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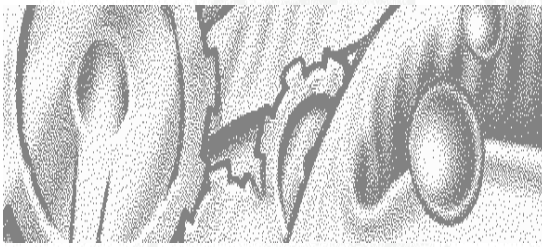
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Autotitrator User manual

MAN 0290 Issue 2.0 July 1999



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Addendum

M A L V E R N I N S T R U M E N T S

MANUAL N°	MAN0290 issue 2.0 and MAN0151 issue 2.0
DATE OF ISSUE	01/01/01
ADDENDUM N°	ADD0052 - 1.1

The design of the pH probe used on the MPT1 Autotitrator and the Zetasizer range has changed. The information given below should be read in conjunction with the maintenance section of MAN0290 (MPT1)

Maintenance instructions for liquid-filled pH probes

Liquid filled electrodes in general exhibit fast response times and stable outputs in difficult environments. However a small amount of maintenance is necessary mainly to ensure that there is always sufficient electrolyte to cover the wire of the reference electrode.

For optimum performance these instructions should be followed.

Preparation before first use

- Remove the screw end-cap and connect to the cable supplied.
- Remove the membrane protection teat. This teat contains a small amount of saturated KCl solution (3.8M).
- To allow air pressure equalization in the reference junction part of the probe, remove the blue rubber cover.
- Check that the reference electrolyte is about 1cm below the level of the opening. Top up with the electrolyte supplied if necessary.
- Check that there are no air bubbles within the inner glass membrane. Bubbles may be removed by shaking the electrode using a similar action used with a clinical thermometer.

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Calibration

The accuracy and speed of the titration is very dependent on correct pH electrode calibration. Weekly calibration is recommended, and should be carried out in accordance with the instructions below:

- Rinse the electrode in deionized/distilled water, and dab dry with a good quality tissue. (one that does not shed fibres).
- From the sample tab within the titrator software, select ‘pH calibration’.
- Enter the two buffer values before starting the calibration. Buffers of pH 4 and pH9.2 are supplied with the electrode. NB. If possible ensure that the calibration buffer solution temperature is the same as the sample under test.
- Start the calibration procedure and follow the instructions given.
- At the end of the calibration, one of three messages will appear:
    1. ‘Calibration complete’, select ‘OK’ and continue with the measurement.
    2. ‘The pH probe response was slow. Please clean your pH probe and recalibrate’, check the advice given later.
    3. ‘The sensitivity of the pH probe is low. The pH probe may need replacing’, check the advice given later or replace the electrode.

Storage

For optimum performance the electrode should be stored upright and with the glass membrane immersed in (an acidic) KCl solution. If the electrode does dry out, then it may be possible to reactivate the membrane by soaking overnight in KCl solution. However if this is not successful then it will be necessary to try the techniques described later. Should these fail then the electrode will need to be replaced.

If the electrode is not being used for a period of, say, more than a week, then it is advisable to protect the glass membrane by replacing the protective rubber teat containing a small quantity of the electrolyte solution. It is best to squeeze the end of the teat first to make it easier to slide on and also to prevent electrolyte being forced back into the probe.

Maintenance

This electrode operates over the range of pH 0 – 14, and 0 – 70 deg. C. It may be used at higher temperatures, but the operating life will be greatly reduced.
Care must always be taken to ensure that the sample and its dispersant do not react with the electrode’s electrolyte, (KCl saturated with AgCl). Problems may be caused by solutions that contain heavy metals and proteins.

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The glass membrane may be dried between measurements by ‘dabbing’ (never by rubbing) with a tissue.

Care must always be taken to ensure that the cable connections are kept clean and dry, and not exposed to corrosive elements.

Common Problems

- pH electrodes are versatile but their performance is dependent on the sample being tested. For example, pH measurement of high purity water or liquids containing sulphur ions will require special electrodes.

- Slow response can often be caused by the build-up of deposits on the glass membrane or frit. Try initially to clean by dipping into concentrated Nitric Acid for 10 seconds. If this is unsuccessful, repeat for one minute. If still unsuccessful then regeneration may be possible by dipping the membrane for one minute into a solution of 2% HF and 5% HCl whilst stirring. Should the frit be blocked by proteins then try soaking the electrode in an appropriate organic solvent, or a solution of 10% pepsin in 1Mol HCl. As such actions usually dehydrate the glass membrane it will be necessary to soak overnight in 3M KCl solution. If these actions are unsuccessful then the electrode will need to be replaced.

- Always ensure that the level of the electrode’s electrolyte is above that of the liquid under test. This will prevent electrolyte contamination, and ensure a good electrical contact. The electrode uses about 1ml per day when the blue rubber cover is removed. When in continuous use, weekly top-ups are recommended.

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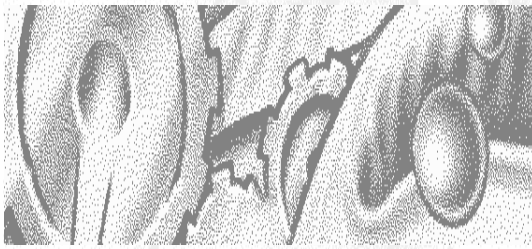
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Introduction



C H A P T E R 1

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Introduction to this manual

This manual covers the operation and maintenance of the MPT-1 multi purpose titrator.

Instrument	*Model number*
MPT-1 multi purpose titrator	*DTS5900*

This manual can be thought of as a stand-alone manual that covers the operation and maintenance of the MPT-1. The aim of this manual is to:

- Identify what the accessory is.
- Explain, in simple terms, how it works.
- Identify the physical features of the accessory.
- Explain how the accessory should be used to make a measurement.
- Identify the user maintenance procedures.

If you have never operated a Malvern particle analyser before, it is recommended that you read this manual fully before you start your first measurement.

If you are more familiar with particle analysers, you may wish to jump straight to chapter 3 which gives a quick guide on making measurements. However, the importance of sample preparation before it is measured can not be over-stated. Therefore it is recommended that the chapters on sample preparation (chapter 3 of MAN0149 & MAN0150) is high on your priority list.



Warning

Use of the system in a manner not specified by Malvern Instruments Ltd may impair the protection provided by the system.

Access to the titrator

Within this manual, reference is made to the various people who will have access to the accessory. Below is a list of these people and their responsibilities:

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Malvern personnel

Malvern personnel (service engineers, representatives etc.) have full access to the accessory and are authorised to perform all service procedures that may require the removal of the covers.

Supervisor

The supervisor is the person responsible for the management/safety of the system and of its operation. The supervisor is responsible for the training of the operators. The supervisor can perform all user maintenance routines identified in chapter 5.

The supervisor must under no circumstances remove the covers of the instrument or accessory.

Operator

An operator is a person trained in the use of the system. The operator can perform all user maintenance routines identified in chapter 5.

The operator must under no circumstances remove the covers of the instrument or accessory.



Warning

Failure to follow these guidelines could result in exposure to hazardous voltages.

Removal of the covers by unauthorised personnel will invalidate the warranty of the accessory.

Assumed information

Naming convention

Within this manual the Zetasizer optical unit will be referred to as the “Optical Unit” or the “Instrument”.

The MPT-1 Autotitrator will be referred to in full or as the “Titrator”.

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The combination of the Optical Unit, one or more accessories and the computer will be referred to as the "System".

Menu commands

Menu commands from the Malvern software are referred to in the form **main menu-menu item**. As an example, the command **Edit-Copy graph**... refers to selecting the **Copy graph**… item in the **Edit** menu. The same rules apply for sub-menus of sub-menus, so that **Edit-Copy table-As picture** refers to the **As picture** item in the **Copy table** sub-menu, which itself is a sub-menu of the **Edit** menu. Menu commands are always shown in **bold** text.

Where to get help

Other manuals

Several manuals are supplied with your system and each one will give information on a different aspect of the system. Below is a list of the typical manuals supplied with your system and details the type of information you will expect to find in each one.

MPT-1 Autotitrator operators guide (this manual)

This manual can be thought of as a basic tutorial. It will take you through the operation of the titrator. After reading this manual you should be a fairly competent operator of the system.

Zetasizer 1000, 2000, 3000 series hardware manual (MAN0151)

Covers all aspects of the main optical unit (except operation). i.e. Health & safety issues, unpacking instructions, system checkouts, identification of features & maintenance. It does not cover any aspect of the Titrator.

Zetasizer 1000/3000 series - making size measurements (MAN0149)

Covers all aspects of making size measurements and interpreting the results using the main software and the optical unit. It does not cover the specific operation of the Titrator.

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Zetasizer 2000/3000 series - making zeta measurements (MAN0150)

Covers all aspects of making zeta measurements and interpreting the results using the main software and the optical unit. It does not cover the specific operation of the Titrator.

Zetasizer software reference manual (MAN0160)

Reference manual that covers all aspects of the main Zetasizer application software it does not cover the Titrator software.

Zetasizer basic manual (MAN0179)

Reference manual covering the Zetasizer programming language.

Zetasizer theory manual (MAN0152)

Manual covering the underlying theory behind the Zetasizer product.

On-line help

The primary function of the on-line help is to give detailed information on the software.

Each dialogue of the titrator software has **Help** and **Advice** buttons that, when pressed, will give help specific to that dialogue.

All help files can be accessed from any **Help** or **Advice** button and then pressing the **Contents** button.

Help desk

All queries regarding your system should initially be directed to your local Malvern representative. You will need to quote:

- Model number of the optical unit and accessories.
- Serial number of the optical unit and accessories.
- Version of the Malvern software.

The serial number of the optical unit and the accessories can be found on the rear panel of each module.

The version number of the Malvern software can be found by starting the software and selecting **Help-About**.

The titrator version can be found by pressing the **About** button in the **Configuration** tab.

