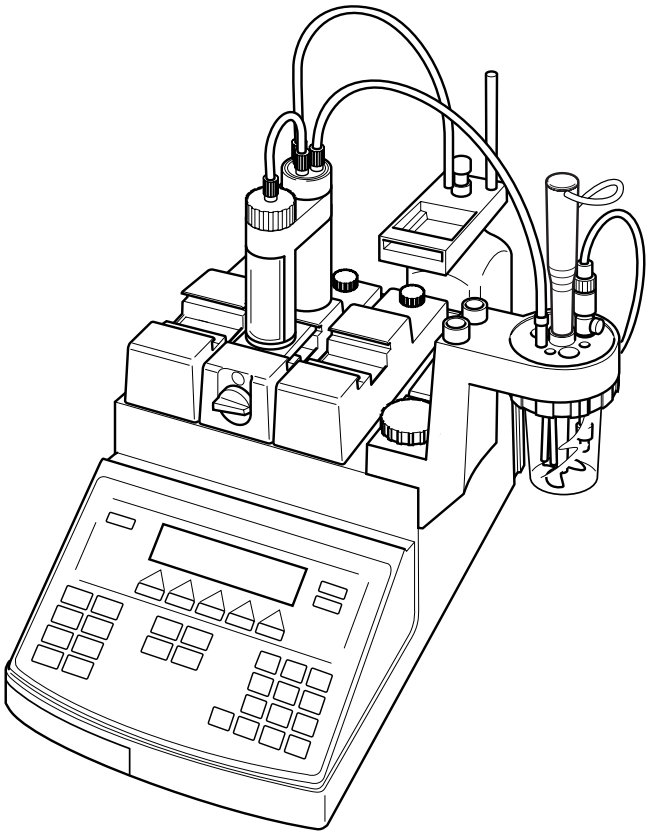
Quick Guide



DL50/DL53/DL55/DL58
Titrators

Contents

1. Introduction

The METTLER TOLEDO DL50, DL53, DL55 and DL58 Titrators are microprocessor controlled analytical instruments which supply accurate and reproducible results thanks to their built-in intelligence.

You can use the titrators to perform end point, equivalence point and pH-stat titrations, to measure the potential and temperature of solutions, as well as to determine TAN/TBN and acid and base capacities. Voltametric and amperometric titrations can be performed using a KF option and the moisture content determined by the Karl Fischer method with a KF titration stand. You can perform conductivity measurements and conductometric titrations with a third-party device equipped with an analog output.

In addition to electrodes, temperature sensors and a stirrer, you can also attach an analog recorder to the titrators. You can attach an external keyboard to the DIN socket which permits not only text entries but also remote control of the titrator. You can also connect a bar-code reader to this keyboard. Maximum two TTLIO sockets are used for the attachment of devices you can control via the inputs and outputs.

With the Centronics option you can attach

- a balance to the RS232 interface, which transfers the sample weight automatically, and
- a printer, which records the desired data.

With an RS option you can attach

- a computer, which interchanges data with the titrator, or a terminal for use as an auxiliary display, and
- a sample changer from METTLER TOLEDO for the automatic analysis of sample series.

The titrators have a slot for a memory card on which you can store your method and measured data. In the DL50 case, only a software update with a card is possible.

While the four titrators are operated in the same manner, they differ in regard to their hardware and software; these differences are indicated in the relevant sections.

Where can I find what information?

1. This **Quick Guide** will help you learn to operate the titrator within a short space of time. You will perform your first analyses with the aid of stored methods.

2. The **Reference Handbook** provides a complete description of the operating principles of the four titrators. You will find the installation instructions in Section 10. The additional commands and functions, which offers the DL58, are described in Section 7. The index in Section 13 includes key words from both the Quick Guide and the Reference Handbook.

3. The application brochure describes 30 METTLER methods; 4 methods are stored in the DL50, 20 in the DL53 and 30 in the DL55 and DL58.

4. The **Computer Interface Description**, namely a detailed explanation of the communication between titrator and computer, can be ordered.

2. Safety measures

The titrators have been tested for the experiments and intended purposes documented in the Quick Guide and the Reference Handbook. However, this does not absolve you from the responsibility of performing your own tests of the products supplied by us regarding their suitability for the methods and purposes you intend to use them for. You should therefore observe the following safety measures.

Measures for your protection

Risk of electric shock

– Ensure that you plug the power cable supplied into a receptacle outlet that is grounded! In the absence of grounding, a technical fault could be lethal.
– Switch the instrument off and disconnect the power cable before you open the housing! An electric shock could be lethal.

Risk of explosion

– Never work in an environment subject to explosion hazards! The housing of the instrument is not gas tight (explosion hazard due to spark formation, corrosion caused by the ingress of gases).

Risk of corrosion

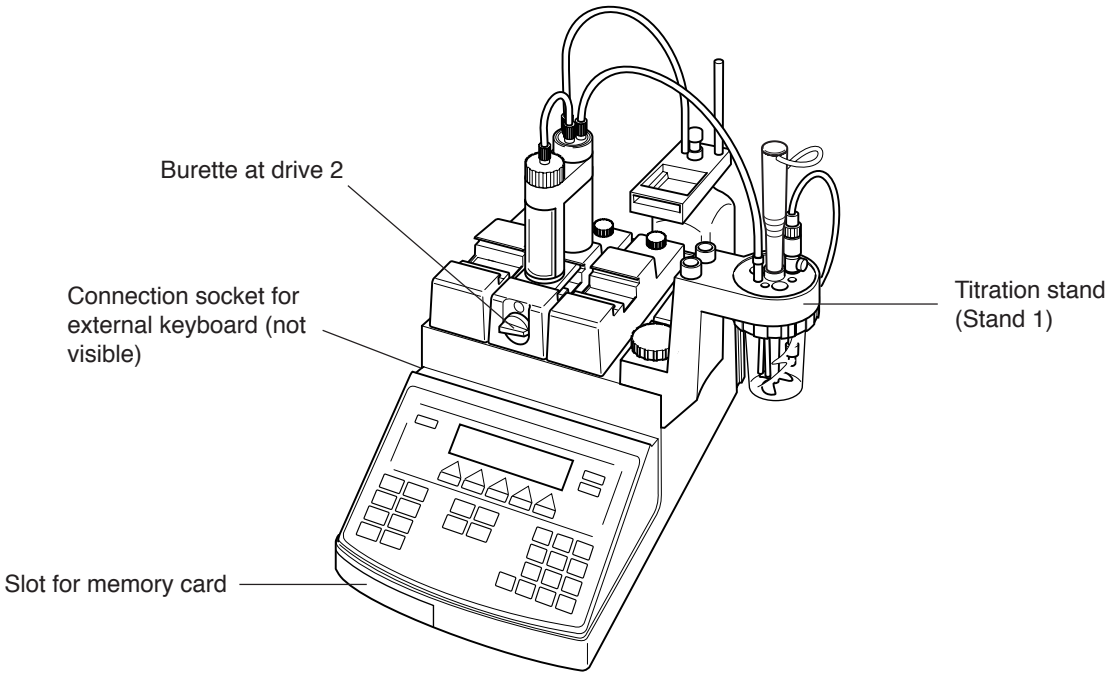
– Always test the titration vessel for firm seating in the titration head! If it falls off, you could injure yourself if working with toxic titrants and solvents or strong acids or bases.
– When using chemicals and solvents, comply with the instructions of the producer and the general lab safety rules!

Measures for operational safety

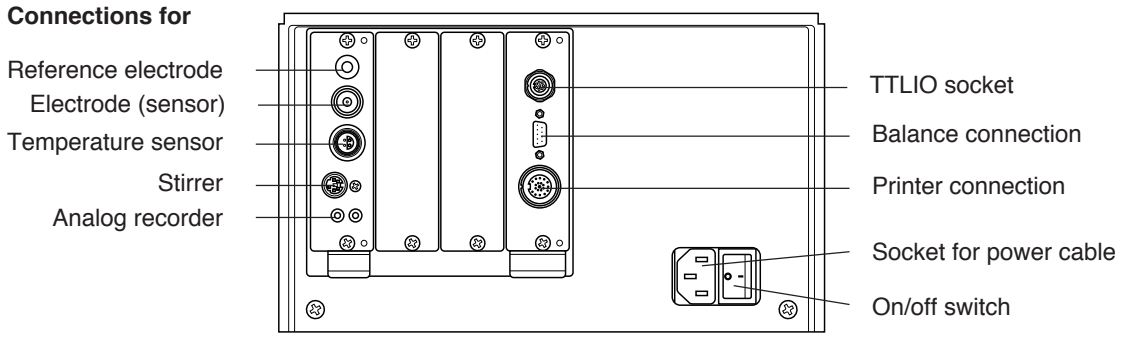
Caution

– Have the instrument serviced only by METTLER TOLEDO Service!
– Always wipe off splashed liquids immediately! The instrument is not water-proof.
– Exclude the following environmental influences:
  • powerful vibrations,
  • direct sunlight,
  • atmospheric humidity greater than 80%,
  • temperatures below 5 °C and above 40 °C,
  • powerful electric or magnetic fields!

3. The titrator



Rear view (the diagram refers to the standard equipment with pH and Centronics options)

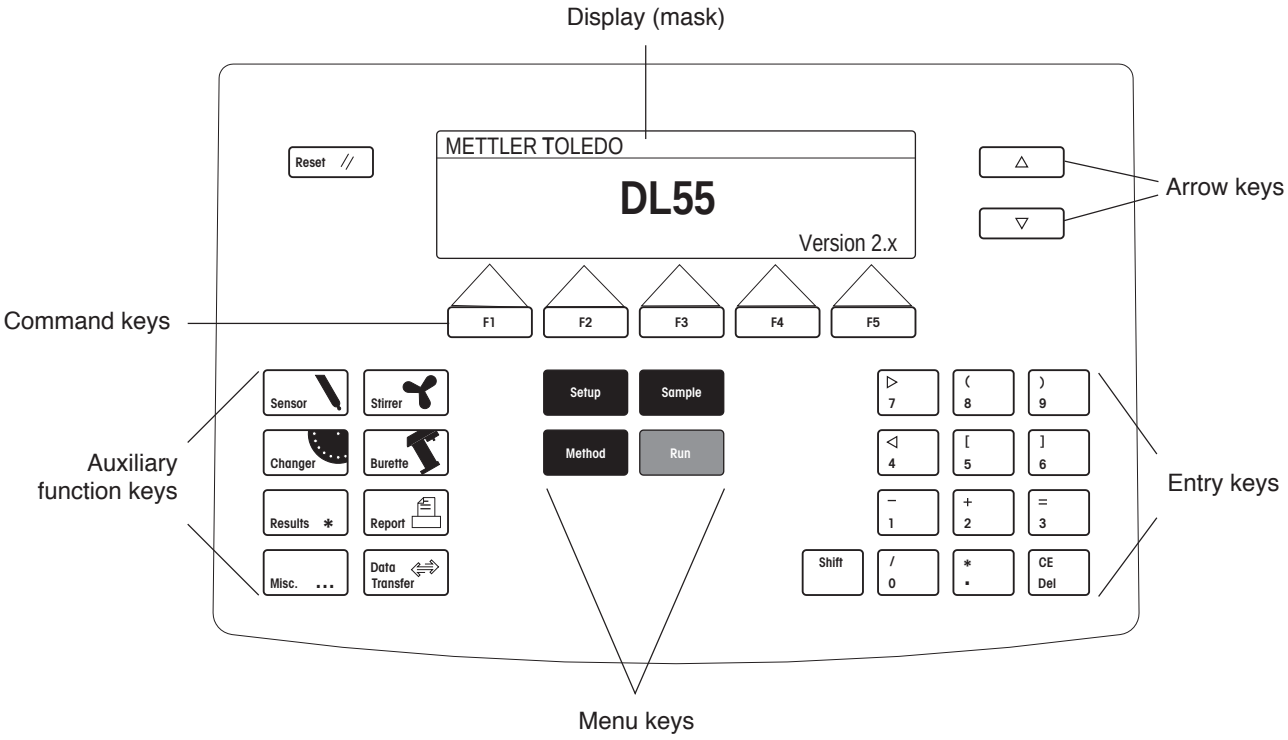


You have installed your titrator (see Section 10 of the Reference Handbook) and would like to start titrating immediately. Before you start, however, you must be familiar above all with the functions of the keys and be capable of following the display in the language of your choice: All texts in the titrator are available in English, German, French, Spanish and Italian.

