Malvern Panalytical
a spectris company

Zetasizer Advance Series Accessories Guide

Malvern Panalytical
a spectris company

Zetasizer Advance Series Accessories Guide

Publication date: Monday, March 20, 2023
MAN0604-05-EN

Disclaimer

Although diligent care has been used to ensure that the information in this material is accurate, nothing herein can be construed to imply any representation or warranty as to the accuracy, correctness or completeness of this information and we shall not be liable for errors contained herein or for damages in connection with the use of this material. Malvern Panalytical reserves the right to change the content in this material at any time without notice.

Copyright notice

Malvern Panalytical Ltd.
Grovewood Road, Malvern,
Worcestershire, WR14 1XZ,
United Kingdom

Tel +44 1684 892456
Fax +44 1684 892789

Malvern Panalytical B.V.
Lelyweg 1, 7602 EA Almelo,
The Netherlands

Tel +31 546 534 444
Fax +31 546 534 598

info@malvernpanalytical.com

www.malvernpanalytical.com

Contents

Chapter 1 Introduction

1.1 About this manual

This manual gives an overview of the accessories that are available for use with the Zetasizer Advance Series instruments.

This manual accompanies the following manuals:

- Zetasizer Advance Series Basic Guide
- Zetasizer Advance Series User Guide

WARNING - General hazard
The accessories or the samples to be measured may be hazardous if misused. Users must read the Health and Safety information in the *Zetasizer Advance Series Basic Guide* before operating the system.

WARNING - General hazard
For information on the features available for each Zetasizer Advance Series instrument, refer to the *Introduction* chapter in the *Zetasizer Advance Series Basic Guide* or *User Guide*.

1.2 Assumed information

To use this manual, you must understand the product naming convention and how menu commands are described.

1.2.1 Naming convention

The ZetasizerAdvance Series instruments are referred to either in full, or as 'the Zetasizer' or ‘the instrument’. The combination of the Zetasizer, accessories and computer is referred to as ‘the system’.

1.2.2 Menu commands

Menu commands in the are always shown in bold text in the form:

main menu > item

As an example, the command File > Options refers to selecting Options from the File menu.

1.3 Where to get help

This section gives information on the various channels in place to get help with your Zetasizer system.

1.3.1 Website - www.malvernpanalytical.com

Our website offers a comprehensive range of resources for use by customers. It gives free access to exclusive content including webinars, presentations, application notes, technical notes, whitepapers, software downloads and more.

1.3.2 Help system

A full help system is supplied with your Malvern Panalytical software system. This provides detailed reference information on all software features. To access this, press F1 or click the Help button within the application (sometimes indicated by a question mark icon).

1.3.3 Technical support

All queries about the system must be directed to your local Malvern Panalytical representative, quoting the following information:

- Model and serial number of the instrument (usually located on the outside casing of the instrument).
- Software version (see Help>About in the software).
- Firmware version (Technical support will inform you how to locate this information).

Visit www.malvernpanalytical.com to find your local Technical Support representative.

1.4 Accessory range

The cells available for use with the Zetasizer are listed in the table below. The accessories you can use depend on the instrument model and measurement type to be performed.

Table 1.1 *Cells overview*

	Cell	Size	Zeta	MADLS	Particle concentration	pH Titration
	DTS0012 Disposable 10 x 10 polystyrene cell	✓		✓	✓	
	ZSU1002* Low volume disposable sizing cell	✓				
	ZEN0040 Low volume disposable cuvette	✓				
	ZEN2112 Low volume quartz batch cuvette	✓		✓	✓	
	PCS8501 Glass cuvette with round aperture	✓		✓	✓	
	PCS1115 Glass cuvette with square aperture	✓		✓	✓	
	DTS1070 Disposable folded capillary cell	✓	✓			✓

	ZEN1010 HC cell	✓	✓		✓
	ZEN1002 Dip cell	✓	✓		

*Zetasizer Ultra and Zetasizer Lab instruments only.

1.4.1 Zeta potential cells

The following table provides further details on the cell options available for zeta potential measurments.

Table 1.2 Zeta potential cells

Part	Description
DTS1070	**Folded capillary cell** - maintenance-free, primarily designed for zeta potential measurements. This cell also allows the patented Diffusion Barrier Method to be used, which allows low volume measurements and reliable zeta potential measurements of fragile materials.
ZEN1002	**Dip cell** - provides repeatable measurements of aqueous, or non-aqueous samples. Use with the DTS0012 or PCS1115 cell.
ZEN1010	**High concentration cell** - intended primarily for the measurement of zeta potential on a concentrated aqueous sample. The cell is suitable for a broad range of conductivities.

1.4.2 Size cells

The following table provides further details on the cell options available for size and concentration measurements.

Table 1.3 Size cells

Part	Description
DTS1070	**Folded capillary cell** - maintenance-free, primarily designed for zeta potential measurements.

ZEN1010	**High concentration cell** - can be used for size as well as zeta potential.
ZSU1002*	**Low volume disposable sizing cell** - for the measurement of low volume samples (3-30 µl) and to extend the size range of DLS measurements.
DTS0012	**Square polystyrene cuvette** - disposable cell for size measurements.
ZEN0040	**Low volume disposable cuvette** - for size measurement at a 173° scattering angle.
PCS8501	**Square glass cuvette with cap (round aperture)** - for size.
PCS1115	**Square glass cuvette with cap (square aperture)** - for size. Also for use with the Dip cell.
ZEN2112	**Low-volume quartz batch cuvette** - for size.

*Zetasizer Ultra and Zetasizer Lab instruments only.

Note: All cuvettes have an outside dimension of 12 mm x 12 mm.

1.5 Selection advice

CAUTION
Polystyrene cells must not be used for measurements above 70 °C, because they may melt.

- Use disposable polystyrene cuvettes (DTS0012) for "easy to measure" samples. These samples will have relatively high scattering intensity, for example latex with 0.01% mass or higher.
- Disposable cuvettes are not resistant to organic solvents.
- Glass and quartz cuvettes have higher optical quality, which is vitally important when performing low concentration protein measurements (derived count rate <100 kcps).
- Glass and quartz cells are resistant to organic solvents and can be used at higher temperatures.
- The polystyrene cells are low cost, disposable, and do not require cleaning.
- Glass and quartz cells require cleaning after a measurement.

Note: Disposable polystyrene cells are easily scratched and should never be used more than once.

1.6 Cuvette options

Any cuvettes not mentioned below are described in detail in later chapters.

Table 1.4 Cuvette options.

Cell type	Application	Typical solvent	Minimum sample volume	Material
DTS0012	Size	Water, water/alcohol	1 ml	Polystyrene
ZEN0040	Size	Water, water/alcohol	40 µl	Polystyrene
PCS1115	Size	Water, most organic and inorganic solvents	1 ml	Glass
PCS8501	Size	Water, most organic and inorganic solvents	1 ml	Glass
ZEN2112	Size	Water, most organic and inorganic solvents	12 µl	Quartz

1.7 Cleaning advice

Note: Clean and carry out any necessary maintenance before using a cell or cuvette. Specific cleaning advice is given in later sections.

- Rinse/clean size cells with filtered dispersant before use.
- Rinse zeta potential cells with filtered dispersant before use.

Note: Use the cell caps to improve sample thermal stability and prevent dust introduction and spillage.

Chapter 2 Standard size cells

2.1 Introduction

This section covers the following size cuvettes:

Table 2.1 Standard size cells

	Cell	Size	MADLS	Particle concentration
	DTS0012 Disposable 10 x 10 plastic cuvette	✓	✓	✓
	ZEN0040 Low volume disposable cuvette	✓		
	ZEN2112 Low volume quartz batch cuvette	✓	✓	✓
	PCS8501 Glass cuvette with round aperture	✓	✓	✓
	PCS1115 Glass cuvette with square aperture	✓	✓	✓

Refer to Chapter 3 for details on the ZSU1002 cell.

The choice of cuvette depends on the type of measurement being performed and the sample being measured. The aspects covered in this section are:

- Identification of each cuvette and measurement type indication.
- How to fill each cuvette and insert it into the instrument.
- Maintenance procedures, cleaning and chemical compatibility information.

2.2 Filling cuvettes

Fill the cuvette with the prepared sample using a pipette or syringe as described below.

Note: Fill the cuvette slowly to avoid creating air bubbles. Ultrasonication can be used to remove air bubbles if suitable.

Note: If using syringe filters, never use the first few drops from the syringe in case there are residual dust particles in the filter that could contaminate the sample. Alternatively, flush the filter with the sample dispersant before use to avoid wasting precious samples.

2.2.1 Standard cuvettes

A minimum sample volume must be provided to make sure that the laser passes through the sample. The sample should fill the cuvette to a minimum height of 10 mm from the bottom of the cuvette (the measurement is made 8 mm from the bottom of the cuvette).

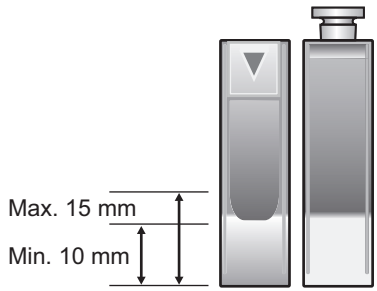


Figure 2.1 Minimum and maximum filling depth

Do not overfill the cuvette. Use a maximum depth of 15 mm, as more can produce thermal gradients in the sample that reduce the accuracy of the temperature control.

- When filling, tilt the cuvette and allow it to fill slowly.
- To stop bubbles forming let the sample flow down the inside wall of the cuvette.

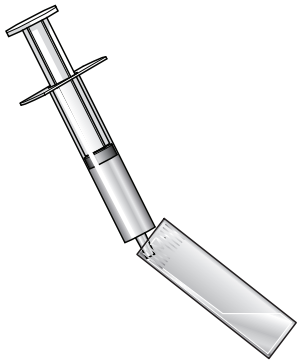


Figure 2.2 Filling a cuvette using a syringe

- When filled, place a lid securely on the cuvette.
- Do not agitate the sample once the cuvette is filled.

2.2.2 Low volume cells

These cells are designed to use the minimum volume of sample possible for a size measurement. Minimum volumes for different measurement types are given below.

Table 2.2 Minimum sample volume for each measurement type

Measurement type	Minimum volume	Cell
Backscatter	12 µl	ZEN2112
Forward	20 µl	ZEN2112
Side	3 µl	ZSU1002
MADLS	20 µl	ZEN2112
Particle concentration	20 µl	ZEN2112

Note: When using the ZEN2112, the sample must be pipetted carefully into the bottom of the cuvette, so it is filled from the bottom up.

2.3 Inserting a cell

Do the following:

1. Open the cell area lid by pushing the button in front of the lid.
2. Push the cell into the cell holder until it stops.

   Note: A polished optical surface must be facing the front of the instrument (towards the button). Most cells have a small triangle or Malvern Hills logo at the top to indicate the side that faces the front.

3. Place the thermal cap over the cell if applicable.
4. Close the cell area lid.

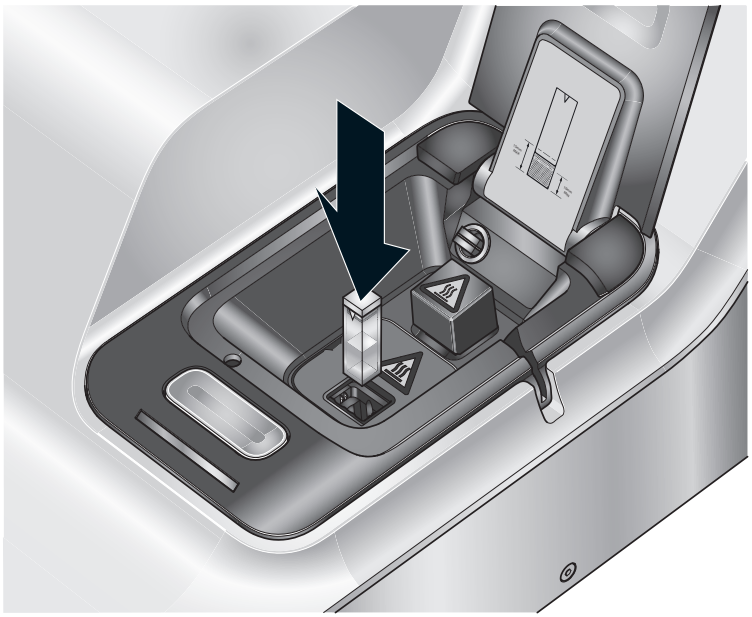


Figure 2.3 *Inserting a standard or low volume cell*

2.4 Cleaning glass or quartz cuvettes

Cleaning procedure depends on the sample measured so specific instructions cannot be given. Follow these guidelines:

- Rinse the cuvette with the dispersant that was used for the measurement.
- If required, submerge the cuvette in an ultrasonic bath containing clean solvent.

CAUTION
Do not put PCS8501 in an ultrasonic bath as this may break the cuvette.

- Once clean, wipe the cuvette with a lint free tissue. Droplets of liquid can also be removed using compressed air.
- Cleanliness is especially important when using small or dilute samples.

2.5 Chemical compatibility

Table 2.3 Component materials for the cells discussed in this section are as follows:

Component	Material
PCS1115	
Cell material	Boron silicate crown glass
Stopper	Polypropylene (Square cap)
PCS8501	
Cell material	Boron silicate crown glass
Stopper	PTFE (Round cap)
ZEN0040	
Cell material	Polystyrene

Component	Material
Stopper	Polypropylene
DTS0112	
Cell material	Polystyrene
Stopper	Low density polythene
ZEN2112	
Cell material	Quartz
Stopper	Polypropylene

Chapter 3 Low volume disposable sizing cell

3.1 Introduction

This section covers the following cell:

Table 3.1 Cell information

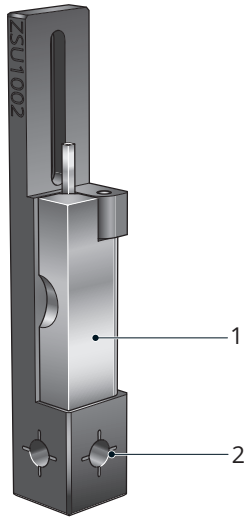
Part	Cell
ZSU1002	Low volume disposable sizing cell

The Low volume disposable sizing cell is used for the measurement of low volume samples and to extend the size range of DLS measurements. It has a sample volume measurement range of 3 - 30 µl.

