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ZETASIZER 4

Hardware Reference Manual

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RECORD OF AMENDMENTS

This manual may receive amendments as the equipment is updated. This page can be used to note the amendments that have been made since the manual was issued. Please quote the issue number and the amendments added with any enquiry or request for further copies.

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Issue	**Date**	**Comments**
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SAFETY CONSIDERATIONS

These safety guide-lines apply to ZetaSizer systems and their accessories. The guide-lines are part statutory and part advisory. They should not be used as a replacement for common sense by the operator.

The instrument is a laser product satisfying the requirements of the Centre for Devices and radiological Health (Class I) and British Standard EN 60825. The user is advised of the possibilities of a laser hazard where the laser beam emerges and the location of safety labels.

In addition, the instrument uses mains power and liquids are used in some cell systems and so further advice is given on the location of the mains powered parts.

Finally, the samples that you may wish to analyse could present further safety problems and some guide-lines are provided.

This instrument contains no user serviceable parts and repairs should be left to the qualified staff of your supplier. The optical components are precisely positioned during manufacture,under no circumstances should these settings be changed. A section later in this manual on maintenance describes all the operations that are required for correct routine use.

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LASER SAFETY PRECAUTIONS

WARNING - LASER RADIATION CAN BE HARMFUL TO THE BODY AND CAN CAUSE PERMANENT EYE DAMAGE. READ THIS SECTION OF THE MANUAL CAREFULLY BEFORE INSTALLING OR OPERATING THE EQUIPMENT.

CAUTION - USE OF CONTROLS OR PERFORMANCE OF PROCEDURES OTHER THAN THOSE SPECIFIED HEREIN MAY RESULT IN HAZARDOUS RADIATION EXPOSURE.

NOTES

- Never look into the direct path of a laser beam or its reflections.
- Never look into the expected path of a laser beam without ensuring by positive means that the laser cannot be excited.
- While carrying out any operations in which the laser beam is exposed, wear protective goggles, ensuring that they are suitable for the power output and wavelength of the laser in use, and avoid even when wearing such goggles, direct viewing along the paths of the laser beam.
- Only authorised personnel should be allowed to operate or enter the area in which the equipment is in use.
- Avoid traversing the laser beam with metal or other reflective objects.
- Warning signs of the presence, or potential presence, of Laser radiation should be posted at the entrance to the hazard area.

Laser warnings on the ZetaSizer Optics Unit

The warning notes of the previous page are general notes that apply to the operation of ANY laser equipment. These notes supplement the general advice with information specifically for the ZetaSizer. They are related to the requirements of the C.D.R.H. safety standards in the U.S. and to EN 60825 in the U.K.

The positions of the laser safety warnings are shown on the next page.

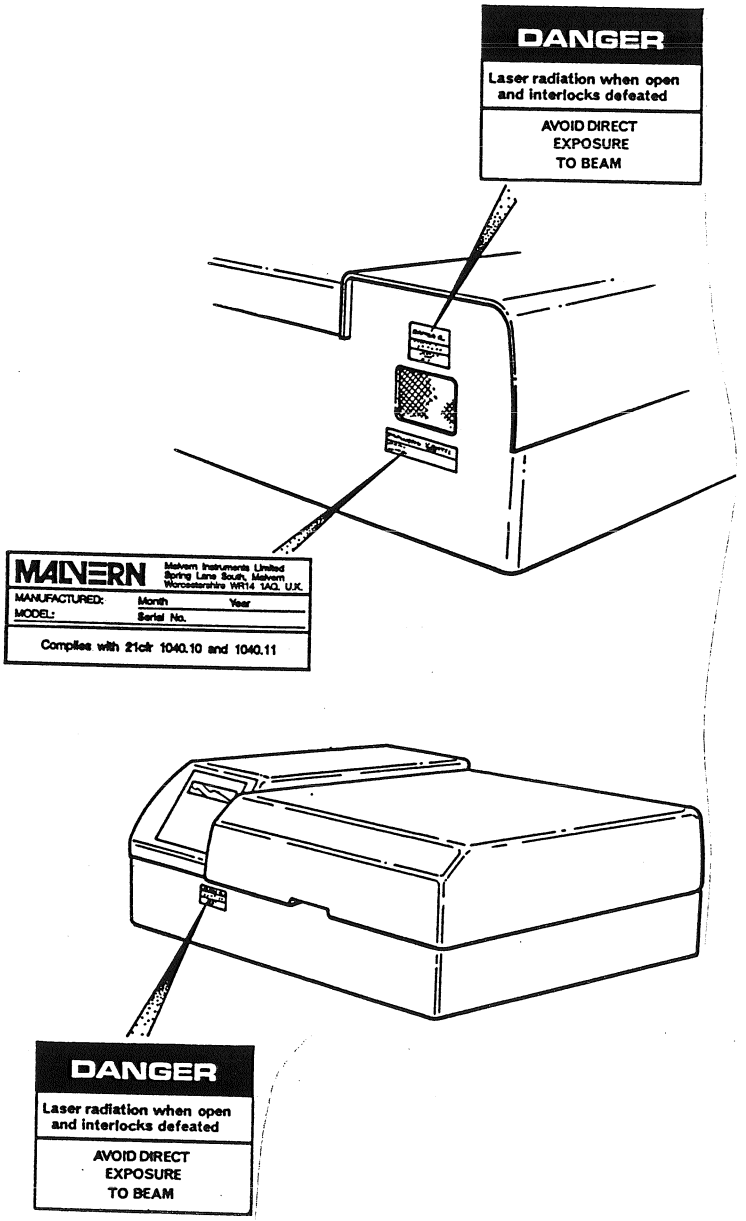
The ZetaSizer optics unit has an automatic cut-out switch fitted under the lid. When the lid is raised the laser beam is interrupted. During normal operation this safety cut-out switch should not be overridden.