Note: Please leave the titrator switched on during the first 48 hours to allow the built-in battery (rechargeable battery) to become fully charged. This battery supplies the internal clock with power when the titrator is switched off. If the titrator is not used for 4 months or more, you may well have to recharge the battery and reset the time.

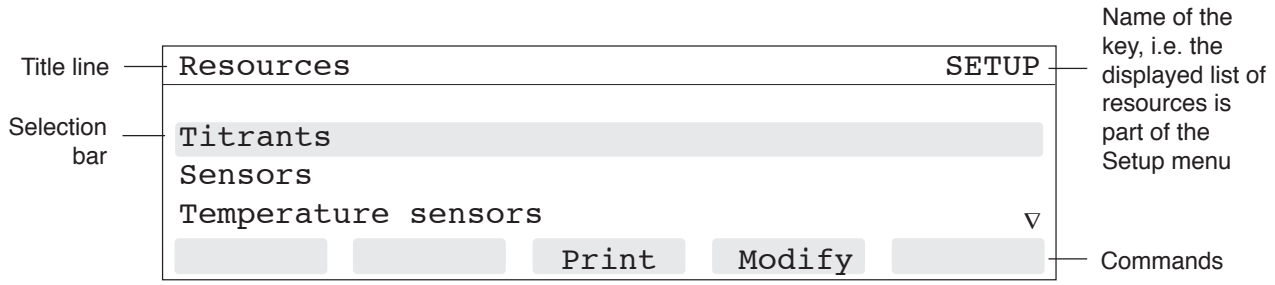
3.1 The operating concept

After you have switched on the titrator, it always performs a self-test before the display of the titrator name appears (example showing DL55):



All Menu and Auxiliary function keys can now be activated.

– Press, e.g. the Setup key:



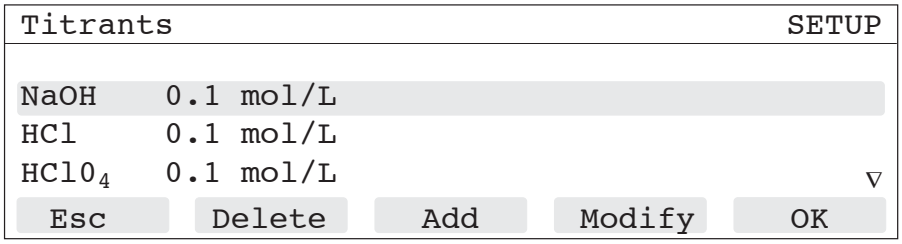
In addition to the Menu and Auxiliary function keys, you can now activate the ∇ key, Reset and Command keys <F3>, "Print" and <F4>, "Modify".

∇: The arrow in the display means that the list contains additional resources. When you press the ∇ key, the lines are scrolled upward, the selection bar is fixed. The commands you can execute always refer to the line that is **selected**.

Reset: The initial mask "METTLER TOLEDO..." reappears: Pressing Reset **aborts** analyses or other actions.

Print (press <F3>): The list of titrants is printed out (if a printer is attached and defined, see Section 2.7 of the Reference Handbook).

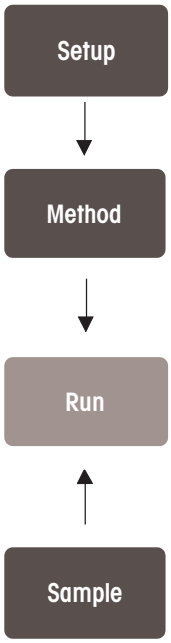
Modify (press <F4>): The list of titrants appears:



– Press the Reset key to display the initial mask again.

3.1.1 The Menu keys

For an analysis of a sample to be performed automatically, the required data must be stored. In the titrator, these data are assigned to sets of particular operations, the menus, and can be accessed with the Menu keys. It is the coordination between these menus that permits automatic analysis.



All resources such as titrants and electrodes needed for the titration are stored in the Setup menu.

The sample is determined using a method. All methods needed for the different analyses are stored in the Method menu. These methods have available all resources defined in the Setup menu.

The titration is performed in the Analysis menu. The method you call up determines the analysis sequence: Before the start of the titration, the titrator checks whether, e.g. the titrant defined in the method is also stored in the Setup menu.

The Sample menu is used for the entry of sample data, primarily for sample series, which can be performed automatically with a sample changer (sample data memory).

Note: Each menu is further subdivided, i.e. it has several sets of operations which, depending on the task, are further subdivided. In this Quick Guide and in the Reference Handbook, these submenus are described as menus, lists or masks.

3.1.2 The Auxiliary function keys

To measure the potential of a solution or rinse a burette, the required commands are also assigned to sets of operations (menus). As they are independent of a sample analysis, but can act in a supporting role, we refer to these as auxiliary functions. They are accessible under the corresponding keys.

You can measure the potential or the temperature of a solution and calibrate the temperature sensors.

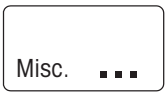
You can switch the stirrer on or off and change the stirring speed.

You can operate the sample changer manually.

You can rinse the burette, dispense a particular volume and titrate manually.

You can view the result list of analyzed samples and modify the statistical evaluation of a series.

You can print out additional reports.



You can, among other things, define the date and language and activate control inputs and outputs.

You can copy data from the titrator to the memory card or transfer data to a computer.

3.1.3 The Command keys

The commands initiated with <F1>...<F5> change in accordance with the selected function. The following commands require an explanation:

Esc: If you have made changes to the current menu or a submenu, they are discarded, in other words the original values/names are retained.

OK: The command is always used as a confirmation for what you
- have done, e.g. changed a value
- have viewed, e.g. checked a list for completeness
- wish to do, e.g. rinse a burette
- wish to adopt, e.g. a name or value from a selection menu.

Modify: When this command appears, <F4> can be used to
- show a submenu that can or must be modified
- show a selection menu from which you can or must adopt values or names
- change an existing parameter value or name directly.

Note: If a value (name) can be modified or entered only with the keyboard, this command does not appear.

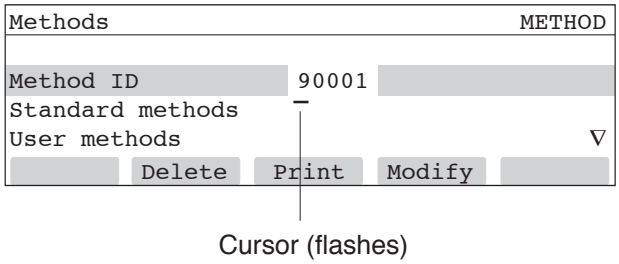
3.1.4 The Entry keys

Shift — With the Shift key you can activate the characters associated with the numeric keys.

CE Del — Del: You delete the number/symbol/letter at the cursor position.
CE: You clear the entry in a line.

Shift + ▷ 7 — You move the cursor to the right

Shift + ◁ 4 — or to the left.



Key combinations

With the Shift and Command keys (key combinations) you can execute the following commands:

Shift + Δ key jumps 4 display lines at a time upward.
Shift + ∇ key jumps 4 display lines at a time downward.

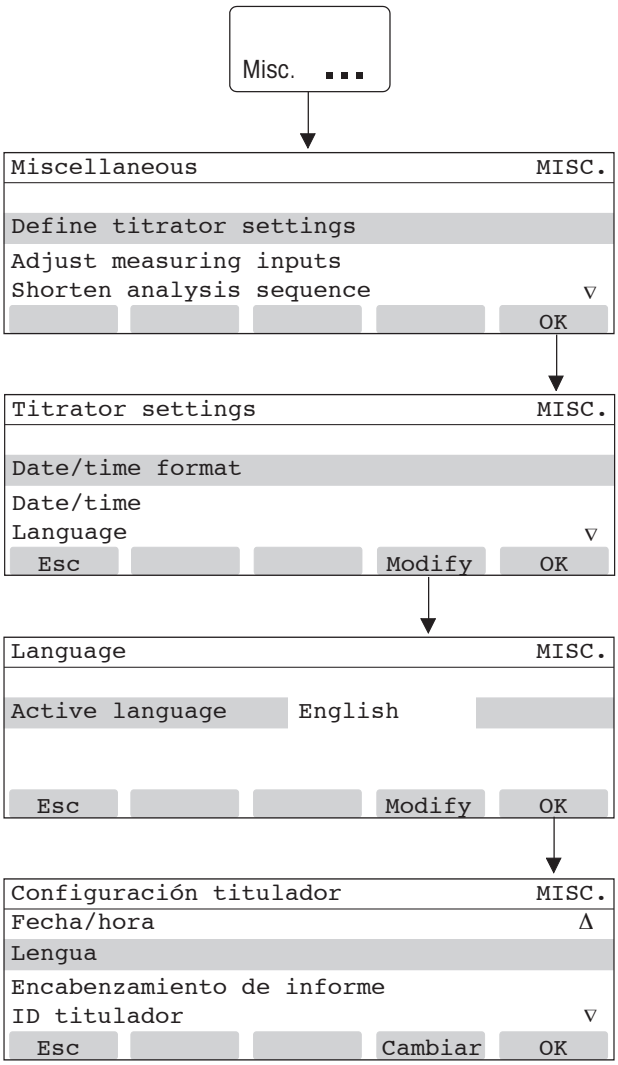
Shift + <F1> initiates a line feed on the printer.
Shift + <F2> initiates a form feed on the printer.
(See Reference Handbook, Section 2.7.1.1)

Shift + <F3>: the current display is printed out (copied).

Shift + <F4>: the system data are printed out (see Reference Handbook, Section 9).

3.2 Changing the language

Should you understand one of the available languages better than the one displayed, select this as follows:



– Press <F5>.

– Press the ∇ key twice to select "Language".

– Then press <F4>.

– Press <F4> repeatedly until, for example, "Español" appears.

With the two commands, **Esc** or **OK**, the mask "Titrator settings" reappears.

Esc: The change you have made is discarded, i.e. the texts remain in English.

OK: The change you have made is confirmed, i.e. the texts appear in Spanish.

4. How to perform your first titration

We will use a simple acid-base titration to explain the sequence of a titration method. The method for this acid determination is stored as the METTLER method entitled "Acid content" with the identification **90001**:

5 mL of an **HCl solution** (concentration = 0.1 mol/L)
are titrated with **NaOH** (concentration = 0.1 mol/L).

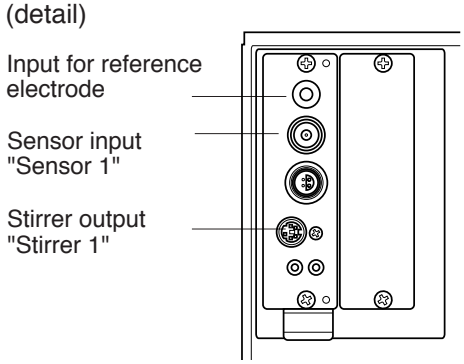
4.1 Preparations

– Prepare hydrochloric acid and sodium hydroxide with the above concentrations. The sodium hydroxide must be free from carbonate.
– Prepare the 10 mL burette for the sodium hydroxide and position on the second drive (see illustration in Section 3).
– To protect the sodium hydroxide against CO_2, place a drying tube on the burette holder of the NaOH bottle filled with, e.g. "sodium hydroxide on support".
– Fasten a titration beaker to the titration stand and insert the dispensing tube of the NaOH in one of the openings of the titration head.
– Fill the burette (see overleaf).

