

P. Wining Dr. Robert Reining Geschäftsführer, Managing Director

Mainz den 21.07.2017

Konf. No.: Titrat 020c

Xylem Analytics Germany GmbH Dr.-Karl-Slevogt-Str. 1 82362 Weilheim Deutschland, Germany, Allemagne, Alemania

Bescheinigung des Herstellers

Wir bestätigen, dass oben genanntes Gerät gemäß DIN EN ISO 9001, Absatz 8.2.4 "Überwachung und Messung des Produkts" geprüft wurde und dass die festgelegten Qualitätsanforderungen an das Produkt erfüllt werden.

Supplier's Certificate

We certify that the above equipment has been tested in accordance with DIN EN ISO 9001, Part 8.2.4 "Monitoring and measurement of product" and that the specified quality requirements for the product have been met.

Certificat du fournisseur

Nous certifions que le produit a été vérifié selon DIN EN ISO 9001, partie 8.2.4 «Surveillance et mesure du produit» et que les exigences spécifiées pour le produit sont respectées.

Certificado del fabricante

Certificamos que el aparato arriba mencionado ha sido controlado de acuerdo con la norma DIN EN ISO 9001, sección 8.2.4 «Seguimiento y medición del producto» y que cumple con los requisitos de calidad fijados para el mismo



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