Note: The Low volume disposable sizing cell can only be used with the Zetasizer Ultra and Zetasizer Lab instrument ranges.

3.2 Cell parts

The Low volume disposable sizing cell consists of a capillary holder, inside which a disposable capillary holding the sample is contained. The capillary is discarded after use, removing the need to clean the accessory between measurements.



1. Lid
2. Measurement area

Figure 3.1 *The Low volume disposable sizing cell*

3.3 Filling the capillary

There are two methods of filling the disposable capillary, depending on sample volume, viscosity and availability:

- Capillary action method - for sample volumes between 5 µl and 30 µl when there is some sample to spare.
- Pipette method - for sample volumes between 3 µl and 10 µl when you only have a small amount of sample available.

WARNING - General hazard
Handle the capillary with care. Do not use too much force as this could break the capillary and cause cutting injury.

Note: Only hold the top end of the capillary. The bottom is used for measurement and should not be touched. Fingerprints and other residues can adversely affect results.

Note: Always clean the capillary before each use. To do this, dip the capillary into water or the chosen solvent, remove and shake off any excess liquid.

3.3.1 Capillary action method

You will need the following additional equipment:

- A container for the sample
- Sealing clay

To fill the capillary:

1. Dip the end of the capillary into the sample. Sample will be drawn up into the capillary. Don't dip the capillary deeper than 5 mm into the sample.
2. Wipe away any excess sample on the capillary with a lint-free wipe.

Figure 3.2 Capillary action filling method

3. Carefully press the sample end of the capillary into the sealing clay. Twist the capillary slightly after pressing it into the clay to make the capillary easier to remove from the clay.

3.3.2 Pipette method

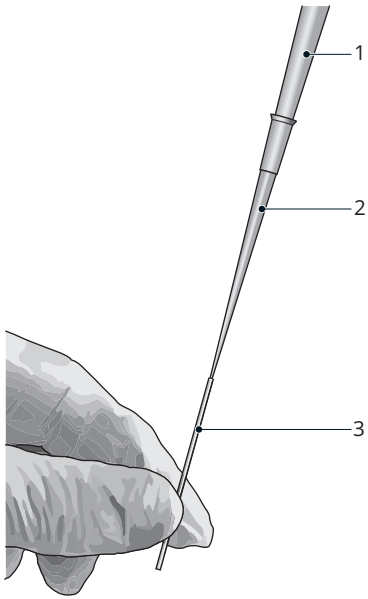
You will need the following additional equipment:

- Calibrated manual pipette
- 1 - 2000 µl gel loading tip
- Sealing clay

To fill the capillary:

1. Load the sample into a calibrated manual pipette fitted with a 1 - 2000 µl gel loading tip.
2. Insert the tip into the bottom of the capillary, holding the capillary at the top.
3. Gently load the sample into the capillary with one push of the pipette.
4. Carefully press the sample end of the capillary into the sealing clay. Twist the capillary slightly after pressing it into the clay to make the capillary easier to remove from the clay.

Note: The sample slug should sit approximately 5 mm from the end of the capillary to make sure the sample will be visible in the measurement area. Refer to the advice later in this chapter.



1. Pipette
2. Gel loading tip
3. Capillary

Figure 3.3 *Loading the capillary using the pipette method.*

3.4 Inserting the capillary

To place the capillary into the capillary holder:

1. Open the lid of the capillary holder.
2. Place the capillary into the holder, inserting the capillary into the hole in the bottom of the holder with the sample at the bottom end of the capillary.

Note: The capillary should be inserted so that its edges lie parallel to those of the capillary holder. The following cross-section shows the correct alignment.

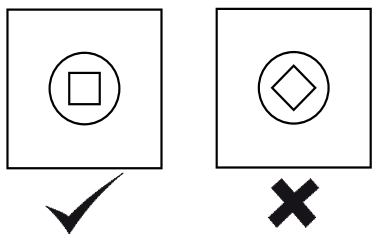


Figure 3.4 Correct alignment of the capillary inside the holder (view from bottom of the cell)

3. Adjust the height of the capillary in the holder so that the sample slug is located within the measurement area. Make sure the sealed end of the capillary is at the bottom. The sealing clay should not be visible in the measurement area.

Note: The capillary must not protrude from the bottom of the capillary holder. If protruding, the capillary may break when inserted.

Tip: The measurement area is indicated by the target markings on the exterior of the capillary holder. This is visible in the illustration below.

Figure 3.5 *Capillary positioned correctly with the sample in the measurement area*

4. Carefully close the lid of the capillary holder.

Note: Hold the capillary in place as you close the lid to prevent the capillary from rotating out of alignment.

CAUTION
Close the lid of the capillary holder slowly and carefully. Closing the lid incorrectly can cause the capillary to break.

3.5 Using and cleaning

Note: When using particularly small volumes, the sample may evaporate very quickly, especially at elevated temperatures. Sealing the capillary with clay at both ends can help to reduce this problem.

Note: When inserting the cell into the Zetasizer, make sure the Malvern Hills logo faces the front of the instrument.

Refer to the Zetasizer Advance Series Basic Guide for instructions on running a measurement.

The capillaries used with the Low volume disposable sizing cell are disposable, so cleaning is not necessary.

If a spillage occurs on the capillary holder, wipe clean with a lint-free wipe.

3.6 Chemical compatibility

With normal usage, only the capillary should come into contact with the sample.

Table 3.2 Component materials

Component	Materials
Capillary	Borosilicate glass

Chapter 4 Folded capillary cell

4.1 Introduction

This section covers the following cell:

Table 4.1 *Cell type and part number*

Part	Cell
DTS1070	Folded capillary cell

Folded capillary cells are maintenance-free and primarily designed for zeta potential measurements. They can also be used for size measurements, without removal and repositioning. To reduce the risk of cross-contamination, they should be discarded after being used for either a single measurement or series of measurements.

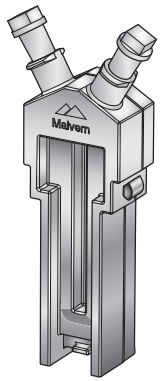


Figure 4.1 *The folded capillary cell*

Cell	Description
Application	Aqueous based samples
Typical solvent	Water, water/alcohol
Minimum sample volume	0.75 ml, 20 µl if using diffusion barrier

Cell	Description
Advantages	• Low cost • Single use disposable (no cleaning) • Use with MPT-3 Multi-purpose Titrator • No sample cross-contamination • Fast sample change over
Disadvantages	• Not resistant to organic solvents • Unsuitable for use at high temperatures (above 70 °C)
Additional notes	• Stoppers can be replaced with Luer connectors for leak-free connection to the optional MPT-3 Multi-purpose Titrator • Sample details can be written on the textured side of the cell with a permanent pen

4.2 Preparing the Folded capillary cell

This cell is intended to be used once then discarded. Before a cell is used for the first time, it should be flushed through with ethanol or methanol to facilitate wetting on the capillary walls. A syringe or a wash bottle can be used. Use only sufficient fluid to wet the surface of the cell and electrodes.

The cell should then be flushed through with water as described below.

1. Fill one syringe with de-ionised water or the dispersant.
2. Place the full syringe in one of the sample ports on the cell and an empty syringe in the other.
3. Flush the contents of the full syringe through the cell into the empty syringe.
4. Repeat the flushing process five more times, flushing the liquid backwards and forwards between the syringes. The cell is then ready for use.

Note: Never attempt to clean the outside of the Folded capillary cell. It causes small surface scratches that give inaccurate results.

4.3 Filling the Folded capillary cell

Folded capillary cells should be filled with the prepared sample as described below. Rinse/clean with filtered dispersant before use. Refer to section 4.2.

1. Prepare the sample in a syringe of at least 1 ml capacity.
2. Place the sample syringe into one of the sample ports.
3. Invert the cell [**1**].
4. Slowly inject the sample into the cell, filling the capillary to just over half way [**2**].
5. Check no air bubbles form in the cell. Tap the cell gently to dislodge any bubbles that do form.

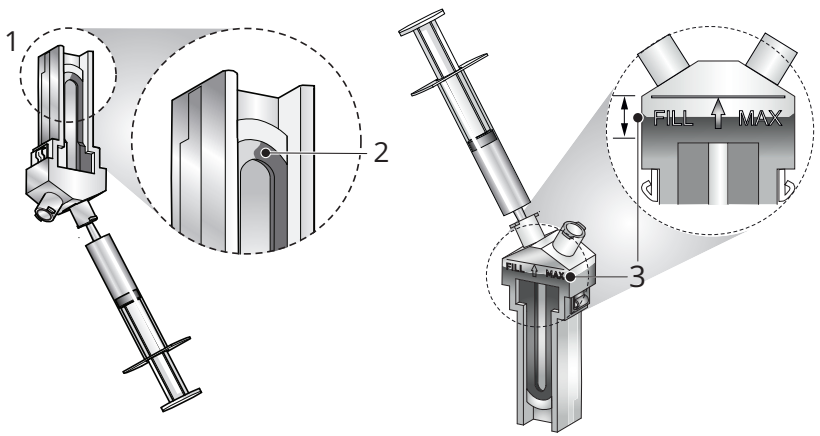
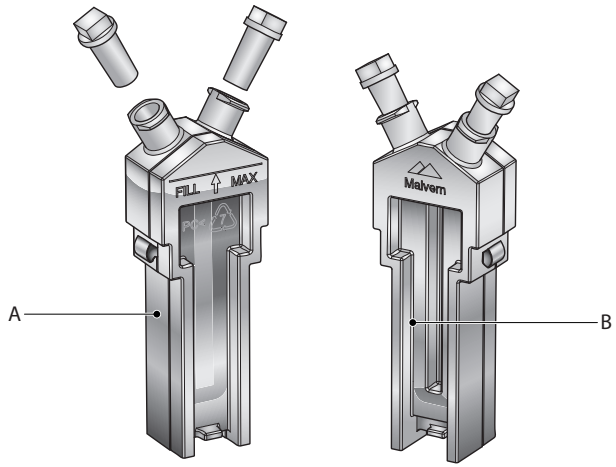


Figure 4.2 *Injecting sample into the folded capillary cell*

6. Turn the cell upright and continue to inject slowly until the sample reaches the fill area shown [**3**]. Fill between the shoulder of the cell and the FILL MAX line.
7. Check again for bubbles in the cell. Tap gently to dislodge any bubbles if present.

8. Check that the electrodes are fully immersed.
9. Remove the syringe and insert a cell stopper in each port.
10. Remove any liquid spilled on the electrode contacts.



A. Without stoppers - rear view
B. With stoppers - front view

Figure 4.3 Inserting cell stoppers

Note: Always fit the stoppers before making a measurement. Ensure that one stopper is fitted firmly, and the other one loosely, to avoid pressurization of the cell.

4.4 Inserting the Folded capillary cell

To insert the Folded capillary cell:

1. Place a thermal contact plate into the recess on either side of the Folded capillary cell. The plates increase temperature stability.

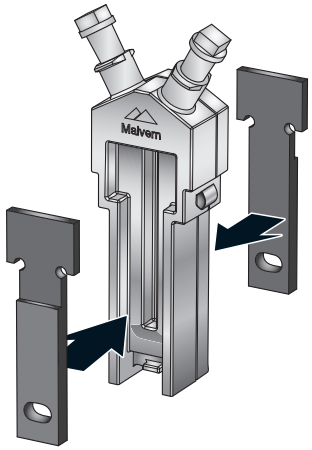


Figure 4.4 *Placing the thermal contact plates*

2. Open the cell area lid by pushing the button in front of the lid.
3. Hold the cell near the top, away from the lower measurement area, and push into the cell holder until it stops.

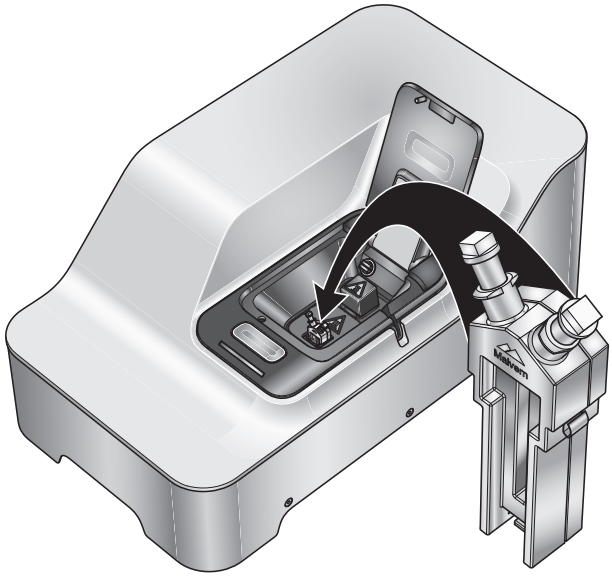


Figure 4.5 *Cell insertion - note direction of cell logo*

Note: Ensure that the Malvern Hills logo faces towards the front of the instrument when inserting.

4. Close the cell area lid.

Note: Different versions of the Folded capillary cells have unique thermal plates. If required, contact your Malvern Panalytical representative for the correct plates for your cell.

4.5 Chemical compatibility

The following table details the materials from which the cell components are manufactured.

Table 4.2 Component materials

Component	Materials
Body	Polycarbonate
Electrodes	Gold plated beryllium/copper
Stoppers	Polypropylene

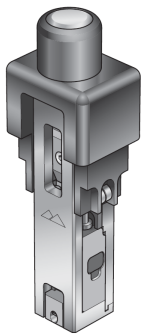
Chapter 5 High concentration cell

5.1 Introduction

This section covers the following cell:

Table 5.1 *Cell type and part number*

Part	Cell
ZEN1010	High concentration cell



Use the High concentration cell for the measurement of zeta potential of concentrated aqueous samples. The cell can be used with the MPT-3 Multi-purpose Titrator for automated titrations.