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Contact the United Kingdom help desk if your local Malvern representative is not available. The direct line to the United Kingdom Helpdesk is +44 (0)1684 891800. It should be noted that this help line is primarily English speaking.

Remote modem support

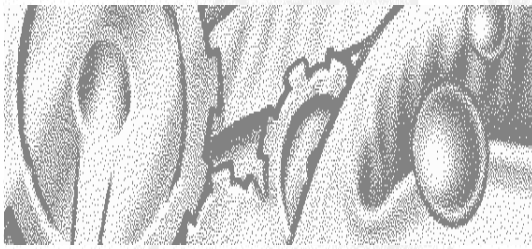
Malvern offers a remote support service, delivered via a modem, for the Zetasizer range. Benefits include fast and efficient fault diagnosis anywhere in the world, reduced costs and less downtime. Online user training is also available, as well as new software or program updates delivered directly via the modem.

The remote modem support package will be installed by the installation engineer, free of charge, providing all necessary site requirements are available when the system is first commissioned.

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Overview



C H A P T E R 2

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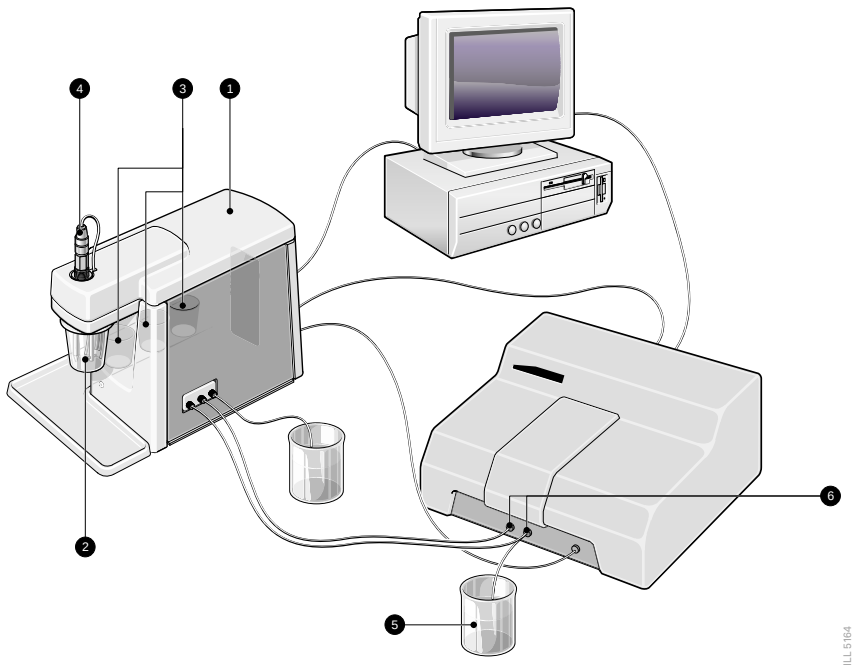
What does the Titrator do?

The principal function of the Titrator is to allow zeta potential, size or intensity measurements to be made (while adding quantities of additives (titrants) to the sample) as a function of another parameter, i.e. pH conductivity or concentration of additive. This allows the effect of adding these titrants on the measurement to be investigated.

The Titrator can also be used (as a separate function) to dispense exact quantities of titrant into a beaker.

How does it do it?

A schematic of the Titrator system is shown below.



The heart of the Titrator is the dispenser unit ①. The sample to be measured is placed in a disposable sample beaker ②. The dispenser unit has an internal pump

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that circulates the sample through the cell in the optical unit. (A size flow cell will be required if size measurements are to be made.)

The dispenser unit has three titrant bottles ③ that can be filled with either acid, base or some other additive. On request from the software, a syringe system can deliver a precise quantity of titrant from one of the titrant bottles into the sample beaker.

The software can be setup so that measurements are made at a number of sample conditions (points) - between points a parameter of the system is changed by adding one of the titrants to the sample. The sample is stirred, pumped through the flow cell and then measured. In this way the effects of adding various amounts of these titrants on the zeta potential, size or intensity can be investigated. This method is known as a titration measurement.

A pH probe ④ continually monitors the pH of the sample, while a temperature controller in the optical unit maintains the sample at a selected temperature.

Once the measurement sequence is complete, the sample can be flushed from the system. A beaker containing flush fluid is attached to the dispersion unit. The fluid is then either recirculated through the system or flushed to a waste container ⑤. Valves on the cell ports on the optical unit ⑥ control the direction of flow of the flush fluid.

For flushing with larger quantities of liquid, the waste tubing should be connected to a larger container using the silicone tubing supplied.

The measurement settings are controlled by a Titration Operating Procedure (TOP). A TOP is a user specified procedure that defines all the settings for a measurement. It will specify, for example:

- The type of measurement that is made (zeta potential, size etc.).
- Which titrant is to be added (and how much).
- Number of titration points etc.

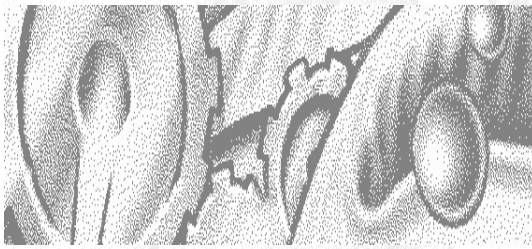
Using a TOP ensures that all measurement settings for a particular measurement type are always consistent. This method is ideal when routine measurements are made by relatively untrained staff as all options are specified in the TOP.

There are two main modes of operation for the Titrator.

- **Single point measurement.** This is where the sample is added to the sample beaker and then a single measurement is made. The sample is pumped through the cell and directly to waste.
- **Titration measurement.** This is where the conditions of the sample are changed and measurements are made at different “points”.

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Using the Autotitrator



C H A P T E R 3

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Introduction

This chapter will cover the basic operation of the titrator which will enable you to make measurements. There are three basic steps involved when making a measurement. These are:

- **Setup.** This will include system setup such as filling the titrant bottles. Additionally, setup may include priming the system or specifying a new TOP (see **Creating/modifying a TOP**).
- **Measurement.** This involves; selecting the TOP to be used, entering sample details and then starting the measurement. Typically (dependant on how the TOP is set up), the system will make the measurement, save the results, print a report and display the results on the computer.
- **Flushing.** This is the process whereby the sample within the system is flushed out, cleaning the system ready for the next measurement.

As can be seen, if a routine measurement is being made then the setup stage can be omitted. In this case, making a measurement is a very quick and easy procedure.

If your system is already setup, and you wish to try making a measurement, jump straight to the section “Making a measurement” later in this chapter.

Setup

Some setup procedures will have to be performed daily, while others will only have to be performed at much longer intervals.

Typically setup involves one or more of the following:

- **Filling the titrant bottles.** Only required when the bottles are nearly empty.
- **Priming the titrant syringe pumps and tubes.** Usually done at the beginning of the day or at the start of a new session.
- **Calibrating the pH probe.** Usually performed at least once a week.
- **Calibrating the conductivity.** Only done if the cell or cell electrodes have been replaced or cleaned.

Note that the pH probe will require special care. Never allow the pH probe to dry out. It is important to read the section on the care of the pH probe in the maintenance chapter (chapter 5).

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Filling and changing the titrant bottles

The three titrant bottles are typically filled with acid, base, salt or other additives. For most applications all that is required is to check that there is enough titrant in the bottles and re-fill if necessary.

Alternatively, if you are continually using different titrants you may need to change one of the titrant bottles prior to the measurement.

Always consult the Materials Safety Data Sheets for any Titrant being used for information on safe handling.

Re-filling the bottles.

The titrant bottles will hold up to 500ml of titrant. When the level reaches approximately 100ml, it is recommended to re-fill the bottle. To refill:

- Remove the Titrant bottle by disconnecting the pipe connector. This requires the coloured part of the connector to be turned anti clockwise about one complete turn.
- Remove the cap from the bottle.
- Dispose of the remainder of the titrant and rinse the bottle.
- Prepare 500ml of titrant to the correct concentration (see “A note on titrant concentration” later in this section).
- Add the titrant to the bottle.
- Re-fit the bottle’s cap.
- Reconnect the pipe to the bottle.
- Prime the titrant tubing using the default settings (see the next section).

Changing bottles

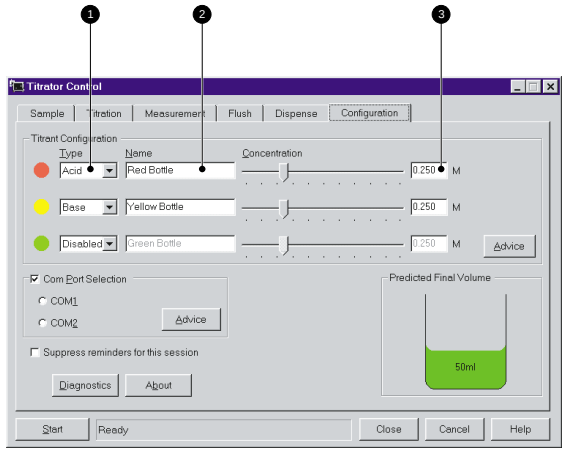
The dispensing unit can hold three titrant bottles. However, in some situations, this may not be sufficient. It may therefore be necessary to change one the titrant bottles. To change a bottle:

- Prepare 500ml of titrant to the correct concentration (see “A note on titrant concentration” later in this section) and add to a clean titrant bottle.
- Remove the titrant bottle that is to be replaced by disconnecting its pipe connector.
- Attach the new titrant bottle.
- Prime the titrant tubing. To avoid cross contamination, it is necessary to either set the **Prime volume** to 9ml (3 x the default (3ml)) or alternatively, press the **Prime** button three times. See the next section for more details on priming the titrator.

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If you are creating a new TOP that requires the titrant to be changed then it is also important to inform the titrator software that the titrants have changed. To do this:

- Open the titrator application by selecting the **Titrator** button 
- Select the **Configuration** tab.



- Change the relevant titrant type ①, titrant name ② and titrant concentration ③ as applicable.

