TitraLab®

TIM550

Volumetric Karl Fischer Titrator

User's Manual

D21T028

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Approved Quality System

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1. Introduction

The TIM550 Volumetric Karl Fischer Titrator from Radiometer Analytical is especially designed for water analysis in many sectors including the pharmaceutical, oil and food industries.

On the TIM550 Volumetric Karl Fischer Titrator, burette, pump for safe solvent handling, titration stand and keyboard are all integrated in one compact unit.

To speed up maintenance and guarantee reproducible titrations, the TIM550 is equipped with the new Bayonet cell. This is designed with a special L-shaped slot so that electrodes fit into place in a single easy movement. The Bayonet concept keeps all parts completely moisture tight during use with no risk of leakage or water entering the cell.

The TIM550 increases analysis throughput by keeping your cell ready for immediate use. An automatic conditioning function avoids long cell reconditioning and continuous cell volume monitoring avoids overflowing. During the conditioning, the moisture drift is measured and automatically taken into account in calculations for faster, more accurate analyses.

To comply with quality control requirements, the TIM550 includes specific QC parameter setting together with High-Low alarms to help operators make the right choice in reviewing results.

The results obtained at the end of a titration can be given directly associated to their uncertainty. Radiometer Analytical offers an Uncertainty Plug-in to allow the uncertainty given for your Karl Fischer reagent titre, burette and instruments used to prepare sample (balance, pipette) to be programmed based on standard manufacturer specifications.

2. Getting started

This instrument has been developed to meet the requirements of electrochemical applications. It is aimed at experienced users who have the knowledge required to operate the instrument and implement the security instructions enclosed. Please remember that this system must not, under any circumstances, be used to perform tests on living beings.
Make sure that the TIM550 mains socket is within easy access, for quick disconnection purposes.

Radiometer Analytical SAS have developed installation kits to make setting up the TitraLab 55 Titration Workstation easy; refer to the Installation Sheet, part no. D26T019 and start mounting your system. If you intend to work with a method which is already stored in the TIM550, you may immediately proceed to chapter 5 page 51.

Rear panel

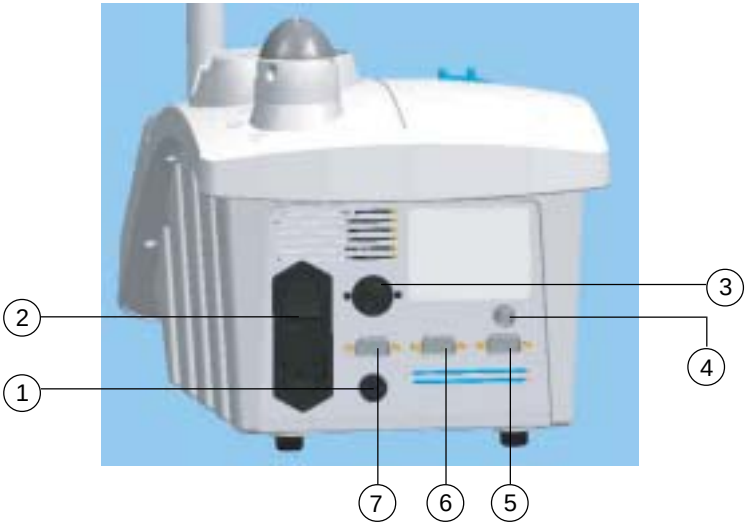


Figure 1: rear panel of the TIM550

1. Check that the voltage selection switch setting (115 V - 230 V) corresponds to the mains voltage. If not use a suitable tool to move the required voltage to the arrow.
2. Connect the mains socket of the TIM550 to the mains supply using the line cord, 230 V Euro or 115 V.

   If the TIM550 is to be used in the USA with a 230 V supply, the tandem blade attachment plug cap must be used, part no. 615-405.
3. When setting up the instrument for the first time it is necessary to install the battery delivered with the system, refer to chapter 7.
4. Connect the M241Pt2-8 Double Platinum Electrode to the socket marked ELECTRODE INPUT.
5. RS232C PC/PRINTER port for connection of printer or a personal computer.
6. RS232C port for TIM550 interconnection.
7. A balance can be connected to the TIM550 via the RS232C BALANCE port. Refer to chapter 6.

Note: an external keyboard or bar code reader can be connected to the TIM550 via the 6-pin mini DIN socket situated on the right hand side of the instrument; refer to chapter 6.

Adjusting the contrast

The contrast of the TIM550's display can be adjusted by means of a screw located on the bottom of the TIM550. By turning the screw clockwise or anticlockwise, the display will turn darker or brighter respectively.

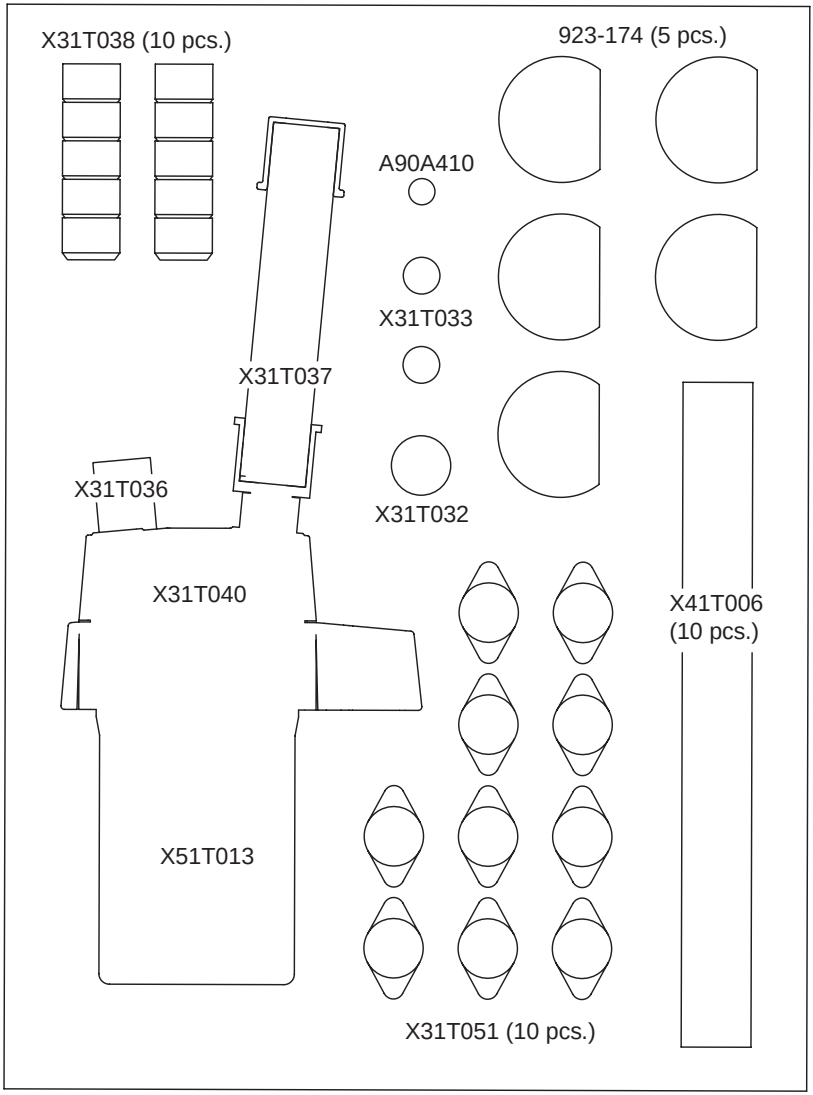
Changing the selected source voltage

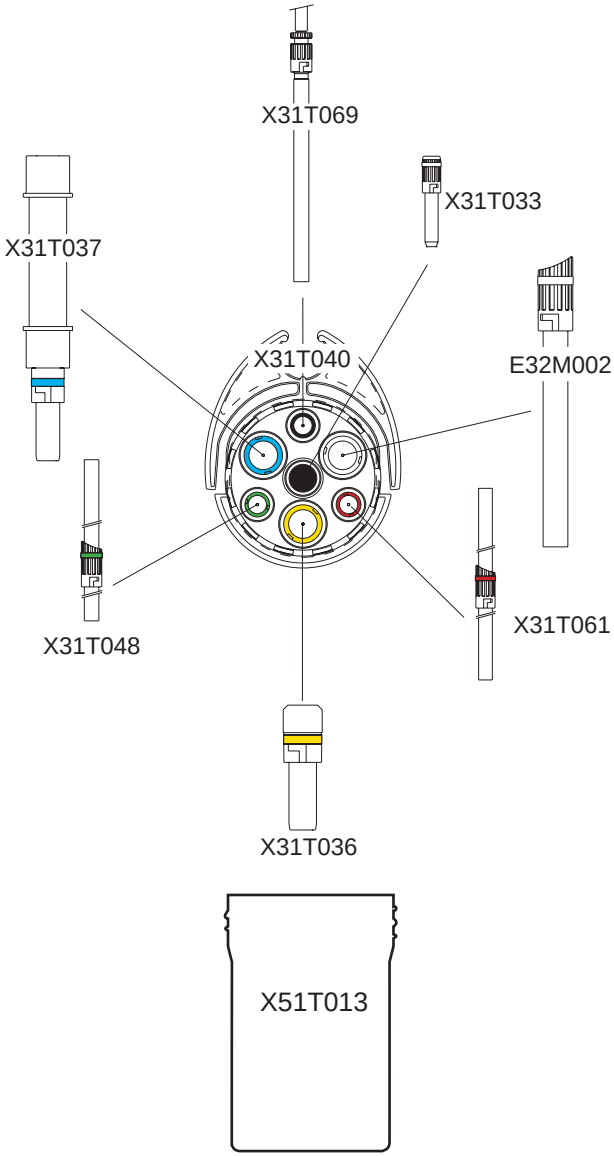
The TIM550 is designed to operate at 115 V and 230 V, depending on the setting of the voltage selection switch located on the back of the instrument.

When changing the input voltage, the fuse has to be changed. Refer to the table chapter 7.

To change the voltage, first unplug the power cord. Pull out the fuse cover and compare the fuse ratings with the desired voltage setting. Using a suitable tool, move the required voltage to the white arrow.

Setting up the Bayonet KF Cell with accessories (X91T003)




X31T069
X31T033
X31T037
E32M002
X31T040
X31T048
X31T061
X31T036
X51T013

Mounting the titration vessel

Place the magnet bar (A90A410) into the titration vessel (X51T013). Screw the titration vessel clockwise onto the electrode head.

Mounting the KF cell onto the TIM550

Press the two clips situated on both sides of the electrode head and slide the head onto the metal rod. Release the two clips to secure the head in place.

Filling the desiccant tube

Fill the tube with molecular seive (S72T004).
IMPORTANT: if you are using the molecular seive for the first time, you must activate it by placing it in an oven (temp. 250 to 300°C) for at least 10 hours, refer to page 104.

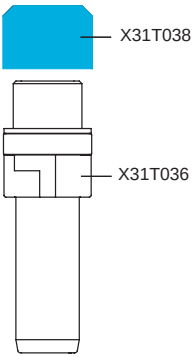
Getting the best results!

Make sure that the two platinum wires of the double platinum electrode are covered by the solvent.

Stirring must be effective without causing air bubbles.

Replacing the used silicon septa

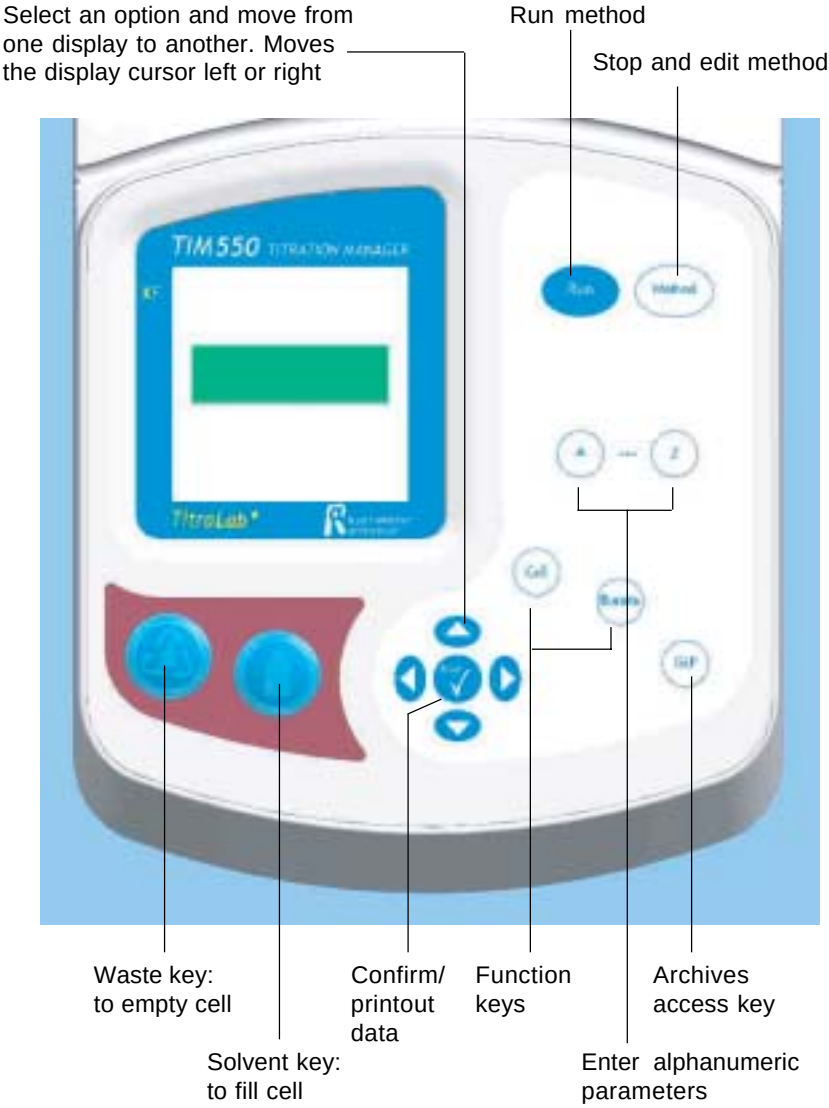
After approximately 20 injections, it is recommended to replace the blue silicon septa in the following way. Simply remove the old one and replace it with a new one (X31T038).



Thermostated KF cell

When working with temperatures above ambient temperature, the release of alcohol vapours and SO_2 contained in the KF solvents is accelerated.
It is therefore recommended to keep the solvent and waste bottles cool to reduce the rate of saturation of the molecular sieve found in the bottle stopper dessiccant tubes.

TIM550 front panel



Description of keys

Key	*Description*
Run	Press to start the method indicated on the TIM550 display (method no. 1 to 50).
Cell	Press to activate the cell's functions, refer to chapter 3. Pump: empty cell, add solvent, Standby: activate/deactivate Stirrer : set the stirrer On/Off and select the stirring speed from 100 to 1100 rpm. Molecular sieve: replace molecular sieve
Burette	Press to select burette's functions: Fill, Empty, Flush, Rinse, Bottle exch., New titrant, Calibrate or Enter titre. Hold down the key for 3 seconds to enter the burette edit mode. The user can now perform the following: 1. Select ***Enter*** for manual entry of the KF reagent titre. 2. Select ***Calibrate*** to determine the KF reagent titre using a calibration procedure.
GLP	The **GLP** key gives access to the following: 1. Last 200 sample results stored in chronological order. 2. Last blank result stored for each method + last 10 blank results in chronological order. 3. Last 10 KF titre results are stored in chronological order for each KF reagent.