The cell has a high precision optical measurement block held within electrode chambers. This is contained in a cuvette sized casing assembly that allows excellent thermal contact with the instrument cell holder.

The cell is supplied with the following components:

- 0.79 mm internal bore silicone tubing with appropriate Luer fittings
- Luer plugs for manual filling
- Interdental brushes for cleaning of the electrode chamber, internal flow paths and optical block

5.2 Filling the High concentration cell

1. Inject sample slowly until the liquid reaches the bottom of the Luer outlet [1].

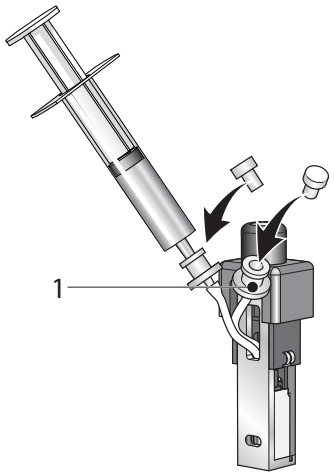


Figure 5.1 Filling the High concentration cell

2. Check the measurement area/optical block for bubbles. If any are present, inject more sample until it is bubble free.
3. Remove the syringe and insert a cell stopper in each port.
4. Remove any liquid spilled on the electrodes.

5.3 Inserting the High concentration cell

Insert the High concentration cell into the instrument as shown below.

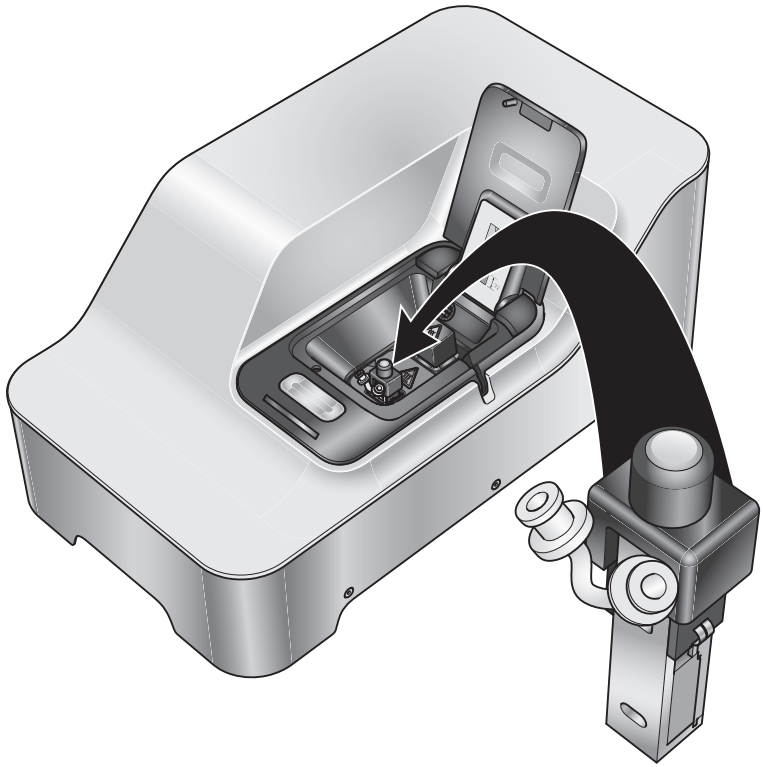


Figure 5.2 Inserting the High concentration cell

The metal face of the cell must face the front of the instrument. This ensures good thermal contact between cell and instrument.

5.4 Cleaning the High concentration cell

- General cleaning - rinse the cell before a measurement by flushing through with de-ionised water. Wipe external surfaces of the assembled cell clean with a weak soap solution.
- Intensive cleaning - disassemble the cell before doing any cleaning.

To disassemble the cell:

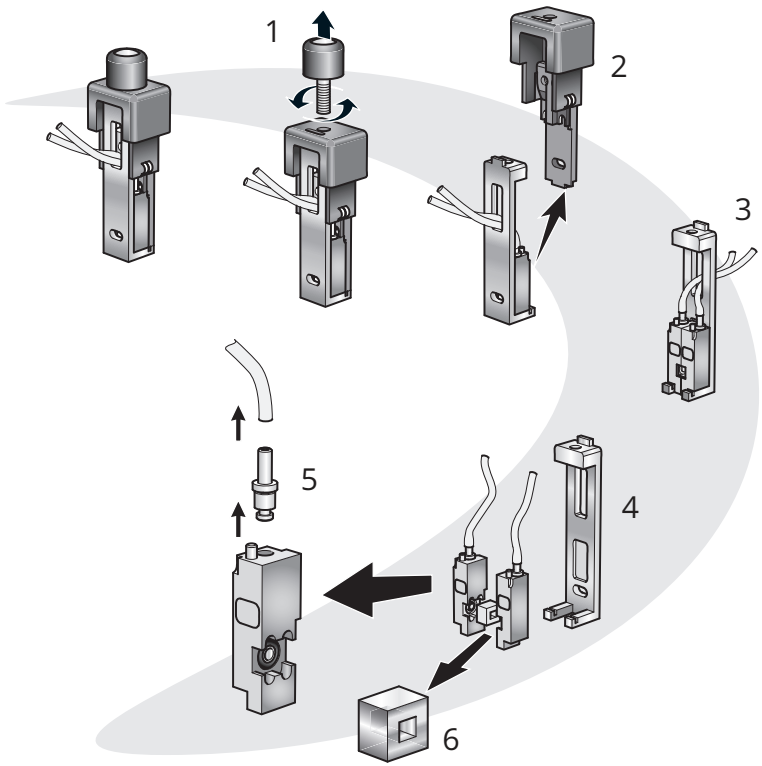


Figure 5.3 Cell disassembly

1. Remove the screw cap [1].
2. Separate the two halves of the cell [2] by pulling the rear casing vertically away from the metal front.
3. Note how the electrode chambers and quartz measurement cell block are assembled [3].
4. Remove the chambers and cell block from the metal front casing [4].
5. Detach the pipework and remove the top port [5].
6. Protect the cell block from damage [6].

Once disassembled, clean the cell as described below.

Table 5.2 Cleaning method for each cell component

Component	Cleaning method
Screw cap	Wipe clean with a mild soap solution.
Outer casing	Black part of casing (Rear - Delrin): Wipe clean with a mild soap solution. Metal part of casing (Front - Stainless steel): Immerse the casing in Hellmanex and place in a gentle ultrasound bath (30 Watts) for 5 to 15 minutes. Rinse with water once cleaned.
Electrode chambers and port	Electrode Chamber: Scrub gently with interdental brush and Hellmanex, then scrub with de-ionised water. Smaller internal bore: Scrub gently with interdental brush and Hellmanex, then scrub with de-ionised water.
Quartz measurement cell block	Scrub both internally and externally with interdental brush. Afterwards brush with water. Be careful not to scratch the block.

Note: Once inserted into the assembly, clean the outside of the cell block with a cotton bud and ethanol.

Once cleaned, leave all parts to dry before re-assembling. Re-assembly is the reverse of disassembly.

CAUTION
Take care not to damage the sprung electrodes located in the rear casing.

5.5 Chemical compatibility

Only the central electrode and measurement section of the High concentration cell should come in contact with sample. The outer components of the cell will only come into contact if spillage or overfilling occurs.

Table 5.3 Component materials

Component	Materials
Electrode chambers	Natural PEEK
O-rings	Nitrile rubber
Electrodes	Palladium
Electrode contacts	Brass
Precision measurement block	Quartz
Tubing	Silicone rubber
Casing	Delrin / Stainless steel 316
Cap	Delrin / Phosphor Bronze
Contacts	Gold plated Beryllium / Copper

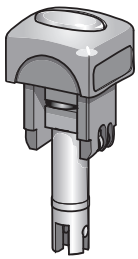
Chapter 6 Dip cell

6.1 Introduction

This section covers the following cell:

Table 6.1 *Cell type and part number*

Part	Cell
ZEN1002	Dip cell



The Dip cell is used to provide a method to measure the zeta potential of both aqueous and non-aqueous samples.

A number of samples can be prepared and the Dip cell inserted to measure each one in turn. For aqueous samples the Dip cell can be used with the disposable polystyrene cuvette (DTS0012) or the Glass square aperture cuvette (PCS1115). For non-aqueous samples use the reusable Glass square aperture cuvette (PCS1115).

Note: If you use the Dip cell for non-aqueous measurements, do not then use the same cell for aqueous measurements. It cannot be guaranteed that the cell will be suitably clean for reliable zeta potential measurements to be performed. Cleanliness of the cell may be assessed by measuring a zeta potential standard (such as the Malvern transfer standard- DTS1235). Instead it is recommended to use two Dip cells: one for aqueous dispersants, and another for non-aqueous dispersants. The Dip cells have colored badges on the top of them to help keep track of which cell is being used for each dispersant.

6.2 Filling cuvettes

This section describes how to fill the cuvette correctly for use with the Dip cell. The Dip cell uses square cuvettes to hold sample. When the Dip cell is inserted, the sample is displaced upwards.

Note: Do not overfill the cuvette - it may overflow when the Dip cell is inserted. Overfilling can also produce thermal gradients in the sample that reduce the accuracy of the temperature control.

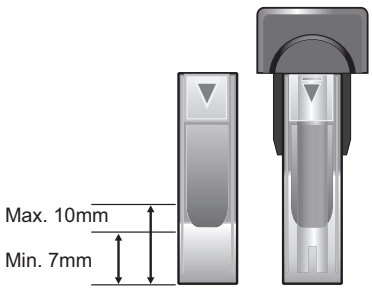


Figure 6.1 Minimum and maximum filling depth

To ensure a sufficient minimum sample volume is provided and protect against overfilling, fill the cuvette to a depth of between 7 mm and 10 mm. This equates to approximately 0.7 - 1.0 ml of sample.

6.3 Inserting the Dip cell

This section describes both how to insert the Dip cell into the cuvette, and also the whole assembly into the instrument. Fill the cuvette with sample before inserting the Dip cell - refer to section 6.2.

Note: Do not fill the cuvette more than the maximum depth of 10 mm [**1**].

To insert the Dip cell into the cuvette:

1. Tilt the cuvette to a maximum angle of 45° [**2**].
2. Slowly insert the cell into the cuvette until the metal electrodes are covered [**3**]. As the cell is inserted it displaces the sample.
3. Once the electrodes are covered, tilt the cell back up to vertical [**4**].
4. Check for any bubbles [**5**]. Gently tap the bottom of the cuvette to dislodge any bubbles. If bubbles remain, repeat the procedure above.
5. The cell can only be inserted one way round. Hold the base of the Dip cell cap and the top of the cuvette simultaneously [**6**]. Ensure the colored band on the label (and cuvette triangle) is facing the front of the instrument. Push the cell into the cell holder until it stops. Check that the cell sits flat on the cell holder.

Note: The measurement face of the cuvette and the colored band on the Dip cell label must face in the same direction.

The following illustration shows the correct insertion of the Dip cell.

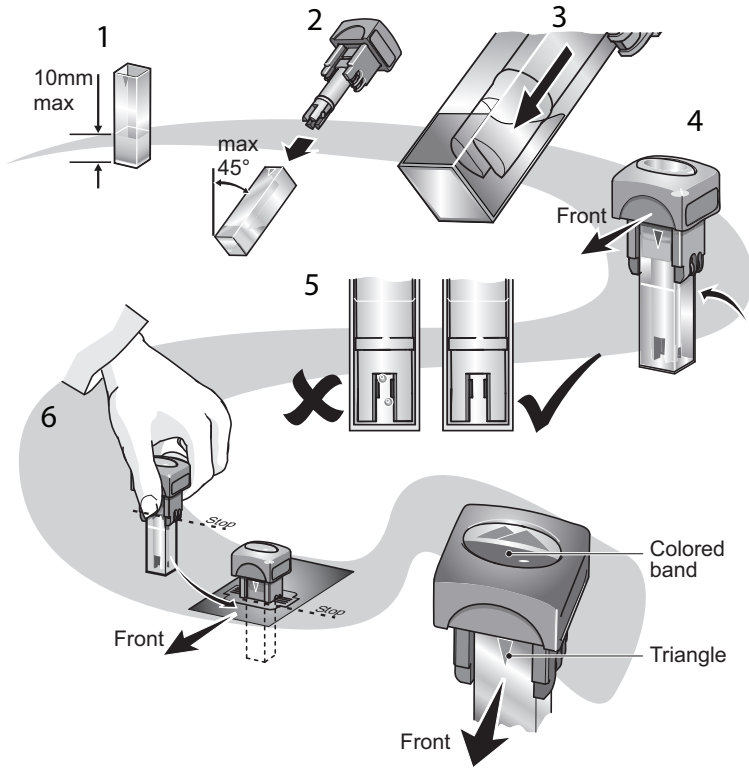


Figure 6.2 *Overview of inserting the Dip cell*

6.4 Removing the Dip cell

Carefully hold the base of the Dip cell cap and the top of the cuvette, and remove both the Dip cell and cuvette together. If you cannot securely grip both parts, do the following:

1. Lift the Dip cell up out of the cuvette, but before completely removing, gently tap the bottom of the Dip cell on the top of the cuvette [1]. This will help prevent drops of sample falling onto the instrument.
2. Place the Dip cell immediately into an empty cuvette for storage. This will prevent any damage occuring to the cell electrodes or the workspace.
3. Remove the sample cuvette afterwards.

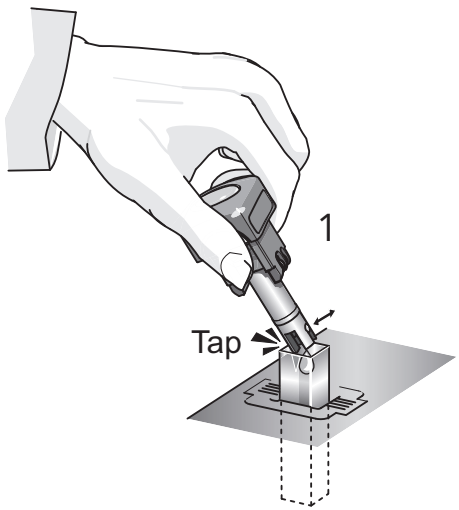


Figure 6.3 *Removing the Dip cell*

Note: Use the Dip cell case for long term storage.