A note on titrant concentration

Preparing and specifying the concentration of the titrants is critical to achieving accurate results. The concentration specified within the **Configuration** tab of the titrator software must be accurate to within 10%.

A concentration of 0.25M is satisfactory for most measurements. In some situations you may wish to vary this. For example, if you intend to use a large number of points in a titration measurement, or perform pH measurements close to pH 7 you may wish to use a lower concentration.

The titrator can accurately dispense small volumes so concentrations of titrant up to 1 molar can be used.

It is recommended that pre-diluted titrants are purchased. Either use titrants that are diluted to the exact concentration you require, or purchase titrants of a known Molar concentration that can be easily diluted further.

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Priming

Priming is the act of ensuring that the titrant tubes are completely full of the titrant. Over time, the titrant may evaporate from the end of the titrant tube and therefore it will not deliver the correct volume when first used. It is recommended that the dispenser unit is primed every morning or at the beginning of a new session. Use the default prime volume (3ml) when performing a daily prime.

The dispenser unit should also be primed when a titrant bottle is changed in order to remove any traces of the original titrant that may me left in the titrant tubes. Use a prime volume of 9ml when changing a titrant bottle (alternatively perform a default prime 3 times).

To prime the system:

- Attach a sample beaker containing clean water to the dispersion head. Water in the beaker is not essential, but will eliminate the effect of surface tension at the end of the titrant tube.
- Open the titrator application by selecting the **Titrator** button .
- Select the **Flush** tab.
- Optionally set the **Prime volume**.
- Press the **Start Prime** button to start the prime sequence.

The titrator tubing and the capillary cell contain about 8ml of fluid. After flushing and before measuring the next sample, this flushing fluid can be left in the tubes or pumped out by removing the sample beaker before the end of the flushing routine.

Calibrating the pH and conductivity

The pH probe should be calibrated on a regular basis. Once a week should be sufficient in normal use. However, it may be done more often if greater accuracy is important, or your internal QC/QA policy specifies it.

The conductivity only needs to be calibrated if the cell or cell electrodes have been replaced or cleaned.

Details on calibration can be found in chapter 5 of this manual.

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Making a measurement

A measurement can be made in two ways. The easiest method is to use the **Start** icon. This will start a measurement based on an existing TOP. This method is used when you are routinely measuring the same sample type.

Alternatively, a measurement can be started from the titrator application (after selecting an existing TOP or creating a new one). This method is used when you are either developing a TOP or you are making "one-off" measurements - it can be thought of as a "manual" measurement.

Both methods are explained below. You may also wish to read the on-line help topic **Demonstration measurement**, which describes an actual measurement using the coffee creamer Coffee-mate®. Coffee-mate® is a cheap, commonly available sample that is ideal for initial measurements.

Making a measurement using the Start icon

This section assumes that a TOP exists for your sample. Refer to the section "Creating/modifying a TOP" for details on creating a TOP. This section also assumes that the Malvern software and the dispenser unit have been setup correctly.

To make a measurement:

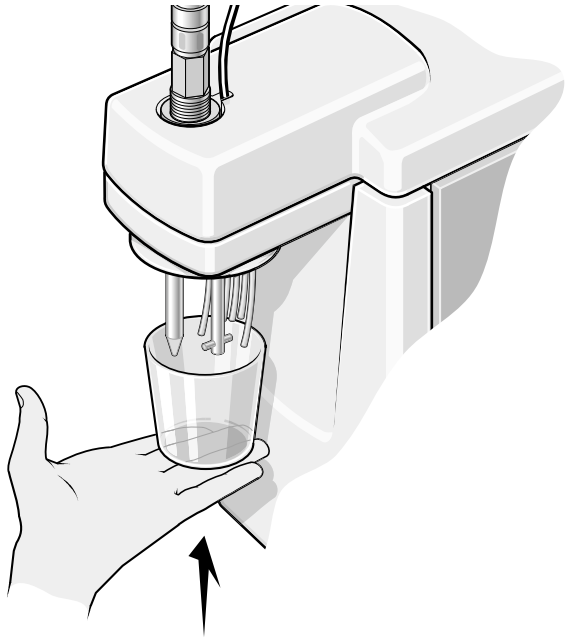
- First prepare the sample to be measured and place a know volume into a sample beaker. A initial sample volume of 50ml is recommended (30ml minimum to 100ml maximum). The preparation of the sample is of prime importance. Read the sections on sample preparation in the **Making a size measurement** and **Making a zeta potential measurement** manuals.

Note

- The sample used should be of a suitable concentration that gives an intensity of scattering within recommended values. This is checked at the beginning of each titration.
  If the concentration is too high the titration may proceed slowly.
  If a message occurs indicating the sample concentration is higher than recommended dilute the sample and restart the titration.

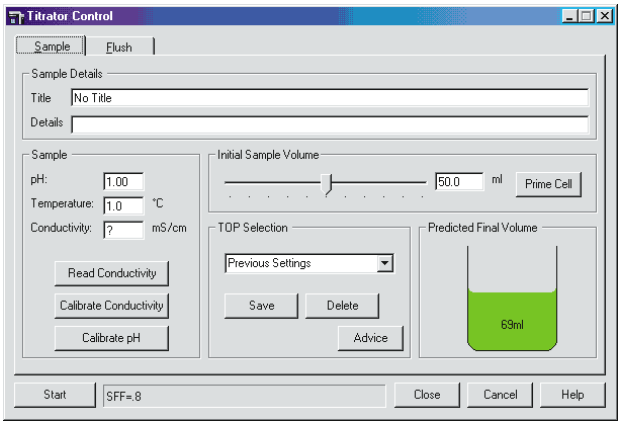
- Attach the sample beaker to the dispersion head by holding the beaker under its base and lifting it vertically into place under the dispersion head (see diagram). The beaker will click into place. Be careful not to trap any of the pipes when attaching the beaker.

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- Start the titrator software by selecting the **Start** icon .
- The **Sample** tab shown below will appear. Note that all functions that are not required are greyed out.



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- Enter the sample title and details.
- From the drop down list within the **Top selection** area, select the TOP to be used. Note that the TOP defines everything about the measurement i.e. whether it is a single point or titration measurement, the titrants to be used, whether reports will be printed etc.
- In the **Initial sample volume** section, enter the volume of sample that is in the beaker.
- Press **Start** button and the measurement will be made.
- Follow the instructions for the position of the valves.
- A graph of the results will be plotted as the titration proceeds.
- At the end of the titration any iso electric points found will be calculated and displayed.

Note
- A titration report can only be printed in full if selected to be printed at the end of the measurement.

Once the measurement is complete the system will require flushing to remove the sample from the tubes and flow cell. The software will ask if you wish to flush the system. If you select **yes** then the system will use the settings from the TOP to complete a flush. Instructions will be given throughout the flush routine.

There are four types of flush routine:

- **Titration flush (Routine)**. A titration flush will automatically be used after a titration measurement. A beaker of clean flush fluid is attached to the dispenser and the contents are recirculated. This method will **dilute** the sample within the system so you may wish to perform more than one flush routine after a titration measurement.
- **Single shot flush (Routine)**. A single shot flush is automatically used after a single shot measurement. A request is given for a beaker of clean flush fluid to be attached to the dispenser. The contents are flushed through the system and then directly to a waste container.
- **Cell flush**. A cell flush is used to pump large volumes of flush fluid through the cell. A cell flush should be performed if you are about to measure a new sample that is substantially different from the the previous sample or you are cleaning the cell prior to making a DTS0050 Zeta Potential Transfer Standard measurement.
  A cell flush can only be performed manually by selecting the **Flush** tab, selecting **Cell flush** and pressing the **Start Flush** button.
  A cell flush will require a large flush reservoir to be attached to the flush connector of the dispenser, and a large waste container to be connected to the outlet tubing.

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- **Continuous flush**. This enables the pump to be turned on and allow flushing until the option is deselected or the titrator switched off.

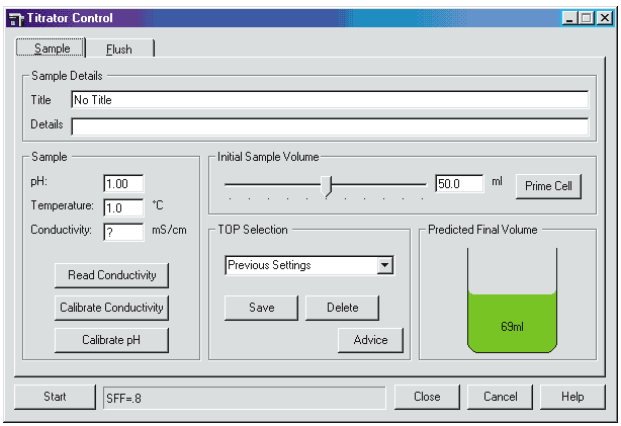
Making a measurement using the titrator application

A measurement can be started from the titrator application. This method is used when you are either developing a TOP or you are making "one-off" measurements.

All setup and measurement procedures are basically the same as that described above, except that this method allows the TOP settings to be changed prior to starting the measurement.

To start a measurement:

- Start the titrator application by selecting the **Titrator** icon .
- The **Sample** tab shown below will appear.



- Either select the TOP to be used from the drop down list, or make any changes to any of the settings within the tabs. The following section and chapter 4 will give more details on the options within these tabs.
- Enter the sample title and details.
- Press the **Start** button and the measurement will be made.

Once complete the system will require flushing - see "Making a measurement using the start icon".

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Creating/modifying a TOP

A new TOP can be created by making changes to the settings within the titrator application. The **Titration** and **Measurement** tabs are specifically used to define the measurement, but all the tab settings are saved within the TOP.

Any change to the tab settings will cause the currently selected TOP name to be blanked out. This prevents accidental changes to a TOP. To create a new TOP, type a new name into the **TOP selection** list box (on the **Sample** tab) and then press **Save**. It is not necessary to add the TOP extension - this will be done automatically.

If changes are made, but not saved, then the settings will be written to a default TOP called **Previous settings**, if **Close** is pressed.