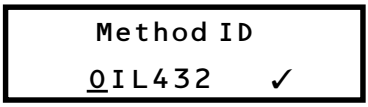
Key	*Description*
Method	This key is used to select one of the 40 programmable methods and one of the 10 pre-programmed methods. Hold down for 3 seconds to enter the Edit mode and edit a method to fit your particular requirements. **IMPORTANT**: the **Method** key is used to stop a titration!
A to Z	Use these keys to enter the following alphanumeric characters: ABC...YZ0123456789 • - / and blank space. 1. Press the **arrow** keys to move the cursor. 2. Press the **A or Z** to change the alpha-numeric data. The **left** and **right arrow** keys are used to: 1. Move the cursor left or right. 2. Select an option which appears within the arrows on the display. **Note**: if a chain of characters has already been entered, pressing the **right arrow** key will position the cursor at the end of the chain, pressing the **left arrow** key will position the cursor at the beginning of the chain. The **up** and **down arrow** keys are used to move up and down in the edit mode displays. **IMPORTANT**: the **down** arrow key can be used to accept a non-stable drift reading, thus continue to the next step (if the reading is inferieur to drift threshold parameter entered during programming).

Key	*Description*
Check mark	The **Check Mark** (✓) is used to : 1. Start a direct access function i.e. Burette Fill. 2. To confirm a display during sample analysis, thus continue to the next step. 3. To accept a result during reagent calibration and sample analysis. 4. Printout data, results and GLP tables.

Entering alphanumeric characters

Using the TIM550 keypad

For example, enter the name of a method:

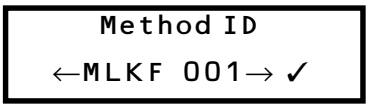


1. Use the right and left arrow keys to position the cursor in the text.
2. Use **A...Z** to enter the alphanumeric characters.

   Note: 16 characters can be stored to identify the method, the last 8 characters are displayed.

The method ID is longer than 8 characters:

Arrows at the beginning and end of the chain indicate that there is hidden text. Use the **right** and **left arrow** keys to move along the chain.



Deleting alphanumeric characters

Deleting an entire chain of characters

1. Use the arrow keys to position the cursor on the first letter in the chain.
2. Press **Z**, to delete the chain.

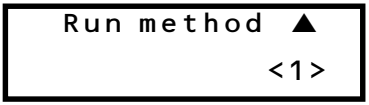
Deleting certain characters in the chain

1. Use the arrow keys to position the cursor on the first letter in the chain.
2. Press **A**, and select the "blank space". Please note that spaces will now replace text.

Please note that a PC keyboard and/or bar code reader can also be used to enter alphanumeric data.

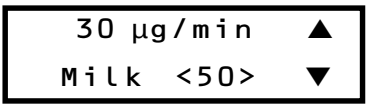
The Setup menu

1. Switch on the TIM550 using the on/off switch situated on the rear panel.
2. The first display you will see will remain for a few seconds and shows that the TIM550 is performing an autotest.
3. Press ✓ to enter the setup menu (when "setup" appears on the display). Refer to page 18 for details on how to program the setup menu.
4. If no keys are pressed, the TIM550 switches to the method display:



Press **Run** to start the titration or use the **arrow** keys to select another method from 1 to 50.

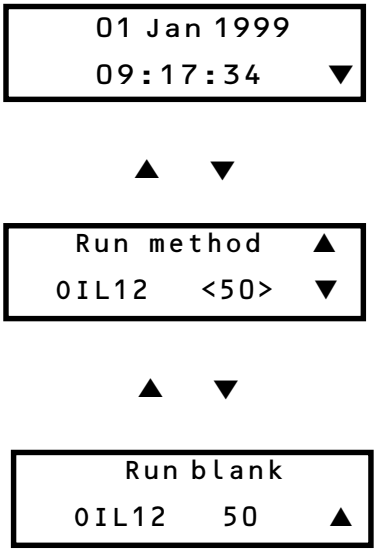
To activate the Stand by, refer to chapter 3. The method display will now show the "live" drift value.



Refer to chapter 4, to calibrate your KF reagent titrant and create your KF titration method.

Displaying the date and time

In the method display, press ▲ to display the date and time:



Running a blank titration

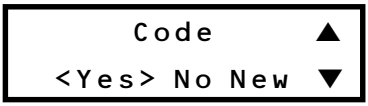
If you have selected the option ***Blank = Yes*** in the edit method menu. Press ▼ then the **Run** key to start the blank titration.

Programming the Setup menu

The TIM550 will perform an autotest as soon as it has been switched on. After 3 seconds, press ✓ to gain access to the setup menu.

Entering a protection code

Use **A...Z** to enter a four character code to lock the TIM550.

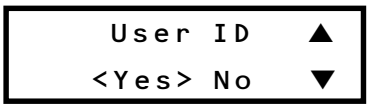


Select New: to enter a new user-defined code.

Select Yes: the user-defined code entered above is maintained. The TIM550 will prompt you to enter this code in the "Enter code" display each time you enter the Edit mode.

Select No: your method will remain unprotected.

Entering a User ID

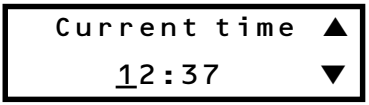


Select Yes: to enter a User ID at the start of each method i.e. after pressing **Run**.

Setting the time

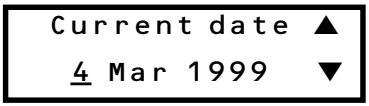
The TIM550 is fitted with an internal clock. It is possible to visualise the date and time by pressing **Method** then ▲.

The actual date and time are entered in the following displays:



Use the arrow keys to position the cursor and **A...Z** to enter the hour and minutes.

Setting the date



1. Using **A...Z**, enter the day. Press ▼ to move to the month.
2. Using the arrow keys enter the month. <Jan>, <Feb>, <Mar>, <Apr>, <May>, etc. Press ▼ to move to the year.
3. Using **A...Z** enter the year.

Choosing the language

Choose your language for displays and printouts from the list available.

Use the arrow keys to select the language desired.

Choosing the keyboard type

An external PC keyboard can be connected to the 6-pin mini DIN socket situated on the right hand side of the TIM550. Using the **arrow** keys, select your keyboard type.

Refer to Chapter 6 for information on keyboard functions.

Selecting printer type and printout format

Select ***80 columns***: printer gives a "detailed" printout of results.

Select ***40 columns***: printer gives a "condensed" printout of results.

Select ***No***: if a printer is not connected.

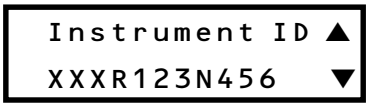
Refer to Chapter 5.

Keyboard beep

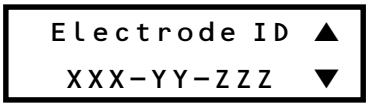
Select Yes: the TIM550 will emit 3 beeps when a result is obtained and 1 beep when the cell is ready to receive the sample.

Select No: to cancel all beeps.

Entering Instrument, Electrode and Burette ID



The instrument ID is defined during manufacturing, therefore it cannot be changed.



For Radiometer Analytical electrodes the ID is given as follows:
XXX = month; YY = batch number; ZZZ = electrode number

Use **A...Z** to enter the identification.

This identification will appear at the top of all "detailed" printouts.

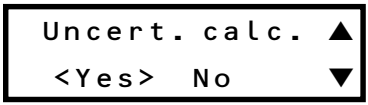
Uncertainty calculations

The results obtained at the end of a titration can be displayed directly associated to their corresponding expanded uncertainty. The uncertainties given for your Karl Fischer reagent titre, burette and instruments used to prepare sample (balance, pipette) are programmed based on standard manufacturer specifications. These calculations comply with the following standard: EN 13005 (GUM)[1].

Radiometer Analytical has developed an Uncertainty Plug-in (R31T009) so that the TIM550 can give results with their expanded uncertainty.

Follow the procedure given below to unlock the uncertainty calculations.

1. Select ***<Yes>***.



2. Use **A...Z** to enter the 6 digit access key from the product package.

   If the correct access key has been entered the uncertainty calculations are unlocked.

   Press ▼ to return to the start of the Setup menu.

   Note: you can select **No** to re-lock the uncertainty calculation option.

[1] *GUM: Guide for the Expression of Uncertainty in Measurement, published by ISO, 1993*

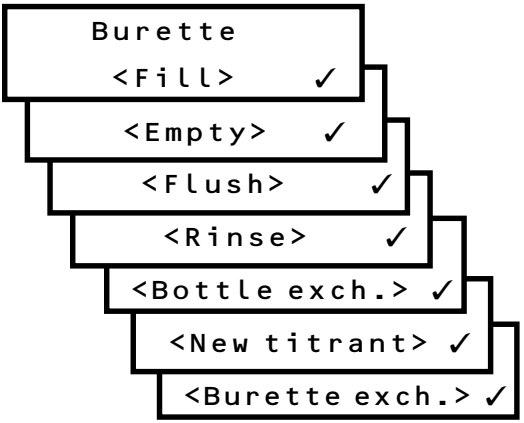
3. Manual functions

Burette functions

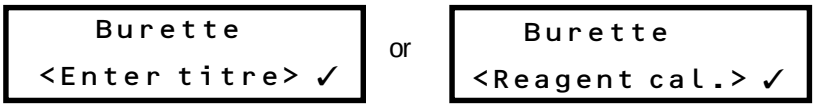
The following burette functions can be controlled by the TIM550 by means of the **Burette** key.

Use the arrow keys to select the burette function and press ✓ to start.

Note: stand by (see page 34) is set to off when a burette function is activated, except when performing a reagent calibration.



One of the following displays will appear depending on the choice made in the Edit Burette menu "Calibrate" or "Enter". Method ID.



The following display appears when a burette function is running. Press ✓ to stop the function and return to the previous display.

Fill procedure

This procedure is used to fill the burette with titrant.

IMPORTANT: prior to dismounting the burette the piston should be taken to its lowest position. Perform a Fill to do so.

Empty procedure

This procedure allows you to empty the contents of the glass cylinder into the cell.

Flush procedure

This procedure ensures complete rinsing of the system and expulsion of air bubbles from the burette, delivery tube etc. It is recommended to carry out this procedure whenever titrant/reagent has been changed or whenever it is suspected that air bubbles have been drawn into the burette.

The flush procedure lasts for approximately 2 minutes. Within this period, the burette will be flushed with titrant/reagent. The flush procedure leaves the burette filled and ready for use.

Note that the length of the flush programme has been adapted to delivery tubes of standard length, 90 cm. If a longer delivery tube is used, start out with the flush programme.

Rinse procedure

For safe reagent handling use this procedure **each** time the burette is to be replaced. It makes sure the delivery tubes are titrant free.

Press ✓ to start the procedure.

The contents of the KF cell are emptied into the waste bottle and the burette starts emptying into the KF cell.

Remove the bottle stopper from the titrant bottle.

Mount the rinse solution bottle. Screw the bottle stopper onto the bottle. The burette starts the flush procedure.

Remove the bottle stopper from the rinse solution bottle.

Replace the titrant bottle and screw the bottle stopper into place.

At the end of the rinse procedure the TIM550 returns to the burette menu display.

Bottle exchange procedure

This procedure allows you to remove the empty titrant bottle and replace it with a new one.

Press ✓ to start the procedure.

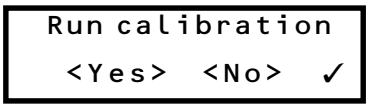
The contents of the KF cell are emptied into the waste bottle. The burette starts emptying into the KF cell.

Remove the bottle stopper from the titrant bottle.

Place the new titrant bottle into the bottle holder (optional) and screw the bottle stopper into place. The KF reagent ID entered in the reagent library is displayed.

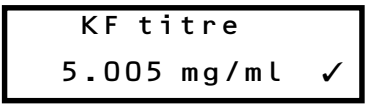
If necessary enter a new KF titre, see below.

If KF reagent titre = calibrate.



Press ✓ to start the calibration procedure. Refer to page 55.

If KF reagent titre = enter.



Enter KF titre. Refer to page 57.

The new KF titre will now be stored in the KF reagent GLP table.

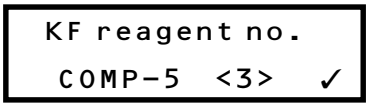
At the end of the procedure the TIM550 returns to the burette menu display.

KF titrant replacement procedure

This procedure allows you to change the titrant in use with a titrant of a *different* type.

Press ✓ to start the procedure.

The contents of the KF cell are emptied into the waste bottle. The burette starts emptying into the KF cell.

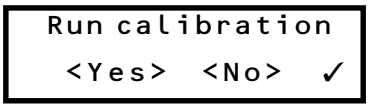


Using the **arrow** keys, select the new KF reagent from the reagent library (numbers 1 to 20); refer to chapter 4.

Place the new titrant bottle into the bottle holder (optional). Screw the bottle stopper onto the bottle.

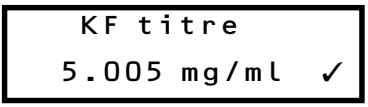
Enter a new KF titre, see below.

If KF reagent titre = calibrate.



Press ✓ to start the calibration procedure. Refer to page 55.

If KF reagent titre = enter.



Enter KF titre. Refer to page 57.

The new KF titre will now be stored in the KF reagent GLP table.

At the end of the procedure the TIM550 returns to the burette menu display.

Dismounting the burette

IMPORTANT: prior to dismounting the burette from the burette stand the burette must be rinsed using the "Rinse" procedure from the burette menu.

1. If necessary, press the **Burette** key and select "Fill". The Fill procedure ensures that the piston is in its lowest position.
2. Run "Burette exc." procedure when prompted to do so; see below.
   - Turn the burette a 1/4 of a turn anti-clockwise.
   - Slide the burette off the stand in order to free the piston shaft.

***Warning**:

The calibration certificate supplied with a calibrated burette will no longer be valid if the burette stand is replaced or any parts dismounted. Please contact your Radiometer Analytical representative to recalibrate the burette.

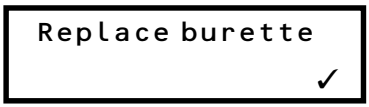
Burette exchange procedure

If the burette stand has been replaced or dismounted the calibration data is no longer valid.

This procedure allows you to reset the calibration parameters of the burette stand if it has been replaced or dismounted. The TIM550 will therefore use the default parameters.

Press ✓ to start the procedure. The Stand by is deactivated.

The burette starts emptying into the contents of the KF cell into the waste bottle. When the emptying is over, the burette is filled with air and the piston returns to its lowest position.



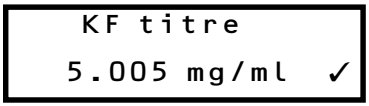
Press ✓ after burette replacement. The default parameters are restored.

Return to the burette menu.