6.5 Cleaning the Dip cell

Clean the cell between measurements, especially between different sample types. Cross-contamination between samples can seriously affect the results.

Clean the Dip cell electrodes regularly. These are made from palladium and can be cleaned physically and chemically. Follow the instructions below:

1. Immerse the electrodes in a gentle ultrasound bath (30 Watts) for 5 to 15 minutes before use. Use the dispersant used for the previous sample as the cleaning fluid. If this dispersant contains additives such as surfactants, follow this by ultrasonicating for 2 minutes in the pure solvent.

WARNING - General hazard
Ultrasonication can produce a fine aerosol of the bath liquid.

CAUTION
Do not immerse the complete cell. Only the sample electrodes must dip in to the dispersant, as shown below.

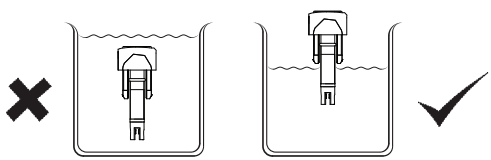


Figure 6.4 *Correct cell immersion for cleaning*

2. Remove the electrodes from the bath and rinse them with pure solvent. Use a pipe cleaner for gentle cleaning of electrodes.
3. To protect the Dip cell after cleaning, store it in an empty cuvette.

Before making a measurement, rinse the electrodes and cuvette with the sample to be measured. When changing sample, rinse the electrodes with pure dispersant.

6.6 Chemical compatibility

With proper use, only the central electrode section of the Dip cell will come into contact with sample. The outer components of the Dip cell will only come into contact if spillage or overfilling occurs.

Table 6.2 Component materials

Component	Materials
Electrode casing	Natural PEEK (polyetheretherketone)
Electrodes	Palladium
Top and side casing	Natural PEEK (polyetheretherketone)
Contacts	Phosphor bronze with nickel plating

Note: The electrode holder is made from Natural PEEK (Polyetheretherketone) which is resistant to a wide range of chemical products. However, seek advice from Malvern Panalytical and the sample manufacturer before using strong acid or base.

Chapter 7 MPT-3 Multi-purpose Titrator

7.1 Introduction

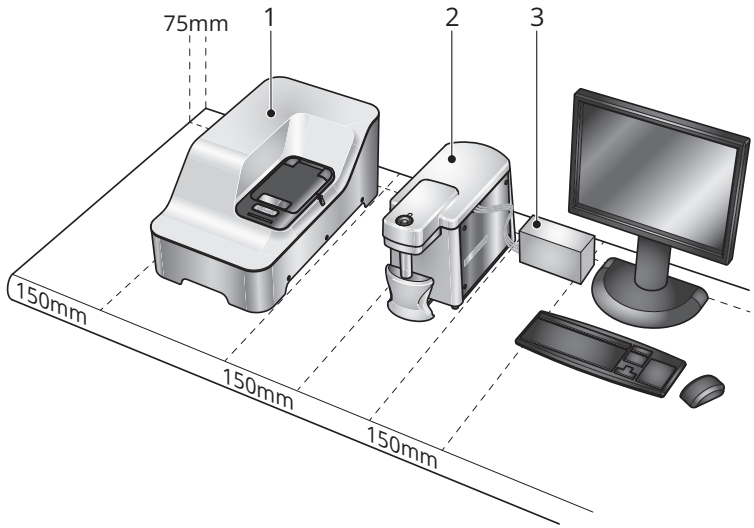
This section gives details on the MPT-3 Multi-purpose Titrator.

Table 7.1 Accessory and part number

Accessory	Part number
MPT-3 Multi-purpose Titrator	ZSU1001

7.2 MPT-3 Multi-purpose Titrator Overview

The MPT-3 Multi-purpose Titrator allows you to make zeta potential, size or intensity measurements while adding titrants to the sample so their effect can be observed over different pH's. This is a typical system setup:



1. The Zetasizer
2. The MPT-3 Multi-purpose Titrator
3. The Auto Degasser

Figure 7.1 System setup

7.2.1 How does it work?

The sample is pumped in circulation from a disposable container through the cell in the optical unit.

The Zetasizer [1] measures particle size and/or zeta potential of particles or molecules in a liquid medium.

The MPT-3 Multi-purpose Titrator [2] allows you to make zeta potential, size or intensity measurements while adding titrants to the sample so their effect can be observed.

The Auto Degasser [3] removes dissolved gasses from the titrants. Refer to Chapter 8 for details.

7.2.2 Control of the MPT-3 Multi-purpose Titrator

The MPT-3 Multi-purpose Titrator can be controlled via the Zetasizer ZS XPLORER software:

- Automatically, through a method. Once a method has been set up and started, the software will control the MPT-3 Multi-purpose Titrator to automatically titrate a sample based on the parameters set in the method.
- Manually, through the Titrator controls window. This features allows you to calibrate the pH probe, prime titrant lines, clean the sample lines and fill an attached cell.

Refer to the *Zetasizer Advance Series User Guide* for details on the software controls for the MPT-3 Multi-purpose Titrator.

7.3 Health and Safety

WARNING - General hazard
The accessories or the samples to be measured may be hazardous if misused. Users must read the Health and Safety information in the *Zetasizer Advance Series Basic Guide* before operating the system.

7.3.1 General safety issues

WARNING - General hazard
Use of the system in a manner not specified by Malvern Panalytical may impair the protection provided by the system.

WARNING - General hazard
Never remove the dispensing area cover during a measurement sequence.

7.3.1.1 Site requirements

The system has specific site requirements that must be met to ensure safe operation of the instrument. Refer to section 7.4.

Positioning the instrument

WARNING - General hazard
Do not position the instrument such that the power cord, where it exits the instrument, is unreachable for disconnection.

Purge warnings (MPT-3 Multi-purpose Titrator)

WARNING - General hazard
If a nitrogen supply is used, the system must be located in a well ventilated environment. Turn **off** the supply when not in use.

WARNING - General hazard
If a nitrogen supply is used, local Health and Safety requirements must be followed.

7.3.1.2 Electrical safety warnings

Take care when measuring samples not to spill liquid on the system covers. Conducting materials or liquids can damage insulation and cause dangerous conditions. If a spillage occurs, disconnect the power and clean up before re-applying power to the system. Users who suspect powder or liquid has entered the covers should call a Malvern Panalytical representative to arrange a service call.

7.3.1.3 Moving the MPT-3 Multi-purpose Titrator

If it is necessary to move the unit, follow these guidelines:

- Disconnect the power supply before attempting to move the unit.
- Disconnect and drain or vent any tubing that carries fluid or compressed air, including sample tubing, before moving the unit.
- Lift the unit by holding it under the base.

WARNING - Lifting hazard
The MPT-3 Multi-purpose Titrator weighs 5.3 kg and the Auto Degasser weighs 2.75 kg. Adopt proper lifting techniques to avoid back injury.

- If moving the unit large distances, Malvern Panalytical recommends repacking the unit in its original packaging.

7.3.2 Sample handling warnings

- Always handle all substances in accordance with the COSHH (Control Of Substances Hazardous to Health) regulations (UK) or any local regulations concerning sample handling safety.
- Before using any substance, check the *Safety Data Sheets (SDS)* for safe handling information.
- Use the instrument in a well ventilated room, or preferably within a fume cupboard, if the fumes from the sample or dispersant are toxic or noxious.
- Wear personal protective equipment as recommended by the Safety Data Sheets if toxic or hazardous samples are being handled, particularly during sample preparation and measurement.
- Wear protective gloves when handling hazardous materials, or those that cause skin infections or irritations.
- Do not smoke during measurement procedures, particularly where inflammable samples are used or stored.
- Do not eat or drink during measurement procedures, particularly where hazardous samples are used or stored.
- Take care when handling glass (e.g. microscope slides and beakers). Hazardous materials may enter a wound caused by broken glass.
- Always test a new sample or dispersant for chemical compatibility before use.
- After measuring hazardous samples, clean the system to remove any contaminants before making another measurement.
- Always label samples for analysis using industry standard labeling, particularly if they are handled by a number of staff or stored for long periods. Clearly label any operator hazard and associated safety precautions that are required for the handling of dangerous materials.
- Keep a record of all hazardous substances used in the system for protection of service and maintenance personnel.

- Always use responsible procedures for the disposal of waste samples. Most local laws forbid the disposal of many chemicals in such a manner as to allow their entry into the water system. The user is advised to seek local advice as to the means available for disposal of chemical wastes in the area of use. Refer to the *Safety Data Sheets*.
- The surfaces of the system may be permanently damaged if samples are spilled on them. If a spillage does occur, disconnect the system from the power supply before cleaning the spillage.

7.4 Site requirements

Note: These requirements should be read in conjunction with the system requirements as stated in the *Zetasizer Advance Series User Guide*.

7.4.1 Nitrogen purge specification

WARNING - General hazard
A nitrogen supply must be used in a well ventilated environment. Turn off the supply when not in use. Always follow local health and safety regulations regarding the use of nitrogen.

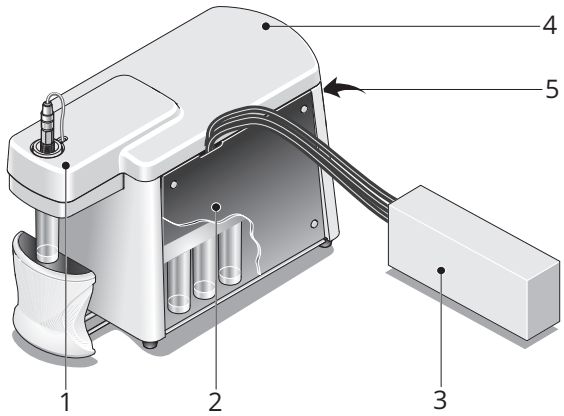
If a nitrogen supply is required then it must conform to the following specifications and flow rate conditions:

- It is important that the nitrogen supply is dry, free from oil and filtered to remove any contaminants that could affect the sample being measured.
- The flow rate should be adjustable between 2 and 20 ml/min.

7.5 Hardware

7.5.1 Overview

This section identifies the main features of the MPT-3 Multi-purpose Titrator accessory.

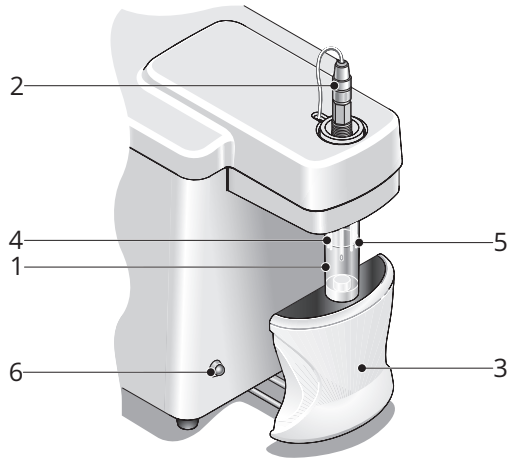


1. Dispersion head - refer to section 7.5.2
2. Dispensing area - refer to section 7.5.3
3. Auto Degasser - refer to Chapter 8
4. Dispensing area cover and tubing connection bracket - refer to section 7.5.4
5. Rear Panel - refer to section 7.5.5

Figure 7.2 *Main features of the MPT-3 Multi-purpose Titrator*

7.5.2 Dispersion head

The Dispersion head mixes sample with titrants, and also houses the pH probe. The illustration below identifies the main features of the dispersion head.



1. Sample container
2. pH probe
3. Magnetic stirrer
4. Sample exit and return tubes
5. Titrant tubes
6. Status indicator

Figure 7.3 *Dispersion head parts*

The above features are discussed in more detail in the following sections.

7.5.2.1 [1] Sample container

The sample container is the mixing and reaction vessel for the measurement. The sample container initially contains only the sample. Titrants are then added to the sample through the titrant tubes, and finally stirred.

To attach/remove the 'standard' sample container, screw the container into the dispersion unit as shown. Unscrew to remove.

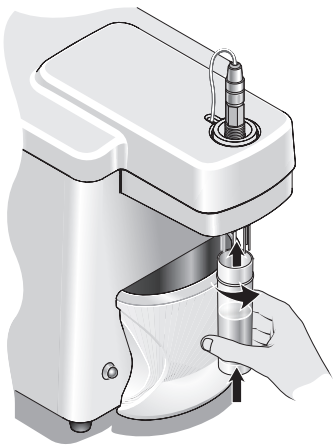


Figure 7.4 Attaching the sample container

Note: Be careful not to splash any sample when inserting and removing the sample container.

Note: The sample containers are disposable - obtain replacements from Malvern Panalytical. There are two sizes of sample container: 'standard' (shown above) and 'large volume' - for dilution titrations.

Large volume container

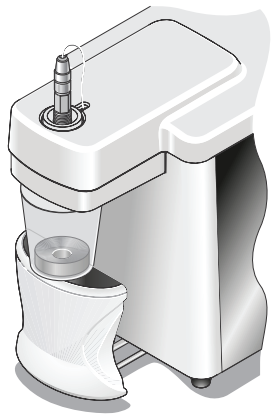


Figure 7.5 *The large volume container*

When using the large volume container, place the displacement ring at the bottom. The ring will make sure the magnetic flea is positioned correctly, and acts as a weight to keep the container in place when resting on the stirrer.

Note: The large volume container is primarily used for dilution titrations.

Note: This manual assumes that a standard sample container is used, unless otherwise stated.

7.5.2.2 [2] pH probe

The pH probe measures the pH of the sample. The performance of the pH probe will deteriorate over time. Correct maintenance will extend its life and ensure rapid and accurate titrations. It is recommended to replace the probe at least once a year.