All Malvern default settings can be re-instated by selecting the **Malvern Defaults** TOP.

Chapter 4 will give details on all settings available within the titrator application.

Distributing a TOP

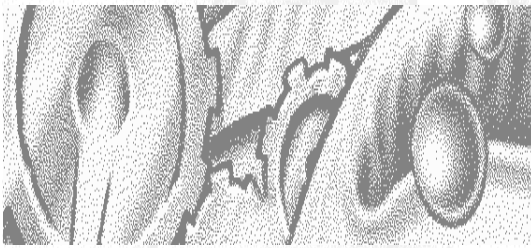
A TOP can be distributed to other users of a titrator system, so that samples can be measured in a consistent way from instrument to instrument. A TOP file (*.TOP) must be copied to the **C:\program files\titrator** directory.

It should be noted that the software has no way of automatically determining what is in the titrant bottles. Make sure that all recipients of the TOP check which titrants are specified within the **Configuration** tab so that the titrant bottles can be changed if necessary.

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Identifying the features of the titrator



C H A P T E R 4

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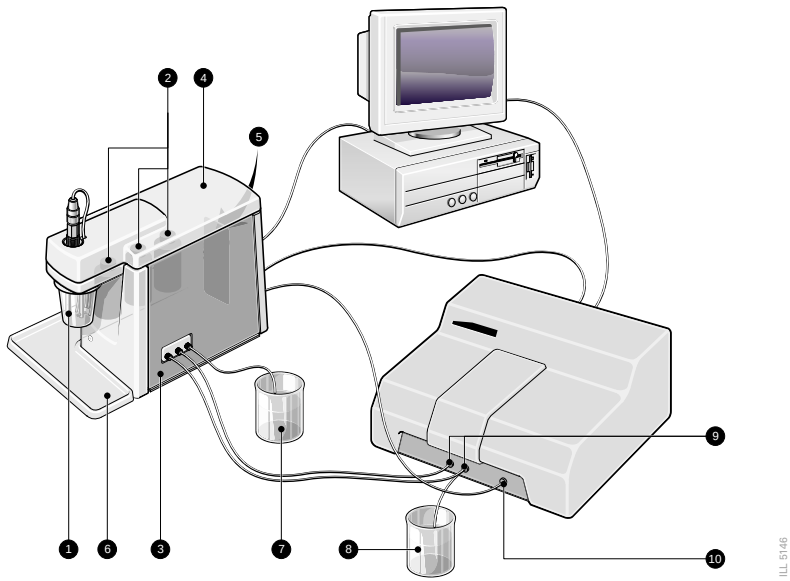
Introduction

The aim of this chapter is to help you to identify the physical features of the Titrator. It can be thought of as a reference guide where you will find an explanation of these features, whether it is an LED indicator or a check box within the software.

This chapter is divided into two sections. Section one will examine the features of the dispenser unit in more detail. Section two will look at the features of the software in more detail.

Features of the dispenser

The illustration below identifies the main features of the dispenser (note that some of the tube and cables are not shown in their final positions for clarity).



① Dispersion head.

The Dispersion head can be thought of as a mixing bowl. The sample to be measured is placed in a beaker that is attached to the Dispersion head. Titrants are added and the sample stirred. The dispersion head also houses the pH probe.

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This area is explained in more detail later in this chapter.

② Titrant bottles.

The titrant bottles contain the solutions that will be added to the sample during a titration measurement sequence. Each bottle can hold either acid, base, salt or an additive. The tubing and bottles are colour coded (red, yellow and green). These colours are matched to LED indicators on the syringes (see later) and also the **Configuration** tab of the software.

③ Dispensing area.

The dispensing area houses the dispensing syringes, valves and the pump head. This area is explained in more detail later in this chapter. For your safety the dispersion area cover must always be in place during a measurement sequence.

④ Dispenser lid.

The dispenser has a removable lid that allows access to the titrant and sample tubes. Removing the lid also allows the dispensing area cover to be removed.

⑤ Rear panel.

The rear panel contains all electrical connections to the computer and optical unit. This area is explained in more detail later in this chapter.

⑥ Drip tray.

The drip tray holds the titrant bottles and collects any spillages from the beaker.

⑦ Flush container.

The flush container contains a supply of flushing fluid (typically clean dispersant) that is used to flush the cell. The container can be any clean plastic bottle.

⑧ Waste container.

The waste container is used to collect the used sample and flush liquids after a Single shot measurement or Cell flush. The container can be any bottle that is chemically compatible with the sample, dispersant, flush fluids and titrants used during the measurement.

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⑨ Cell inlet valves.

The inlet valves control the flow of sample through the cell. The valves are opened/closed during some flush sequences. The valves also allow a manual measurement, using a syringe, to be performed without the necessity to disconnect the titrator.

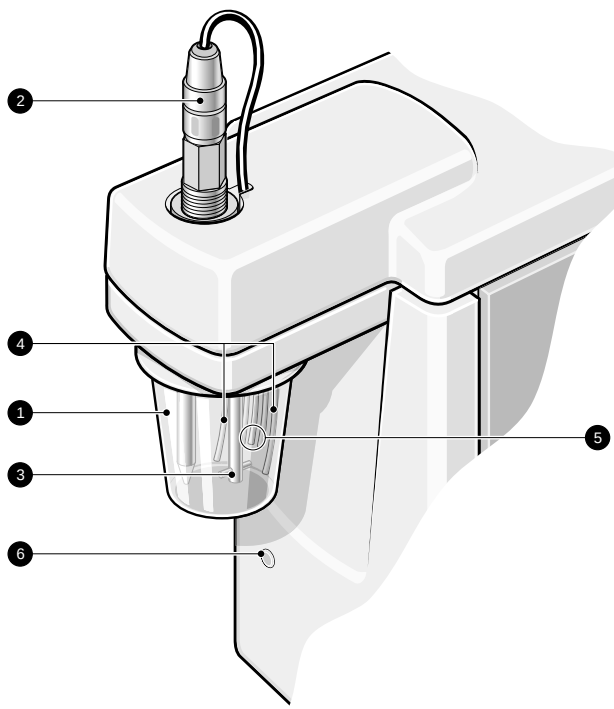
⑩ pH probe connector.

The pH probe on the titrator is attached to this connector

Dispersion head

The sample to be measured is placed in a sample beaker that is attached to the dispersion head. Titrants are added and the sample stirred. The dispersion head also houses the pH probe.

The illustration below identifies the main features of the dispersion head.

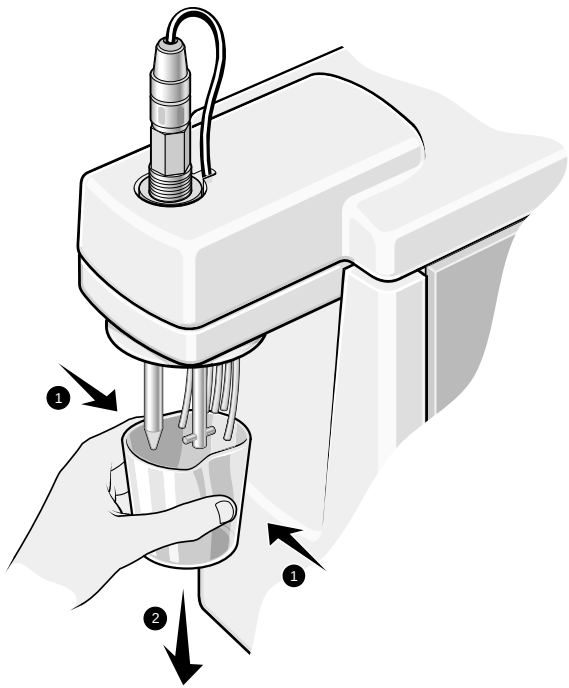


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① Sample beaker.

The sample beaker is the mixing vessel for the measurement. It initially contains the sample to be measured. Titrants are added to the sample through the titrant pipes and the dispersion is stirred.

The sample beaker is attached to the dispersion head by simply pressing the beaker into the holder (see “Making a measurement using the start icon” in chapter 3). The beaker is released by gently squeezing its sides. See the illustration below:



Note ▪ Squeezing the beaker in any other orientation will not allow the beaker to be released.

Be careful not to splash any sample when removing the sample beaker.

The sample beakers are disposable - replacement beakers can be obtained from your Malvern representative.

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② pH probe.

The pH probe measures the pH of the sample. The performance of the pH probe will deteriorate over time. It is recommended that the probe is replaced at least once a year.



Caution

The probe must never be allowed to be dry out. If it does it may need replacing. See chapter 5 for details on maintaining the probe.

③ Stirrer.

The stirrer agitates the sample and titrant dispersion. The speed of the stirrer is set through the **Measurement** tab of the titrator software. Care should be taken when adjusting the stirrer speed as air may be introduced into the sample. If in doubt, set the stir speed check box to '**Auto**'. Autostir will set the stirrer to a value dependent on the sample volume.

④ Sample exit and return pipes.

The sample is pumped to and from the cell through these pipes (during a single shot measurement sample is only pumped **to** the cell).

⑤ Titrant pipes.

The measured dose of titrant from the titrant bottles enters the sample beaker through these pipes. The most accurate volumes of titrant are dispensed if the titrant pipes are below the surface of the sample.

⑥ Status indicator.

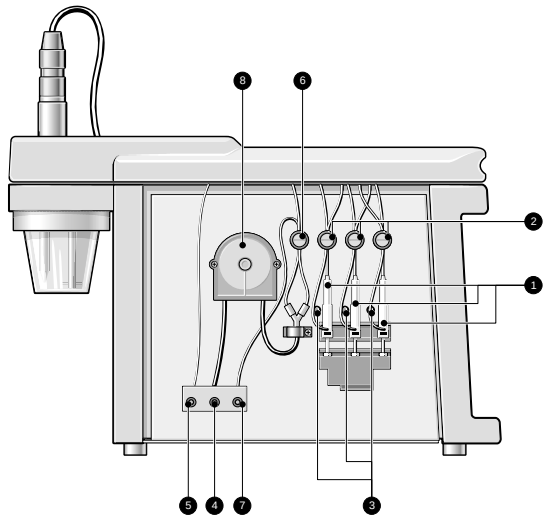
This indicator shows the status of the titrator. The indicator will show:

Red if the titrator has encountered an error condition.
Green if the titrator is in operation.
Amber if the titrator is in standby mode.