During normal use of the ZetaSizer this interlock system gives a Class I level of protection. Higher levels of hazard should only arise during maintenance and service operations.

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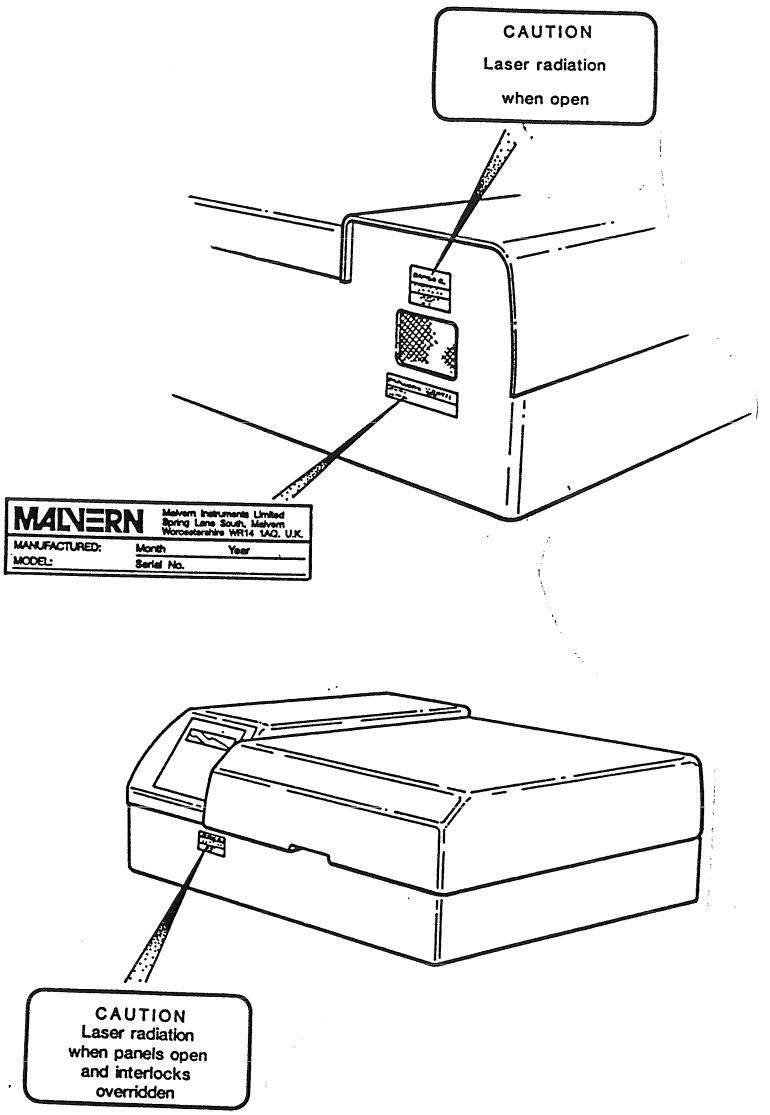
The laser used within the ZetaSizer is a nominal 5mW Helium Neon continuous power model having a nominal beam diameter before expansion of 1mm. It is rated for safety purposes as 10mW maximum power and is marked by the supplier as complying with C.D.R.H. Class IIIb. A label is fitted to the laser body recording this information.