CAUTION
Never allow the probe to dry out. If it does it may need replacing. Refer to section 7.8.2 for probe maintenance details.

7.5.2.3 [3] Magnetic Stirrer

The stirrer motor sits directly below the sample container. A magnetic stirrer 'flea' placed in the sample container agitates the sample during a titration or pH calibration and aids dispersion.

Set the stirrer speed in the *Titrator controls* window or when setting up a method. Refer to the *Zetasizer Advance Series User Guide* for details on the software controls for the MPT-3 Multi-purpose Titrator.

7.5.2.4 [4] Sample exit and return tubes

The sample circulates through these tubes to the cell. Make sure the ends of the sample exit and return tubes are below the surface of the sample. This ensures that the sample is pumped and circulated as well as reducing the bubbles produced and entering the sample line.

7.5.2.5 [5] Titrant tubes

The measured dose of titrant from the titrant containers enters the sample container through these tubes. The ends of the titrant tubes must always be below the surface of the sample. This makes sure that small amounts of titrant are dispensed fully.

If required they can be extended to use a larger volume titrant container. Channels are provided at the top of the dispersion area cover for the exit of the tubes.

Note: For protein measurements, where only a minimum sample volume is available, loosen the tubing clamp and carefully pull both sample and titrant tubing down towards the bottom of the container. Refer to section 7.5.4. Do not use the stirrer.

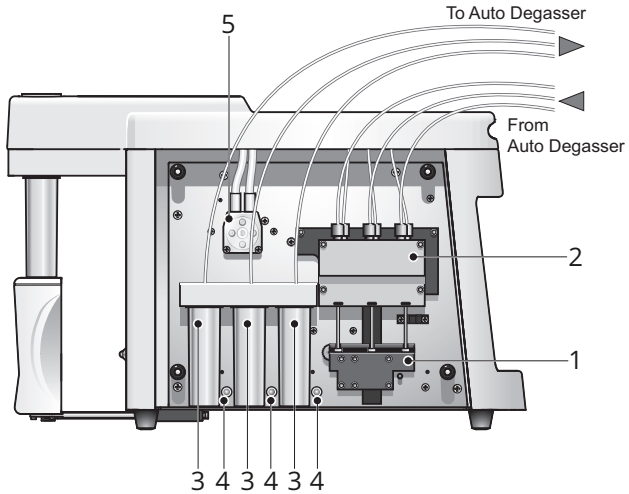
7.5.2.6 [6] Status indicator

This indicator shows the status of the MPT-3 Multi-purpose Titrator:

- Green — during operation.
- Amber — standby mode.
- Red — an error condition.

7.5.3 Dispensing area

WARNING - General hazard
Never remove the dispensing area cover during a measurement sequence.



1. Dispensing syringes and syringe drive
2. Manifold assembly and solenoid valves
3. Titrant containers
4. Titrant indicators
5. Pump head

Figure 7.6 The dispensing area (and cover)

The rest of this section gives further information about the indicated parts.

7.5.3.1 [1] Dispensing syringes and syringe drive

The syringes collect and dispense precise amounts of titrants from the titrant containers to the sample container.

7.5.3.2 [2] Manifold assembly and solenoid valves

The manifold assembly and solenoid valves control the flow of sample through the cell. These valves open or close to control the flow of the titrant from the titrant container, through the manifold assembly, to the sample container.

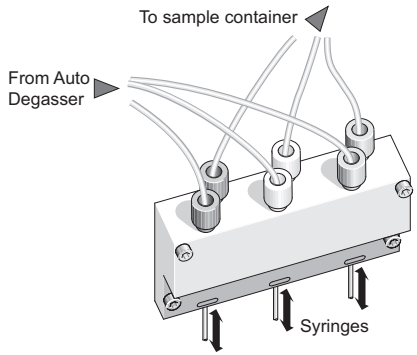


Figure 7.7 Manifold assembly

7.5.3.3 [3] Titrant containers

The titrant containers contain the solutions that will be added to the sample during a titration measurement sequence. Each container can hold either an acid or base. The tubing connectors from the top of the titrant containers are color coded at the manifold to match the LED indicators (below).

Refer to section 7.7 for information on how to fill, change and insert the titrant containers.

7.5.3.4 [4] Titrant indicators

Each titrant container has a corresponding LED that comes on whenever that titrant is being dispensed. The three LEDs are color coded (red, yellow and green).

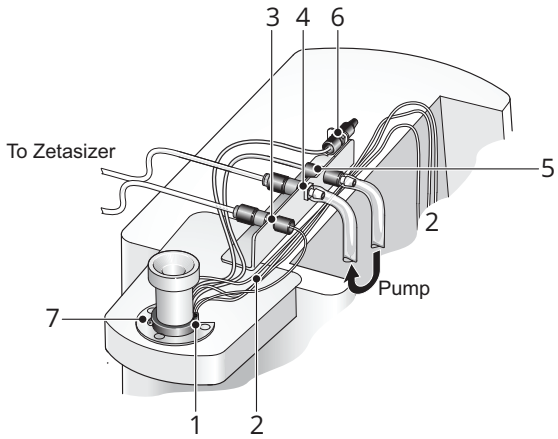
7.5.3.5 [5] Pump head

The pump head pumps the sample (or cleaning fluid) through the cell. The speed of the pump is set in the *Titrator controls* window. refer to the *Zetasizer Advance Series User Guide* for details on the software controls for the MPT-3 Multi-purpose Titrator.

7.5.4 Dispensing area cover and tubing connection bracket

A removable cover allows access to both the titrant and sample tubing. The cover pulls up and off the unit. The sample and titrator tubing is routed around the tubing connection bracket underneath the dispenser lid. To access the bracket, pull the dispensing area cover off. The illustration below shows the various connections.

For more information refer to section 7.6.



1. Tubing entry
2. Titrant tubes
3. Sample in connector
4. Sample out connector
5. Sample to pump
6. Purge connector
7. Clamping screw

Figure 7.8 Dispensing area cover and tubing connection bracket

7.5.4.1 [1] Tubing entry and tubing clamping ring

All tubing enters the sample container through channels in the container holder. The tubing includes three titrant tubes, purge tube, sample in and sample out tubing.

Adjusting the tubing length

Depending upon your sample volume and the type of titration, the tube length can be altered to higher or lower within the sample container:

- Loosen the clamping screw to loosen the tubing clamping ring.
- Carefully pull the tubing either up or down to the required height.
- Tighten the clamping screw to secure the tubing in position.

Note: The clamping ring can be used for securing both thin and thick tubing diameters in place. Thick tubing (3.2mm) is supplied as standard for connecting the sample flow in and out of the sample container.

7.5.4.2 [2] Titrant tubes

The titrant tubes are fed directly from the manifold to the sample container.

7.5.4.3 [3] Sample in connector (from Zetasizer)

The sample that has been measured is re-circulated to the sample container via this connector.

7.5.4.4 [4] Sample out connector (to Zetasizer)

The sample to be measured is recirculated through this connector to the cell in the instrument.

7.5.4.5 [5] Sample to pump

This connection feeds the sample from the sample container to the pump before passing to the sample out connector.

7.5.4.6 [6] Purge connector

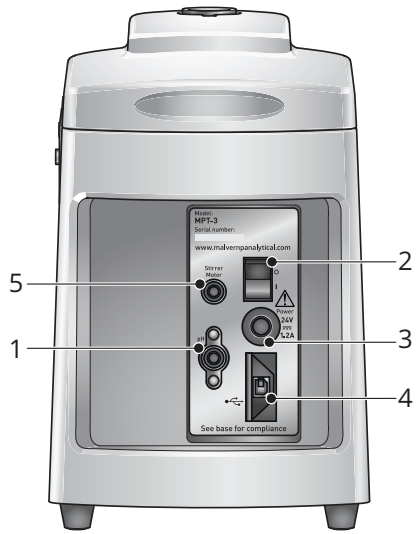
WARNING - General hazard
If a nitrogen supply is used the system must be located in a well ventilated environment. Turn off the supply when not in use. Refer to section 7.4 for a requirement specification for the nitrogen supply.

The purge connection enables connection of a nitrogen purge supply. This prevents any unwanted interactions with the atmosphere.

The amount of nitrogen needed to prevent oxygen absorption should be just enough to cover the immediate space above the sample. The nitrogen can be introduced into the container by blanketing the area directly above the sample or by bubbling the nitrogen through the sample. If too much nitrogen is introduced, or the flow rate is too high, there is a risk of bubbles being generated in the sample which can enter into the sample line and affect measurement results.

7.5.5 Rear panel

The rear panel contains all electrical connections to the computer and optical unit.



1. pH probe connection — where the pH probe is connected to the MPT-3 Multi-purpose Titrator. The probe connections must be kept clean and dry at all times. Contaiminated connectors may affect the performance of the probe.
2. On/off switch — the power on/off switch for the MPT-3 Multi-purpose Titrator.
3. Power input — the external power supply is connected here.
4. Communications connection — the connection cable to the computer is connected here. This allows the Zetasizer software to control the MPT-3 Multi-purpose Titrator.
5. Stirrer connection — the magnetic stirrer motor is connected here.

Figure 7.9 MPT-3 Multi-purpose Titrator rear panel

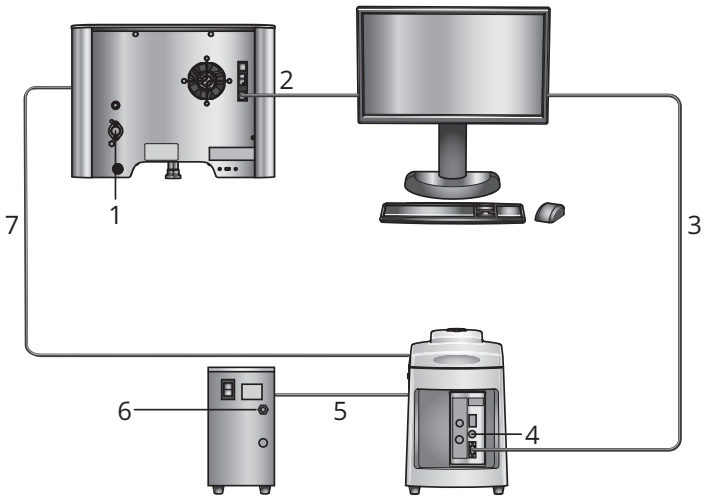
CAUTION
Take care when inserting the communications cable as it is a high retention cable. Forceful insertion or bending of the cable during insertion may damage the adapter at the rear panel connection.

CAUTION
Only use the power supply provided by Malvern Panalytical. Substitution may impair the safety or performance of the unit.

7.6 Connecting the MPT-3 Multi-purpose Titrator

Note: When moving the accessory short distances, attach a sample container containing an appropriate pH probe storage solution to the dispersion head to prevent the pH probe from drying out. If moving the accessory greater distances, completely remove the pH probe and its protective cover and store it in a suitable solution to stop the probe from drying out, refer to section 7.8.2.

Connect the MPT-3 Multi-purpose Titrator as described below:



1. Power cable to instrument (from Zetasizer PSU)
2. Communication connection — from the instrument to the computer (USB)
3. Communications connection — from the computer to the MPT-3 Multi-purpose Titrator
4. Power cable to MPT-3 Multi-purpose Titrator (from MPT-3 Multi-purpose Titrator PSU)
5. Titrant tubing — to and from the Auto Degasser
6. Power cable to Auto Degasser (from Auto Degasser PSU)
7. Sample tubing — to and from the Zetasizer cell

Figure 7.10 MPT-3 Multi-purpose Titrator connections

7.6.1 Tubing connections

7.6.1.1 MPT-3 Multi-purpose Titrator to Auto Degasser connection

Connect the titrant lines to be degassed to the Auto Degasser's front panel ports, as shown below:

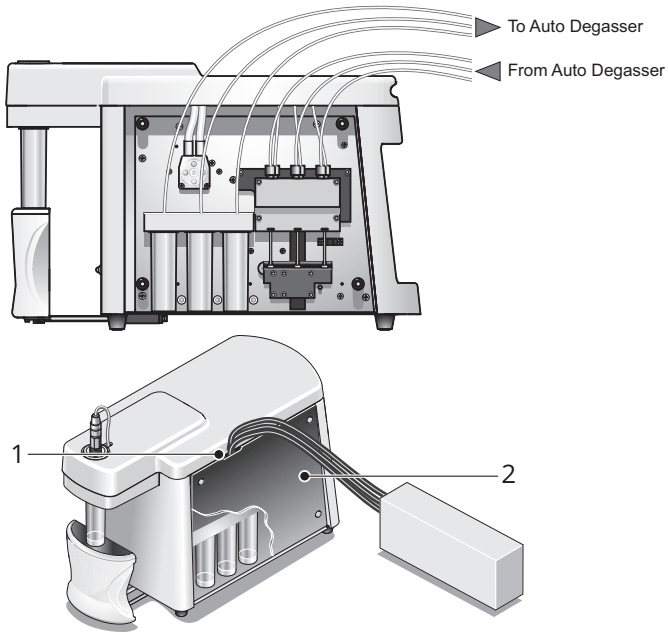


Figure 7.11 Connecting the MPT-3 Multi-purpose Titrator and Auto Degasser tubing

Refer to Chapter 8 for details on the Auto Degasser.

Note: Unused ports must be plugged to enable the Auto Degasser to operate correctly.

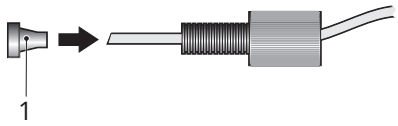
Feed titrant tubes from the titrant containers through the space [1] at the top of the dispensing area cover towards the Auto Degasser input ports, and then from the Auto Degasser outlet ports back again towards the manifold assembly [2].

End connections

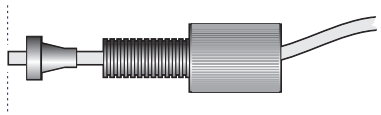
1. Make sure the tubing lengths will easily reach the Auto Degasser from the MPT-3 Multi-purpose Titrator.
2. Push tubing through the titrant container clamp above the titrant containers until it

touches the base of the container.