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Dispersing area

The illustration below identifies the main features of the dispersing area.



① Dispensing syringes.

The syringes are the mechanism for dispensing precise amounts of titrants from the titrant bottles to the sample beaker.

② Dispensing syringe valves.

The syringe valves open and close to control the flow of the titrant from the bottle to the sample beaker.

③ Dispensing syringe indicators.

Each syringe has an LED indicator that illuminates if that syringe is being used to dispense. The three LEDs are colour coded (red, yellow and green) to reflect the colour coding of the titrant bottles.

④ Sample out connector.

The sample to be measured is pumped through this connector to the cell in the optical unit.

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⑤ Sample in connector.

The sample that has been measured is returned to the sample beaker via this connector (titration measurements only).

⑥ Flush valve.

The flush valve automatically controls the flow of flush fluid during a Cell flush routine. Note that the valves on the inlet ports of the optical unit must be manually controlled during some flush routines.

⑦ Flush connector.

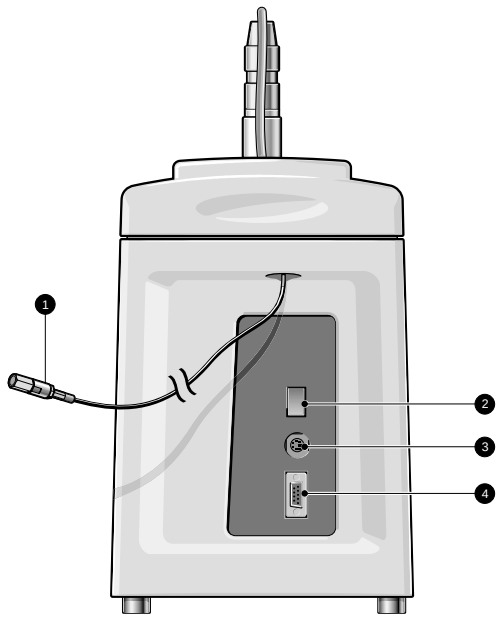
This is connected to a container that holds a quantity of flush fluid (typically clean dispersant) that is used during a cell flush routine.

⑧ Pump head.

The pump head is used to pump the sample (and flush fluid) through the cell. Its speed is controlled through the **Measurement** tab of the titrator software.

Rear panel

The illustration below identifies the main features of the rear panel.



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① pH probe connector.

This is connected to the pH probe connector on the front of the optical unit. The pH cable is attached to the pH probe via a screw connector that can be unscrewed if the pH probe is to be removed.

The pH probe connectors must be clean at all times. Contaminated connectors may affect the performance of the probe.

② On/off switch.

The power on/off switch for the titrator.

③ Power input.

The power input lead from the **Aux. Output** socket on the rear panel of the optical unit connects to this socket to provide power for the titrator.

④ COM connector.

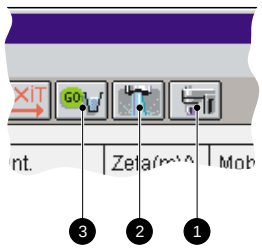
Connects to a COM port on the host computer to allow the software to control the titrator.

Features of the software

Software features can be separated into two areas; those that appear within the main application software (the three buttons in the button bar) and the titrator application.

Main application software features

All functions of the titrator are initially accessed through the main application software, in particular the three buttons on the toolbar.



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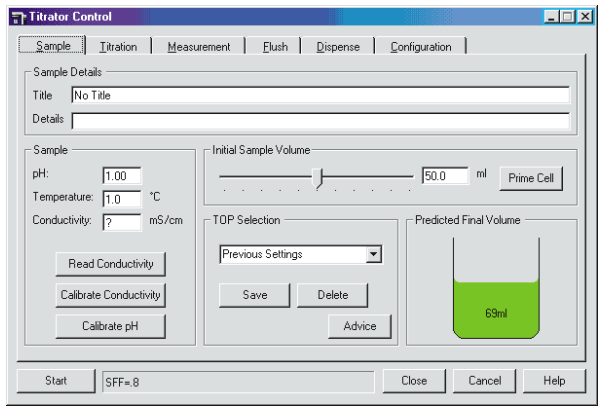
Pressing the **Titrator** button ① will start the titrator application (explained in the next section). The titrator application is the main interface for the titrator that allows you to set up a TOP. Measurements can also be run from the titrator application.

Pressing the **Flush** button ② will display the flush tab, allowing a flush routine to be started. A flush routine will clean the system ready for the next measurement. The operator will be given instructions through out the routine to ensure that it is completed successfully.

Pressing the **Measure** button ③ will allow a measurement to be made using a previously defined TOP. Pressing this button will start the titrator application, but all features that allow a TOP to be altered will be disabled.

Titrator application

The titrator application is accessed by pressing the **Titrator** button. Once enabled the titrator application shown below will appear. This may take a few seconds depending on the speed of your computer.



As a brief overview:

- The **Sample** tab can be thought of as a place where mandatory information must be entered before starting a measurement.
- The **Titration, Measurement** and **Flush** tabs are used to specify a TOP.
- The **Configuration** tab is used to define what is contained in the titrant bottles and their concentration, the COM port used etc.

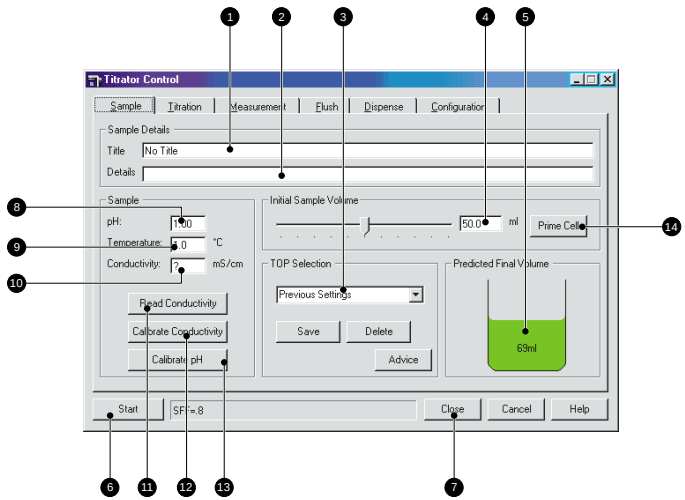
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- The **Dispense** tab allows the dispenser unit to accurately dispense titrants into a sample beaker. This feature is independent of the normal use of the titrator.

Sample tab

The **Sample** tab is used to specify information that must be entered before starting a measurement i.e. sample name, any sample details and the TOP to be used for the measurement.

Use the diagram below to identify the features of this tab.



① Title

Use this to type in the sample name that will be saved with the measurement file(s).

② Details

Details entered here, such as how the sample was prepared or the batch number of the sample etc, appear in the first line of the sample details section of the measurement file.

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③ TOP selection.

Use this list box to select a Titrator Operating Procedure (TOP) that will be used during a measurement. A TOP is a user specified procedure that defines all the settings for a titration measurement.

Selecting a TOP resets all fields (including sample title and details) to that which is defined in the TOP.

If any of the setting within the tabs are changed then the TOP name will disappear, allowing you to rename the TOP and then save it.

If you wish to modify an existing TOP, then after making modifications, select the TOP to overwrite, answer **No** to the message **Discard changes to settings** then save and select **Yes** to **Overwrite the existing file**.

If changes are made, but not saved, then the settings will be written to a default TOP called **Previous settings**.

The **Malvern Default** TOP is a standard TOP supplied by Malvern Instruments. It can be used as a base for creating other TOPs. The Malvern Default TOP can't be re-named.

④ Initial sample volume.

This is used to inform the software how much sample is initially added to the sample beaker. The more accurately this volume is specified, the closer the titration points will be to the required values.
It is recommended that an initial sample volume of 50ml is used (30ml minimum and 100ml maximum).

⑤ Predicted final volume.

This display will calculate the total volume that will eventually end up in the sample beaker (initial sample volume plus all the titrant additions), to ensure that the beaker does not overflow and is full enough to be stirred correctly.

The maximum volume of the beakers supplied by Malvern is 125ml. The display will show red if the predicted volume is less than 30ml (24%) or greater than 113ml (90%). A message will appear, warning that the beaker will overflow if the predicted volume is greater than 119ml (95%).

⑥ Start.

Pressing the **Start** button will start the measurement. A progress window will appear giving details on the progress of the measurement.

The **Start** button is always displayed at the bottom of the titrator application.

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⑦ Close/Cancel.

Pressing the **Close** button will close the titration application but will save any changes made to the **Previous setting** TOP.

Pressing **Cancel** will close the titrator application without saving any changes.

⑧ Sample pH.

This section continually displays the current pH of the sample. (Updates every 10 seconds, unless focus is in **Title**, **Detail** or **TOP** name box).

⑨ Sample Temperature.

This continually displays the temperature of the sample within the sample cell.

⑩ Sample Conductivity.

This displays the last recorded conductivity of the sample. This value is updated when a measurement is performed. Alternatively, the user can request the value to be read by pressing the **Read conductivity** button ⑪, (this may take up to 30 seconds).

⑪ Read conductivity.

Press to update the value reported in the sample conductivity display ⑩.
Also see **Prime cell** ⑭.

⑫ Calibrate conductivity.

The conductivity should be re-calibrated prior to measuring samples that are substantially different from the previously measured sample or if the system has be altered in any way i.e. the cell electrodes have been replaced or cleaned.

Pressing the calibrate button will start a calibrate procedure.

⑬ Calibrate pH.

The pH probe will require regular calibration. Typically a calibration should be performed once a week or how often your company QA/QC specifies.

Pressing the calibrate button will start a calibrate sequence (see chapter 5 for more details).