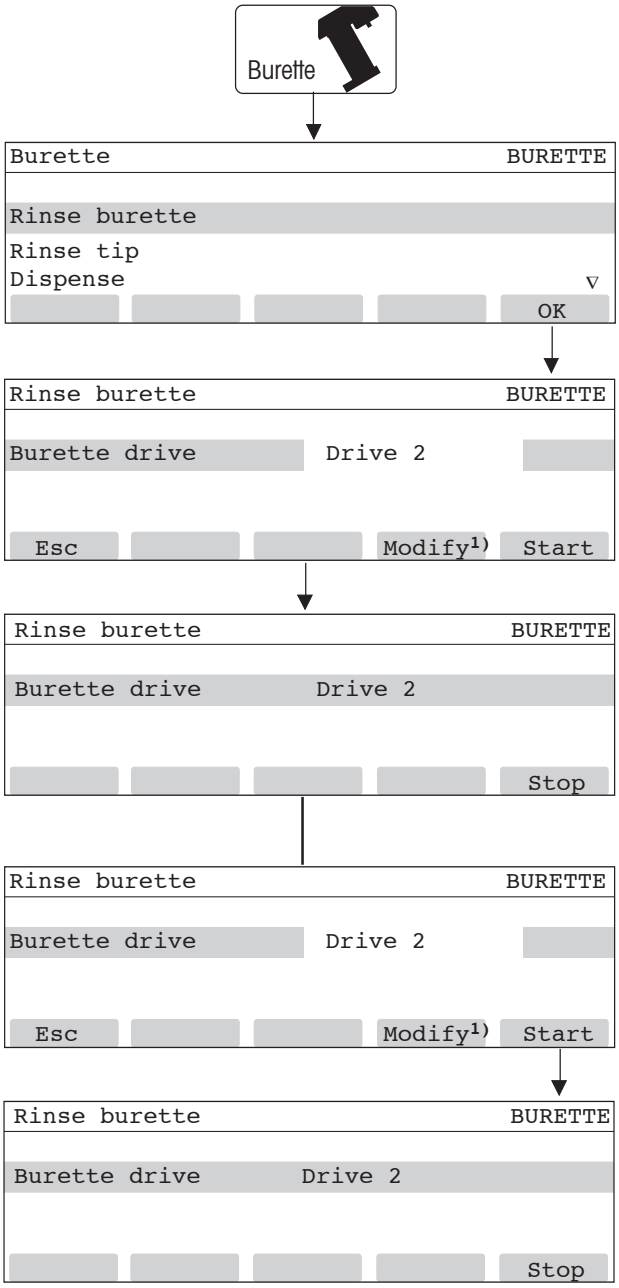
Wait until you have filled the burette before
– attaching a pH electrode with a Lemo cable to the sensor input and the stirrer to the output.
(In the Setup menu, inputs and outputs are defined with the names "Sensor 1" and "Stirrer 1")

The attachment of a printer and/or a balance is described in Section 2.7 of the Reference Handbook. For the actual analysis, there is no need to attach either.

Rear view of the titrator with pH option (detail)



4.1.1 Filling (rinsing) burette



– Press <F5>.

1) appears only with DL55 and DL58, as two burette drives can be installed.

– Press <F5>.

The piston of the burette is moved upward and the air expelled. An initial small amount of sodium hydroxide is siphoned in when the piston returns to its initial position. The display again shows:

– Repeat the rinsing operation twice more to ensure the burette is completely filled and the tubes are well rinsed.

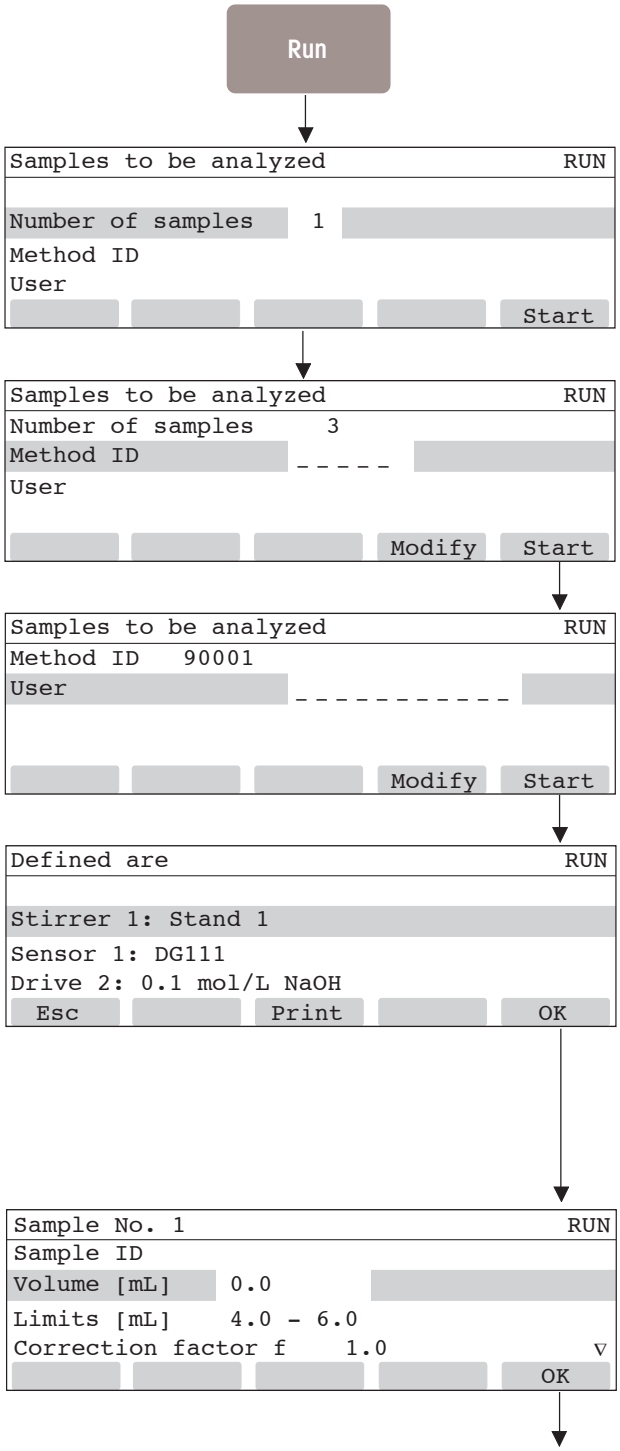
– Then remove the titration beaker and rinse the propeller stirrer and dispensing tube with deionized water.

– Press the Reset key: The initial mask appears.

You can stop the rinsing operation at any time with <F5>.

4.2 Performing titration method 90001

– Add approx. 50 mL deionized water to a titration beaker, pipette in 5 mL of the prepared hydrochloric acid and fasten the beaker to the titration head.



The following sequence is described for a series of three samples to allow the calculation of statistical data.

– Enter **3** for the number of samples.

– Press the ∇ key.

– Enter **90001** for the method identification and then press <F5> or the Run key.

(The identification of the method used to perform the last analysis is displayed.)

– Enter your **name** (if a keyboard is attached) and press <F5> or the Run key.

(The name used to perform the last analysis is stored as a suggestion.)

Using this information, you should check whether

- the stirrer of titration "Stand 1" is attached to output "Stirrer 1",
- a pH electrode is attached to the input "Sensor 1" (DG111 is the METTLER TOLEDO pH electrode),
- the burette with the NaOH is located at "Drive 2".

– Press <F5> or the Run key.

Sample ID: You can enter an identification for this sample.

The **volume** is the amount of sample that should be titrated.

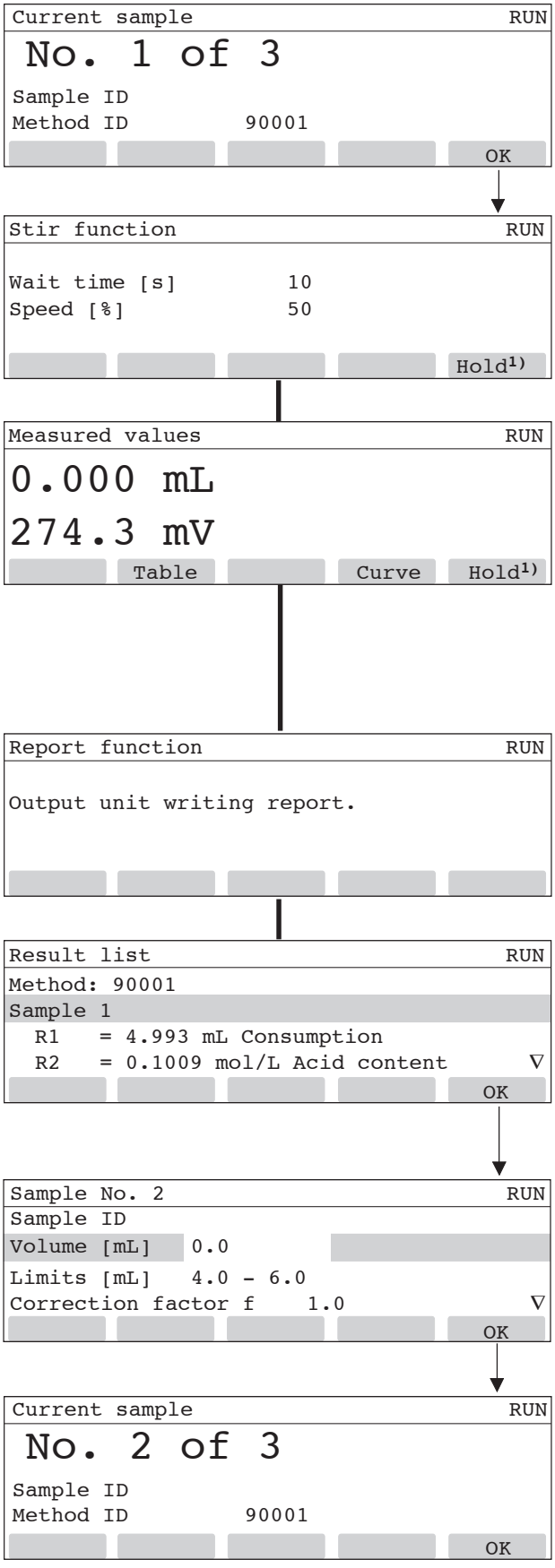
The limit values are defined in the method and indicate that there are lower and upper limits to the amount of sample which should not be violated.

Correction factor f: see Section 4.1 of the Reference Handbook.

You can enter the **temperature** (line not shown) of the solution to be titrated.

– Enter **5.0** as the volume of the first sample.

– Press <F5> or the Run key.



This is the prompt or the last opportunity to fasten the beaker with the first of the three defined samples to the titration head.

– Press <F5> or the Run key.

The titrator stirs for 10 seconds at a speed of 50% to mix the solution (the elapsed time is displayed).

1) appears only with DL55/DL58, i.e. you can press <F5> to interrupt the titration.

The titrator
- dispenses **2 mL** in 3 steps and then
- titrates up to the specified maximum volume of **7 mL.**

Pressing <F2> displays the table of measured values, pressing <F4> the titration curve "Potential vs Volume".

If you have defined and attached a printer, the titration curve and the table of measured values of this determination are printed out (see following pages). The mask appears during this time.

The results of the first sample are then displayed.

You can view the third result with the ∇ key.

The formulas for the calculations of these **3** results **R1**, **R2** and **R3** are defined in the method.

– Press <F5> or the Run key.

– Remove the titration beaker with the first sample.
– Rinse electrode, stirrer and dispensing tube with deionized water.
– Prepare the second sample and fasten the beaker to the titration head.
– Enter **5.0** for the volume of the second sample.
– Press <F5> or the Run key.

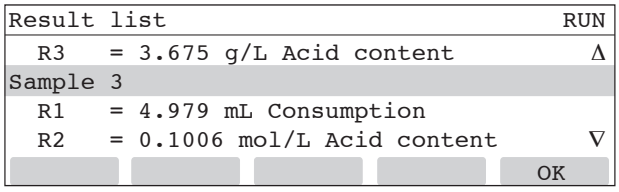
This is the prompt or the last opportunity to fasten the second sample beaker to the titration head.

– Press <F5> or the Run key: The sequence for the determination of the second and third samples is the same as for the first.

When the third sample has been analyzed and you have defined and attached a printer, the following are printed out:

- the results of all three samples
- the table of measured values for the third sample and
- the Potential-Volume curve for the third sample.