Alignment checks and/or re-alignment should only be carried out by fully qualified service personnel who fully understand the dangers of laser radiation. On completion of service operations, the override facility should be removed and retained by the service department and not left with the operator.



CDRH Laser Safety Labels

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BSI Laser Safety Labels

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Electrical Safety Warnings

The ZetaSizer and all accessories are mains powered and their input power cables should be treated accordingly.

The metal parts of the instrument are earthed via the mains earth connection. Never run the equipment without an earth connection. If servicing the units, never remove permanently any earth connections within the housings.

Be careful when measuring samples to avoid liquid or powder spillages over the equipment. Conducting materials or liquids can break down the insulation and cause dangerous conditions within the instrument. Should such spillages occur, disconnect the power and scrupulously clean up.

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Sample Handling Warnings

Use the instrument in a well ventilated room if noxious samples or dispersants are to be analysed.

Wear a protective respiratory mask if noxious samples or dispersants are being handled, particularly in their dry state during sample preparation or measurement.

Do not smoke during measurement procedures, particularly where inflammable samples or dispersants are used or stored.

Do not eat during measurement procedures, particularly where poisonous samples or dispersants are used or stored.

Wear rubber protective gloves when handling poisonous materials, or those that cause skin infections or irritations.

Always label samples for analysis, particularly if they are handled by a number of staff or stored for long periods. Clearly mark any operator hazard and associated safety precautions that are required for the handling of dangerous materials.

Always adopt responsible procedures for the disposal of waste dispersant and sample mixtures. Many chemicals are forbidden by law to be disposed of 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.

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c) No delay or omission of either party in exercising any right hereunder shall impair such right or to be constructed as a waiver thereof.

d) This Agreement shall, in all respects, be construed as and operate as an English Contract, conform to English Law and be subject to the jurisdiction of the English Courts.

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b). The Company's warranty provided in this sub-paragraph extends for a period of 90 days from the date of the receipt of the equipment by the (original) buyer and this period is automatically extended to 1 year from that date provided that appropriate invoices are paid within 30 days after receipt of the equipment.

c) All representations, warranties, guarantees and conditions (other than as to title and those herein expressed or specifically referred to) and whether statutory or otherwise are hereby expressly excluded. Without prejudice to the generality of the foregoing and to the extent permissable by law, the Company will not be liable for consequential loss or damage however caused resulting directly or indirectly from the sale or supply of goods or services by the Company or for any loss or damage whatsoever arising from stoppage or breakdown of or damage to or caused directly or indirectly by any of the goods or services or in any other way for the performance of any of the goods or services. The purchaser shall indemnify the Company against all claims by third parties in connection with the goods.

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g) This warranty shall in all respects be construed and operate as an English contract, conform to English Law and be subject to the jurisdiction of the English Courts

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CHAPTER 1

Introduction

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The ZetaSizer 4 combines the advantages of a cross beam laser anemometer, for optimum electrophoresis, and a parallel beam along the axis of the cell for optimum sizing, particularly of larger particles. The multi-angle positioning of the receiver, in conjunction with the parallel and cross beam modes, gives the instrument considerable flexibility for measurements over a large range of particle sizes. Electrophoresis, using reference beam mode, is also available for high resolution of samples containing mixtures of mobilities.

This chapter first explains, in a simple way, the principles of the particle sizing and electrophoresis measurements carried out by the ZetaSizer 4. The equipment is then described in sufficient detail to understand the way in which it works.

1.1 Principles of Operation

The following paragraphs give a brief outline of the principles of operation for particle size and electrophoresis measurement. A more mathematical treatment of the theory underlying the operation of the instrument is in the "Size and Zeta Potential Appreciation" manuals.

1.1.1 Particle Size Measurement

The ZetaSizer 4 can measure the size of particles with diameters in the range from 5 to 5000nm, using the method of photon correlation spectroscopy (PCS). This technique is also referred to by a number of other names, such as quasi-elastic light scattering (QELS) or dynamic light scattering. Particles in this size range are often called colloids. One of their characteristics is that they are in constant random thermal, or Brownian, motion. This motion causes the intensity of light scattered from the particles to vary with time. Large particles move more slowly than small particles, so that the rate of fluctuation of the light scattered from them is also slower. Photon correlation spectroscopy uses the rate of change of these light fluctuations to determine the size distribution of the particles scattering light.