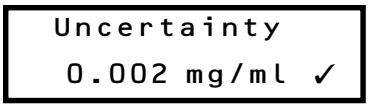
Manual entry of KF titre

Available if ***KF reagent titre = Enter*** is selected in the KF reagent library. Refer to chapter 4.

Press ✓ to continue.



Enter the KF titre.
Range available: 0.001 to 9999.999



If ***Uncertainty calc. = Yes***
Enter the standard uncertainty of the titre. Refer to bottle indications or titrant certificate (R31T009).
Range available: 0 to 9999.999

The entered KF titre will be automatically stored in the KF reagent GLP table.

Reagent calibration procedure

This procedure allows you to determine the Karl Fischer reagent titre.

Available if ***KF reagent titre = Calibrate*** is selected in the KF reagent library. Refer to chapter 4, page 38.

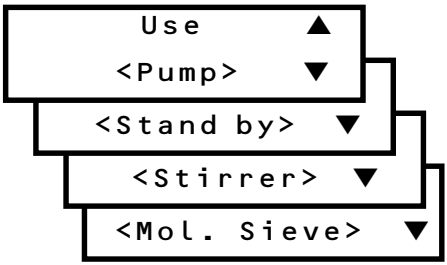
Press ✓ to start the calibration.

The titre determined at the end of the calibration procedure is automatically transferred to the KF reagent GLP table.

An overview of the calibration procedure is given in chapter 4 page 55.

KF cell functions

The following functions are controlled by the TIM550 by means of the **Cell** key:



Pump functions: empty cell and add solvent.

Stand by: on/off

Stirrer functions: stirrer on/off, set stirring speed

Molecular sieve: replace molecular sieve

Use the arrow keys to select one of the cell functions. Press ▼ to continue to the next level of displays.

Once the pump has been activated, use this key to add solvent to the cell.

Once the pump has been activated, use this key to empty the cell.

Pump functions

Emptying the KF cell

Pump <Empty cell> ✓

Press ✓ to activate the pump.

The stirrer starts at medium speed.

Press  to empty the cell.

To stop the pump press ✓

Press ✓ to stop the pump.

The pump will automatically stop after 60 seconds.

Cell full empty cell ✓

This message will appear when the maximum level in the cell is reached. Press ✓ to activate the pump and the Waste key to start emptying the cell. Refer to chapter 7, section error messages.

Adding solvent to the KF cell

Pump
<Add solvent> ✓

Press ✓ to activate the pump.

Press  to add approximately 35 to 40 ml of solvent.

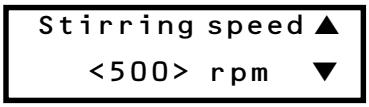
Radiometer Analytical have marked the KF cell to indicate the minimum level to which solvent must be added. Refer to chapter 9.

To stop the pump
press ✓

Once the fill mark is reached, release the solvent button to stop the pump.

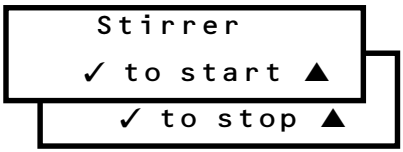
The pump will automatically stop after 60 seconds.

Stirrer functions



Select a stirring speed from one of the 11 available speeds: 100, 200, 300, 400, 500 (default value), 600, 700, 800, 900, 1000, 1100 rpm.

Press ▼ to valid the stirring speed and continue to the next step.

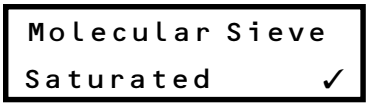


Press ✓ to start/stop stirring.

Replacing the molecular sieve

Press ▼ to valid the replacement of the molecular sieve and continue to the next step.

When ✓ is pressed, the internal counters "empty cell" and "molecular sieve replacement date" will be reset:



This message will appear when the Empty cell function is activated and the number of empty cells is greater than 50 or the molecular sieve replacement date has exceed 7 days.

Automatic moisture drift compensation "Stand by"

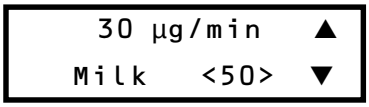
Stand by remains activated at the end of titrations.

The stand by function maintains the cell ready for the next analysis.

Follow the procedure given below to activate the stand by.

Press ✓ to start the stand by.

When stand by is activated the parameter values of the current KF titration method are used.



The TIM550 displays the drift reading in the "method" display.

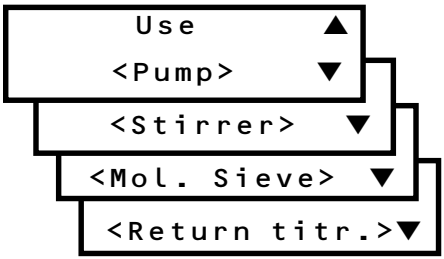
Press ✓ to stop the stand by.

Stand by is stopped automatically when:

1. A manual pump function is started.
   Note: the stand by is reactivated if the titration is continued.
2. A manual burette function is started, except for a reagent calibration procedure.
3. The TIM550 is switched off.
4. The method key is pressed to stop a titration, while the TIM550 is busy compensating the drift.
5. The cell volume is above the maximum "authorised" level to avoid overflowing.

Accessing KF cell functions during a titration

It is possible to access the following functions during a titration i.e. before introducing the sample to the KF cell or before confirming a repeat analysis.



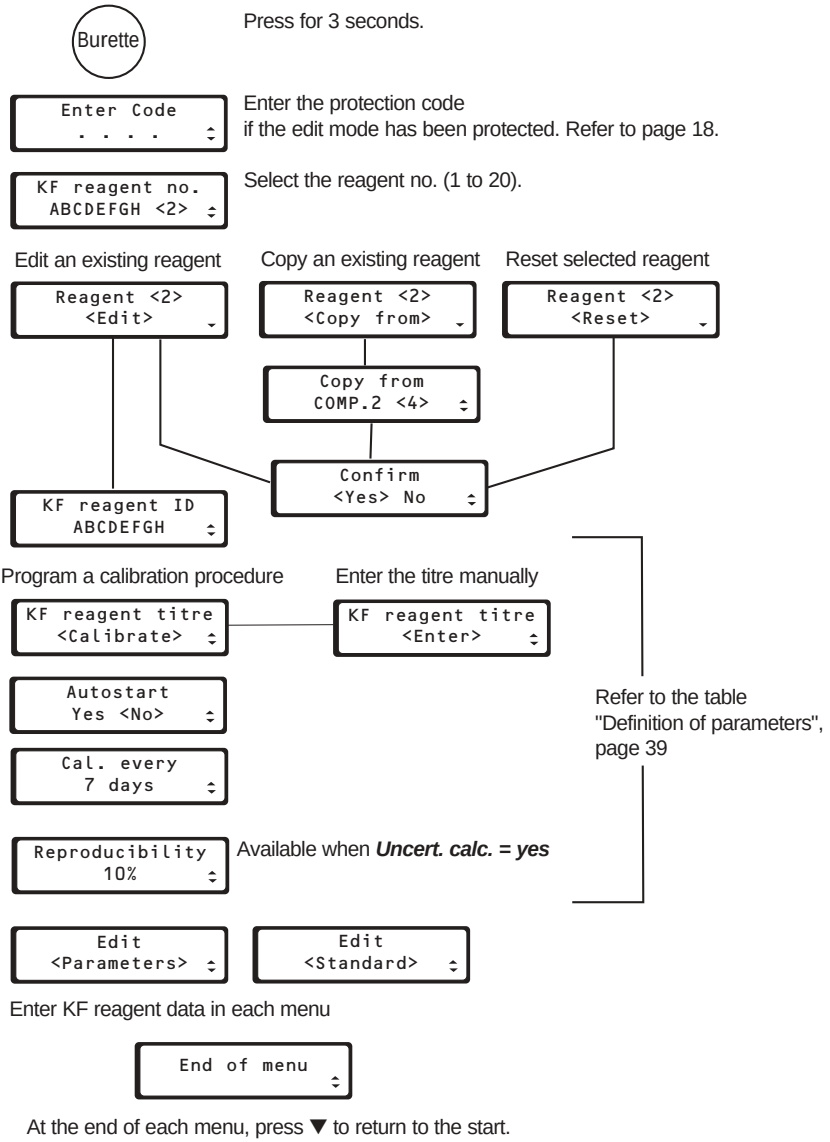
Press **Cell** and select the required function ***<Pump>***, ***<Stirrer>*** **or** ***<Mol. Sieve>***.

You can then empty the KF cell, add solvent, and/or change the stirring speed.

Select ***<Return titr.>*** to return to the ongoing titration.

4. Programming and running analysis

Programming the KF reagent library



KF reagent library

The KF reagent library has the possibility to store calibration data for up to 20 different Karl Fischer reagents.

Important: whichever titrant is selected is used for all methods.

To change titrant, refer to the KF titrant replacement chapter 4. Do not forget to select your titrant from the reagent library (no. 1 to 20) and if necessary determine the titre.

You have two possible ways to introduce the titre:

1. **Perform a calibration**.

   Select ***<Calibrate>*** when programming the KF reagent library. Start the calibration procedure in the burette menu, refer to page 55.

2. **Enter the titre manually**.

   Select ***<Enter>*** when programming the KF reagent library. Enter the KF reagent titre in the burette menu, refer to chapter 3.

As the KF reagent quickly deteriorates with time, Radiometer Analytical recommend frequent calibrations to ensure optimal accuracy.

Definition of parameters

Parameter	*Calibration type*	*Range available*	*Default value*	*Description*
Autostart	Calibrate	Yes/No	No	When activated the ""Autostart"" function enables the TIM550 to automatically detect the addition of sample to the KF Cell. The titration will start as soon as the water in the sample is detected.
Cal every	Calibrate	1 to 99	7 days	Enter a calibration reminder. The TIM550 will prompt you to perform a new calibration if the current calibration is older than an ""x"" number of days.
KF reagent ID	Calibrate/ Enter	A to Z. 0 to 9, •, -, /, blank space		Use A...Z to enter the ID.
Reproducibility	Calibrate	0 to 999.99	10%	Appears if Uncert. calc. = Yes in the setup menu. If you are to perform only one titration enter the reproducibility of the result. This corresponds to the the relative standard deviation, in %, estimated from a previous series of measurements, refer to chapter 5.

Press the ▼ key and Edit **<*Standard*>** and Edit **<*Parameters*>**.

Calibration procedure parameters

The following parameters are available when you press the **Burette** key for 3 seconds.
Press ▼ to accept the values and continue to the next display.

Edit <Parameters> menu

Parameter	*Range available*	*Default value*	*Description*
Drift Threshold	0 to 999 µg/min	50 µg/min	This is the maximum drift allowed to start the titration.
Max. burette speed	1 to 200	150 %/min	The maximum speed at which the burette will deliver KF reagent.
Min. titr. time	00:01 to max. titr. time	00:30 (min:s)	This is the minimum time the titration will take. The timer will start as soon as the sample is introduced into the KF cell (if autostart) otherwise as soon as ✓ has been pressed.
Max titr. time	Min. titr. time to 99:59	00:05 (h:min)	This is the maximum time the titration will take. This parameter is used to avoid never ending titrations.
Max. volume	1 to 99	10 ml	This setting should be used as a stop criterion corresponding to the maximum volume of titrant to be added during the titration. The titration will stop when the volume reaches the entered value.
Quality control	Yes or No	Yes	This is the quality control option. It will help you determine whether or not you should accept or reject a result. Select <Yes> to enter an acceptance range. See the minimum and maximum titre values on the next page.

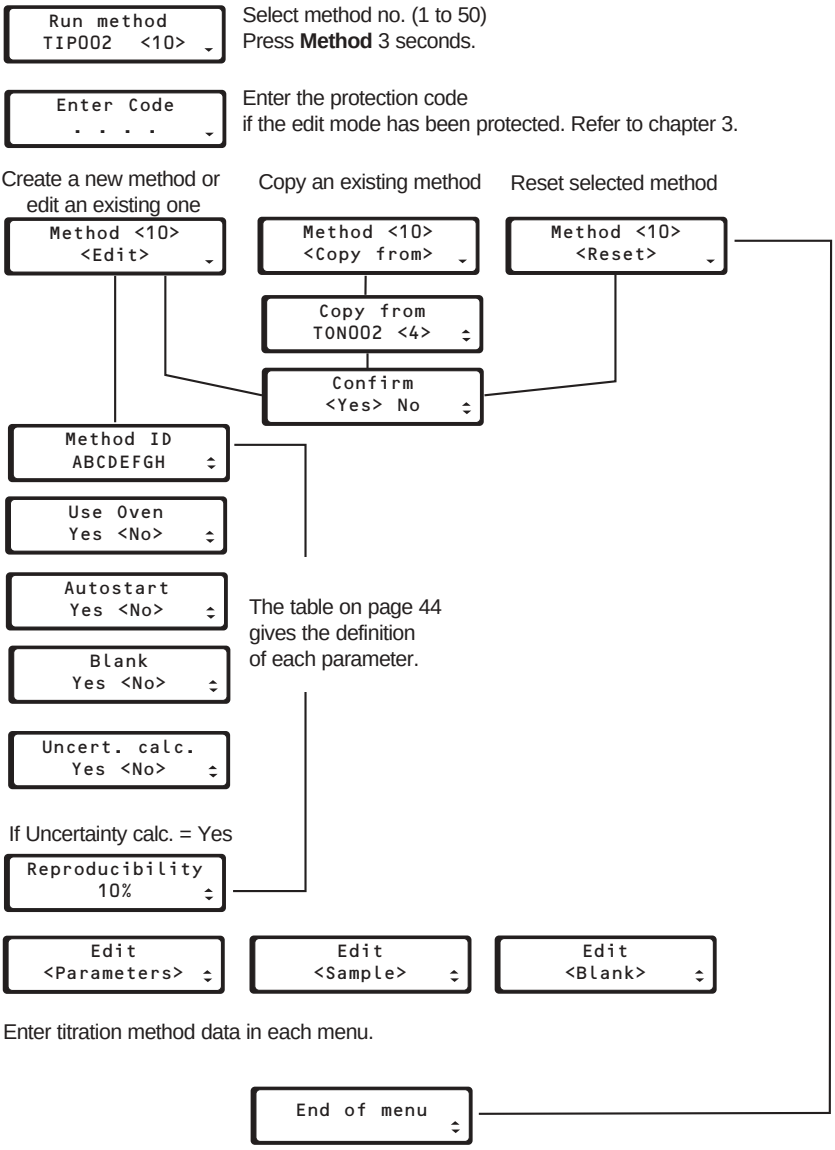
Parameter	*Range available*	*Default value*	*Description*
Minimum titre	0.001 to maximum titer	0.001 mg/ml	This is a Quality control parameter setting, Quality control = Yes. Set the minimum acceptance value for the KF reagent titer. An alarm message will appear if the titre lies below this value. Refer to chapter 7.
Maximum titre	minimum titer to 9999.999	9999.999 mg/ml	This is a Quality control parameter setting, Quality control = Yes. Set the maximum acceptance value for the KF reagent titre . An alarm message will appear if the titre lies above this value. Refer to chapter 7.