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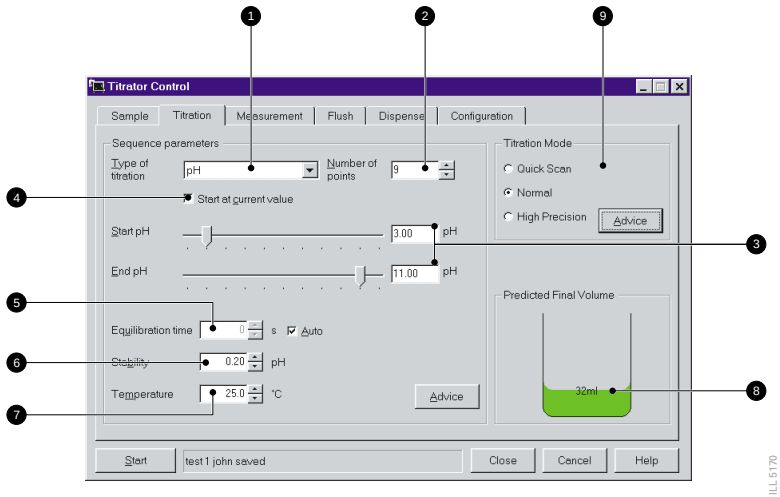
⑭ Prime Cell.

Pressing the prime cell button will start the pump and stirrer for 45 seconds. This will ensure that the sample in the beaker has circulated to the cell.
This only needs to be done if the conductivity is required before the titration has been started.

Titration tab

The **Titration** tab is used to control the settings for the points of titration, such as the number of points, the range of the titration (e.g. the range of pH).

Use the diagram below to identify the features of this tab.



① Type of titration.

This specifies which parameter you are controlling during the titration measurement. The choices are pH, conductivity, additive or single measurement.

The option selected here will determine what is displayed in the **Start/end pH/conductivity** area ③.

If an **Additive** titration is specified, the software will assume that the sample has 0% concentration of the additive at the start of the measurement.

If a **Single measurement** is chosen then the number of points ② will appear as 1.

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② Number of points.

Sets the number of different sample conditions at which the sample will be measured (points).

A maximum of 30 points can be set.

③ Start/end pH/conductivity/concentration.

This display will change, depending on the type of titration set in ①.

If pH **titration** is selected then two sliders will appear that will allow the initial pH and the final pH for the titration to be specified. At the start of the titration the titrator will change the pH of the sample to match that requested for the start point.

If a **conductivity** titration is specified then two sliders will appear that will allow the initial conductivity and the final conductivity for the titration to be specified. At the start of the titration the titrator will change the conductivity of the sample to match that requested for the start point.

Note ▪ A titration cannot be started at a lower conductivity than the initial sample conductivity.

If an **additive** titration is specified then a single slider will appear allowing the end concentration to be specified. The application will assume that the starting concentration is always 0%.

④ Start at current value.

Selecting this check box will override the **start** value. Instead the titrator will use the current value and equally space the titration points between this value and the **end setting**.

When the **Current value** check box is selected the **Start pH** slider will be greyed out but will indicate the current sample pH.

⑤ Equilibration time.

Once the pH has been set, the system will wait for this period of time to allow the pH to stabilise. Typically the **Auto** check box will be used as default. The **Auto** function will monitor the stability until it is within an acceptable limit.

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⑥ Stability.

The pH/conductivity is compared before and after a measurement to see if it has altered. If the difference between the two measurements is greater that the value set here, then the results are deleted and the measurement is repeated.

⑦ Temperature.

This is the temperature at which the measurement is made. The sample cell is heated or cooled to achieve this temperature. The value set here should be in the range of 20-30°C.

⑧ Predicted final volume.

This display will calculate the predicted total volume that will eventually end up in the sample beaker (initial sample volume plus all the titrant additions), to ensure that the beaker does not overflow and is full enough to be stirred correctly.

The maximum volume of the beakers supplied by Malvern is 125ml. The display will show red if the predicted volume is less than 30ml (24%) or greater than 113ml (90%). A message will appear, warning that the beaker will overflow if the predicted volume is greater than 119ml (95%).

⑨ Titration modes.

These modes allow a balance between a fast titration and obtaining points close to the requested valve to be selected. These modes are **not** applicable to additive titrations. The sample may have a buffering effect that will cause the pH or conductivity to drift during measurement. The sample must be allowed to equilibrate to a stable value before measurement.

Typical times for a pH titration in the situation where; there is little sample buffering, the pH probe is well calibrated and the titrant concentrations are accurately specified, are given in the table below.

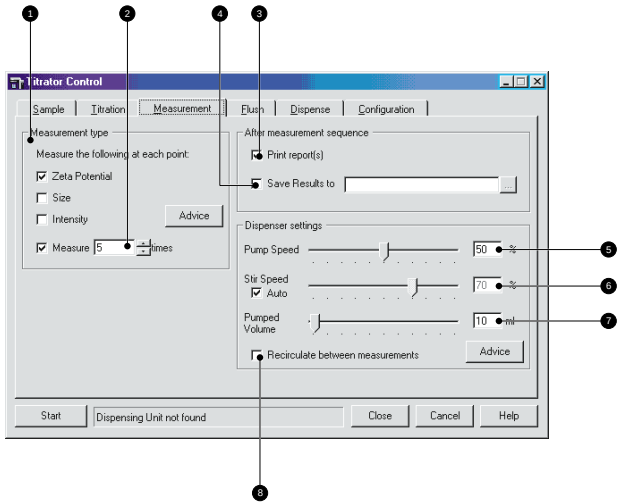
Titration mode	*Time per measurement point*
Quick scan	*2 minutes*
Normal	*3 minutes*
High precision	*4 minutes*

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Measurement tab

The **Measurement** tab specifies the measurement options, i.e. what type(s) of measurement is to be made at each titration point, pump and stirrer speeds, whether a report should be printed etc.

Use the diagram below to identify the features of this tab.



① Measurement type.

This defines what will be measured at each point. The options are; measure the zeta potential, measure the particle size or measure the intensity. More than one measurement type can be selected.

The intensity measurement is in effect a short size measurement where no analysis is performed, so either a size or an intensity measurement can be selected, but not both.

② Measure n times.

This will repeat the measurement at each point n number of times. The maximum value for n is 100.

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③ Print.

With this check box enabled, once the measurement sequence has been completed, a report will be printed for each measurement type.

④ Save results to.

Enables the results to be saved to a set of specific files once the measurement sequence is complete.

If more than one measurement type is selected in ① then a separate file will be created for each type i.e.

If you call it **default**, you get **default.ZET** and **default.SZ2**.

⑤ Pump speed.

Sets the speed of the pump that recirculates the sample through the flow cell.

⑥ Stirrer speed.

Sets the stirrer speed. Care should be taken not to introduce bubbles into the measurements by setting the stirrer speed too high. Selecting the **Auto** check box will set the stirrer speed to a speed dependent on the sample volume.

⑦ Pumped volume.

This specifies the amount of sample that is passed through the cell before a measurement.

⑧ Recirculate between measurements.

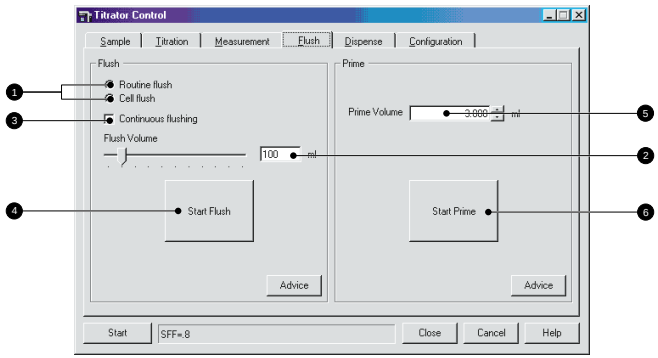
If more than one measurement is being made at one titration point then this check box gives the option of either making all measurements on the sample within the cell or recirculating the sample between the measurements.

Flush tab

The **Flush** tab is used to specify and start the flush sequence. It also contains the prime function that is used to prime the titrant tubes before use.

Use the diagram below to identify the features of this tab.

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① Routine/Cell flush

A **Routine flush** will flush the titrator after a measurement. The type of flush will depend on whether the TOP specifies a Single measurement or Titration measurement.
Cell flushing is used to pump large volumes of flush fluid through the **cell** to clean it.

② Flush volume.

Specifies the amount of flush fluid to be pumped through the cell.

③ Continuous flush.

Circulates the flush fluid continuously until the box is unchecked or the application is closed.

④ Start flush button.

Pressing this button will start the flush sequence.

⑤ Prime volume.

Priming is the process of ensuring that all the titrant delivery tubes are free of air and full of liquid. The prime volume is the amount of titrant that is passed through the tubing to remove the air or previous titrant. It is important that a waste sample beaker is in place on the dispersion head to catch the titrant.

⑥ Prime button.

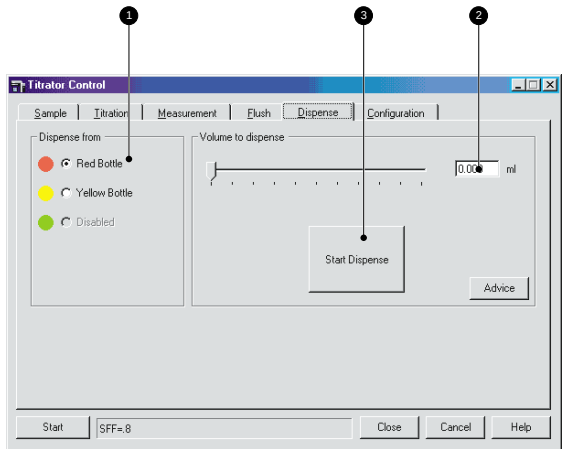
Pressing the **Start prime** button will start the prime procedure.

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Dispense tab

The Dispense tab is an added feature that is independent of the normal operation of the titrator. It allows the user to dispense precise amount of liquid from any of the titrant bottles. Ensure that a clean sample beaker is in place on the dispersion head or there is a container to add the titrant to.