The ZetaSizer 4 is normally delivered with a correlator incorporating 64 channels, although 128 and 256 channel correlators can also be used. Each channel measures changes in light fluctuation over one particular time span (called the sample time or delay time). The correlator is digital, that is, it works by counting photons rather than measuring light intensities. For very short delay times, the change in light level is small, as the particles have not had time to move very far. In statistical terms, it can be said that the positions of the particles over short times are highly correlated. For very long delay times, the positions of the particles will be completely random when compared with their initial positions. Statistically speaking, the positions of the particles for long delay times are not correlated at all.

In the simplest case, all 64 correlator channels are arranged to look at successively larger time spans, for instance if channel one has a delay time of 1 µs, then channel 64 will have a 64 µs delay (this is called serial mode). For particles of a single size in Brownian motion, a plot of counts per correlator channel against delay time produces a single exponentially decreasing function. If the particles are small, then this function decreases quickly, whereas if they are large the function decreases more slowly. This plot is called the autocorrelation function. It can be seen in the live display which is shown during size measurements. Each channel of the correlator works by multiplying together light intensities separated by its delay time. Correlation measurements are started at delay times which are short (100ns or so) compared with the fluctuations, so that the product of intensities at short delays is high i.e. the degree of correlation is high. At long delay times, when there is no correlation, the product of the two intensities will be equal to the square of the average light intensity. This can be seen as the long flat baseline to the right of the live display during size measurements. These channels have long delay times.

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It is difficult to carry out accurate size distribution measurements using "serial" mode. This is due to the fact that each particle size produces an exponentially decaying correlation function. If the fundamental delay time is optimised for small particles, i.e. it is short, then data will be missed for large particles, whose correlation function decays slowly, and vice versa. This problem is overcome by using "parallel" mode, the Malvern correlator's variable time expansion (VTE). A 64 channel correlator is divided into four 16 channel sub-correlators, each of which has a fundamental delay time which is a geometric factor (called the dilation) greater than the previous one. The dilation is usually set to 4. For example, if the first sub-correlator has delay times from 1 to 16μs, the second will have times from 4 to 64μs, the third from 16 to 256μs and the fourth from 64 to 1024μs. Data gathered using parallel mode contains both information about small particles in the first block of delay times, and very large particles in the last block. When particle sizing data is being carried out, the live display shows the correlation functions of the third and fourth sub-correlators building up.

Analysis of the auto-correlation function obtained over a period of time, a sufficient number of data points, enables the translational diffusion coefficient of particles undergoing Brownian motion to be calculated. From the diffusion coefficient, together with the temperature and viscosity of the suspending liquid, the particle size can be calculated. The particle size measured is the hydrodynamic diameter.

1.1.2 Electrophoresis Measurement

Microelectrophoresis, called simply electrophoresis from now on, is the measurement of the movement of colloidal particles when they are placed in an electric field. The measurement can be used to determine the sign of the charges on the particles and also their electrophoretic mobility, which is related to the surface charge and zeta potential.

Traditionally, electrophoresis is a laborious measurement. The sample is highly diluted, so that individual particles can be observed using a microscope. A voltage is placed across the cell and the direction and velocity of the particles is observed. The velocity is measured by timing individual particles between marks on a grid placed in the microscope eyepiece. To get good statistics many particles need to be measured, and there is always a tendency to choose the brighter particles. A single measurement takes at least 30 minutes, and possibly considerably longer.

The experiment is complicated by the fact that the applied voltage causes the liquid in the cell to move. This movement, called electro-osmosis, occurs because the glass walls of the cell are negatively charged. In fact, there is only one particular point between the cell wall and the centre of the cell, the stationary layer, where the particles move with a velocity which is due solely to their own charge. To get a true result, the microscope must be focused in this layer and the particles measured must be in focus.

Because the measurement of each particle is slow, the sample must be subjected to DC voltages for a long time. If the sample is highly conducting, i.e. if the salt concentration is high, then this will cause heating in the sample. The voltages that can be applied are limited in these cases, so the measurement of the mobility is inaccurate. Applying DC voltages for a long time also leads to problems of electrode polarisation, so that the electrode voltages become asymmetrical and applying the same voltage in both directions results in different measured voltages and errors in the results.

When the mobility is very low, accurate measurements are difficult, as the particles move randomly under Brownian motion and the effect of the applied field is hard to measure accurately.

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The ZetaSizer 4 incorporates technology which solves these problems which traditionally beset electrophoresis.