Edit <Standard> menu

Parameter	*Range available*	*Default value*	*Description*
Advised amount	0.001 to 9999.999	1.000 ml	The recommended amount of standard to be added to the KF cell during the titration. The value will appear on the "Introduce standard" display in the calibration procedure.
If standard unit = volume			
H_20 in std.	0.001 to 9999.999	5.000 mg/ml	Enter the amount of water the standard contains. This data is given on the standard bottle.
± H_20 in std.	0 to 9999.999	0.02 mg/ml	Enter the standard uncertainty (u) on the amount of water in the standard. The ± value given on the bottle corresponds to the tolerance (T). Use the following equation to calculate the uncertainty. u = T /√ 3 For additional information, refer to "Uncertainty Plug-in" (R31T009).
If standard unit = weight			
H20 in std.	0 to 100	15.66%	see above.
± H20 in std.	0 to 100	0.05%	see above.
Standard ID	Yes or No	No	When selected, this option allows you to identify the standard during the calibration.
Standard unit	µl, ml, µg, mg, g	ml	The unit used for the standard.
Uncertainty	0.001 to 9999.999	0.01 ml	Enter the standard uncertainty u of the standard used for the calibration. u = U/k or u =T/√ 3 U = expanded uncertainty k = coverage factor

Programming a KF Titration

Creating or editing a titration method



Definition of parameters

Parameter	*Range available*	*Default value*	*Description*
Autostart	Yes or No	No	The ""Autostart"" function enables the TIM550 to automatically detect the addition of sample to the KF Cell. The titration will start as soon as the water is detected in the sample whichever option is selected Yes or No. When No is selected and water is not detected after sample injection (slow release of water), then titration can be started manually by pressing ✓.
Blank	Yes or No	No	Set to yes to create a titration method using a blank.
Method ID	A to Z.	0 to 9 -, •, /, blank space	Use A...Z to enter the ID.
Reproducibility	0 to 999.99	10%	Appears if Uncert. calc. = Yes in the method. If you are to perform only one titration enter the reproducibility of the result, estimated from a previous series of measurements, refer to chapter 5.
Uncert. calc.	Yes or No	No	This option appears if the uncertainty calculations have been unlocked in the setup menu. Select Yes, results will be given directly associated to their related uncertainty. The calculations will also take into account all the parameters involved in your analyses. Select No, results for a series of measurements will be given with their standard deviation (±....).
Use Oven	Yes or No	No	Set to yes if you are working with an oven.

Method menu parameters

The following "general" parameters are available when you press **Method** for 3 seconds.

Edit <Parameters> menu

Parameter	*Range available*	*Default value*	*Description*
Drift Threshold	0 to 999 µg/min	50 µg/min	This is the maximum drift allowed to start the titration.
Max. burette speed	1 to 200	150 %/min	The maximum speed at which the burette will deliver KF reagent.
Min. titr. time	00:01 to max. titr. time	00:30 (min:s)	This is the minimum time the titration will take. The timer will start as soon as the sample is introduced into the KF cell (if autostart) otherwise as soon as ✓ has been pressed.
Max titr. time	Min. titr. time to 99:59	00:05 (h:min)	This is the maximum time the titration will take. This parameter is used to avoid never ending titrations.
Max. volume	1 to 99	10 ml	This setting should be used as a stop criterion corresponding to the maximum volume of titrant to be added during the titration. The titration will stop when the volume reaches the entered value.
Quality control	Yes or No	Yes	This is the quality control option. It will help you determine whether or not you should accept or reject a result. Select <Yes> to enter an acceptance range. See the minimum and maximum titre values on the next page.

Parameter	*Range available*	*Default value*	*Description*
Minimum titre	0.001 to maximum titer	0.001 mg/ml	This is a Quality control parameter setting, Quality control = Yes. Set the minimum acceptance value for the KF reagent titer. An alarm message will appear if the titre lies below this value. Refer to chapter 7.
Maximum titre	minimum titer to 9999.999	9999.999 mg/ml	This is a Quality control parameter setting, Quality control = Yes. Set the maximum acceptance value for the KF reagent titre . An alarm message will appear if the titre lies above this value. Refer to chapter 7.

Edit <Sample> menu - *general parameters*

Parameter	*Range available*	*Default value*	*Description*
Sample ID	Yes or No	Yes	When selected, this option allows you to identify the sample, during the titration.
Sample unit	µl, ml, µg, mg, g.	ml	Unit used for the sample.
Sample factor	0.001 to 9999.999	1.000	The sample factor is used when it is necessary to correct a result. Example: 10 g of sample containing two components“(5 g A + 5 g B) are added to the KF cell: A - contains no water. B - water content unknown. In order to calculate the water content of B, enter a sample factor of 0.5.
Result unit If sample unit = µl, ml	g, mg, µg, mg/ml, g/l, µg/ml	g	The result unit will appear with the result at the end of titrations.
Result unit If sample unit = mg, g	g, mg, µg, %, g/kg, mg/g, µg/g, ppm, mg/kg	g	The result unit will appear with the result at the end of titrations.
Number of digits	3 to 6	5	Select the number of digits to be displayed in the result at the end of the titration.
Quality control	Yes or No	Yes	This is the quality control option. It will help you determine whether or not you should accept or reject a result. Select <Yes> to enter an acceptance range. See minimum and maximum values on next page.

Parameter	*Range available*	*Default value*	*Description*
Minimum value	0.001 to maximum value	0.001 ml	This is a Quality control parameter setting, Quality control = Yes. Set the minimum acceptance range for results. An alarm message will appear if the result lies below this value. Refer to chapter 7.
Maximum value	minimum value to 9999.999	9999.999 ml	This is a Quality control parameter setting, Quality control = Yes. Set the maximum acceptance range for results. An alarm message will appear if the result lies above this value. Refer to chapter 7.

Sample menu *(Blank = No)*

Parameter	*Range available*	*Default value*	*Description*
Advised amount	0.001 to 9999.999	1.000 ml	Enter the recommended sample amount to be added to the KF cell during the titration. The value will appear on the "Introduce sample" display in the run procedure.
Uncertainty	0 to 9999.999	0.01 ml	Enter the standard uncertainty of the sample. This parameter comprises different components, for example the uncertainty of the syringe and/or balance. For additional information, refer to "Uncertainty Plug-in" (R31T009).

Sample menu *(Blank = Yes)*

Parameter	*Range available*	*Default value*	*Description*
Sample amount	0.001 to 9999.999	1.000 ml	This is the amount of sample that has been added to the solvent. Refer to "Blank samples".
Sample uncert.	0 to 9999.999	0.01 µl	Appears if Uncert. calc. = Yes. Enter the standard uncertainty of the sample amount entered.
Dilution volume	0.001 to 9999.999	10 ml	Enter the volume of solvent used to dissolve the sample. Refer to "Blank samples".
Dilution uncertainty	0.001 to 9999.999	0.2 ml	Appears if Uncert. calc. = Yes has been selected in the setup menu. Enter the standard uncertainty of the dilution volume.
Advised aliquot	0.001 to 9999.999	1.000 ml	Enter the recommended amount of sample to be added to the KF cell during the titration. The value will appear on the "Introduce sample" display in the run titration procedure.
Aliquot uncertainty	0.001 to 9999.999	0.01 ml	Appears if Uncert. calc. = Yes has been selected in the setup menu. Enter the standard uncertainty of the recommended amount of sample aliquot to be added to the KF cell during the titration.

Edit <Blank> menu

Parameter	*Range available*	*Default value*	*Description*
Advised amount	0.001 to 9999.999	1.000 ml	The recommended blank amount to be added to the KF cell during the titration. The value will appear on the "Introduce blank" display in the run procedure.
Uncertainty	0 to 9999.999	0.01 ml	Enter the standard uncertainty of the blank. This parameter comprises in general different components, for example the uncertainty of the syringe and/or balance. For additional information,refer to "Uncertainty Plug-in" (R31T009).
Blank res. unit	mg/ml or µg/ml	mg/ml	The blank result unit will appear with the blank result at the end of titrations.
Quality control	Yes or No	Yes	This is the quality control option. It will help you determine whether or not you should accept or reject a blank result. Select <Yes> to enter an acceptance range. See minimum and maximum blank.
Maximum blank	minimum blank to 9999.999	9999.999 mg/ml	This is a Quality control parameter setting, Quality control = Yes. Set the maximum acceptance range for results. An alarm message will apear if the result lies above this value.
Minimum blank	0.001 to maximum blank	0.001 mg/ml	This is a Quality control parameter setting, Quality control = Yes. Set the minimum acceptance range for results. An alarm message will apear if the result lies below this value

Performing a titration

Procedure

1. Setup up the titration system as described in the Installation Sheet, part no. D26T019.
2. Prepare the burette, refer to chapter 3.
3. Add solvent to the "fill mark" on the KF cell, refer to chapter 3.
4. Prepare your sample, refer to page 52.
5. Press the **Method** key and select the method using the **arrow** keys.
6. Press **Run** to start the method.

- To perform a calibration, refer to page 55.
- To perform a blank titration, refer to page 56.
- To perform a KF titration, refer to page 57.
- To perform a KF titration using an oven, refer to page 58.
- To perform one of the 10 pre-programmed methods, refer to page 59.

Weighing the sample

For the connection of a balance, refer to chapter 6.

Liquid samples, using the weight by difference method

Liquid samples can be injected into the KF cell with a syringe through the exchangeable septum stopper.

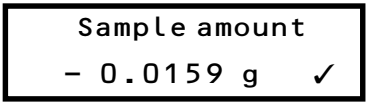
1. Fill the syringe with sample.
2. Place the syringe onto the balance and set the balance to zero "tare".
3. When the message "Introduce sample" appears on the display, inject the contents of the syringe.

   It is recommended to inject your sample as soon as the message appears on the display.

   If the auto detection function is activated, the titration will start immediately on detection of the sample injection.

   If this is not the case, press ✓ to start the titration.
4. Weigh the syringe.

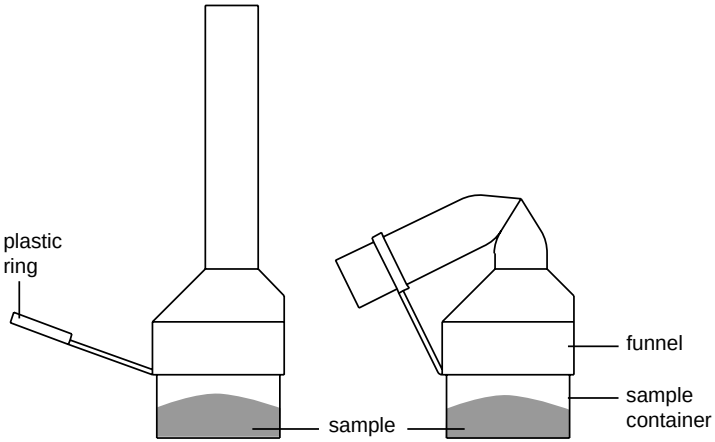
   The value shown on the TIM550's display has been sent from the balance.

   This weight is expressed as an absolute value for calculation purposes.
5. Press ✓ to accept the sample amount indicated on the stable balance reading and continue the titration.

Solid samples

A special sample container with funnel, part no. 923-174 is available for weighing solid samples.



1. Weigh the sample in the sample container and fit the funnel.
2. Bend the funnel and secure into place with the plastic ring.
3. Put back on the balance and set the balance to zero "tare".
4. When the message "Introduce sample" appears on the display, remove the septum stopper from the electrode head and introduce the sample into the cell.

   It is recommended to inject your sample as soon as the message appears on the display.

   If the auto detection function is activated, the titration will start immediately on detection of the sample.

   If this is not the case, press ✓ to start the titration.
5. Weigh the empty sample container and funnel.

   The value shown on the TIM550's display has been sent from the balance.

   ```
   Sample amount
   - 0.0159 g   ✓
   ```
6. Press ✓ to accept the sample amount and continue the titration.

Blank samples

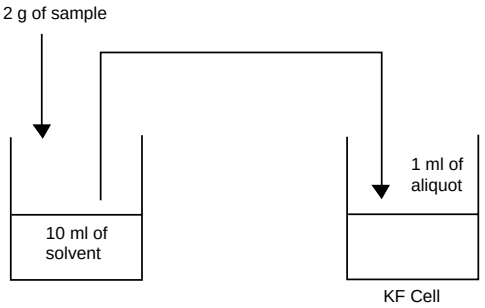
Remove an aliquot of solvent to be used for the dilution of the sample. Follow steps 1 to 5 on page 52. The blank titration procedure is described on page 56.

How to use the dilution volume

Dissolve the sample in the appropriate KF solvent. When throughly dissolved, remove an "aliquot" quantity of sample for analysis.

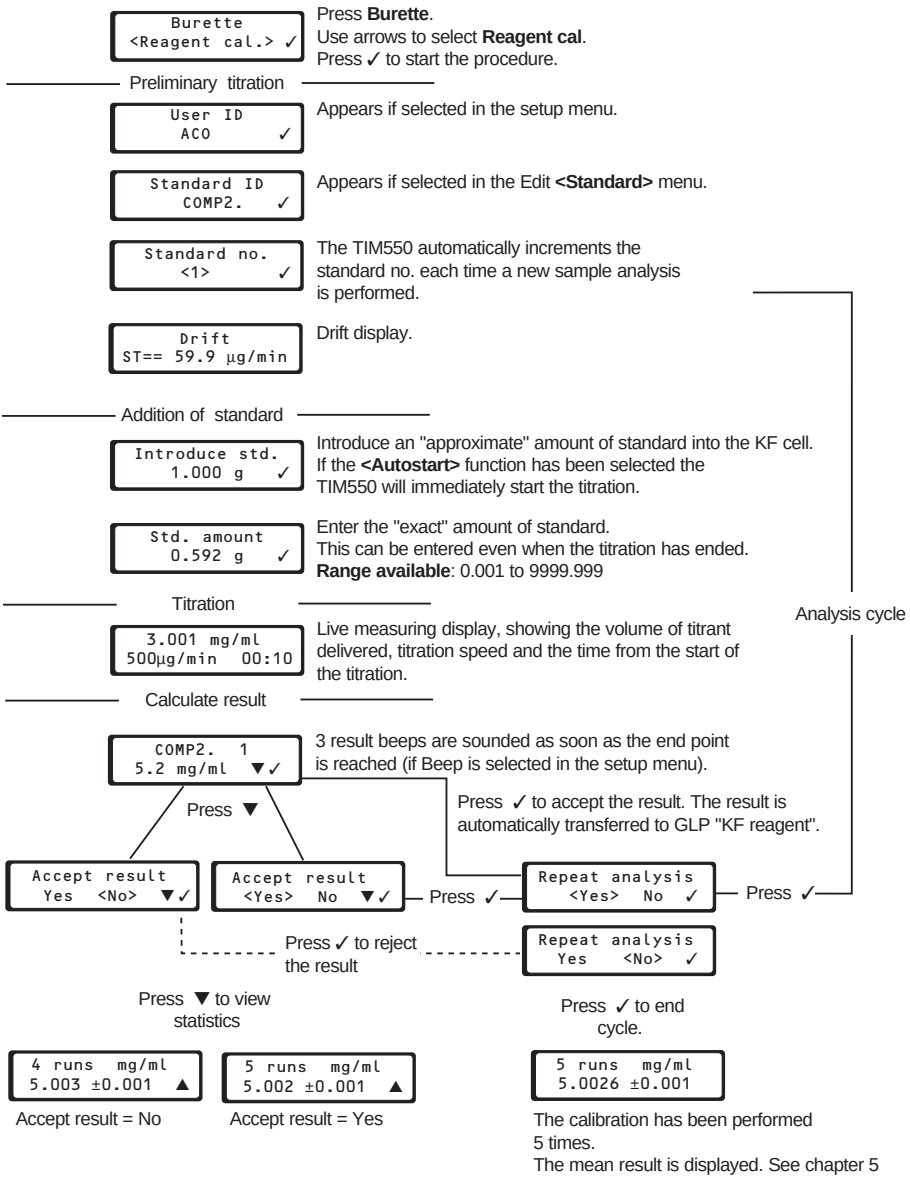
The ***dilution volume*** and ***dilution uncertainty*** is entered in the edit sample menu, when programming a titration using a blank.