As soon as all data have been sent to the printer, the results of all samples and the statistical calculation for the NaOH consumption and HCl content appear in the display:



Use the arrow keys to view all results.

You can then press either <F5> or the Run key: The analysis menu remains active.

Report of all results of the titrated samples

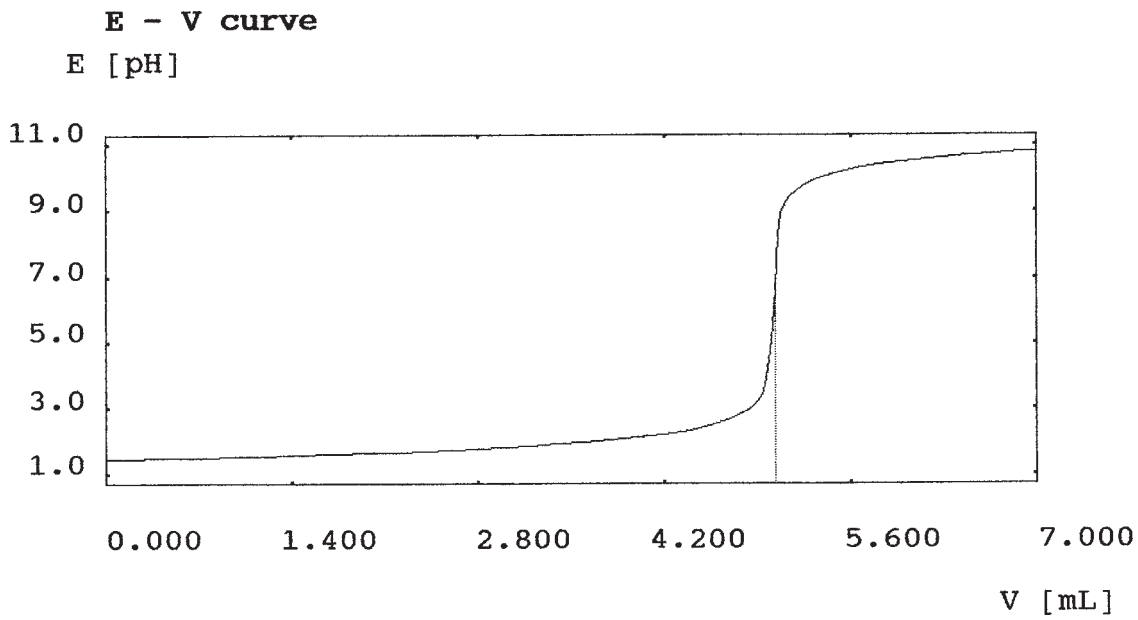
```
Method       90001 Acid content              01-Jul-1995  12:00
Measured     18-Oct-1996  11:48
User         C. De Caro

ALL RESULTS

 No.  ID     Sample size and results
  1   HCl          5.0       mL
                 R1 = 4.993    mL         Consumption
                 R2 = 0.1009   mol/L      Acid content
                 R3 = 3.680    g/L        Acid content
  2   HCl          5.0       mL
                 R1 = 4.987    mL         Consumption
                 R2 = 0.1008   mol/L      Acid content
                 R3 = 3.675    g/L        Acid content
  3   HCl          5.0       mL
                 R1 = 4.979    mL         Consumption
                 R2 = 0.1006   mol/L      Acid content
                 R3 = 3.669    g/L        Acid content

STATISTICS
  Number results       R1       n = 3
  Mean value                    x̄ = 4.986    mL     Consumption
  Standard deviation            s = 0.00738  mL     Consumption
  Rel. standard deviation    srel = 0.148    %

STATISTICS
  Number results       R3       n = 3
  Mean value                    x̄ = 3.675    g/L    Acid content
  Standard deviation            s = 0.00544  g/L    Acid content
  Rel. standard deviation    srel = 0.148    %
```

Report of the table of measured values of the last titrated sample

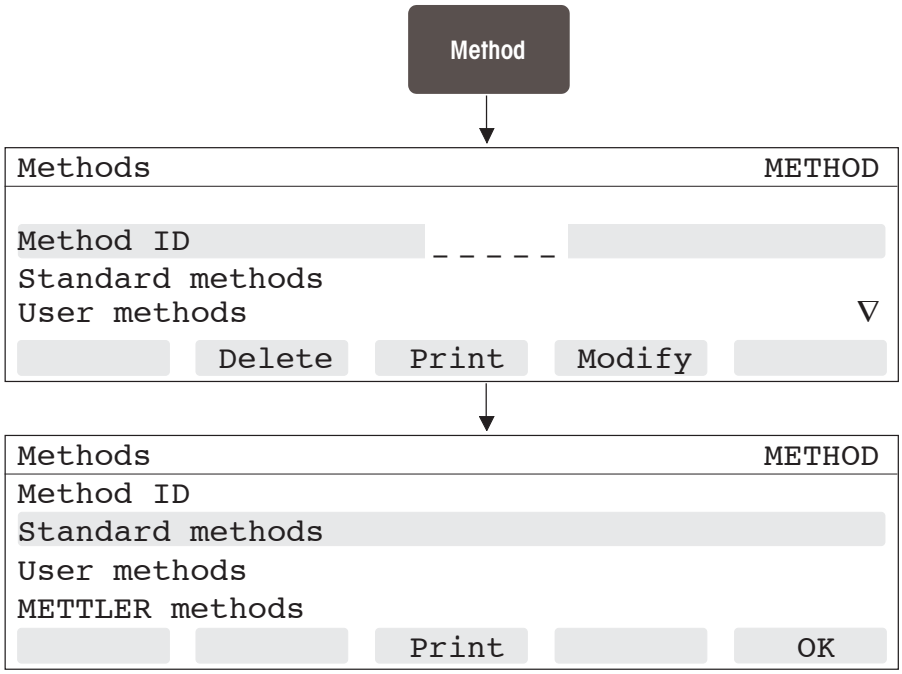
Method 90001 Acid content 01-Jul-1995 12:00
Measured 18-Oct-1996 11:48
User C. De Carlo

	Volume mL	Increment mL	Signal pH	Change pH	1st deriv. pH/mL	Time min:s
ET1	0.0000		2.176			0:03
	1.1420	1.1420	2.236	0.059	0.052	0:10
	1.7130	0.5710	2.287	0.052	0.091	0:15
ET2	2.0000	0.2870	2.318	0.031	0.107	0:19
	2.2000	0.2000	2.345	0.026	0.132	0:23
	2.4000	0.2000	2.370	0.025	0.127	0:26
	2.6000	0.2000	2.399	0.029	0.143	0:30
	2.8000	0.2000	2.432	0.033	0.165	0:34
	3.0000	0.2000	2.467	0.035	0.176	0:38
	3.2000	0.2000	2.506	0.040	0.198	0:42
	3.4000	0.2000	2.554	0.047	0.237	0:47
	3.6000	0.2000	2.608	0.054	0.270	0:52
	3.8000	0.2000	2.672	0.065	0.325	0:57
	4.0000	0.2000	2.748	0.076	0.380	1:02
	4.2000	0.2000	2.843	0.095	0.473	1:08
	4.4000	0.2000	2.971	0.128	0.638	1:14
	4.5690	0.1690	3.121	0.151	0.892	1:21
	4.6870	0.1180	3.275	0.154	1.305	1:28
	4.7660	0.0790	3.426	0.151	1.908	1:34
	4.8200	0.0540	3.564	0.138	2.547	1:40
	4.8620	0.0420	3.722	0.158	3.772	1:46
	4.8890	0.0270	3.864	0.142	5.256	1:53
	4.9090	0.0200	3.982	0.118	5.886	1:59
	4.9290	0.0200	4.204	0.222	11.111	2:06
	4.9490	0.0200	4.711	0.507	25.358	2:15
	4.9690	0.0200	6.044	1.333	66.667	2:24
EQP1	4.9890	0.0200	8.753	2.708	135.423	2:47
	5.0090	0.0200	9.356	0.603	30.143	2:59
	5.0290	0.0200	9.581	0.226	11.276	3:07
	5.0490	0.0200	9.746	0.165	8.251	3:14
	5.0750	0.0260	9.911	0.165	6.347	3:21
	5.1050	0.0300	10.031	0.120	3.997	3:27
	5.1650	0.0600	10.208	0.177	2.952	3:34
	5.2360	0.0710	10.351	0.143	2.014	3:40
	5.3620	0.1260	10.525	0.174	1.380	3:47
	5.5440	0.1820	10.691	0.166	0.913	3:55
	5.7440	0.2000	10.820	0.129	0.644	4:01
	5.9440	0.2000	10.918	0.098	0.490	4:07
	6.1440	0.2000	10.998	0.080	0.402	4:13
	6.3440	0.2000	11.063	0.065	0.325	4:18
	6.5440	0.2000	11.120	0.057	0.286	4:23
	6.7440	0.2000	11.171	0.051	0.253	4:28
	6.9440	0.2000	11.216	0.045	0.226	4:32
	7.0000	0.0560	11.234	0.018	0.314	4:36

Report of the titration curve



5. The method concept



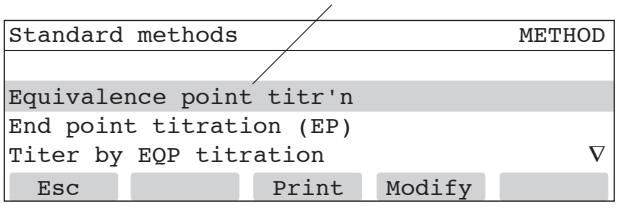
METTLER and standard methods are stored in the titrator in the factory. You can adapt the methods of both groups to meet the requirements of your analyses. The modified methods are always stored as **User methods**.

Standard methods

These methods have been entered by us and can not be recalled for an analysis directly.

- They do not have a method ID(entification) needed by the Analysis menu.
- The method parameters are stored with default values which you first have to modify in accordance with your application.

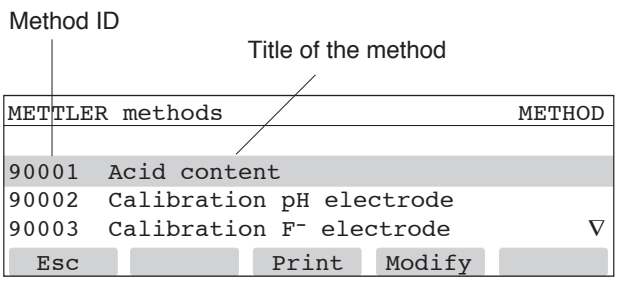
– Confirm "Standard methods" with OK:



METTLER methods

These methods have been developed by us for particular applications to allow the appropriate analyses to be run immediately: You can recall each METTLER method using the method ID(entification) in the Analysis menu, e.g. method 90001 (see Section 4.2).

– Use the ∇ key to select "METTLER methods" and press <F5>:

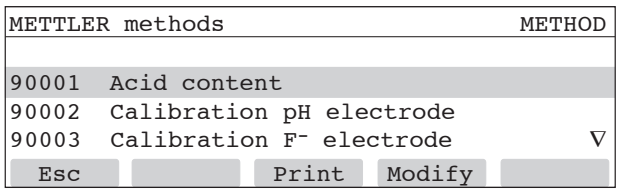


Each method comprises several substeps which we refer to as **Functions**. These functions are executed in succession in the analysis (see Sections 6.2 and 7.3).

Each function comprises **Parameters**, which define the actual task of the function. You can modify these parameters.

5.1 METTLER methods

The DL50 has 4, the DL53 20 and the DL55 and DL58 30 METTLER methods stored. All these methods are described in the enclosed brochure "30 Selected Applications for METTLER TOLEDO Titrators DL50/DL53/DL55/DL58".



Print: Method 90001 is printed out with its functions and parameters.

Modify: The functions of method 90001 appear.

Press <F1>, Esc: The method groups reappear.

5.2 Standard methods

In all four titrators, 21 standard methods are stored under a title. Number and sequence of the functions are given for each method.