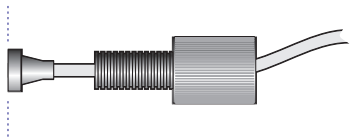
3. Push the tubing through the connector and slide a ferrule [1] over the tubing end. Note the direction of the ferrule taper.



4. Cut the tubing so the end is flat.



5. Move the tubing so its end is flush with base of the ferrule.



6. Screw the connector into one port on the front of the Auto Degasser. The direction of flow through the Auto Degasser is not critical.

Note: Only tighten plastic connectors by hand. Do not over-tighten.

7. Repeat the steps above to connect additional titrant lines to the Auto Degasser.

7.6.1.2 Auto Degasser to MPT-3 Multi-purpose Titrator connection

CAUTION
Never connect the Auto Degasser to the output side of the MPT-3 Multi-purpose Titrator pump. The high pressure may cause permanent damage to the degassing membrane.

1. Run the tubing from the titrant containers in the MPT-3 Multi-purpose Titrator to the Auto Degasser input.
2. Follow the instructions above on how to fit the connector and ferrule to the tubing end.
3. Connect the completed tubing assembly to the Auto Degasser and the MPT-3 Multi-purpose Titrator manifold input.

Note: Connect tubing to the manifold in the same order as they exit the titrant containers.

Titrant connections example shown in the following illustration. The direction of flow through the Auto Degasser is not critical.

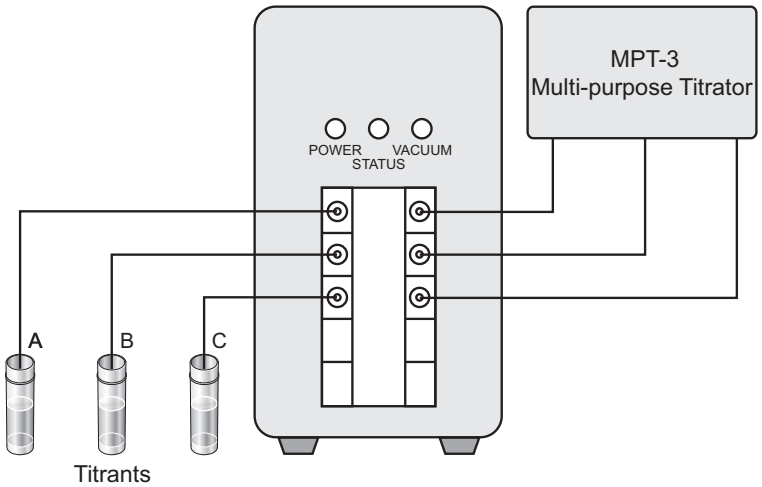


Figure 7.12 Titrant connections

4. When all required titrant lines are connected, plug any unused ports with the spare plugs supplied. Push these in by hand.

7.6.1.3 MPT-3 Multi-purpose Titrator to Zetasizer connection

The cell used, and the measurement being performed dictates how the tubing is connected. For each cell, minimize the tubing within the cell area before inserting into the tubing channel.

1. Folded capillary cell (DTS1070) - attach the tubing to the Luer connectors and screw them onto the Luer ports on the top of the DTS1070 cell (do not overtighten).
2. Once the cell is filled, insert the cell into the cell chamber.
3. Then insert the tubing into the tubing channel.

Note: For guidelines on filling a folded capillary cell for the MPT-3 Multi-purpose Titrator, refer to section 7.7.

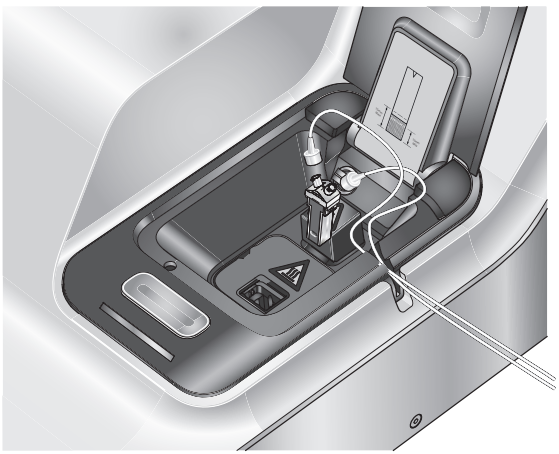


Figure 7.13 Connecting a DTS1070 cell

7.6.1.4 Zetasizer to MPT-3 Multi-purpose Titrator connection

Connect the tubing from the cells mentioned above, to the tubing connection bracket as shown.

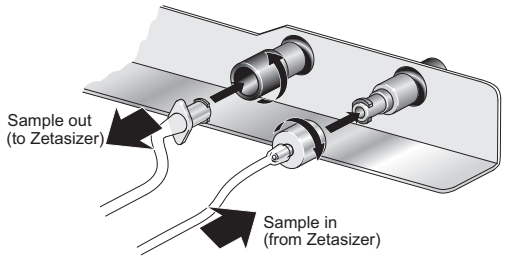


Figure 7.14 Tubing connection bracket

7.7 Preparing for measurements

Before making a measurement:

- If the titrant containers are nearly empty with approximately 5 ml or less left, fill the containers.
- Calibrate the pH probe, before each titration session.
- Make sure the titrant lines are fully primed and free from air bubbles before starting a titration.
- Prepare the sample, before each titration session.
- Fill the measurement cell, before each titration session.

Use the *Titrator controls* window to do any of these - refer to the *Zetasizer Advance Series User Guide* for more information.

7.7.1 About titrant concentration

To get accurate results you must prepare titrants. A concentration of 0.25 M is satisfactory for most measurements. In some situations it may be wise to vary this, therefore we recommend using two titrants of differing concentrations. For example, if performing pH measurements close to pH 7, you may need a lower concentration.

Titrant concentrations up to 1 M can be used, however it is usually more appropriate to use weaker titrant concentrations, such as 0.25 and 0.025 M.

Pre-diluted titrants are easier to use. Either use titrants that are diluted to the exact concentration or dilute titrants of a known Molar concentration.

For titrations that cover a wide pH range, use two concentrations of titrant, e.g. 0.25 M of acid and 0.025 M of the same acid. This ensures that the pH values achieved are closer to the requested values.

7.7.2 Calibrating the pH probe

The pH probe must be correctly calibrated. If the pH probe has not been calibrated the pH values will be incorrect. Calibrate the pH probe before each titration session. To calibrate the pH probe, refer to the *Zetasizer Advance Series User Guide*.

7.7.3 Priming the titrant lines

The titrant lines must be primed to make sure they are free of any bubbles before a measurement can take place. To prime the titrant lines, refer to the *Zetasizer Advance Series User Guide*.

7.7.4 Preparing the sample in the container

Before starting the measurement, prepare the sample and attach the container to the dispersion head.

- First prepare the sample to be measured and place an alliquot of known volume into a sample container. An initial sample volume of 10 ml is recommended (5 ml minimum to 20 ml maximum - for titrants over a small pH range). Refer to *Sample preparation* in the *Zetasizer Advance Series User Guide*.

Note: The sample should be of a suitable concentration to give an intensity of scattering within recommended values. Dilute the sample and restart the titration if the concentration is too high.

- Attach the sample container to the dispersion head as described in section 7.5.2.

7.7.5 Preparing cells for titration measurements

Fill the cell so that no bubbles are left within the sample path. This is especially important for Zeta potential measurements. Depending on the cell type, the filling instructions are different. Refer to the instructions for different cell types below.

7.7.5.1 Preparing the Folded capillary cell

To fill a folded capillary cell DTS1070, follow the instructions below:

1. Attach the tubing to the Luer connectors and screw them onto the Luer ports on top of the DTS1070 - do not over tighten.
2. Open the *Titrator controls* window from the **Titrator** tab on the **Instrument** window.
3. Select the pump speed, stirrer speed and number of cycles required. Then click the **Fill** button to begin the operation. Each cycle will pump the volume of the sample tubing and cell.

Note: Fill the cell at a lower speed than during a normal titration e.g. at 50%. This decreases the chance of bubbles entering the system. If bubbles are present, invert the cell while still pumping and tap it lightly to dislodge them.

4. Invert the cell and watch as the cell until it is filled to just over half way.
5. Check no air bubbles form in the cell. Tap the cell gently to dislodge any bubbles that do form.
6. Turn the cell upright until the cell is full.
7. Check again for bubbles in the cell. Tap gently to dislodge any bubbles if present.
8. The pump will circulate the sample through the sample tubing and into the cell until the number of cycles has been completed or when the **Cancel** button is pressed.
9. Insert the cell into the Zetasizer cell chamber, making sure the tubing is inserted into the tubing channel.

7.7.5.2 Preparing the High concentration cell

To fill a High concentration cell, ZEN1010, follow the instructions below:

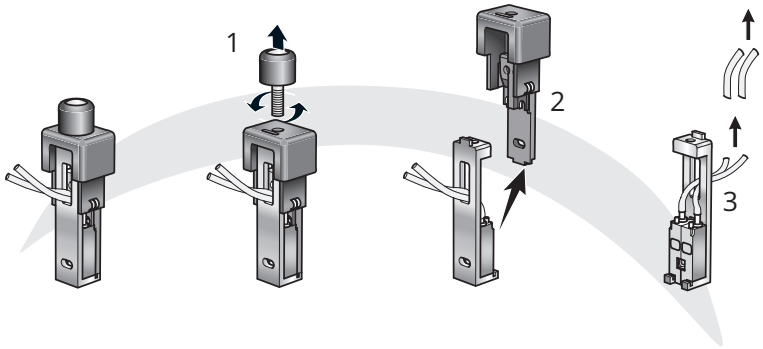


Figure 7.15 *Disassembly of the ZEN1010*

1. Remove the screw cap [**1**].
2. Separate the two halves of the cell [**2**] by pulling the rear casing vertically away from the metal front.
3. Detach and remove the tubing from the electrode chambers [**3**].
4. Attach the sample tubing from the MPT-3 Multi-purpose Titrator to the electrode chambers.
5. Re-assemble the cell in the reverse order.
6. Open the *Titrator controls* window from the **Titrator** tab on the **Instrument** window.
7. Select the pump speed, stirrer speed and number of cycles required. Then click the **Fill** button to begin the operation. Each cycle will pump the volume of the sample tubing and cell.

   Note: Fill the cell at a lower speed than during a normal titration e.g. at 50%. This decreases the chance of bubbles entering the system. If bubbles are present, invert the cell while still pumping and tap it lightly to dislodge them.

8. Check for bubbles in the cell. Tap gently to dislodge any bubbles if present.
9. Insert the cell into the Zetasizer cell chamber, making sure the tubing is inserted into the tubing channel.

7.8 Maintenance

CAUTION
Only a qualified Malvern Panalytical representative is allowed access to the inside of the MPT-3 Multi-purpose Titrator. There are no user serviceable parts inside the MPT-3 Multi-purpose Titrator.

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

- Cleaning the titrator
- Maintaining the pH probe
- Replacing the Dispensing syringes and O-ring
- Changing tubing

For more information refer to section 7.8.5.

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

7.8.1 Cleaning the MPT-3 Multi-purpose Titrator

WARNING - General hazard
Make sure the accessory is disconnected from the power supply before cleaning.

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

7.8.1.1 Cleaning the titrator surfaces

- Periodically, clean the covers using a mild soap solution on a damp cloth.
- Use a small amount of liquid only.
- Avoid the electrical components on the rear panel.
- Make sure the accessory is completely dry before applying power.
- Never use a solvent-based solution to clean the accessory as this could damage the painted surface.

7.8.1.2 Cleaning the sample and titrant lines

Keep the sample line clean to avoid cross-contamination of samples. Always flush the sample after each measurement session.

Note: Choose an appropriate cleaning fluid for the sample or titrants being used (e.g. deionised water). Make sure that it will reliably clean the sample or titrants used from the tubing.

7.8.1.3 Cleaning the sample tubing

1. Remove the sample container and replace with container filled with cleaning fluid.
2. Click the **Titrator** tab from the **Instrument** window. The **Clean** settings will be shown.
3. Select the required pump and stirrer speeds. Then click the **Clean** button to start the operation. The pump will circulate the cleaning fluid through the sample tubing.

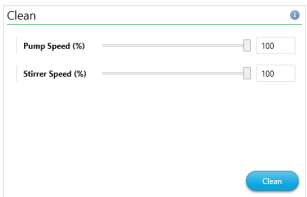


Figure 7.16 The clean settings

4. Run the cleaning cycle at least three times to make sure the tubing is properly cleaned. This will involve refreshing the cleaning fluid and repeating.
5. If you want to cancel a cleaning cycle when it is still running, click **Cancel**.

Tip: Use a larger cleaning fluid container for more effective cleaning.

Tip: To aid cleaning, pump air into the tubing before the cleaning fluid. The air will act as a buffer, first driving the sample through the tubing before the cleaning fluid cleans the tubes. To do this remove the cleaning fluid while leaving the pump running.

7.8.1.4 Cleaning the Titrant tubes

Insert containers of cleaning fluid in place of the titrant containers and use the **Prime Titrants** to pump the cleaning fluid through the selected titrant lines.

7.8.2 Maintaining the pH probe

Maintain the pH probe to ensure that the MPT-3 Multi-purpose Titrator performs correctly. The pH probe will also degrade over time and with usage pattern. It usually needs to be replaced annually, but this also depends on:

- How well the probe is maintained
- How often it is used
- The types of sample being measured

Important advice:

1. **Do not let the pH probe dry out**.
2. **Calibrate the pH probe regularly**.
3. **Clean the pH probe regularly**.

The pH probe operates over a range of pH 1 to pH 14, and temperature range of 0 to 70 °C. It is recommended that the titrations are performed at ambient temperature conditions, i.e. by setting the measurement temperature to equal the ambient temperature. This is because:

- The pH Probe is not temperature compensated, so the sample in the container must be the same temperature as the cell.