Example:

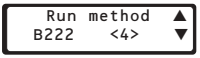


1. Dissolve 2 g of sample in 10 ml of solvent.
2. 1 ml of the diluted sample "aliquot" is added to the KF cell for titration.
3. Enter a dilution volume = 10 ml.
4. For uncertatinty calculations, enter the standard uncertainty of the dilution volume, aliquot and sample amount.

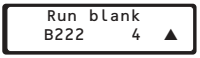
Reagent calibration



Blank titration



Select method no. (1 to 50).
Press ▼ if the option Blank = Yes is selected.



Press **Run** to start the blank determination.

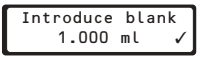
Preliminary titration

Appears when selected in setup menu.

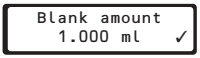
The TIM550 automatically increments the blank no. each time a new blank analysis is performed

Drift display.

Addition of blank

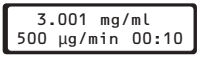


Introduce an approximate amount of blank into the KF cell.
If the **<Autostart>** function has been selected the TIM550 will immediately start the titration.



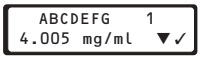
Enter the "exact" amount of blank.
Range available: 0.001 to 9999.999

Titration



Live measuring display, showing the volume of titrant delivered, titration speed and time from the start of the titration.

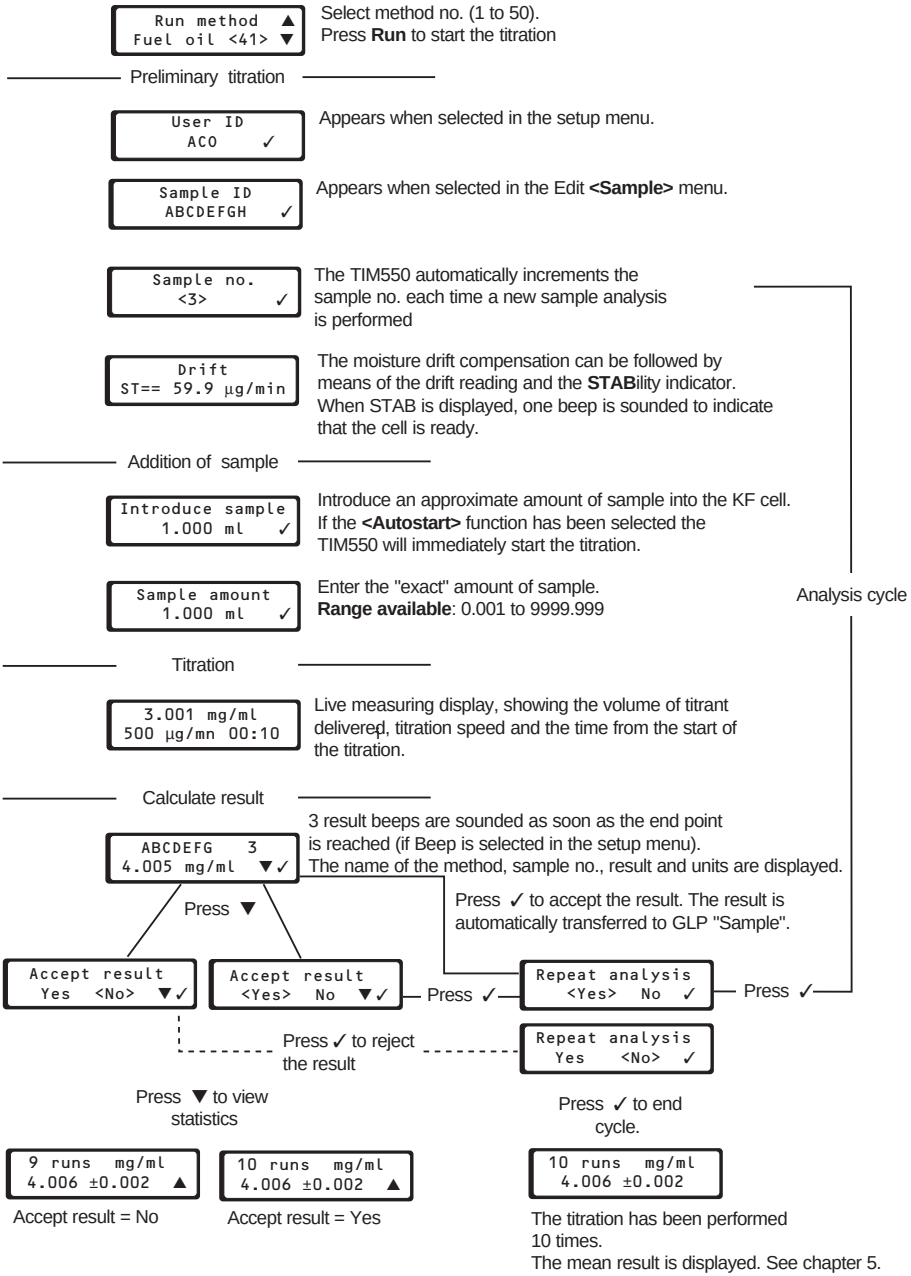
Calculate result



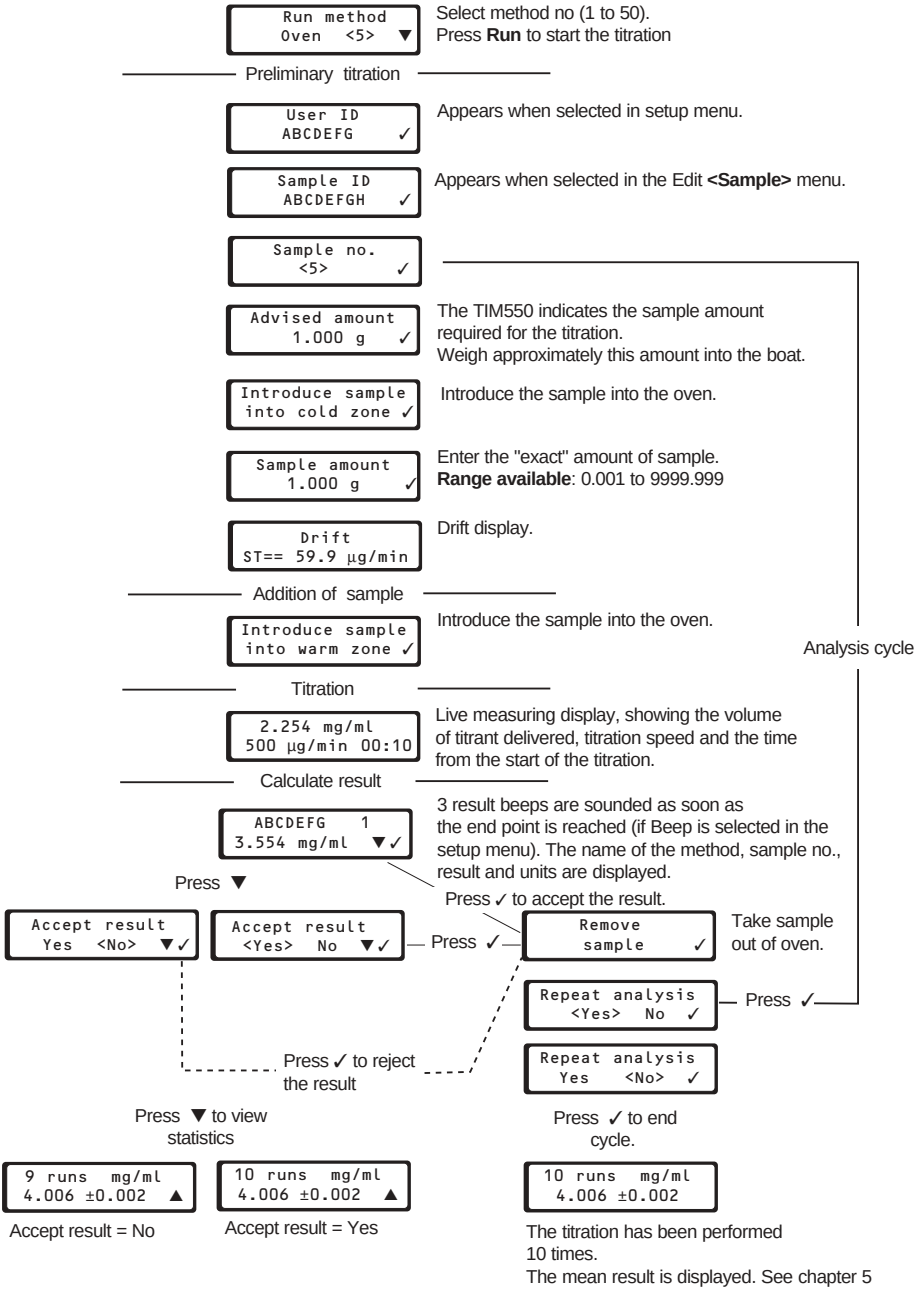
3 result beeps are sounded as soon as the end point is reached (if Beep is selected in the setup menu).
The name of the method, blank no., result and units are displayed.

Follow the procedure given on page 55.
To repeat an analysis cycle.
To accept/reject a result.
To view statistics.

KF titration



KF titration with oven



Pre-programmed methods

The TIM550 offers the following pre-programmed methods. Simply select the method number, press Run and inject the sample when the message telling you to do so appears on the display. The TIM550 will take care of the rest.

These methods are pre-programmed but can be modified to suit your specific needs.

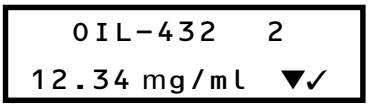
Method no.	***Method ID***	***Titrant***	***Solvent***
41	KNO_3	HYDRANAL® -Composite 2	Mixture of 30 ml HYDRANAL® -Solvent + 15 ml formamide
42	Salicylic acid	HYDRANAL® -Composite 2	HYDRANAL® -Solvent
43	Melamin	HYDRANAL® -Titrant 5	40 ml HYDRANAL® -Solvent + 10 g Salicylic acid at 50°C
44	EDTA	HYDRANAL® -Titrant 5	mixture of 30 ml HYDRANAL® -Solvent + 15 ml formamide
45	Toothpaste	HYDRANAL® -Composite 5	40 ml methanol
46	Skin cream	HYDRANAL® -Titrant 5	40 ml HYDRANAL® -Solvent
47	Acetic acid	HYDRANAL® -Titrant 5	40 ml HYDRANAL® -Solvent
48	Cetone	HYDRANAL® -Composite 5K	40 ml HYDRANAL® -Working Medium K
49	Mayonnaise	HYDRANAL® -Titrant 5	40 ml HYDRANAL® -Solvent CM or 15 ml HYDRANAL® -Solvent + 30 ml chloroform
50	Cereals	HYDRANAL® -Titrant 5	40 ml HYDRANAL® -Solvent

5. Results and archives

The TIM550 automatically calculates the water content of the sample in the chosen units. The drift measured from the start of the titration, the quantity of water introduced by the solvent and the dilution parameters are also taken into account during calculations. If a series of measurements is performed, the TIM550 will calculate the mean value, the standard deviation and the uncertainty on the mean value.

The user is able to accept or refuse the last result obtained and check the impact it might have on the mean result.

Accepting

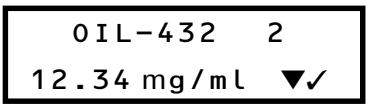


Press ✓.

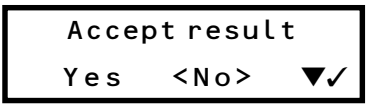
The result is automatically transferred to the GLP table.

Rejecting

From the "result" display at the end of the titration:



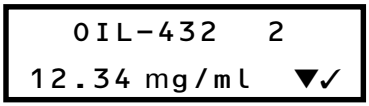
Press ▼.



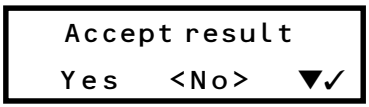
Select No and press ✓.
The rejected result is automatically transferred to the GLP table for consultation.

Viewing statistics

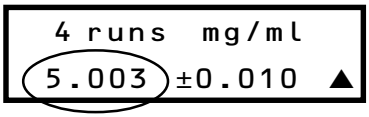
From the "result" display at the end of the titration:



Press ▼.



Press ▼.



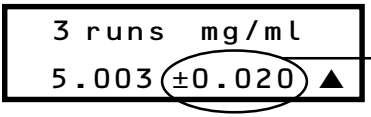
The mean result is given with the corresponding units.

Depending on whether Yes or No is selected above, the mean is displayed for n or n + 1 results.

Press ▲ to return to accept result display.

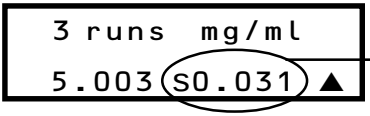
The following results are possible for a series of titrations (no. of runs >1):

1. If ***Uncert. calc. = Yes***



"±" indicates that this is the expanded uncertainty.

2. If ***Uncert. calc. = No***



"s" indicates that this is the standard deviation of the mean (Sm).

Theory

Let R be the concentration to be found during the titration.

The distribution of R (estimated as normal), has the following characteristics:

μ	mean value of the distribution of R
σ	standard deviation of the distribution of R

By definition, these characteristics are limits of mathematical functions and correspond to an infinite number of determinations of R.

Their estimations, *m* and S_{dis} respectively are determined by population sampling, i.e. from *n* titrations, with results *R1*, *R2*, ..., *Rn*:

$$m = \frac{\sum_{i=1}^{n} Ri}{n}$$

$$S_{dis} = \sqrt{\frac{\sum_{i=1}^{n} (Ri - m)^2}{n - 1}}$$

The estimations of the characteristics of the distribution of μ, mean of R are:

m	mean value of the distribution of μ
s_m	standard deviation of the mean of μ where

$$S_m = \sqrt{\frac{S_{dis}^2}{n}}$$

For a series of n titrations where the mean *m* is expressed, the standard deviation of the mean, s_m, is used to represent the dispersion of the results around *m*.