1

Equivalence point titr'n
Title
Sample
Stir
EQP titration
Calculation
Calculation
Calculation
Report

2

End point titration (EP)
Title
Sample
Stir
EP titration
Calculation
Calculation
Calculation
Report

3

Titer by EQP titration
Title
Sample
Stir
EQP titration
Calculation
Titer
Report

4

Titer by EP titration
Title
Sample
Stir
EP titration
Calculation
Titer
Report

5

Sensor calibration
Title
Sample
Stir
Measure
Calculation
Calibration
Report

6

Sensor measurement
Title
Sample
Stir
Measure
Calculation
Calculation
Report

7

Learn titration
Title
Sample
Stir
Learn titration
Calculation
Calculation
Calculation
Report

8

Stat titration
Title
Sample
Stir
pH/mV-stat
Calculation
Calculation
Calculation
Report

9

Blank by EQP titration
Title
Sample
Stir
EQP titration
Calculation
Calculation
Auxiliary value
Report

10

Blank by EP titration
Title
Sample
Stir
EP titration
Calculation
Calculation
Auxiliary value
Report

11

2 Step titration (EQP)
Title
Sample
Dispense
Stir
EQP titration
Calculation
Calculation
Report
Dispense
Stir
EQP titration
Calculation
Calculation
Report

12

2 Step titration (EP)
Title
Sample
Dispense
Stir
EP titration
Calculation
Calculation
Report
Dispense
Stir
EP titration
Calculation
Calculation
Report

13

EQP titr'n with dispense
Title
Sample
Dispense
Stir
EQP titration
Calculation
Calculation
Calculation
Report

14

EP titration with dispense
Title
Sample
Dispense
Stir
EP titration
Calculation
Calculation
Calculation
Report

15

Combined EP/EQP titr'n
Title
Sample
Stir
EP titration
Calculation
Calculation
Report
EQP titration
Calculation
Calculation
Report

16

EQP titration (Ipol/Upol)
Title
Sample
Stir
EQP titration (Ipol/Upol)
Calculation
Calculation
Calculation
Report

17

Titer (EQP Ipol/Upol)
Title
Sample
Stir
EQP titration (Ipol/Upol)
Calculation
Titer
Report

18

Blank (EQP Ipol/Upol)
Title
Sample
Stir
EQP titration (Ipol/Upol)
Calculation
Calculation
Auxiliary value
Report

19

EP titration (Ipol/Upol)
Title
Sample
Stir
EP titration (Ipol/Upol)
Calculation
Calculation
Calculation
Report

20

Titer (EP Ipol/Upol)
Title
Sample
Stir
EP titration (Ipol/Upol)
Calculation
Titer
Report

21

Blank (EP Ipol/Upol)
Title
Sample
Stir
EP titration (Ipol/Upol)
Calculation
Calculation
Auxiliary value
Report

Note: You can perform the standard methods 16 through 21 only if you have installed a KF option (see Section 10.5 of the Reference Handbook).

Application examples of the standard methods	
1 Equivalence point titration (EQP)	For titrations to the equivalence point, e.g. acid/base, redox, argentometric and complexometric titrations
2 End point titration (EP)	For titrations to the end point, e.g. acid/basetitrations
3 Titer by EQP titration	Titer determination with an equivalence point titration, e.g. acid/base, redox, argentometric and complexometric titrations
4 Titer by EP titration	Titer determination with an end point titration
5 Sensor calibration	Calibration of pH and ion selective electrodes
6 Sensor measurement	Concentration measurement with ion selective electrodes
7 Learn titration	(see Section 3.3.8 of the Reference Handbook)

8	Stat titration	pH- or mV-stating, e.g. solution and enzyme kinetics
9	Blank by EQP titration	Blank value determinations with an equivalence point titration, e.g. blank value of a solvent
10	Blank by EP titration	Blank value determinations with an end point titration, e.g. standard titrations TAN/TBN, polymer solutions
11	2 Step titration (EQP)	Two equivalence point titrations in the same sample, e.g. acid mixture (HCl/H_3BO_3)
12	2 Step titration (EP)	Two end point titrations in the same sample, e.g. lactone and formol number
13	EQP titration with dispense	Equivalence point titration with preceding dispensing of a reagent or titrant for the performance of a back titration, e.g. iodimetric titrations with sodium thiosulfate (dispensing of KI solution), photometric determination of the water hardness (dispensing of borate buffer)
14	EP titration with dispense	End point titration with preceding dispensing of a reagent or titrant for the performance of a back titration, e.g. carbonate determination
15	Combined EP/EQP titration	Equivalence point titration with preceding adjustment of a pH or mV value, e.g. calcium determination in water (adjustment to pH 12)
16	EQP titration (Ipol/Upol)	Vitamin C determination in foods and beverages (with voltametric or amperometric indication)
17	Titer by EQP titration (Ipol/Upol)	Titer determination of DPI (2,6-dichlorophenol indophenol)
18	Blank by EQP titration (Ipol/Upol)	Blank value determinations with an equivalence point titration, e.g. blank value of a solvent
19	EP titration (Ipol/Upol)	Bromine number in mineral oils; SO_2 content in wine; Karl Fischer titration
20	Titer by EP titration (Ipol/Upol)	Titer determination of I_2 solution and Karl Fischer titrant
21	Blank by EP titration (Ipol/Upol)	Blank value determination of the solvent for the bromine number; drift determination for Karl Fischer titration

5.3 Generating a method

The METTLER and standard methods are composed of the 16 functions listed below. Not all functions are needed for every method; some occur more than once.

Title _ _ _ _ _ _ _ _ _ _ _ _	identifies the method
Sample _ _ _ _ _ _ _ _ _ _	defines data for the sample determination
Stir _ _ _ _ _ _ _ _ _ _ _ _	stirs at defined speed and for defined time
Measure _ _ _ _ _ _ _ _ _ _	measures the potential of a solution
Dispense _ _ _ _ _ _ _ _ _ _	dispenses a defined volume of a titrant
EQP titration _ _ _ _ _ _ _	titrates to an equivalence point
EP titration _ _ _ _ _ _ _ _	titrates to an end point
Learn titration _ _ _ _ _ _ _	finds parameters for an equivalence point titration
EQP titration (Ipol/Upol) _ _	titrates to an equivalence point by means of polarized electrodes
EP titration (Ipol/Upol) _ _ _	titrates to an end point by means of polarized electrodes
pH/mV-stat _ _ _ _ _ _ _ _ _	keeps a defined potential constant (pH-stating)
Calculation _ _ _ _ _ _ _ _ _	calculates results of the analyzed samples
Calibration _ _ _ _ _ _ _ _ _	calibrates electrodes and calculates their calibration data
Titer _ _ _ _ _ _ _ _ _ _ _ _	assigns the result of a titer determination to the titrant
Auxiliary value _ _ _ _ _ _ _	assigns the result of a titration to a value which can be incorporated in the calculations
Report _ _ _ _ _ _ _ _ _ _ _	determines the printout of results, tables and curves

Standard methods

You can modify the parameters of all functions of a standard method. Preset parameters of the *Sample* function must always be modified, whereas those of the *EQP/EP titration* functions are suitable for many analyses.

To save the method, you must **enter an identification** under the *Title* function.

After being saved, the methods are available as user methods. You will find an example in Section 7.2.1: "*Modifying a standard method".*

METTLER methods

You can modify the parameters of all functions. To save these, you must **change their identification** under the *Title* function.

After being saved, the method is available as a user method.

6. How to calibrate a pH electrode

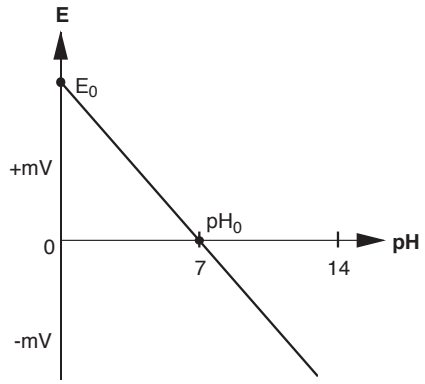
You can perform a calibration with the aid of METTLER method 90002. Three buffer solutions (pH: 4.01, 7.00 and 10.00) from METTLER TOLEDO are defined in the method. If you have other buffer solutions, you must modify the buffer type and the pH values (refer to Section 3.3.13 of the Reference Handbook).

6.1 Zero point and slope

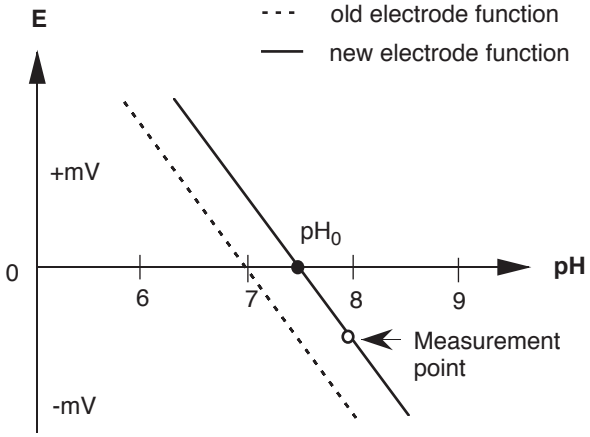
The calibration parameters of a pH electrode are the zero point pH_0 (pH value at a potential of 0 mV) and the slope.
The theoretical values of a pH electrode are stored in the titrator:

- Zero point = 7.0 [pH] and
- Slope = -59.16 [mV/pH].

You perform a calibration to obtain correct values for your electrode. The theoretical values are then automatically overwritten by the calibration data determined by measurement.

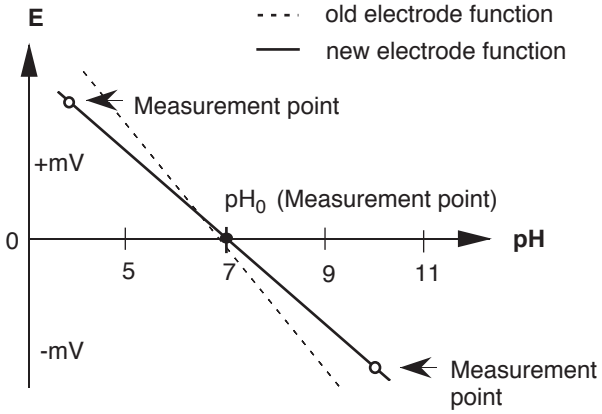


If you calibrate the electrode with just one buffer solution, only the electrode zero point is recalculated.



In a two-point or multipoint calibration, zero point and slope are calculated by linear regression.

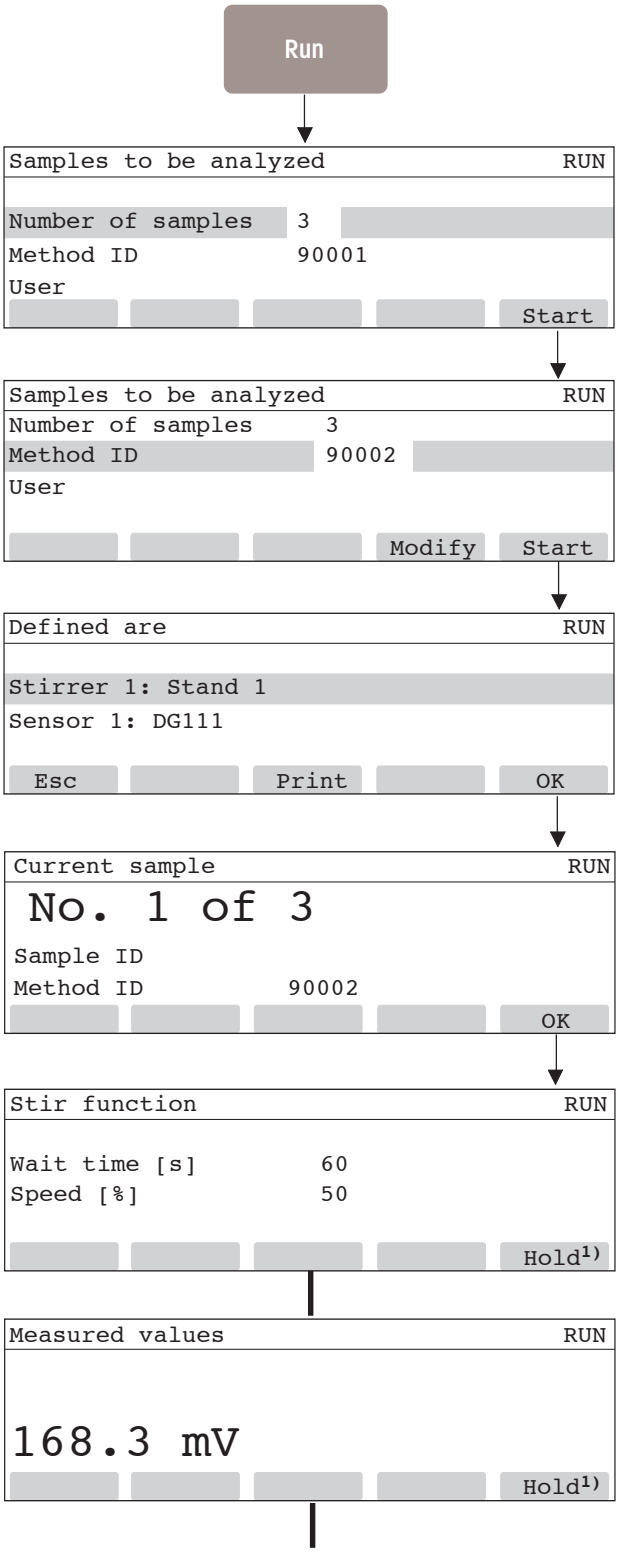
Note
The slope of an electrode is temperature dependent. The temperature is not considered in the following sequence. See Section 3.3.13 of the Reference Handbook.