7.8.2.1 Preparation before first use

1. Remove the screw end-cap and connect to the cable supplied.
2. Remove the membrane protection teat. This teat contains a small amount of saturated KCl solution (3.8 M).
3. To allow air pressure equalization in the reference junction part of the probe, remove the rubber cover, or sleeve that covers the reference junction vent.
4. Check that the reference electrolyte is no more than 1 cm below the level of the opening. Top up with the electrolyte supplied if necessary.
5. Check for air bubbles within the inner glass membrane - if there are any, remove them by gently agitating the electrode.

7.8.2.2 Keep the pH probe wet

CAUTION
Never let the pH probe dry out, as this could severly affect its performance or irreparably damage the probe.

When the probe is immersed in a buffer solution, the pH should be attained (±0.1 of the pH) within 30 seconds. If this time exceeds 40 seconds, the probe should be replaced.

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

7.8.2.3 pH probe storage

When not in use, store the pH probe in water or a soaking solution. Also follow these guidelines:

- Short periods (few hours) — the probe can be immersed in sample solution.
- Up to a day — the probe can be immersed for up to a day in tap water.

CAUTION
Do not use deionised water as it will destroy the charge equilibrium of the glass electrode.

- Long periods (more than a day) — immerse the probe in either a dilute acid solution (pH 2 to pH 4) or an acidic buffer such as pH 4.
- Longer than a week — protect the glass membrane by adding a small quantity of the electrolyte solution to the rubber teat. If the probe has become dry, it may be possible to recover the performance by soaking overnight in a KCl solution. However, the performance may not recover and it will be necessary to replace the pH probe.

7.8.2.4 Cleaning the pH probe

Clean the probe periodically. The calibration procedure will indicate whether the probe needs cleaning.

Note: The wiping of the pH probe can cause a static charge which can alter pH readings. Therefore do not clean the pH probe just before making a measurement.

1. Soak the probe overnight in a solution of 0.1 M hydrochloric acid.
2. Use a soft tissue soaked in isopropanol and gently wipe over the end of the probe.
3. Once cleaned, re-calibrate the probe to check performance.

Note: Probes with a fibrous PTFE 'wick' can become clogged by very fine particles. When this happens, the pH probe will not respond to pH changes in the sample. Carefully brush the wick to recover performance.

7.8.2.5 Common problems

The performance of pH electrodes is dependent on the sample being tested. For example, pH measurement of high purity water or liquids containing sulfur ions will require special electrodes.

A slow response is often caused by the build-up of deposits on the glass membrane or frit. To get rid of the deposits:

1. Try cleaning the probe by dipping into concentrated Nitric Acid for 10 seconds.
2. If this is unsuccessful, repeat for one minute.
3. If still unsuccessful, regeneration may be possible by dipping the membrane for one minute into a solution of 2% HF and 5% HCl while stirring.
4. If the frit is blocked by proteins, soak the electrode in an appropriate organic solvent, or a solution of 10% pepsin in 1 M HCl. This will usually dehydrate the glass membrane, so it will be necessary to soak overnight in 3 M KCl solution.
5. If these actions are unsuccessful then the electrode will need to be replaced.

To provent **electrolyte contamination**:

1. Make sure the electrode's electrolyte level is above that of the liquid under test. This prevents electrolyte contamination, and ensures a good electrical contact.

Note: With the rubber cover removed, the electrode uses a small amount of electrolyte per day. When in continuous use, top up weekly.

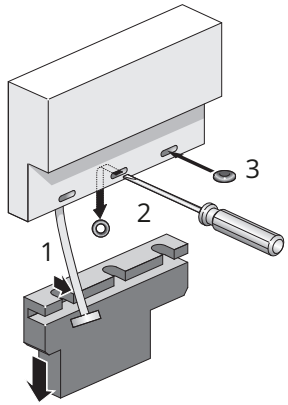
7.8.3 Replacing the dispensing syringes and O-ring

Aim to replace all three dispensing syringes and O-rings at least every 12 months, or sooner if leaks develop.

Contact Malvern Panalytical to obtain replacement syringes and O-rings (refer to section 7.8.6).

To remove a syringe and O-ring:

1. Turn off the accessory and remove the side cover.
2. Manually push down the syringe mount.
3. Pull the base of the syringe **[1]** out of the syringe mount, and then out from the O-ring.
4. Using a small screwdriver push the o-ring out of the manifold assembly **[2]** onto the base of the dispensing area.



1. Removal of syringe
2. Removal of o-ring
3. Replacement of o-ring

Figure 7.17 Replacement of syringe and o-ring

Replacement is the reverse of this procedure.

1. Push the o-ring into the recess on the manifold **[3]**.
2. Slide the syringe mount up, checking that the syringes smoothly enter the manifold assembly - make sure the syringe does not bend severly.
3. Prime the system once all the syringes have been replaced.

7.8.4 Changing the tubing

The illustration below shows how the connections are made.

Connect the tubing at the tubing connection bracket, as shown in the following illustration.

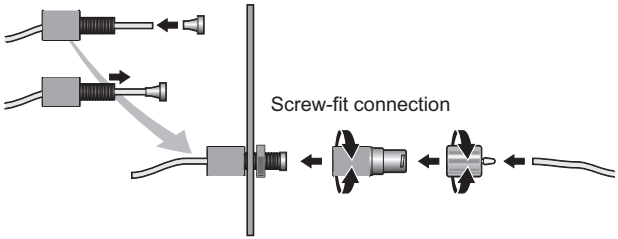


Figure 7.18 A screw-fit connection

7.8.5 Maintenance schedule

Follow this maintenance schedule to ensure correct accessory operation.

Table 7.2 Procedures for maintaining the accessory

Procedure	Period
Prime the system	Before every titration.
Calibrate the pH probe	Before every titration.
Clean the pH probe	Once a week or according to in-house QC procedures.
Clean the covers	Once a month.
Replace dispensing syringe and o-ring	All three dispensing syringes should be replaced every 12 months.
Replace pH probe	At least every 12 months.

7.8.6 Consumables and spares

For a full list of spares contact your Malvern Panalytical representative or use the estore at malvernpanalytical.com.

7.9 Specification

Table 7.3 General specifications

Parameter	Value
Dimensions (excluding pH probe)	Width: 170 mm Height: 260 mm Depth: 390 mm
Weight (dispenser unit only)	5.3 kg
Power requirements	100 - 240 V, 50 - 60 Hz, 24 Vdc via external power supply
Power rating	30 VA
Recommended maximum fill capacity of a standard sample container: Filled with titrant Filled with sample	 25 ml 20 ml
Modes of operation	Automatic via Malvern Panalytical software

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

7.10 Environmental conditions

Table 7.4 Environmental specifications

Parameter	Required value
Operating temperature	+10 to +35 °C
Storage temperature	-20 to +50 °C

Humidity	10 - 90 % (non-condensing)
Altitude	Up to 2000 m
Pollution degree	2
Mains supply voltage fluctuations	± 10 % of nominal voltage

7.11 Chemical compatibility

The sample flow path in the MPT-3 Multi-purpose Titrator and Auto Degasser 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 used is chemically compatible with the materials that they will come into contact with within the accessory.

The sections below detail all materials that come into contact with the sample and titrants in the normal operation of the accessory.

Table 7.5 Component in the accessory and the material it is made of

Component	Materials
Sample/titrant containers (25 ml)	Polypropylene
Sample/titrant containers (125 ml)	Polypropylene
Sample container mount (i.e. dispersion head)	Polypropylene
Titrant container mount	Polypropylene
Magnetic stirrer 'flea'	'Teflon'
Manifold	PEEK (Polyetheretherketone)
Manifold valves	PEEK and FFKM (seal)
Dispensing syringes	PTFE

Peristaltic pump tubing	Silicone rubber
Internal Tubing	PTFE
Tubing to optical unit and cell	Silicone rubber
Tubing connectors	Polypropylene / PVDF
pH probe	Glass
Displacement ring	PEEK

Chapter 8 Auto Degasser

8.1 Introduction

This section gives details on the Auto Degasser. This is used with the MPT-3 Multi-purpose Titrator.

Table 8.1 Accessory type and part number

Accessory	Part number
Auto Degasser (for the MPT-3 Multi-purpose Titrator)	DEG0003

8.1.1 How does it work?

The Auto Degasser removes dissolved gases from titrants. You can degas up to three titrant lines simultaneously with one unit.

- Dissolved gasses migrate across the tubing wall under a concentration gradient produced by the vacuum as the titrant flows within the coil.
- Gasses removed are expelled, and the chamber is maintained at a constant, preset vacuum level by varying the vacuum pump speed as needed.

8.2 Health and Safety

WARNING - General hazard
The accessories or the samples to be measured may be hazardous if misused. Users must read the Health and Safety information in the *Zetasizer Advance Series Basic Guide* before operating the system.

8.2.1 General safety issues

WARNING - General hazard
Use of the system in a manner not specified by Malvern Panalytical may impair the protection provided by the system.

WARNING - General hazard
Never remove the cover from the Auto Degasser.

8.2.1.1 Site requirements

The system has specific site requirements that must be met to ensure safe operation of the instrument. Refer to section 8.3.

Positioning the instrument

WARNING - General hazard
Do not position the instrument such that the power cord, where it exits the instrument, is unreachable for disconnection.

8.2.1.2 Electrical safety warnings

Take care when measuring samples not to spill liquid on the system covers. Conducting materials or liquids can damage insulation and cause dangerous conditions. If a spillage occurs, disconnect the power and clean up before re-applying power to the system. Users who suspect powder or liquid has entered the covers should call a Malvern Panalytical representative to arrange a service call.

8.2.1.3 Moving the Auto Degasser

If it is necessary to move the unit, follow these guidelines:

- Disconnect the power supply before attempting to move the unit.
- Disconnect and drain or vent any tubing that carries fluid or compressed air, including sample tubing, before moving the unit.
- Lift the unit by holding it under the base.

WARNING - Lifting hazard
The MPT-3 Multi-purpose Titrator weighs 5.3 kg and the Auto Degasser weighs 2.75 kg. Adopt proper lifting techniques to avoid back injury.

- If moving the unit large distances, Malvern Panalytical recommends repacking the unit in its original packaging.

8.2.2 Sample handling warnings

- Always handle all substances in accordance with the COSHH (Control Of Substances Hazardous to Health) regulations (UK) or any local regulations concerning sample handling safety.
- Before using any substance, check the *Safety Data Sheets (SDS)* for safe handling information.
- Use the instrument in a well ventilated room, or preferably within a fume cupboard, if the fumes from the sample or dispersant are toxic or noxious.
- Wear personal protective equipment as recommended by the Safety Data Sheets if toxic or hazardous samples are being handled, particularly during sample preparation and measurement.
- Wear protective gloves when handling hazardous materials, or those that cause skin infections or irritations.
- Do not smoke during measurement procedures, particularly where inflammable samples are used or stored.
- Do not eat or drink during measurement procedures, particularly where hazardous samples are used or stored.
- Take care when handling glass (e.g. microscope slides and beakers). Hazardous materials may enter a wound caused by broken glass.
- Always test a new sample or dispersant for chemical compatibility before use.
- After measuring hazardous samples, clean the system to remove any contaminants before making another measurement.
- Always label samples for analysis using industry standard labeling, particularly if they are handled by a number of staff or stored for long periods. Clearly label any operator hazard and associated safety precautions that are required for the handling of dangerous materials.
- Keep a record of all hazardous substances used in the system for protection of service and maintenance personnel.
- Always use responsible procedures for the disposal of waste samples. Most local laws forbid the disposal of many chemicals in such a manner as to allow their entry into the water system. The user is advised to seek local advice as to the means available for disposal of chemical wastes in the area of use. Refer to the *Safety Data Sheets*.

- The surfaces of the system may be permanently damaged if samples are spilled on them. If a spillage does occur, disconnect the system from the power supply before cleaning the spillage.

8.3 Site requirements

8.3.1 Space requirements

The Auto Degasser is designed to be placed on a standard laboratory bench. It is connected to the MPT-3 Multi-purpose Titrator system between the titrant supply and titrator pump.

Allow enough space for easy access to all components of the system.

Table 8.2 Dimensions for the Auto Degasser

Parameter	Description
Dimensions	73 mm, 249 mm, 127 mm (W,D,H)

Allow additional space both in front, to accommodate the tubing connected to the unit, and behind to accommodate the power lead.

8.3.2 Electrical power requirements

The mains power supply must be clean and filtered. If necessary, fit an un-interruptible power supply (UPS) to remove any spikes or noise. The power requirement for the Auto Degasser is indicated below.

Table 8.3 Power requirements for the Auto Degasser

Parameter	Description
Power requirement	100 - 240 Vac, 1 A, 50 - 60 Hz (refer to section 8.9)

CAUTION
Use the correct power lead for the territory.

A set of four interchangeable adaptor plugs are is included to allow the AC adapter to be plugged into the standard electrical wall sockets in North America, Japan, the U.K., most countries in continental Europe, and Australia.

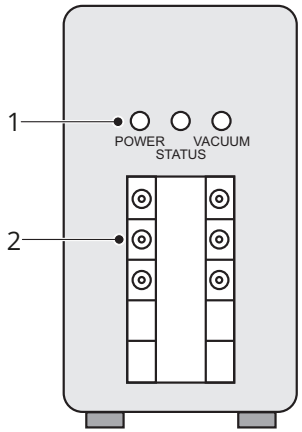
If it is necessary to replace the AC Adapter, contact Malvern Panalytical.

Note: Refer to the **Health and Safety** section in the *Zetasizer Advance Series Basic Guide* for more information.

8.4 Auto Degasser features

This section identifies the main features of the Auto Degasser accessory.

8.4.1 Front panel connections



1. LEDs
2. Titrant line input / output ports

Figure 8.1 Auto Degasser front panel

8.4.1.1 Titrant line ports

The three channels on the front of the Auto Degasser consist of both an input and output port through which to connect lines to the MPT-3 Multi-purpose Titrator.

Note: Flow direction is not critical.