Use the diagram below to identify the features of this tab.



① Dispense from.

Specifies the titrant bottle that will be used to dispense.

② Volume to dispense.

Specifies how much titrant will be dispensed. (Maximum 10ml).

③ Start dispense button.

Pressing the dispense button will deliver the specified volume to the sample beaker.

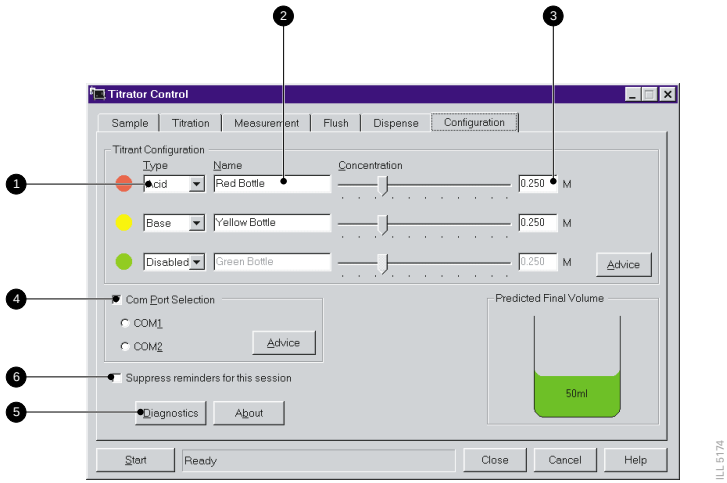
It should be noted that the most accurate volumes will be dispensed if the titrant tubes are below the level of the liquid in the sample beaker.

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Configuration tab

The **Configuration** tab is used to set up some of the features of the titrator (e.g. what is contained in the titrant bottles and their concentration, the COM port used etc).

Use the diagram below to identify the features of this tab.



① Type of titrant.

Specifies the type of titrant in each of the bottles. Use the drop down list to select either acid, base, salt, additive or disabled. The colour coding to the left of the list corresponds to the colour of the titrant bottles and the dispensing syringe LED indicators.

If salt is selected, then the **Salt type** dialogue will appear. This dialogue allows you to select from 50 preset salts. It also allows you to specify your own salt.

② Titrant name.

This is a title for the titrant bottle. By default they are called red, yellow or green - but any other name can be entered into this area. The name entered here will be printed on the titrant report and also used on the dispense tab.

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③ Concentration.

Specifies the concentration of the titrant in the titrant container.

④ COM port selection.

Allows the COM port to be specified for the connection to the computer.

⑤ Diagnostics.

Used to assist Malvern Instruments service engineers during installation and setup.

⑥ Suppress reminders for this session

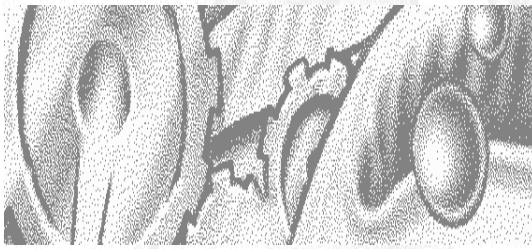
Selecting this check box will suppress most messages and reminders (e.g. reminders to add the sample, or fit a waste beaker when priming) during a measurement. More experienced users will benefit from this feature as it will speed up operation by removing routine dialogues.

When the Autotitrator software is restarted this check box will be turned off.

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Maintenance



C H A P T E R 5

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Introduction

The titrator has been designed so that supervisor/operator maintenance is kept to a minimum. It should be fully understood that no one, except a qualified Malvern representative, should undo any screws to get access to the inside of the titrator. There are no user serviceable parts inside the titrator.

This section explains the routine maintenance procedures that the supervisor/operator can perform. These procedures are:

- Cleaning the titrator.
- Maintaining the pH probe.
- Calibrating the conductivity.
- Replacing the Dispensing syringes.

A summary of how often these procedures should be carried out is detailed in the maintenance schedule later in this chapter.

Additionally, this section contains a list of parts and part numbers of consumables and items that can be replaced by the user in the event of failure.

Cleaning the accessory

Cleaning the titrator surfaces

Caution

The surfaces of the system may be permanently damaged if samples or titrants are spilt onto them. If spillages should occur, then the system should be disconnected from the power supply before scrupulously cleaning any spillage.

Periodically, the covers should be thoroughly cleaned using a mild soap solution on a damp cloth. Always ensure that the accessory is disconnected from the power supply before cleaning.

Never use excess liquid to clean the accessory and always avoid the electrical components on the rear panel.

Always ensure that the accessory is completely dry before applying power.

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Never use a solvent-based solution to clean the accessory as damage to the painted surface may result.

Cleaning the sample path

To avoid cross-contamination of samples, the sample flow path should always be kept scrupulously clean. Always flush the sample after each measurement (see the "Making a measurement" section in chapter 3).

The measurement cell should always be kept clean - see the maintenance section of the **Hardware manual** (MAN0151) for details.

Maintaining the pH probe

The pH probe is essential to ensure performance of the Titrator, and its maintenance is of paramount importance. As with any pH probe, its performance will degrade over time and will eventually have to be replaced. The frequency of replacement will be determined by; the maintenance of the probe, how frequently it is used and the types of sample being measured. In this respect a pH probe can be thought of as a "Consumable" item. Malvern recommends that the probe is replaced every year - but this frequency may vary depending on the factors detailed above.

There are three important points that should always be remembered when using a pH probe. Firstly, the performance of the pH probe will be severely affected if it is allowed to dry out (even for short periods). Secondly, the probe should be cleaned regularly. Finally, the pH probe should be calibrated at regular intervals.

The pH calibration routine includes a check box on the responsiveness of the pH electrode.

If the probe responds slowly a message to clean the electrode will be given.

If the calibration slope has changed significantly a suggestion will given to replace the electrode.

Keeping the pH probe wet

If the pH probe is allowed to dry out, even for short periods, then the performance of the probe can be severely affected. It may not be possible to recover a degraded pH probe.

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Typically, when the probe is immersed in a buffer solution, the pH should be attained (to within 0.1 of the pH) within 10 seconds. If this time exceeds 30 seconds, and the probe can not be recovered, then the probe should be replaced.

When changing the sample beaker do not allow the probe is to be exposed to air for more than 20 seconds. Make sure that the next sample beaker is ready to attach, or alternatively, have a spare beaker of soaking solution available.

What is a soaking solution? A soaking solution is a liquid that the probe can be immersed into to prevent it from drying out. The following guidelines should be followed when using a pH probe:

- The probe can be immersed in the **sample** solution for short periods (a few hours).
- The probe can be immersed in **tap water** for the medium term. (Do not use de-ionised water as it will destroy the charge equilibrium of the glass electrode).
- For long periods (more than a day) the probe should be immersed in either a dilute acid solution (pH 2 to pH4) or an acidic buffer such as pH 4.

If the probe has become dry, then it may be possible to recover the performance by soaking the probe overnight in a solution of 0.1M hydrochloric acid. However, the performance may never fully recover and there may be no alternative except to replace the pH probe.

Cleaning the pH probe

The probe should be periodically cleaned. The calibration procedure detailed below will indicate if the probe requires cleaning.

Chemical cleaning is usually sufficient. To chemically clean, soak the probe overnight in a solution of 0.1m hydrochloric acid, then use a soft tissue soaked in isopropanol and wipe over the end of the probe. Once cleaned, re-calibrate the probe to see if the performance of the probe has recovered.

Some probes have a PTFE 'Wick' which is fibrous. This is more easily wetted than the ceramic junction but can become clogged by very fine particles in the dispersion.

When this happens the pH probe will not respond to pH changes in the sample. Carefully brushing this wick can recover performance.

This effect is reduced by using more dilute samples.

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Calibrating the pH probe

The performance of the Titrator relies on the pH probe being correctly calibrated. If the pH probe has not been calibrated then the points of the measurement may not be evenly spaced throughout the pH range.

It is recommended that the pH probe is calibrated at least once a week.

To calibrate the pH probe you will need:

- Two pH buffers. The default buffers are pH 4 and pH 9.2 (these are supplied with the probe). See "Choosing the buffers when calibrating" later in this section for advice on selecting buffers.
- A beaker of clean, distilled water, to rinse the probe between measurements.

The pH probe is ideally calibrated while still fitted to the system and with pH buffers in the usual sample beakers supplied.

To calibrate the probe:

- Open the Titrator application by pressing the Titrator button .
- Press the **Calibrate pH** button within the **Sample tab**.
- Choose the upper and lower buffers from the drop down lists. The defaults are pH 4 and pH 9.2. See "Choosing the buffers when calibrating" later in this section for advice on selecting buffers.
- Press the **Start** button.
- The software will give instructions throughout the calibration. Initially you will be asked to rinse the probe and stirrer in the clean water and then to add the lower buffer solution (default pH 4).
- Press **OK** and the first set of measurements will be made.
- The software will then ask for the stirrer and probe to be rinsed again and then the upper buffer solution (default pH 9.2) to be attached.
- Again, press **OK** and the second set of measurements will be made.
- The software will display a message if the calibration was successful. Pressing **Cancel** will return you to the **Sample** tab of the Titrator application.

During the calibration procedure one or more of the following conditions may occur:

- If the measurement time for each buffer solution takes more than 1 minute, then the message "The electrode response is slow. Please clean your pH probe and recalibrate". Follow the guidelines described above for cleaning the probe.

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- The pH calibration measures each buffer 10 times. If the reported value at each measurement has drifted by more than 0.02 then the message "pH reading has drifted by n. Remeasure?" Pressing **Remeasure** will re-measure the current buffer again. The measurement can be repeated several times until the drift messages disappears. If the message does not disappear then either:

  1) Clean the pH probe (see section earlier in this chapter) and then recalibrate.

  2) If the drift is still there after cleaning, the probable cause will be a contaminated buffer solution. Dispose of the buffer solution and mix a fresh batch.