6.2 Calibration

– Prepare the buffer solutions and three titration beakers.

The pH electrode must be attached to the input "Sensor 1", the stirrer to the output "Stirrer 1".



– Enter **3** for the number of samples (corresponds to the 3 buffer solutions).

– Press the ∇ key.

– Enter **90002** for the method identification and press <F5> or the Run key.

– Press the ∇ key to enter your name (if a keyboard is attached).

Using this information, check whether

- the stirrer is attached to titration "Stand 1" at output "Stirrer 1"
- a pH electrode is attached to input "Sensor 1" (DG111 is the METTLER TOLEDO pH electrode; you can change its name, see Section 2.2 of the Reference Handbook).

– Press <F5> or the Run key.

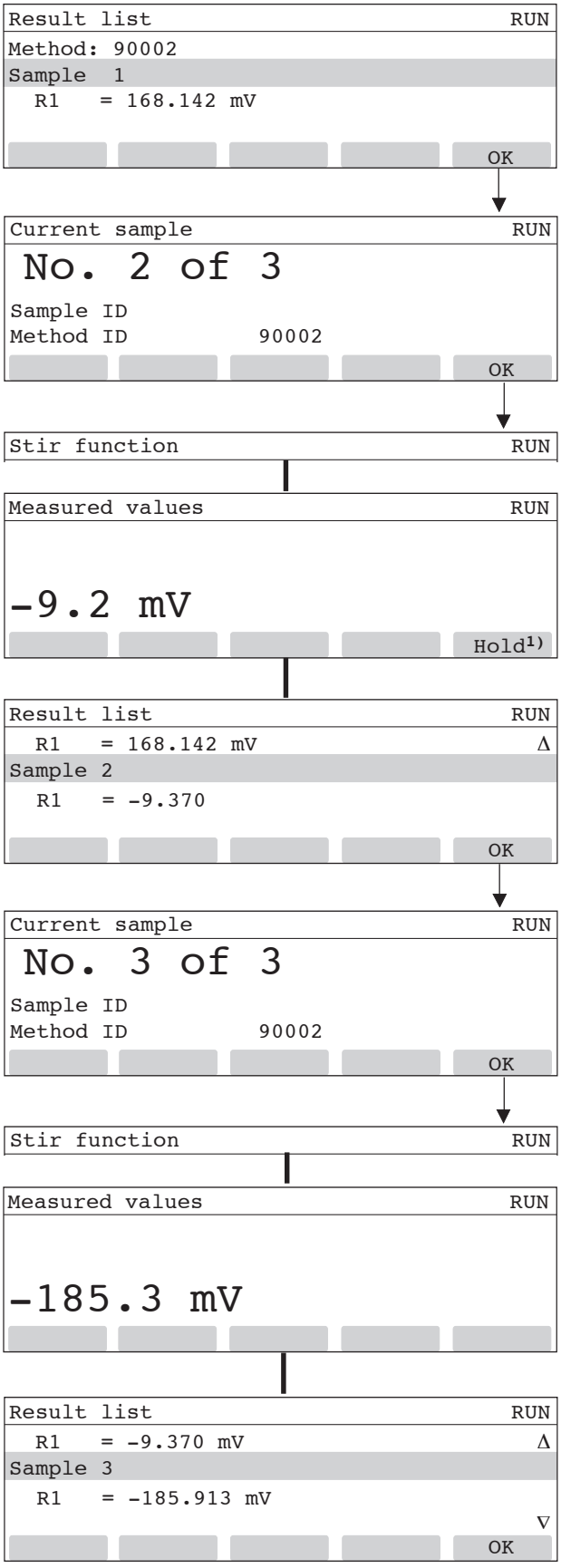
– Add **50 mL** of the buffer solution **"pH 4.01"** to a titration beaker and fasten to the titration head.

– Press <F5> or the Run key.

The titrator executes the **Stir** function: The solution is stirred for 60 seconds to condition the electrode (the elapsed time is displayed).

1) appears only with DL55and DL58, i.e. you can press <F5> to interrupt the measurement.

The titrator executes the **Measure** function and displays the changing potential value of the buffer solution. As soon as the measured value is stable, it is acquired.



The measured potential of the first buffer solution is displayed as the result.

– Press <F5> or the Run key.

– Remove the titration beaker.
– Rinse electrode and stirrer with deion. H_2O.
– Add **50 mL** of the buffer solution **"pH 7.00"** to a titration beaker and fasten to the titration head.

– Press <F5> or the Run key.

The **Stir** function reappears followed by the **Measure** function. As soon as the measured value is stable, it is acquired.

In addition to the result of the first measurement, the measured potential of the second buffer solution is displayed. (The number 1 of the result **R** is an index and refers to the number of calculation functions.)

– Press <F5> or the Run key.

– Remove the titration beaker.
– Rinse the electrode with deion. H_2O.
– Add **50 mL** of buffer solution **"pH 10.00"** to a titration beaker and fasten to the titration head.

– Press <F5> or the Run key.

The **Stir** function reappears followed by the **Measure** function. As soon as the measured value is stable, it is acquired.

If you have defined and attached a printer, the results are printed out before the result list with all three measured values is displayed. During this time, "Output unit writing report" appears".

If you scroll the display with the ∇ key, the calculated calibration data appear (see overleaf).

```
Result list                                  RUN
  R1    = -185.913 mV                          Δ

pH0   = 6.850
S     = -59.11 mV/pH
                                            OK
```

The two values of zero point and slope are stored as parameters of the pH electrode "DG111", i.e. the old values are automatically overwritten (see Section 2.2 of the Reference Handbook).

```
Method          90002          Calibration pH electrode
                Version        01-Jul-1995      12:00

Title
   Method ID ..... 90002
   Title ..... Calibration pH electrode
   Date/time ..... 01-Jul-1995
Sample
   Sample ID .....
   Entry type ..... Fixed volume
      Volume [mL] ..... 50.0
   Molar mass M ..... 100
   Equivalent number z ..... 1
   Titration stand ..... Stand 1
   Temperature sensor ..... Manual
Stir
   Speed [%] ..... 50
   Time [s] ..... 60
Measure
   Sensor ..... DG111
   Unit of meas. ..... mV
   ΔE [mV] ..... 0.5
   Δt [s] ..... 1.0
   t(min) mode ..... Fix
      t(min) [s] ..... 3.0
   t(max) [s] ..... 30.0
Calculation
   Formula ..... R1=E
   Constant .....
   Decimal places ..... 3
   Result unit ..... mV
   Result name .....
   Statistics ..... No
Calibration
   Sensor ..... DG111
   Buffer type ..... pH (METTLER TOLEDO)
     First buffer ..... pH 4.01
     Second buffer ..... pH 7.00
     Third buffer ..... pH 10.00
     Fourth buffer ..... pH 2.00
     Fifth buffer ..... pH 2.00
     Sixth buffer ..... pH 2.00
     Seventh buffer ..... pH 2.00
     Eighth buffer ..... pH 2.00
   Result R ..... 1
   Minimum slope [mV/unit] ..... -55.0
   Maximum slope [mV/unit] ..... -65.0
Report
   Output unit ..... Printer
   Results ..... No
   All results ..... Yes
   Raw results ..... No
   Table of measured values ..... No
   Sample data ..... No
   E – V curve ..... No
   etc.
```

Sample function: Only the parameters "Titration stand" and "Temperature sensor" are important in the calibration (see Section 3.3.2 of the Reference Handbook). The definition of the vol- ume indicates that approx. 50 mL buffer solution should be used.

Stirring speed and the time needed to condition the electrode are defined.

The parameters of the **Measure** function are responsible for the measured value acquisition of the electrode potential. This measured value is called **E**.

Calculation function: The result is the measured potential **E** of the buffer solution in question. All three measured values are stored.

Calibration function: The three measured values of the **Measure** function are assigned to the standard concentrations of the three buffer solutions, the zero point and the slope are calculated by linear regression.

In the **Report** function, only "All results" are defined for a printout.

7. How to determine the titer of an NaOH solution

You can use the standard method "Titer by EQP titration" (EQP is the abbreviation for equivalence point) to determine the titer of a sodium hydroxide solution of concentration 0.1 mol/L without changing the preset parameters. However, you have to enter a method identification in order to recall it into the Analysis menu (see Section 5).

7.1 Titer t

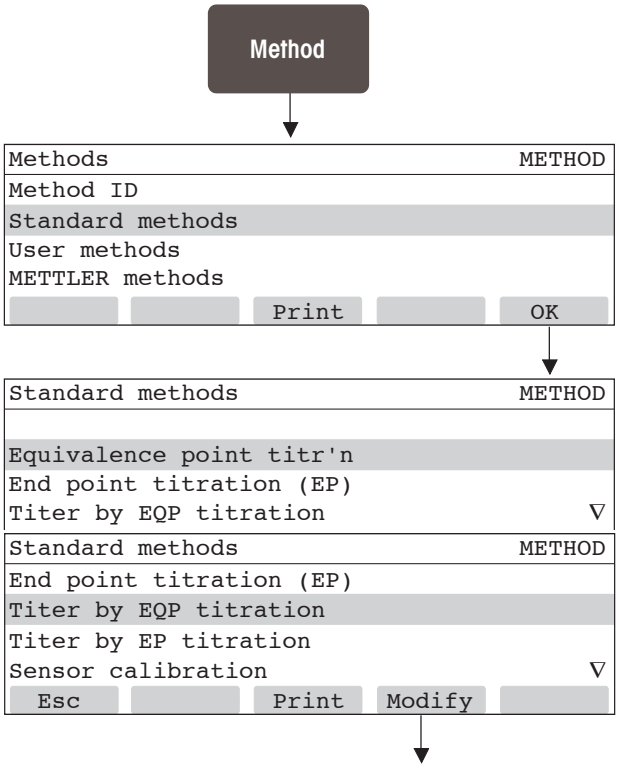
The titer of a titrant is the quotient of the actual concentration and the nominal concentration.

$$t = \frac{c_{actual}}{c_{nominal}}$$

If, for example, you prepare a sodium hydroxide solution of concentration 0.1 mol/L and an error occurs on dilution, the accuracy required for correct content determinations is not achieved. You thus determine the actual concentration with the aid of primary standards.
In the titrator, the titer is stored with the default value of **1.0**. It is automatically overwritten after the determination by the new value (see Section 2.1 of the Reference Handbook).