To calculate the standard deviation of the distribution (S_{dis}) using the standard deviation of the mean (S_m) obtained from n measurements, use the following equation :

$$S_{dis} = S_m \times \sqrt{n}$$

Reproducibility

Use the following equation to calculate the reproducibility:

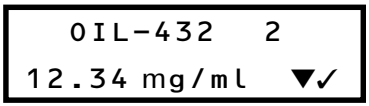
$$Reproducibility = \frac{S_{dis}}{mean\ result} \quad x \quad 100$$

This value can be entered in the reproducibility display in the Edit mode, if the Uncertainty calculation option has been selected. Refer to pages 39 and 44.

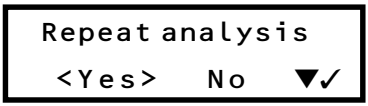
For further details refer to the Uncertainty Plug-in (R31T009).

Successive titrations

1. From the "result" display at the end of the titration:

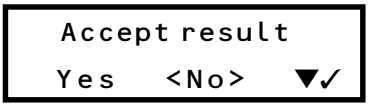


Press ✓.

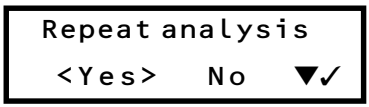


Select Yes and press ✓.

2. From the Accept result display:



Select Yes or No and press ✓.



For more analysis, select yes and press ✓.

Select no to stop the titration cycle.

GLP Table

The GLP table is divided into 3 groups:

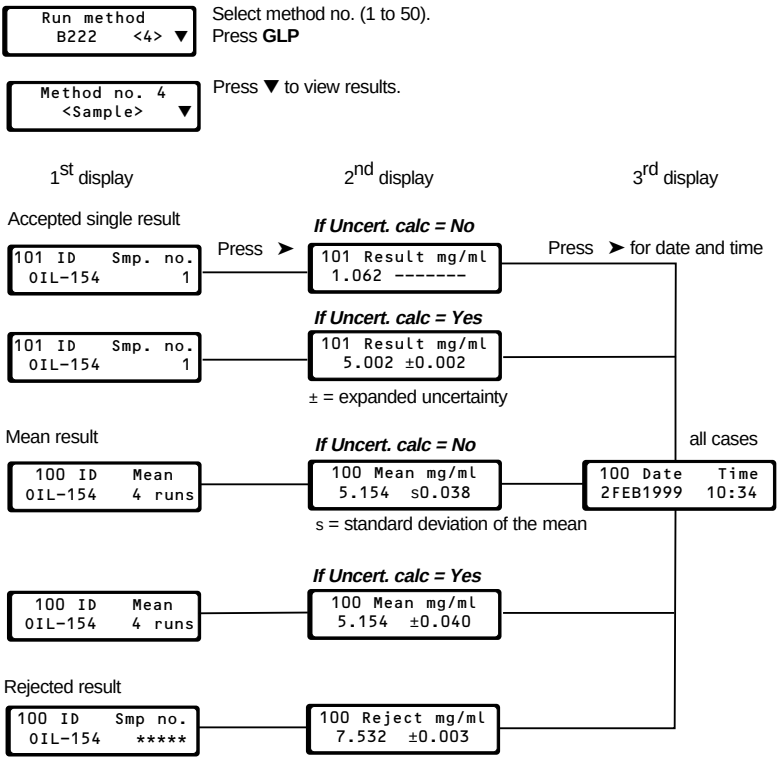
- **Sample**: groups titration method results for all the methods.
  - Last 200 results are stored in chronological order.
- **Blank**: groups blank results.
  - Last 10 blank results are stored in chronological order.
  - Last blank result is stored for each titration method.
- **KF reagent**: groups manually entered or calibrated KF reagent titres.
  - Last 10 calibration results are stored in chronological order for each reagent.

When the **GLP** key is pressed followed by the **Print** key a summary of either the sample, blank or KF reagent data is printed out; refer to Printouts.

The GLP data are accessible in the following way:

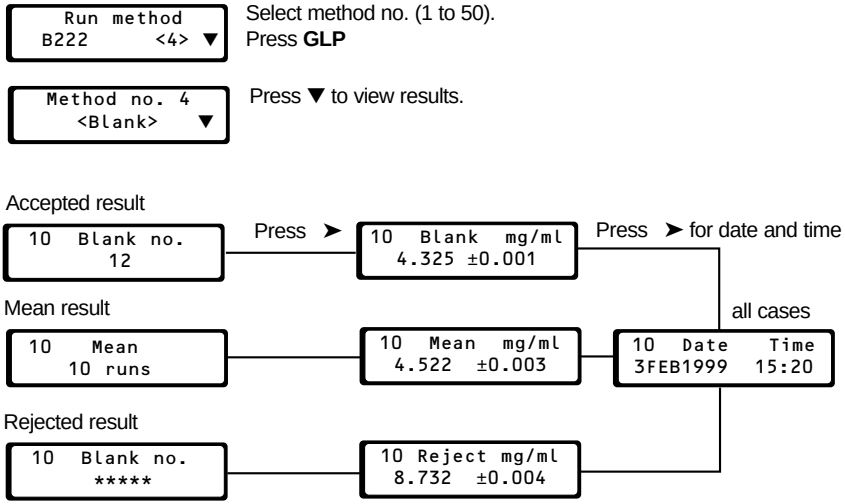
1. Press **Method** and select a method using the **arrow** keys.
2. Press **GLP**.
3. Using the **arrow** keys, select **Sample**, **Blank** or **KF reagent**.
4. Press ▼ to view the **GLP** data.

Sample GLP

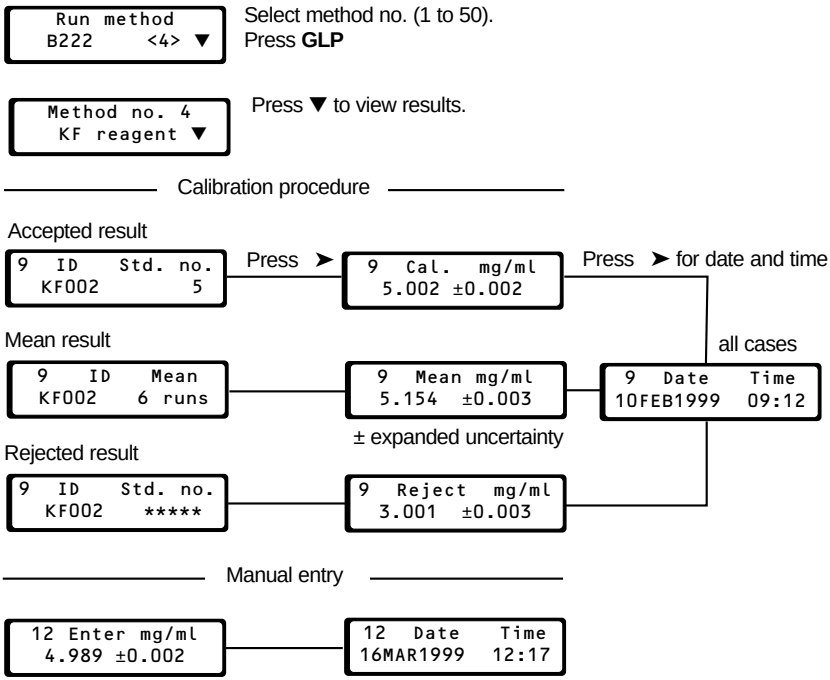

Run method
B222 <4> ▼
Select method no. (1 to 50).
Press GLP
Method no. 4
<Sample> ▼
Press ▼ to view results.
1st display
2nd display
3rd display
Accepted single result
101 ID Smp. no.
OIL-154 1
Press ➤
If Uncert. calc = No
101 Result mg/ml
1.062 -------
Press ➤ for date and time
101 ID Smp. no.
OIL-154 1
If Uncert. calc = Yes
101 Result mg/ml
5.002 ±0.002
± = expanded uncertainty
Mean result
100 ID Mean
OIL-154 4 runs
If Uncert. calc = No
100 Mean mg/ml
5.154 s0.038
s = standard deviation of the mean
all cases
100 Date Time
2FEB1999 10:34
100 ID Mean
OIL-154 4 runs
If Uncert. calc = Yes
100 Mean mg/ml
5.154 ±0.040
Rejected result
100 ID Smp no.
OIL-154 *****
100 Reject mg/ml
7.532 ±0.003

Blank GLP

Only available when Blank = Yes



KF reagent GLP



If the titre is **entered manually**, the uncertainty values which appear in the GLP table are double those entered during programming. This can be explained by the fact that the uncertainty entered is the **standard uncertainty (u)** whereas, the uncertainty displayed in the GLP table is the **expanded uncertainty (U)**.

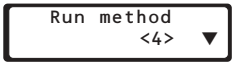
$$U = u \times k$$

k = coverage factor

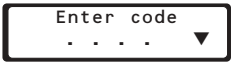
Refer to Guide to Uncertainty Calculations (R31T009)

Resetting the GLP table

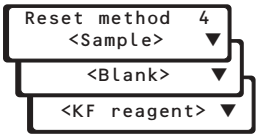
Follow the procedure given below to delete results stored in the Sample, Blank, and/or KF reagent menus of your selected method.



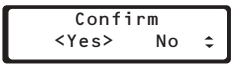
Select the method no. (1 to 50).
Press **GLP** 3 seconds.



Enter your protection code if the edit mode has been protected. Refer to chapter 2.



Use arrow keys to select the menu in which you wish to delete the results.
Press ▼ to continue.



Select yes.
Press ▼ to confirm.

Printouts

An explanation is given here of the printouts available when a printer is connected to the TIM550. Specific information can be found on the following pages, and in the order shown concerning:

- KF titration results
- Blank titration results
- KF reagent titration results
- GLP data
- Edit menu data

How to obtain printouts:

Use this key in association with other keys to obtain your printouts.

When **Method** is pressed followed by **Print**, method menu parameters are printed out.

When **Burette** is pressed for 3 seconds followed by **Print**, KF reagent parameters are printed out.

When **GLP** is pressed followed by **Print**, the results stored in the GLP table are printed out.

Two types of printout formats exist. You can select 80 columns (detailed) or 40 columns (condensed) in the setup menu; refer to Chapter 3.

KF titration, calibration and blank results are printed automatically when a printer is connected.

KF titration results

The following printout is obtained during a KF titration. Each line in the table is printed as soon as the result is obtained.

Detailed - 80 columns

```
*** TIM550 Volumetric Karl Fischer Titrator 679R00N007    10May 1999 16:35
*** Burette              xxxR000N000
*** Electrode            768-11-008

***************************** SAMPLE ************************************

Method  7, VINC

KF reagent titre: 2.000 ±0.040 mg/ml      KF Reagent 5      Date: 07MAY1999

Sample ID: AQUA
```

no.	Sample result mg/g	QC	Time h:min	Sample amount g	Volume ml	Drift µg/min	Dur. min:s
8	4.3689±0.0992	High	16:37	1.015±0.001	2.217±0.012	17± 3	01:07
9	3.8848±0.0903	Low	16:40	1.032±0.001	2.005±0.012	15± 3	01:07
10	4.0629±0.0938	OK	16:43	1.028±0.001	2.088±0.012	46± 4	01:12
**	3.6364±0.0862	Rej.	16:45	1.021±0.001	1.856±0.012	28± 3	01:04

```
Mean: 4.1055 ±0.3435 mg/g          3 runs          10 May 1999       16:46

*******************************************************************************
```

Volume (ml): this corresponds to the volume delivered by the burette during the titration. The uncertainty given is the uncertainty of the volume delivered.

Drift (µg/min): this corresponds to the stable drift reading before the sample is introduced to the KF cell. The uncertainty given is the uncertainty of the drift. This drift value will be used for calculation purposes.

Condensed - 40 columns

```
*** TIM550          10 May1999 16:46
*** Burette         xxxROOONOO
*** Electrode                        DOUBLE Pt

************ SAMPLE **************

Method    7, VINC

KF reagent titre: ±0.040 mg/ml

Sample ID: AQUA
```

no.		Sample result mg/g	Time h:min
12		4.1864± 0.957	16:49
13	H	4.4257± 0.1007	16:52
**	R	4.1099± 0.0955	16:55
15	L	3.9432± 0.0941	16:57

```
Mean: 4.1851 ±0.3430 mg/g

R=Rejected
L = Alarm low          H = Alarm high

************************************
```

Blank titration printouts

The following information is obtained at the end of a blank titration:

Blank no.

Blank result

QC messages: OK; Rej (rejected); Low; High

Time (h:min)

Blank amount (ml)

Volume (ml) of titrant used

Drift (µg/min)

Dur. (min:s)

Mean result

KF reagent calibration printouts

The following information is obtained at the end of a calibration procedure:

Standard no.

KF titre (mg/ml)

QC messages: OK; Rej (rejected); Low; High

Time (h:min)

Std. amount (ml)

Volume (ml)

Drift (µg/min)

Dur. (min:s)

GLP Printouts

To obtain the printout:

1. Press **Method** and select a method from 1 to 50.
2. Press **GLP**.
3. Select ***<Sample>***, ***<Blank>***, ***<KF reagent>***.
4. Press **Print**.

The following information is obtained:

GLP no.

Sample ID

Sample no.

Sample result

QC messages

Date

Time (h:min)

Note: If any of the following data for the selected method change then a new table will be printed out:

a. Burette serial no.

b. Electrode serial no.

c. KF reagent titre

d. Blank

Edit method data

1. Select the Method (1 to 50).
2. Press **Method** for 3 seconds to enter the edit mode.
3. Press **Print**.

You will obtain a printout of the method menu parameters.

Edit KF reagent calibration data

1. Press **Burette** for 3 seconds to enter the edit mode.
2. Select the KF reagent titre (1 to 20).
3. Press **Print**.

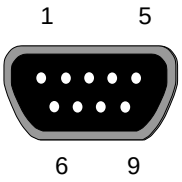
You will obtain a printout of the parameters used for the calibration procedure.

6. Connection of peripherals

The TIM550 Volumetric KF Titrator is equipped with terminals for connection of Printer/PC, balance, PC keyboard/bar code reader and platinum electrode. The terminals in the rear panel are illustrated in chapter 2.

Connection of PC

RS232C specifications: 9-pin, sub D-connector



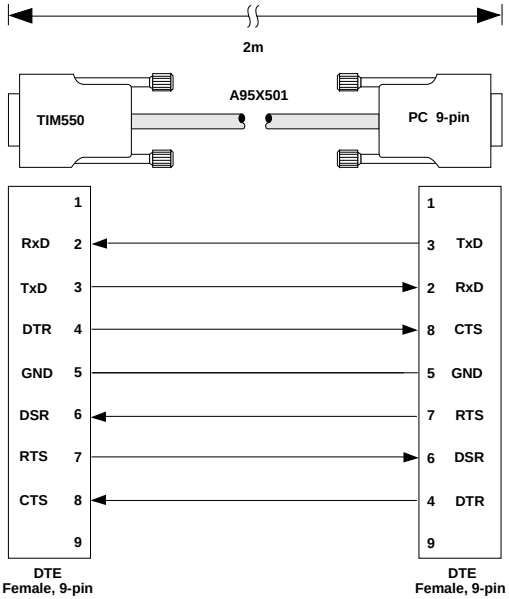
Pin connections:

1. No connection	
2. Receiving Data (input):	RxD
3. Transmitted Data (output):	TxD
4. Data Terminal Ready (output):	DTR
5. Ground:	GND
6. No connection:	DSR
7. Request to Send (output):	RTS
8. Clear to Send (input):	CTS
9. No connection	

For the connection of a personal computer, use the cable, 9-9 pin, part no. A95X501.