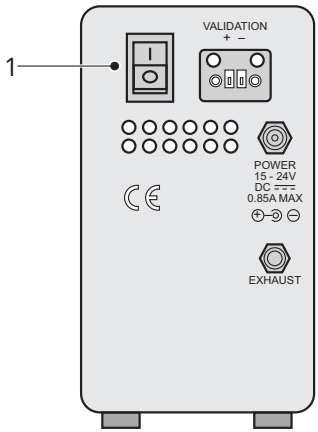
Note: To ensure the Auto Degasser works properly, use the plugs provided to seal any unused channels.

8.4.1.2 Front panel indicators

The LEDs on the front of the Auto Degasser indicate the following:

- Power (Green) — power is applied to the Auto Degasser - cable is plugged in and Power switch turned ON.
- Status (Yellow) — vacuum level is outside the acceptable operating range. Lights at initial power-up and remains on during pump-down. It will turn off after a few minutes, when the vacuum level goes below 100 mm of Hg absolute. In case of error, the LED will flash in one of two modes:
  On and off in even 1-second intervals - pump was not able to reach vacuum set point, indicating a possible leak
  On for 1 second and off for 2 seconds - indicates a vacuum signal error.
- Vacuum (Green) — vacuum level is within the acceptable operating range. The LED will light after the initial pump-down, and remain on as long as the Auto Degasser is powered up and the vacuum level is below 100 mm of Hg absolute.

8.4.2 Rear panel connections



1. Power switch

Figure 8.2 *Rear Panel connections and controls*

8.4.2.1 Power connector

CAUTION
Only use the AC power adapter as supplied from Malvern Panalytical.

Connect the output plug from the AC power adapter to this connector. For further Power requirement information, refer to section 8.3.

8.4.2.2 Power switch

The On/Off power settings are:

O = Off

I = On

8.4.2.3 Exhaust port

Gas expelled from the vacuum chamber exits through the exhaust port.

8.4.2.4 Validation connector

Depending on the model, there may be a 2-pin receptacle labelled "Validation" located next to the power switch. This receptacle and its associated screw-lock plug allow a validation signal from the Auto Degasser's control circuit to be sent to a computer or data system. This validation output indicates vacuum level. Refer to section 8.9 for more details.

8.5 Using the Auto Degasser

WARNING - General hazard
Use appropriate care when handling flammable solvents. Make sure that there are no leaks in the titrant lines and that hazardous exhaust gases are properly vented.

8.5.1 Powering up the Auto Degasser

1. Make sure the Auto Degasser is plumbed into the system and the power lead is installed, refer to section 7.6
2. Turn on the rear panel power switch - the green power LED lights.
3. The system now makes several checks and increases the vacuum pump to high RPM. It will get to the required level but the RPM will vary slightly to maintain a constant vacuum level.

8.5.2 Initial pump-down

During initial pump-down: status LED will be lit yellow.

Normal operating level: status LED will go off and green vacuum LED will light.

Start the titrant flow through the system and check for leaks around the connectors.

Note: If a leak occurs tighten the connection 1/8 turn. If the leak persists, disconnect and inspect the fitting. If the nut and ferrule are in good condition, reconnect the fitting. If the leak persists, replace the nut and ferrule and repeat the procedure until leaks stop.

The Auto Degasser maintains a constant vacuum pressure of 50 mm Hg absolute (nominal) by varying the speed of the vacuum pump as needed depending on the degassing load in the system.

If a leak occurs within the chamber, the controller increases the pump RPM to maintain the vacuum level. If the pump cannot maintain the vacuum level within 30 minutes, the yellow LED flashes, indicating a possible leak. If this happens the system will shut down and go into a "safe" mode.

Also refer to section 8.6.1.

Note: Refer to section 7.6 for details on Auto Degasser connections.

8.5.3 Powering off

Turn off the Auto Degasser when the MPT-3 Multi-purpose Titrator is not in use. The vacuum chamber(s) will slowly return to atmospheric pressure when the unit is powered off.

8.5.3.1 Shutdown

There are two types of shutdown procedures: long-term and short-term.

Short-term shutdown (overnight and weekends)

WARNING - General hazard
Obey all precautions pertaining to hazardous titrants and/or those titrants that form harmful deposits or by-products.

CAUTION
Damage caused by precipitating buffer salts in capillary tubing, or damage resulting from this condition, is specifically excluded from warranty.

- Remove harmful titrants from the Auto Degasser and other instruments in the Zetasizer system.
- Prime the MPT-3 Multi-purpose Titrator with demineralised water in the titrant container to flush buffer salts from the system. Evaporation can leave salt crystals that may form harmful deposits. Remove chloroform or titrants that can decompose to form hydro-

chloric acid from the system.

- For weekend storage flush 60/40% MeOH/Water through the Auto Degasser, then turn off the Auto Degasser.

Long-term shutdown and storage

- Follow the first two *Short-term shutdown* steps above.
- Disconnect the tubing from the MPT-3 Multi-purpose Titrator and direct the output tubing to a beaker. Flush the Auto Degasser, first with water and then with Isopropanol.
- Turn off the Auto Degasser. Then disconnect the tubing between the Auto Degasser and titrant containers, and the Auto Degasser and the MPT-3 Multi-purpose Titrator. Plug all of the ports on the Auto Degasser.
- Store the Auto Degasser in a clean, dry location.
- Before using the Auto Degasser, purge it with the correct titrant before reconnecting the MPT-3 Multi-purpose Titrator and restarting the system.

8.5.4 Flushing the titrant lines

When flushing a tritrant line, the tubing inside the Auto Degasser contains a very small amount of titrant (approximately 480 µl). When changing titrants - if the final titrant is immiscible with the first - use an intermediate titrant that is miscible with both. Once any air bubbles have been cleared from the titrant line, any further bubbles may be caused by the titrant container or a leaking fitting.

Note: Prime the MPT-3 Multi-purpose Titrator after the Auto Degasser has reached equilibration. This ensures that the titrant in the tubes from the Auto Degasser to the sample tube is fully degassed.

8.6 Principles of operation

The Auto Degasser consists of a vacuum chamber, degassing tube, variable speed vacuum pump, controller, sensor, and check valves. The titrant flows into a degassing tube, which is inside a vacuum chamber. Decreased pressure in the chamber causes the outward movement of gas, dissolved in the titrant, across the tube wall, thus degassing the titrant, in accordance

with Henry's Law. The pressure in the vacuum chamber is established by the vacuum pump and monitored by the controller through an integrated sensor. Degassed titrant exits the Auto Degasser and enters the pump.

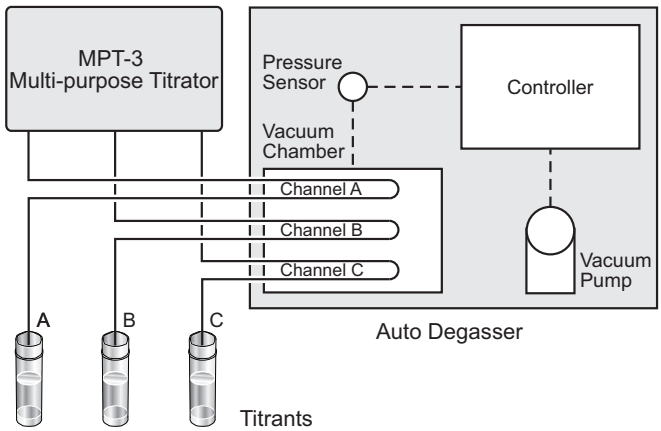


Figure 8.3 *Block diagram of a Auto Degasser*

8.6.1 Extending the degassing flow rate range

Certain organic titrants outgas upon mixing with water, if not properly degassed. These titrants are generally alcohols (e.g. methanol), acetonitrile and tetrahydrofuran. To degas these titrants without outgassing, pass a water (25%) and methanol (75%) mixture through a single channel using a pump flow rate of 2mL/min. At higher flow rates, you usually only need to pass the organic portion of the titrant through a second degassing channel. This is because organic titrants (e.g. methanol) can hold at least 10 times more dissolved atmosphere than water.

To more thoroughly degas a titrant, connect the outlet of the organic channel to the inlet a second channel and the outlet of the second channel to the pump. This places the two channels in series and doubles the degassing capacity for the organic portion of the titrant.

8.7 Maintenance

This section covers both the routine and preventative maintenance steps required for the Auto Degasser.

8.7.1 Preventative Maintenance

To maintain the Auto Degasser in the best condition:

- Obey standard laboratory cleanliness practices. Use only high-purity titrants - preferably High Performance Liquid Chromatography (HPLC) Grade. Water should be bottled HPLC Grade, or filtered and deionised water. Filter all titrants to prevent particulate contamination and tubing blockages.
- Use only high-purity gases when drying contact areas. Follow the short- and long-term shutdown procedures described.
- Clean external surfaces of the instrument with a clean, damp cloth. Immediately clean any spills on or near the instrument using appropriate methods. Some titrants can damage the appearance and function of the instrument.

8.7.2 Routine Maintenance

Routine maintenance is defined as replacing the normal wear items when degradation in performance is noticed.

If a problem arises, refer to the section 8.8.

WARNING - General hazard
Never remove the cover from the Auto Degasser.

8.8 Troubleshooting

Table 8.4 Common troubleshooting problems and their solution

Problem	Probable cause	Solution
Power switch is on, but all 3 LEDs are off	The AC Adapter is not plugged into the AC wall socket. Blown fuse.	Plug the AC Adapter into the AC outlet. Contact your Service Representative.
Yellow Status LED is on steadily, pump running, RPM seems high.	Pump is in initial pump-down phase or system's degassing demand has increased.	This is normal operation. If pump speed audibly continues to rise for an extended period, this could indicate a fault.
Yellow Status LED flashing - 1 second on/off. Vacuum pump not running.	Possible system leak.	Contact your Service Representative.
Yellow Status LED flashing - 2 seconds off, 1 second on. Vacuum pump not running.	Possible sensor or Control Board fault.	Contact your Service Representative.
Pump can't be heard but Power and Vacuum green LEDs are illuminated.	The pump is virtually silent at low RPM, even though vacuum is good and degassing is normal.	Place a hand on the top of the unit. A slight vibration can be felt indicating the pump is operating at low RPM.
Bubbles appear through the output tubing.	Loose fitting(s).	Tighten the input and output fittings.
No titrant flow.	If a buffer titrant was left in the Auto Degasser for some time after use, it may block the degasser elements.	Use a different channel, or connect the channel to a beaker of the titrant without the buffer. Draw the titrant through the channel to dissolve the buffer. Do not push the titrant through the channel. If this fails, contact your Service Representative.

8.9 Specification

8.9.1 General

Table 8.5 General specifications

Parameter	Description
Dimensions	75 mm, 250 mm, 130 mm (W,D,H)
Weight	2.75 kg
Channels	Three independent
Degassing process	Gas permeation through a fluoropolymer membrane
Maximum flow rate	10 ml/min
Degassing capacity	2 ppm at 1 ml/min
Dead volume	480 microliters per channel for standard channel
Materials in contact with titrant-s/solvents	PEEK, glass-filled PTFE, Teflon AF®, PTFE
Power requirement using supplied AC adapter	100 - 240 Vac, 1 A, 50 - 60 Hz
Interchangeable adapter plugs	4 supplied with AC Adapter Interchangeable to AC Adaptor: North America/ Japan, U.K., Continental Europe, Australia
Installation over-voltage category	II (IEC 60664)

8.9.2 Environmental

Table 8.6 Environmental specifications

Parameter	Description
Ambient temperature	Operating: +10 to 35 °C Storage: -20 to +60 °C

Ambient Relative Humidity	20 to 80 % RH (without condensation)
Altitude	Operating: Up to 2000 m Storage: Up to 12000 m
Indoor vs Outdoor use	Indoor
Pollution degree	2

8.9.3 Validation output

Table 8.7 Validation specifications

Parameter	Description
Signal	5 mVdc / 1 mm Hg absolute from 20 to 800 mm Hg (0.100 Vdc at 20 mm Hg; 4.000 Vdc at 800 mm Hg)
Accuracy	±1.0% of reading ±0.010 Vdc from 20 to 800 mm Hg

8.9.4 Connector and tubing sizes

Table 8.8 Connector and tubing sizes specifications

Parameter		Description
Connectors	Material	PEEK
	Size/description	1/4-28 flangeless fittings for 1.6mm 'Outside Diameter' tubing
Connecting tubes from MPT-3 Multi-purpose Titrator	Material	PTFE
	Size/description	1.6 mm x 0.8 mm (Outside Diameter x Inside Diameter)

8.10 Chemical compatibility

Use only HPLC grade solvents in all analyzes.

CAUTION
The degassing membrane in the Auto Degasser is manufactured from Teflon AF®. Use of inappropriate solvents in the Auto Degasser will result in the dissolution and hence destruction of the membrane.

Teflon AF® Solvent Compatibility is:

- Teflon AF® is inert to all solvents normally used in HPLC.
- Teflon AF® is soluble in perfluorinated solvents such as Fluorinert® FC-75 and FC-40 and Fomblin perfluoro polyether solvents from Ausimont.
- Freon® solvents will adversely affect Teflon AF®.

Note: If using high concentration buffers, crystals may eventually form within the channel. To prevent this, follow the same procedures as for the MPT-3 Multi-purpose Titrator - refer to Short-term shutdown procedures in section 8.5.

WARNING - General hazard
Use appropriate care when handling flammable solvents. Make sure that there are no leaks in the titrant lines and that hazardous exhaust gases are properly vented.

All parts that contact the titrant (solvents) are made of PEEK, Kel-F®, Tefzel® or Teflon AF®, PTFE or glass-filled PTFE.

PEEK is sensitive to sulfuric acid and certain solvents.

Malvern Panalytical Ltd.
Grovewood Road, Malvern,
Worcestershire, WR14 1XZ,
United Kingdom

Tel. +44 1684 892 456
Fax. +44 1684 892 789

Malvern Panalytical B.V.
Lelyweg 1,
7602 EA Almelo,
The Netherlands

Tel. +31 546 534 444
Fax. +31 546 534 598

MAN0604-05-EN

www.malvernpanalytical.com