- During the calibration procedure, the software measures the "gain" of the probe. If this gain reaches a factory set limit then the software issues the message "The responsivity of the pH probe is low. The pH probe may need replacing". You may try cleaning the probe first and then recalibrating to see if this has any effect. If not you must replace the pH probe.

Choosing the buffers when calibrating

The default buffers for calibrating are pH 4 and pH 9.2. Other values may be used (they must be chosen from the lists in the pH calibration dialogue) but they should be at least 3 pH values apart. As an example, if you find that all your titration measurements are in the lower end of the pH table you may wish to use pH 2 and pH 5 buffers to calibrate the probe.

The pH probe for the Titrator is supplied with sachets of pH4 and 9.2 buffers. Follow the instructions supplied with the sachets for the best methods of making the buffer solutions.

As buffers are typically low cost items, it will be more economical for you to source your own local supplies. If your have difficulty finding a convenient source, contact your local Malvern representative who will be able to give you advice.

Replacing the dispensing syringes

It is recommended that all three dispensing syringes are replaced every 12 months. Replacement syringes are available through your Malvern representative (see "User consumable and spares" at the end of this section).

It is recommended that you remove one syringe at a time so that the other syringes can be used as a reference on the correct plumbing method.

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To remove a syringe:

Disconnect the titrant pipe ①.

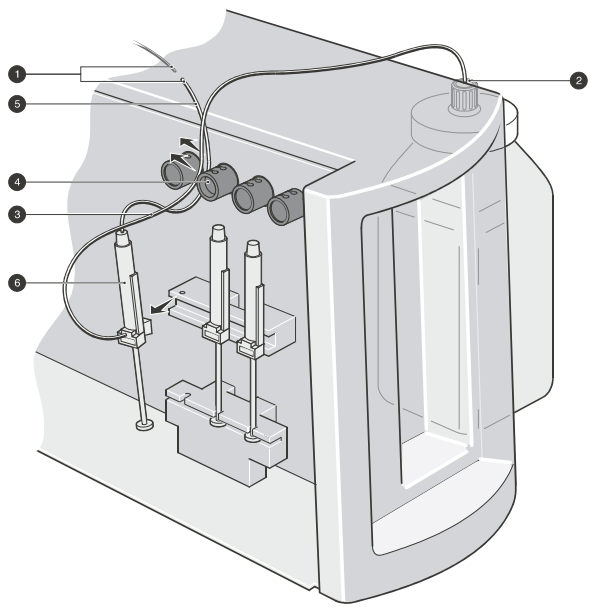
Disconnect the titrant pipe from the connector on the titrant bottle ②.

Pull the pipe from the front of the dispensing valve ③.

Push the front of the dispensing valve ④ and pull the rear pipe ⑤ from the valve.

Pull the syringe and syringe plunger off the mounting block ⑥.

Take care when replacing the syringe not to severally bend the PTFE plunger.



Replacement is the reverse of the above procedure. Prime the system once all the syringes have been replaced.

Calibrating the conductivity

The conductivity should be calibrated every time the cell electrodes are replaced or after the cell is dismantled for cleaning.

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To calibrate the conductivity you will require a Conductivity Calibration Solution. These solutions are readily available from third party suppliers. It is recommended that a solution with conductivity of 1.413mS/cm is used.

To calibrate the conductivity, you must first empty the sample tubes. To do this:

- Remove the sample beaker from the dispersion head and place on the sample tray, (directly under the sample pipes).
- Start the titrator application by pressing the titrator button .
- Press the **Prime Cell** button in the **Sample** tab. The sample pipes will empty into the sample beaker.
- Repeat this procedure first flushing the system with demineralised water.

To calibrate the conductivity:

- Add 50ml of the Conductivity Calibration Solution to a sample beaker and attach to the dispersion head.
- From the **Sample** tab, press the **Calibrate Conductivity** button.
- Type in the conductivity of the calibration solution (the default is 1.413mS/cm).
- Press **Start**.

The conductivity calibration will then be performed - this may take a few minutes. Once complete, the software will report the calibration coefficient. This coefficient is used by the software to correct any calibration reading.

For a final check, read the conductivity of the Conductivity Calibration Solution by pressing the **Read Conductivity** button in the **Sample** tab. The reported conductivity should be within 5% of calibration solution. If it is over 5%, then repeat the calibration procedure. Contact your Malvern representative if the reading still fails to be within 5%.

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Maintenance schedule

The following maintenance schedule should be followed to ensure the accessory continues to correctly function.

Procedure	Period
Priming the system.	*The system should be primed every morning or at the beginning of a new session.*
pH probe calibration.	*At least once a week or according to in-house QC procedures.*
pH probe cleaning.	*Once a week or according to in-house QC procedures.*
Cleaning the covers.	*Once a month.*
Dispensing syringe replacement.	*The three dispensing syringes should be replaced every 12 months.*
pH probe replacement.	*The pH probe should be replaced at least every 12 months.*
Calibrating the conductivity.	*Should only need to be done if the cell electrodes are cleaned or replaced.*

User consumables and spares

This section lists all user consumables and spares. Should you require any of these items, contact your Malvern Instruments representative, quoting the part number listed below.

Part	Part number
Sample beakers (1000).	*BOT0014*
Titrant bottles (3pk - coloured).	*DTS2035*
pH probe	*SEN0026*
Dispensing syringes (3pk) including pipes.	*DTS4044*
Top cover.	*DTS4039*
Side cover.	*DTS4040*

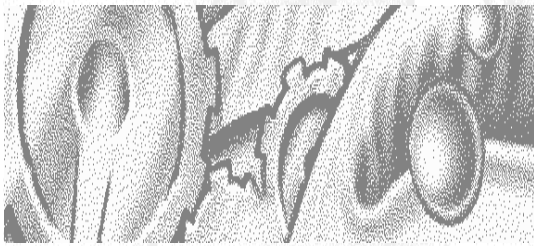
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Drip tray.	*DTS4041*
Stirrer bar.	*DTS4042*
External manual valves (pair).	*DTS2034*
Power cable.	*DTS2029*
RS232 cable.	*CAB0020*
Peristaltic pump head.	*DTS4043*

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Specification



APPENDIX A

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Specification

Dimensions (excluding sample tray and pH probe).	*Width: 170mm* *Height: 253mm* *Depth: 388mm*
Weight (dispenser unit only).	*5.3Kg.*
Power.	*24Vdc via Zetasizer optical unit.*
Power rating.	*30VA*
Maximum capacity of sampler beaker.	*125ml.*
Titrant bottles.	*3 x 500ml (colour coded).*
Modes of operation.	*Automatic via Malvern software.*

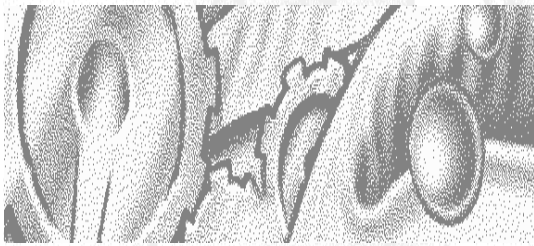
The accessory has been designed to be stored and operated in the following conditions:

Operating temperature.	*+10 to 35°C (+50 to +95°F)*
Storage temperature.	*-20 to +50°C (-104 to +122°F)*
Humidity.	*10 to 90% (non-condensing)*

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Chemical compatibility



APPENDIX B

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Introduction

The Sample flow path in the titrator has been manufactured from materials that are considered to give the widest protection from chemical attack. However, it is important to check that any sample or titrant you use is chemically compatible with the materials that they will come into contact with within the accessory.

This appendix lists all materials that comes into contact with the sample and titrants in the normal operation of the accessory.

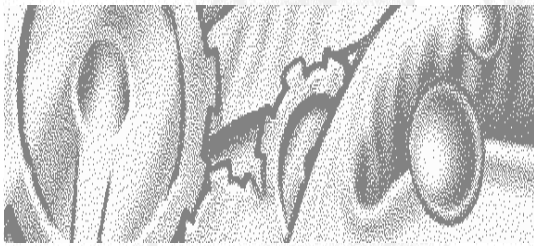
Materials list

Component	Materials
Peristaltic pump tubing.	*Silicone rubber*
Dispensing syringe.	*Polypropylene* *PTFE* *Viton*
Stirrer.	*PVC*
Beaker mounting.	*Polypropylene*
Output manifold and input/output connectors.	*Polypropylene*
Tubing to sample beaker.	*PTFE*
Tubing to titrant bottles.	*Silicone*
Tubing to optics.	*PTFE*
Sample beaker	*Polypropylene*
pH probe	*Glass*

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Installation



APPENDIX C

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Installation

This section gives instructions on how to reconnect the accessory should you wish to move it once it has been commissioned. The accessory **must** be initially commissioned by a Malvern Instruments representative.

Ensure that the new location of the system has enough space before attempting to move the accessory.

If the accessory is to be moved short distances, then a sample beaker containing water should be attached to the dispersion head to prevent the pH probe from drying out. If the accessory is to be moved greater distances then the pH probe should be removed and its protective cover attached to prevent the probe from drying out.

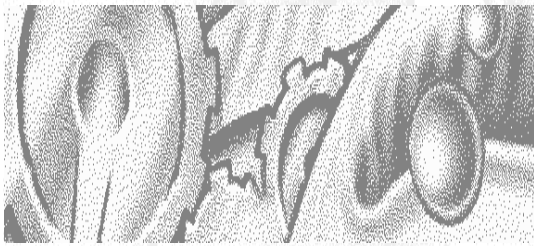
The installation of the Titrator can be summarised by the schematic below.

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Regulatory statements



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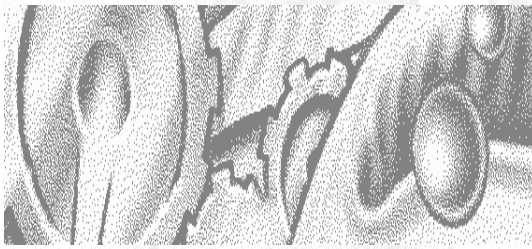
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