See page 78 for the pin layouts of the cables.

Pin layout of the A95X501 cable

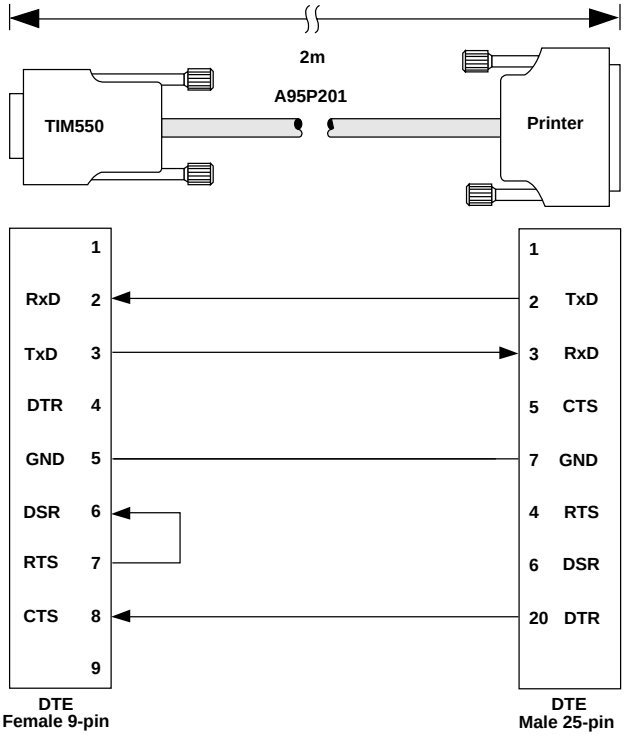


Connection of a printer

For the connection of a printer, use the cable, 9-25 pin, part no. A95P201.

The printer must have the following characteristics for connection to the TIM550.

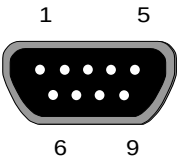
- Printer 40 or 80 characters
- RS232C interface
- 9600 baud, no parity, 8 data bits, 1 stop bit.
- Flux control via the DTR line (pin 20 on the 25-pin plug).
- Printout of tables
- IBM Fonts; character sets



Note: to connect the Kyoline MTP640 Thermal Pocket Printer, part no. A70P020 (230 V), A70P021 (115 V), use the cable 5-9 pin, part no. A95X506.

Connection of a balance

RS232C specifications: 9-pin, sub D-connector: 2400 baud, even parity, 7 data bits, 1 stop bit.



Pin connections:

1. No connection
2. Receiving Data (input): RxD
3. Transmitted Data (output): TxD
4. Data Terminal Ready (output): DTR
5. Ground: GND
6. No connection: DSR
7. Request to Send (output): RTS
8. Clear to Send (input): CTS
9. No connection

Balance configuration: automatic transmission (autosend or autotransmit).

Communication may need some programming on the balance; please refer to balance operating instructions.

The following balances can be connected to the TIM550:

Balance	Type	Cable part no.	Cable length
Mettler	PE + option 016	A95Z201	1 m
	AE + option 012 or 011	A95Z205	2 m
	AM, PM, SM, AT, MT, UMT, PJ, AJ	A95Z206	1 m
Sartorius	MasterPro	A95Z202	1 m
	QC MP8-4	A95Z204	2 m

See page 82 for the pin layouts of the cables.

Note: cables can be made to your desired specifications. Please contact our sales representative if you require information regarding the type of cable to use with your balance.

Format of messages accepted by the TIM550:

- Messages end: LF or CR, LF.
- Units: g, mg, kg.
- The character “g” of the unit is required to mark the end of the data recognised by the instrument.
- Spaces are ignored.
- The number of characters situated between the first character in the message and the "g" in the unit must not be more than 30. (spaces not included).
- The number of characters received after the "g" and before the end of the message LF, is insignificant.
- The numeric data corresponding to the weight must be transmitted before the unit.

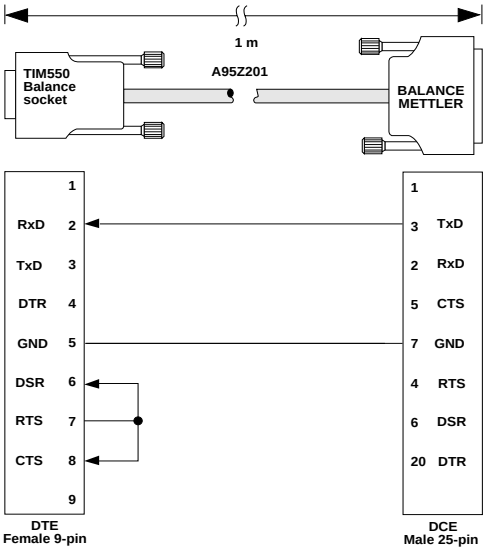
Example of a message transferred from a balance, type Mettler.

S	I		1	2	.	3	4	5	6	g	CR	LF
			data received: 12.3456 g									

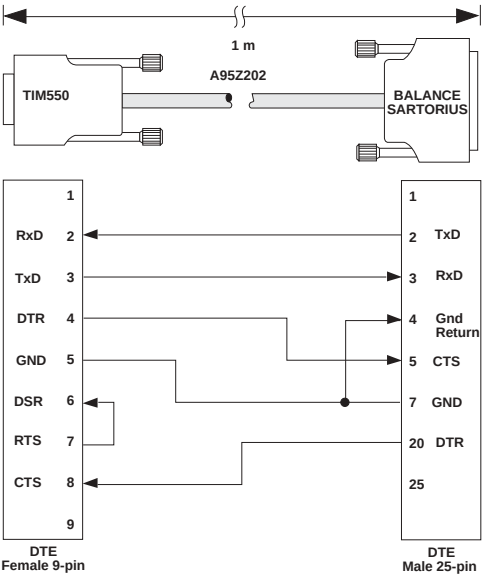
IMPORTANT!

The units selected in the TIM550 must be identical to the units used by the balance.

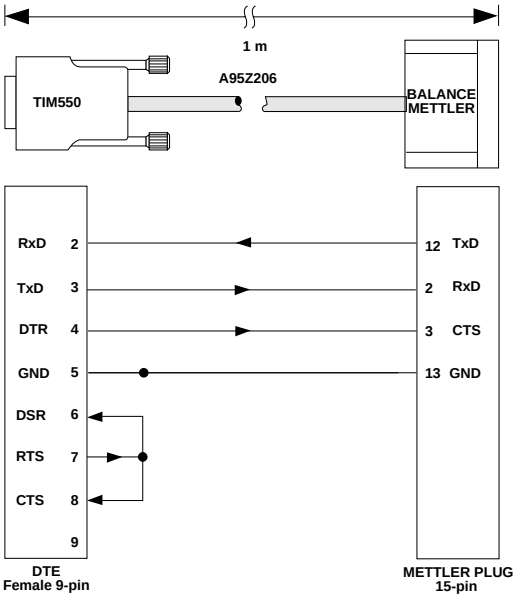
Pin layout of the A95Z201 cable



Pin layout of the A95Z202 cable



Pin layout of the A95Z206 cable

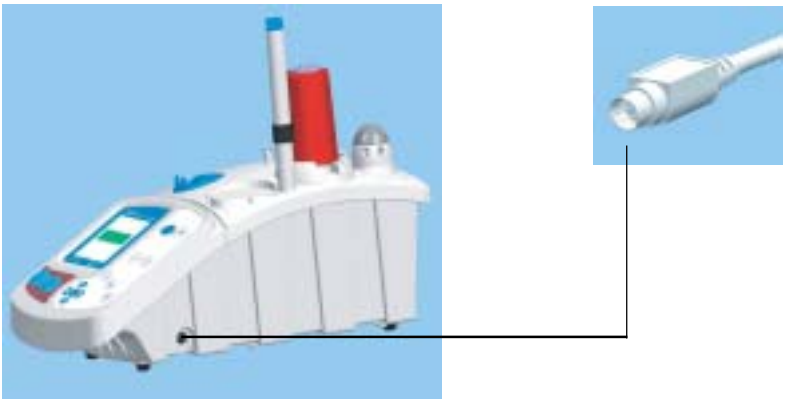


Connection of keyboard and bar code reader

A keyboard and/or a bar code reader can be connected to the TIM550 via the 6-pin mini DIN port situated on the right hand side of the instrument.

Note: you can also connect the keyboard to the 6-pin female socket on the bar code reader.

Keyboard type: PCT or compatible, with a 6-pin mini DIN connector.



Radiometer Analytical offers two types of keyboards:

The **Notebook Keyboard**, used for alphanumeric entry. Is a full 83-key layout with 101 key functionality, available in various languages.

The **Numeric Keypad**, used for numeric entry only, allows you to program 4 keys for a dedicated function (e.g. Run, Cell).

A Notebook Keyboard Mask, part no. X31T108 indicating keyboard functions (see next page) is available for use with the mini keyboards.

Keyboard functions

In combination with the TIM550 the keys of the PC keyboard perform predefined functions. Refer to table below.

Keyboard command	*TIM550 key action*	*TIM550 operation*
<F1>	Run	Starts the displayed method
<F4>	<Enter titre>	Manual entry of KF titre
<F5>	Method	Display method
<F6>	Cell	Operate cell functions
<F7>	Burette	Operate burette functions
<F8>	GLP	Access GLP table
<F9>	Press ✓ during autotest	Edit Setup menu
<F10>	Method key 3 seconds	Edit method
<F11>	Burette key 3 seconds	Edit burette
<F12>	GLP key 3 seconds	Edit GLP
<Enter>	Check mark/Print	Confirmation of the input
<Esc>	Method key	Stop titration
<Pause>	Press and release ✓ key	Stop solvent addition
<Up arrow>	Up arrow	Within the edit menu, parameters can be scrolled
<Down arrow>	Down arrow	Within the edit menu, parameters can be scrolled
<Left arrow>	Left arrow	Select an option which appears within the arrows on the display.
<Right arrow>	Right arrow	Select an option which appears within the arrows on the display.
<Inser>	-	Insert text
<Del.>	-	Delation of an input digit/ character under which the cursor is positioned.
<Home>	-	Go to start of text
<End>	-	Go to end of text
<Numeric keypad>	-	Numeric input is possible.
Page up/page down	A...Z keys	Alphanumeric input
Print screen	Print	Printout data
QWERTY Keyboard		Alphanumeric input possibleparameters

7. Troubleshooting

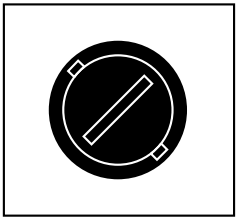
Changing the battery

The 3.6 V lithium battery, part no. 430-127, which has a lifetime of minimum 2 years, should be replaced when the following message appears:

```
BATTERY LOW!
                ✓
```

The battery's function is to ensure the titrator's permanent memory facility. **The battery must be changed while the instrument is switched on to avoid loss of data in the memory.**

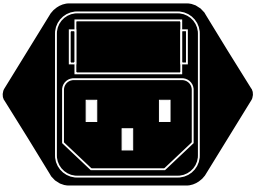
Change the battery in the following way:



1. The cap on the rear plate of the TIM550 behind which the battery is positioned is shown in the illustration.
2. Insert the edge of a coin into the slit on the cap surface and turn.
3. The cap will now open providing access to the old battery which should be removed. The new battery should be positioned with the **+** sign facing towards the **+** sign on the inside of the cap.

WARNING: only replace with a battery of the same type and rating.

Replacing the fuses



1. Disconnect the TIM550. The fuse is positioned just above the power socket on the rear panel.
2. When the two vertical flaps on the cover are gently squeezed together, the cover can be pulled out and the old fuse changed.

   WARNING: for continued protection, only replace the fuse with one of the same type and rating, see table below.
3. Re-insert the cover with the new fuse.

Voltage rating	Fuse description	Part no.
115 Vac	2 x fuses, slow blow, 0.4 A	450-016
230 Vac	2 x fuses slow blow 0.2 A	450-013

   Note: a secondary fuse is mounted on the printed circuit board. If necessary, contact a Radiometer Analytical service representative for replacement of the 2 A, picofuse, UL recognised.
4. Put the cap back in place.

Dismounting the pump module

1. Remove the transparent tubes from the outlets:

2. Unscrew the screws on the pump.
3. Lift out the pump, see figure 1 below.
4. Insert the new module into place and tighten the screws.

Refer to the Pneumatic Module Operating Instructions (D26T021) for further information regarding the dismounting and installation.

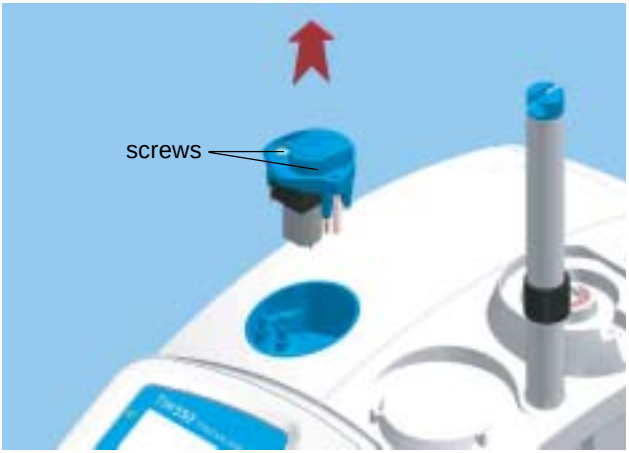


Figure 1: Dismounting the pump module

Error and warning messages

A number of tests are automatically performed when the TIM550 is switched on, during calibration and during titration. If a problem is detected, an error/warning message will appear on the display. The messages are listed below.

By pressing ✓, the TIM550 will normally return to the operational state in which the error was detected. This makes it possible to correct the error and immediately resume operation.

If any of the error messages cannot be cleared by actions listed, contact your Radiometer Analytical representative at the adress given on page 103.

Message	***Problem***	***Action***
Battery low!	The battery is run down and must be replaced as described on page 87.	Replace the battery. Press ✓ to continue.
Wrong access key!	This message is displayed when an incorrect access key has been entered to unlock the uncertainty calculations.	Enter the correct code and press ▼ to continue.
Invalid entry!	This message appears when an entry is made which lies outside the available range.	The entry is replaced by the previous value.
Memory error!	This message is displayed at power on if the TIM550 detects an error in the data stored in the non volatile memory. This message is generally displayed after a battery failure.	Press ✓. Default values are used instead of the erroneous data. If this error remains on the display after the battery has been changed, contact your Radiometer Analytical representative.
Wrong code!	This message is displayed when an incorrect protection code has been entered.	Enter the correct code in the Enter code display. Press ▼ to continue.