7.2 Preparations

7.2.1 Modifying a standard method

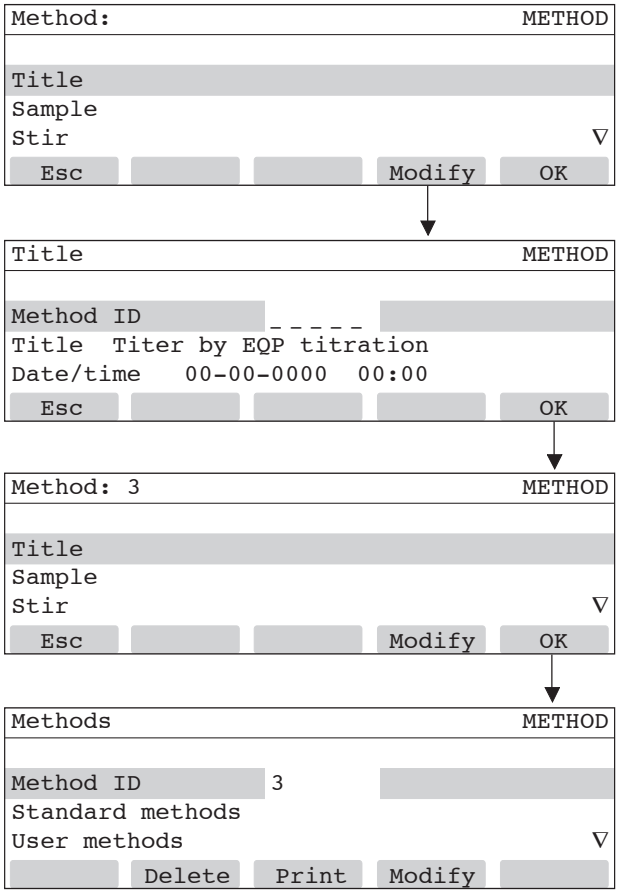


You have pressed the ∇ key to select "Standard methods".

– Press <F5>.

The standard methods appear.

– Select "Titer by EQP titration" using the ∇ key and press <F4>.



The functions of the method appear.

– Press <F4>.

The parameters of the **Title** function appear.

– Enter a number as identification, e.g. **3** and confirm with OK.

The list of functions reappears, this time with identification **3** for the method.

– Press <F5>.

Method **3** is stored and is now a user method. (You can select **User methods** to ensure that it is stored in this group with the title "Titer by EQP titration".)

7.2.2 Titrant and primary standard

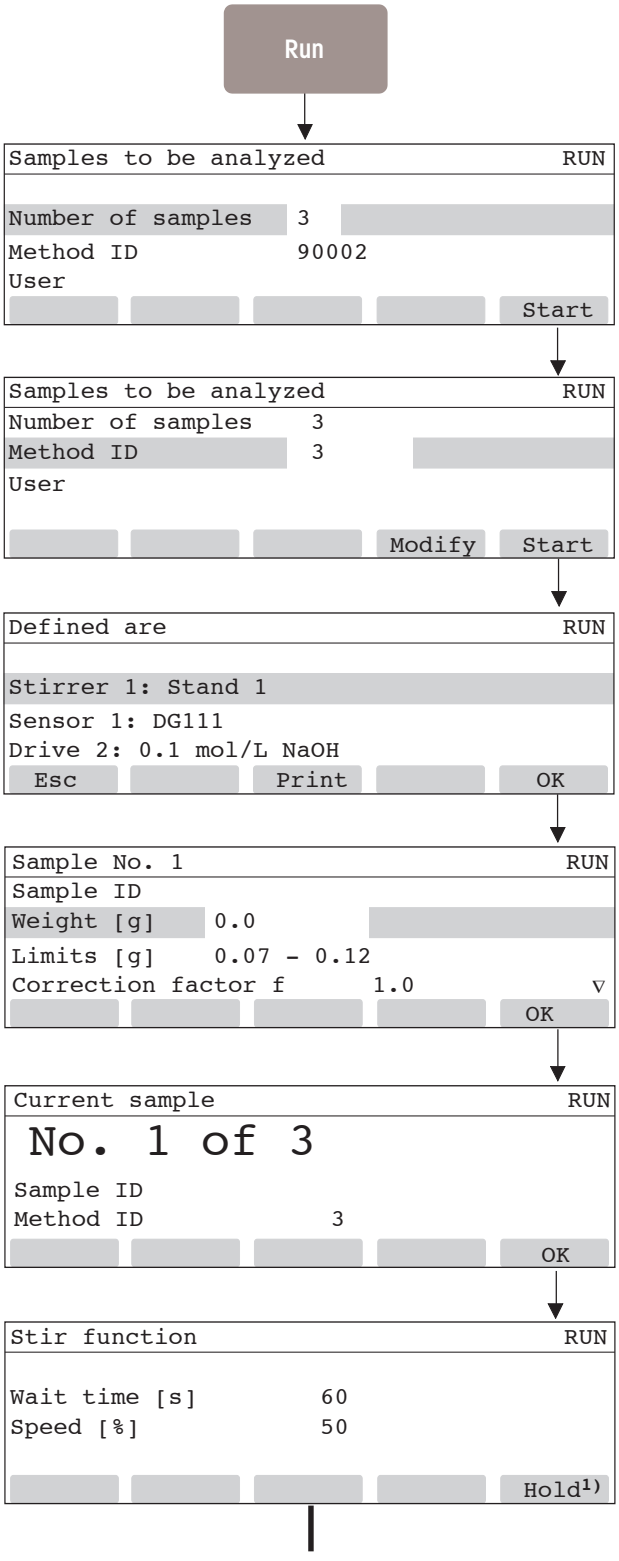
– Fasten an empty titration beaker to the titration stand and insert the dispensing tube for sodium hydroxide in one of the openings of the titration head.
– Rinse the tubes to ensure there are no air bubbles and remove the titration beaker.

A pH electrode must be attached to the input "Sensor 1", the stirrer to the output "Stirrer 1".
– Insert the electrode in the opening of the titration head – opposite the dispensing tube.

Potassium hydrogen phthalate (KHP) is used as the primary standard for the titer determination of the sodium hydroxide.
– Prepare three glass titration beakers and weigh between **0.07** and **0.12** g KHP into each. Note the weight of each sample. (We recommend glass beakers to avoid weighing errors due to electrostatic effects.)
– Add approx. 50 mL deionized water and fasten the first beaker to the titration head.

Note: If you have defined a balance and attached it to the titrator, the weight of each sample will be transferred automatically (see Sections 2.7.2 and 4.2 of the Reference Handbook).

7.3 Determining the titer



– Enter **3** for the number of samples.
– Press the ∇ key.

– Enter **3** for the method identification and press <F5> or the Run key.
– Press the ∇ key to enter your name (if a keyboard is attached).

Using this information, you should check whether you have attached or installed the stirrer, electrode and sodium hydroxide solution in accordance with these settings.

– Press <F5> or the Run key.

– Enter the weight for the first sample, e.g. **0.08451.**

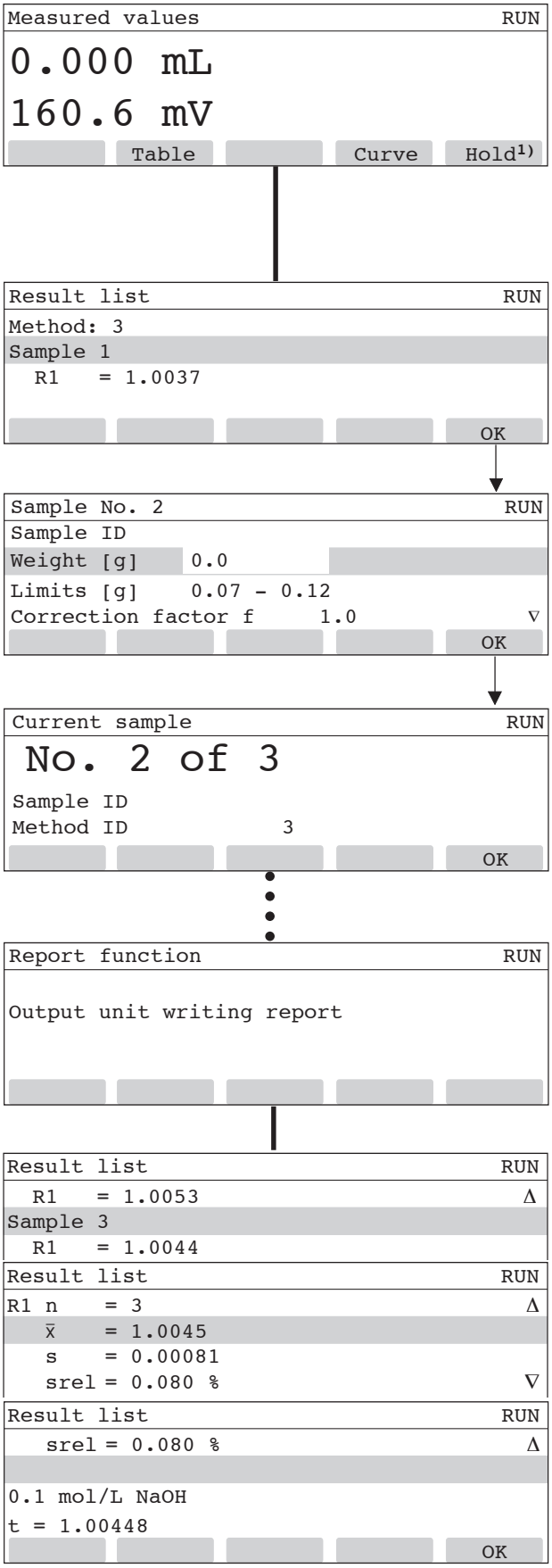
– Press <F5> or the Run key.

This is the prompt or the last opportunity to fasten the beaker with the first of the three prepared samples to the titration head.

– Press <F5> or the Run key.

The titrator stirs for 60 seconds to dissolve the potassium hydrogen phthalate.

[1)] appears only with DL55 and DL58, i.e. you can press <F5> to interrupt the titration.



The titrator executes the **Titration** function:
- it dispenses **2.5 mL** in 3 steps and
- ends the titration when it has found an equivalence point.

If you press <F2>, the table of measured values is displayed, <F4> shows the titration curve "Potential vs Volume".

The result of the first sample is displayed.

– Press <F5> or the Run key.

– Remove the titration beaker with the first sample.
– Rinse electrode, stirrer and dispensing tube with deionized water.
– Fasten the beaker with the second sample to the titration head.
– Enter the weight of the second sample, e.g. **0.08893.**
– Press <F5> or the Run key.

This is the prompt or the last opportunity to add the second sample.

– Press <F5> or the Run key: The **Stir** function reappears etc.

When the third sample has been analyzed and you have defined and attached a printer, the results of all three samples and the statistical data for the titer are printed out. This mask appears during this time.

The result list with the titer value of the third sample then appears.

If you scroll the display with the ∇ key, the statistical data appear: Mean value, standard deviation and relative standard deviation.

If you continue to scroll the display you will see the titer value, which is stored as a parameter of the titrant NaOH (c = 0.1 mol/L) (see Section 2.1 of the Reference Handbook).

```
Method          3               Titer by EQP titration
                Version         23-Oct-1996      13:30

Title
   Method ID ..........................3
   Title ..............................Titer by EQP titration
   Date/time ..........................13-Jul-1995
Sample
   Sample ID ..........................
   Entry type .........................Weight
      Lower limit [g] .................0.07
      Upper limit [g] .................0.12
   Molar mass M .......................204.23
   Equivalent number z ................1
   Titration stand ....................Stand 1
   Temperature sensor .................Manual
Stir
   Speed [%] ..........................50
   Time [s] ...........................60
EQP titration
   Titrant/Sensor
      Titrant .........................NaOH
      Concentration [mol/L] ...........0.1
      Sensor ..........................DG111
      Unit of meas. ...................mV
   Predispensing ......................to volume
      Volume [mL] .....................2.5
      Wait time [s] ...................0
   Titrant addition ...................Dynamic
      ΔE(set) [mV] ....................8.0
      ΔV(min) [mL] ....................0.02
      ΔV(max) [mL] ....................0.2
   Measure mode .......................Equilibrium controlled
      ΔE [mV] .........................0.5
      Δt [s] ..........................1.0
      t(min) [s] ......................3.0
      t(max) [s] ......................30.0
   Recognition
      Threshold .......................500.0
      Steepest jump only ..............No
      Range ...........................No
      Tendency ........................None
   Termination
      at maximum volume [mL] ..........10.0
      at potential ....................No
      at slope ........................No
      after number EQPs ...............Yes
         n = ..........................1
      comb. termination conditions .....No
   Evaluation .........................
      Procedure .......................Standard
      Potential 1 .....................No
      Potential 2 .....................No
      Stop for reevaluation ...........No
Calculation
   Formula ............................R=m/(VEQ*c*C)
   Constant ...........................C=M/(1000*z)
   Decimal places .....................4
   Result unit ........................
   Result name ........................Titer
   Statistics .........................Yes
Titer
   Titrant ............................NaOH
   Concentration [mol/L] ..............0.1
   Formula  t = .......................x̄
Report
   Output unit ........................Printer
   Results ............................No
   All results ........................Yes
   aso.
```

Sample function: The limits for the sample weight are matched to the maximum volume of the Titration function. The molar mass of potassium hydrogen phthalate and its equivalent number are used in the Calculation function.