Burette errors

Message	*Problem*	*Action*
Burette not available	This message is displayed if a burette function Fill, Empty, Flush, Rinse, Bottle exchange or New titrant is requested after pressing the Run key or when the Stand by is activated.	Press ✓ to continue.
BURETTE ERROR 2 BURETTE ERROR 3 BURETTE ERROR 4 BURETTE ERROR 5	These errors may occur when the burette is performing a Fill, Empty, Flush, Rinse, Bottle exchange or New titrant procedure and an operational error has occured to the burette. 2 - Stopcock sensor ON position not found 3 - Stopcock sensor OFF position not found 4 - Piston sensor ON position not found 5 - Piston sensor OFF position not found	If any of the following error messages occur contact your Radiometer Analytical representative or Radiometer Analytical at the address given on page 103. Please remember state the error number 2, 3, 4 or 5.

Cell errors

During a titration cycle

Message	*Problem*	*Action*
Cell almost full <Empty cell>	This messages warns the user that it is time to empty the cell.	Press ✓ to empty the cell.
Cell almost full <Continue>	The user can either empty the cell or continue the titration.	Press ✓ to continue the titration.
Cell full empty cell	This message appears when the contents of the KF cell reach maximum level. The user must empty empty the cell, before continuing the titration.	Press ✓ to empty the cell. Select *Return titr.* to continue the titration.

After a manual key press, e.g. add solvent

Message	*Problem*	*Action*
Cell full empty cell	This message appears when the contents of the KF cell reach maximum level. The user must empty empty the cell, before continuing the titration.	Press ✓ to empty the cell.
Molecular sieve saturated	This message appears when the Empty cell function is activated and the number of empty cells is greater than 50 or the molecular sieve replacement date is greater than 7 days. It is time to replace the molecular sieve.	Replace molecular sieve and press ✓ to reset the empty cell and replacement date counters.

Titration start errors

Message	*Problem*	*Action*
Blank titration <Yes> Blk = 0	This message appears at the start of a sample titration. A blank value is not available for the titration i.e. Blank GLP is empty.	Select <Yes> and press ✓ key to start the blank procedure. Select <Blk = 0> to perform the titration using a blank value = 0.
Cal time elapsed <New Cal> Cal time elapsed <Continue>	This message appears when the option KF reagent titre = Calibrated. A new calibration is required. The delay entered in the "Cal every" display has elapsed; see page 39.	Select <New Cal.> and press ✓ to start the calibration procedure. Select <Continue> and press ✓ to continue the titration using the current reagent titre. Note: this choice is not available if the user has entered a "protection code" in the setup menu. The user is therefore forced to perform a new calibration by pressing ✓.
Check solvent level	There is no solvent in the KF cell.	Press Cell and add solvent. Select <Return titr.> to continue the titration.
Enter titre <Yes> No	This message appears when the option KF reagent titre = Entered. Either the KF reagent GLP is empty or the titre stored in the GLP was determined using the calibration procedure.	Select <Yes> and press ✓ to enter KF titre manually. Select <No> and press ✓ to return to the method menu.
H20 displayed in the drift display.	Excess water in KF cell.	The TIM550 will add reagent until the drift reading is stable. One beep is sounded to indicate that the cell is ready for titration.

Message	*Problem*	*Action*
I2 displayed in the drift display	Excess iodine in KF cell. This generally indicates that the maximum burette speed is too high.	If conditioning of the KF cell takes some time: 1. Inject a few µl of water into the cell. 2. Press Cell and add some solvent to the cell. Select <Return titr.> to continue the titration. 3. Decrease the burette speed (see page 45).
Perform Cal. <Yes> No	This message appears when the option KF reagent titre = Calibrated. Either the KF reagent GLP is empty or, the titre stored in the GLP was entered manually.	Select <Yes> and press ✓ to start to the calibration procedure. Select <No> and press ✓ to return to the method menu.
Cell not available	This message is displayed at the start of a titration if the KF cell is in the process of emptying.	Wait until the KF cell has finished emptying. Press Run to start the titration.
Printer not available	This message is displayed if the printer is off-line.	Turn the printer on-line and press ✓ to continue.

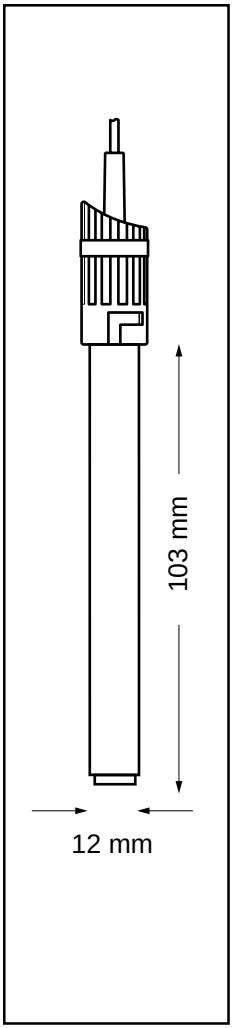
Titration end errors

Message	*Problem*	*Action*
Maximum volume reached!	This message is displayed if thec maximum volume of titrant is delivered before the end point is reached. No result obtained.	Change the Maximum volume setting in the edit method menu or the KF reagent library or decrease the sample amount. Press Run to perform a new titration.
Maximum time reached! Alarm Tmax	This message is displayed if the maximum titration time (time entered in the edit menu) edit has elapsed before the titration has ended. A result is obtained with the alarm message Tmax.	Change the Maximum titration time setting in the method menu or the KF reagent library. Press Run to perform aµ new titration.
Alarm low Alarm high	This message is displayed if the Quality Control option has been selected in the edit menus for a titration or a calibration. The final result is above (Alarm high) or below (Alarm low) the QC parameter setting.	If necessary, check the mininum and maximum values entered in the edit menus. For a reagent calibration refer to page 41. For a KF titration refer to page 48. Press ▼ to accept or reject the result.

The use of this instrument may jeopardise the security of all living beings (uses that may, for example, cause burns and electric shocks...). These are considered as being improper and are therefore forbidden.

8. M241Pt2-8 Double Platinum Electrode

The M241Pt2-8 Double Platinum Electrode is specially designed for use with the TIM550 Volumetric KF Titrator.



Preparing for measurement

1. Remove the protection cap from the electrode.
2. Before starting a measurement, rinse the electrode with a suitable solvent.
3. Check that the electrode body and sensing element are not damaged.
4. Place the electrode in the KF cell.
5. When stirring, make sure that the platinum wires are covered by the solvent.

Cleaning and maintenance

Platinum wires

In order to have reproducible and accurate results with the electrode, treatments may be required. These treatments will depend on the last measurement performed:

Mild cleaning

- Place the electrode overnight in RENOVO•N Normal Cleaning Solution.

Degreasing

- Remove mineral oils by rinsing the electrode with trichlorethylene and dabbing dry with a soft tissue.

After cleaning

- Dab the electrode dry with soft tissue, before performing the titration.

Chemical cleaning

- The electrode can be cleaned with a sulphochromic solution*, or with a boiling solution of hydrochloric acid*.

* **WARNING**: hazardous corrosive chemical!

Storage

- Replace the protection cap, and store the electrode in it's box at room temperature.

Accessories

Description	Part No.
○ RENOVO•N Normal Cleaning Solution, 250 ml	S16M001

Specifications

Temperature range:	-10 to 100°C
Sensing element:	2 platinum wires
Diameter of Pt wire:	0.5 mm
Distance between the Pt wires:	4 mm
Connection:	BNC plug
Cable length:	0.55 cm
Min. immersion depth:	22 mm

9. General information

Specifications

Measuring ranges:	0.1 to 100%
Resolution:	0.5 µg of water
Uncertainty:	refer to Guide to Uncertainty Calculations (R31T009)
Electrode Input (BNC):	1 AC polarised electrode input ±56 µA ±5%
	Voltage range: ±2000 mV Resolution: 0.1 mV Uncertainty: < ±4 mV Resistance: 71.5 kΩ
Inputs/Outputs:	2 RS232C serial ports for Printer/PC and additional TIM550 connections
	1 serial port for balance
	1 Mini-DIN port for PC keyboard and/or bar code reader
Methods:	Volumetric KF titration
	KF reagent standardisation
	Blank determination
Data storage:	50 user-programmable methods with alphanumeric name and protection facility including 10 pre-programmed applications
GLP Functions:	TIM550 storage capacity for 20 KF standards, 20 KF titrants, 20 titrant standardisation 200 results, 10 blank determinations
Stirring:	11 reproducible speeds: 100, 200, 300, 400, 500, 600, 700, 800, 900, 1000 and 1100 rpm
Typical titration time:	2 to 5 minutes
Printouts:	Condensed and detailed GLP result bulletins.
	Printout of methods, GLP tables
Working temperature:	5 to 40°C
Ambient temperature:	Metrological characteristics guaranteed between 15 and 30°C

Relative humidity: 20 to 80%

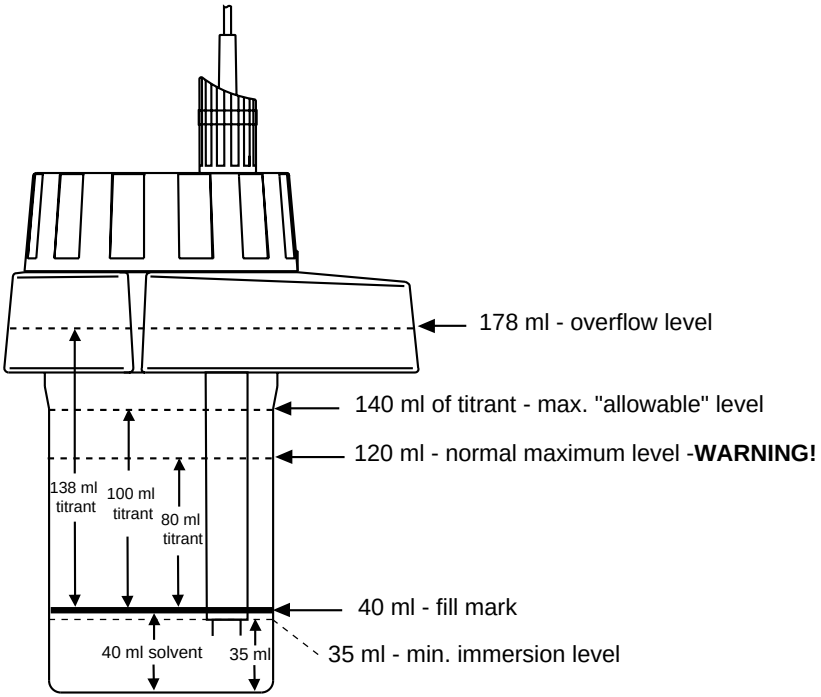
Fuse requirements: Main fuses: slow blow 0.2 A (230 V)
Secondary fuse: 2 A picofuse, UL recognised

Power requirements: 47.5 - 63 Hz; 115/230 Vac +15 -18%

Position of selector switch	*Mains supply voltage range (-18% ... +15%)*	*Typical current (to +15%)*	*Max. current*
115 V	[94 V ... 133 V]	200 mA	400 mA
230 V	[188 V ... 265 V]	100 mA	200 mA

Level of pollution: 2
Transitory overvoltage: level II
Display: 2 x 16 character, alphanumeric LCD display
Finish: chemical resistant, splashproof cabinet
Weight: 5 kg (excluding bottles)
Dimensions (H x W x D): 380 x 230 x 450 mm (excluding tubings)
Languages: English, French, German, Danish, Spanish and Italian

KF cell



Pneumatic module

Approximate filling time:

Working cell volume 35 ml: approximately 5 seconds

Approximate emptying time:

Total cell volume 140 ml: approximately 10 seconds

Minimum needle length required: 115 mm

Functions:

Solvent addition
Moisture protected by dessicant cartridge
Protected against KF corrosive vapours
Solvent/waste push button commands
Timer on cell emptying function

Burette

Syringe volume: 10 ml

Resolution: over 18 330 steps

Trueness: <± 20 µl

Trueness: the closeness of agreement between the average value obtained from a large series of test results and an accepted reference value (*).

Precision: ±7 µl

Precision: the closeness of agreement between independent test results obtained under stipulated conditions (*).

Maximum filling time:

Total burette volume: 20 seconds

Maximum empting time:

Total burette volume: 30 seconds

Functions:

Flush, Fill, Empty, Rinse

Titrant exchange procedure

Glass syringe with plastic cover UV protected

PTFE piston

(*) *According to ISO 5725-1, 1994 edition and ISO 3534-1, 1993 edition*

Transporting the TIM550

Always use the packaging supplied by the manufacturer.

IMPORTANT!

Remove the metal rod before transporting the TIM550.

Never pick-up or carry the TIM550 by the metal rod.

Servicing

DO NOT ATTEMPT TO SERVICE THIS PRODUCT YOURSELF, except as noted in this manual. For servicing, please contact your Radiometer Analytical representative at the address given below or:

_____

_____

_____

or

Radiometer Analytical SAS
72, rue d'Alsace
69627 Villeurbanne Cedex - France

Tel: +33 (0) 4 78 03 38 38
Fax: +33 (0) 4 78 68 88 12
E-mail: radiometer@nalytical.com

Warning: we accept no responsibility when using the TIM550 and its peripheral devices under conditions that are not specified in this User's Manual.

Disposal of Karl Fischer reagents

Dispose of Karl Fischer reagent and cleaning solutions in a proper manner. Refer to the saftey data sheets for the reagents to identify chemical hazards. Follow all local regulations regarding the disposal of chemical wastes.

Cleaning the TIM550

The exterior surface of the TIM550 can be cleaned with a soft cloth and tepid water. In the case of spillages from the titration cell or waste bottle, it is recommended to clean the surface with the KF solvent e.g. Hydranal solvent.

Regenerating the molecular sieve

If you are using a one-component reagent, *it is necessary to regenerate the molecular sieve once a week*. For two-component reagents, regenerate the molecular seive after having performed 50 fill/empty cycles with the KF cell.

The regeneration procedure takes place in two stages:

1. Wash with deionised water to eliminate SO_2.
2. Place in an oven (temp. 250 to 300°C) to eliminate the water.

Procedure

1. Empty the molecular sieve into a tall beaker.
2. Slowly pour deionised water over the molecular seive until fully immersed. WARNING: risk of splashing, due to the release of SO_2.
3. Shake the beaker.
4. Pour off the water.
5. Repeat steps 2 to 4, four to five times.
6. Dab the molecular sieve with an absorbent paper towel.
7. Place the molecular sieve in a crystallising dish.
8. Place the crystallising dish in a hot oven (temp. 250 to 300°C) for at least one night.
9. Place the molecular sieve in an airtight flask, ready to be re-used.
10. Activate the Molecular Sieve function in the Cell menu, refer to page chapter 3.

International Standards

EMC Directive (89/336/EEC)

The TIM550 complies with the following standards:

Emission:	EN 55022, Class B (for conducted emission)
	EN 55011, Class A (for radiated emission)
Immunity:	EN 61000-4-2 level 2 for contact discharge and level 3 for air discharge.
	EN 61000-4-3 level 2
	EN 61000-4-4 level 2
	EN 61000-4-5 level 2
	EN 61000-4-6 level 2
	EN 61000-4-11

Low Voltage Directive (73/23/EEC)

The TIM550 complies with the following standard:

Reference standard: EN 61010-1