The stirring speed and the time needed for dissolution of the potassium hydrogen phthalate are defined.

The **EQP titration** (equivalence point titration) is responsible for all titration parameters.
The amount of potassium hydrogen phthalate must lie within, e. g. the limits entered in the **Sample** function. If it is too small, the equivalence point may possibly lie in the range of the **predispensed** volume of **2.5 mL** and will not be evaluated. If it is too large, more than **10 mL** could be required to find the equivalence point. However, the titration will be terminated at the **maximum volume**.

The result is calculated from the mL consumption (VEQ) of the titrant NaOH, its concentration c and the molar mass of potassium hydrogen phthalate.

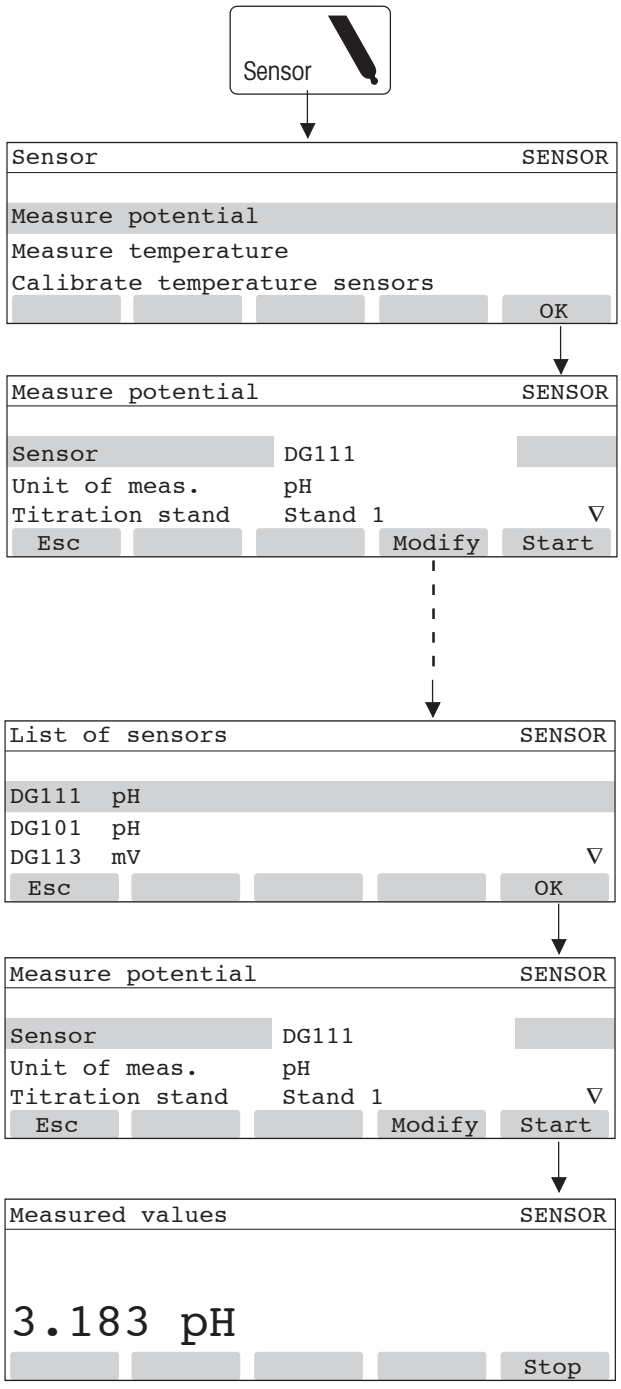
The **Titer** function assigns the calculated mean value $\bar{x}$ of the titer value to the titrant NaOH: The old value is automatically overwritten.

In the **Report** function, only "All results" are defined for a printout.

8. How to measure the pH value of a solution

You can perform a pH measurement with the auxiliary function "Measure potential". The sequence described below requires attachment of the electrode to the input "Sensor 1" and the stirrer to the output "Stirrer 1".

– Fasten the sample beaker to the titration head and immerse a pH electrode in the solution.



– Confirm this prompt with OK.

The parameters of this auxiliary function appear.

– Press <F5>.

The solution is measured and the pH value displayed (see mask at bottom).

If a different electrode or measurement unit is displayed, you must change this:

– Press <F4>.

This list shows all sensors that have been defined in the Setup menu. As the DG111 electrode has been used to illustrate the performance of a calibration (see Section 6), its calibration data are stored, i.e. the resulting pH value is correct.

– Press <F5>.

The defined unit of measurement is adopted simultaneously with the sensor.

– Press <F5>.

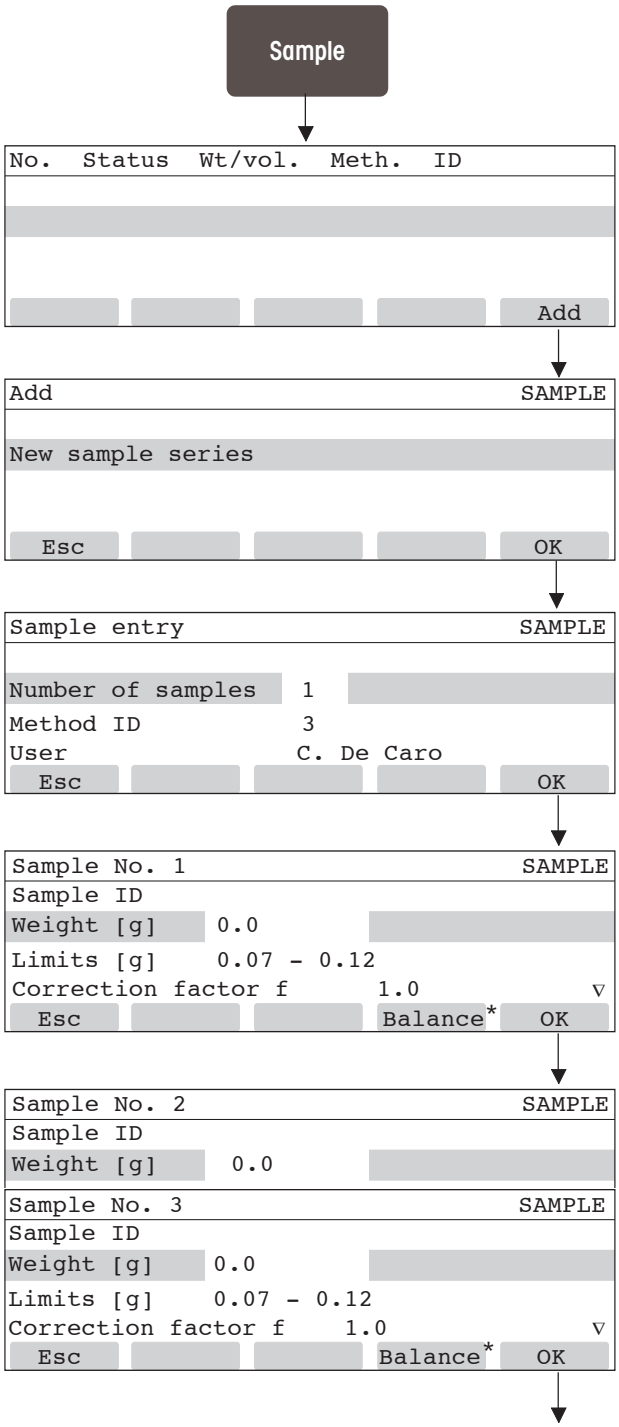
The solution is measured and the pH value displayed.

– Press <F5> to stop the measurement: The mask "Measure potential" reappears.

For an explanation of the other parameters of this auxiliary function, see Reference Handbook, Section 6.1.

9. Storing sample data

In the Sample menu you can enter the weight or volume of all samples of a series before the start of an analysis. The titrator then processes all samples in succession without you having to enter data for each sample (see example of the titer determination in Section 7.3). You can enter data for maximum 60 samples and these data remain stored even if you switch off the titrator.



The blank "sample data list" appears.

– Press <F5>.

– Press <F5>.

Enter the number of samples, e.g. **3.**

The method last executed was the titer determination with the identification **3** (as example in Section 7.3). The method ID and your name remains stored as a suggestion for the next analysis.

– Press <F5>.

The "sample data mask" appears.

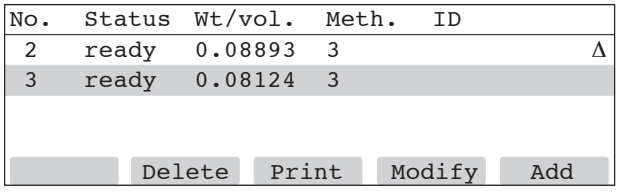
– Enter the weight of the first sample, e.g. **0.08451**.

* appears if you have defined a balance (see Sections 2.7.2 and 4.2 of the Reference Handbook).

– Press <F5>.

The "sample data mask" for the second sample appears, followed by that for the third.

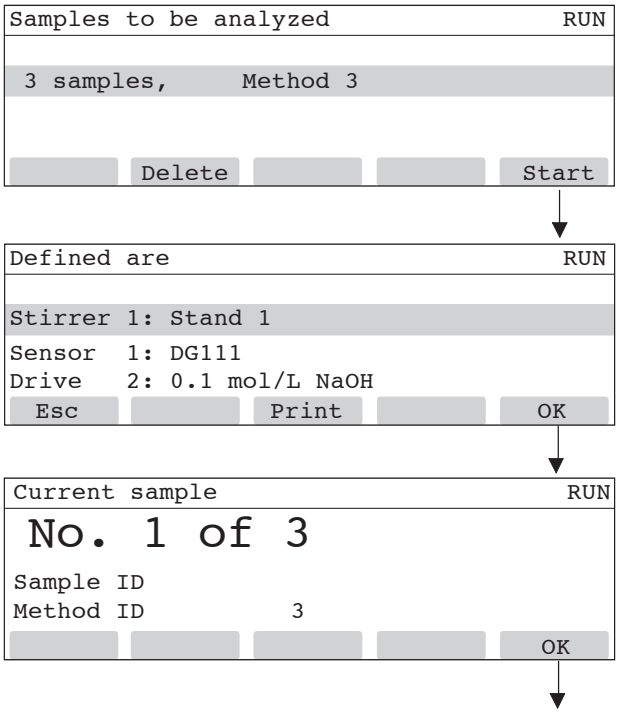
– Enter the weight each time and confirm with OK (press <F5>).



The sample data list containing the entered data reappears: The samples are classed as **ready** in the sample data memory!
You can add additional samples to this list, modify the entries of each sample, print out all sample data and delete any sample.

You will find a detailed explanation of the Sample menu in Section 4 of the Reference Handbook.

If you now press the Run key to titrate the samples, the following appears:



Number of samples and the method are shown. You still have the possibility to delete these at this point.

When the sequence is continued, the mask in which you should enter the weight of the sample no longer appears (see sequence of the titer determination in Section 7.3).

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Subject to technical changes and to the availability of the accessories supplied with the instruments.

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