Malvern has sold instruments for the accurate measurement of particle velocities for a long time. They are called laser anemometers or velocimeters. These have been used extensively for measuring air velocities, dust in the air is used as a marker for the air flow. The ZetaSizer 4 uses exactly the same technology to measure the velocities of particles during electrophoresis experiments. Laser velocimetry is very rapid, the ZetaSizer 4 can carry out a complete electrophoresis experiment in 20 seconds or less.

As the velocity must be measured in the stationary layer, a small volume is defined by the crossing point of two laser beams. Where the two beams cross they produce interference fringes. Particles inside the scattering volume interact with these fringes to produce scattered light which oscillates in time in a way which depends on the speed of the particles. The light scattered by the particles is collected by the photomultiplier. The number of particles in the scattering volume is large, so that the statistics of the measurement are very good, compared with the traditional instruments.

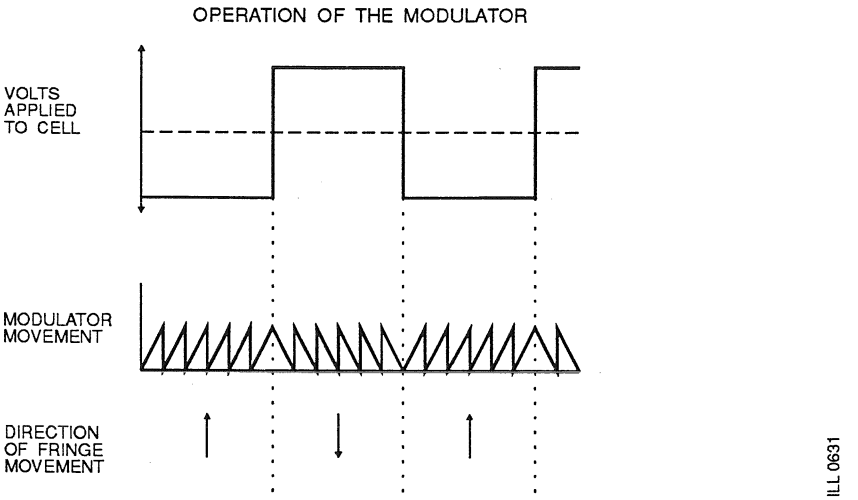


Fig. 1.1 Functioning of the Modulator

The problem of accurately measuring low particle velocities (low mobilities) has been overcome by oscillating (modulating) one of the laser beams. This is done by reflecting one of the beams off a mirror which is attached to a piezo-electric crystal. A varying voltage with a sawtooth waveform is applied to the crystal causing it to move. The sawtooth has a comparatively slow rise and the fastest possible drop, so that the effect is to move the interference fringes across the cell in one direction only. The frequency of the sawtooth wave, the modulation frequency, is between 100 and 1000Hz. The modulation frequency is always present in the signal, so that if the particles have no net velocity, the scattered light collected by the photomultiplier will have exactly that frequency. In this way, particles with low or zero mobility can be accurately measured.

If the particles move, then the collected signal will be Doppler shifted to higher or lower frequencies depending on whether they are positively or negatively charged. In this way, the sign of the charge on the particles can be determined. The measured autocorrelation function is first converted, using a Fourier Transform, into a frequency spectrum. The frequencies are then converted successively to velocities, electrophoretic mobilities and finally zeta potentials.

The problem of electrode polarisation is overcome by reversing the direction of the applied field at about 2 Hz. This is possible because the measurement of particle velocity from many field reversals can be added to obtain a good signal to noise ratio. In order to do this, the direction in

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which the fringes are moved by the modulating frequency must be reversed whenever the direction of the applied voltage is reversed. To do this the phase of the modulator is reversed, i.e. the direction in which the fringes move up the cell is reversed.

The problem of sample heating has been overcome in the ZetaSizer 4 by allowing the voltage to be switched off for a short time between each cycle, if the current flowing in the cell is too high. This is called "duty-cycling". Switching off the applied voltage allows the heat in the sample to dissipate. Obviously data must not be collected during the time that the voltage is switched off. The strobe cable between the optical unit and the correlator is used to send a signal which stops the transmission of data when no voltage is being applied to the cell. When duty-cycling is working, the normally smooth movement of the live display will become jerky, as data collection is switched on and off.

When high resolution electrophoresis measurements are necessary, the ZetaSizer optics can be realigned to use "reference beam mode". In this mode, light coming directly from the laser is mixed with light scattered by the sample. Using this method, particles with several different electrophoretic mobilities can be resolved into separate peaks.

1.2 System Components

The following paragraphs describe briefly the equipment which makes up the ZetaSizer 4 system. The setting-up and connection of these units is described in chapter 2.

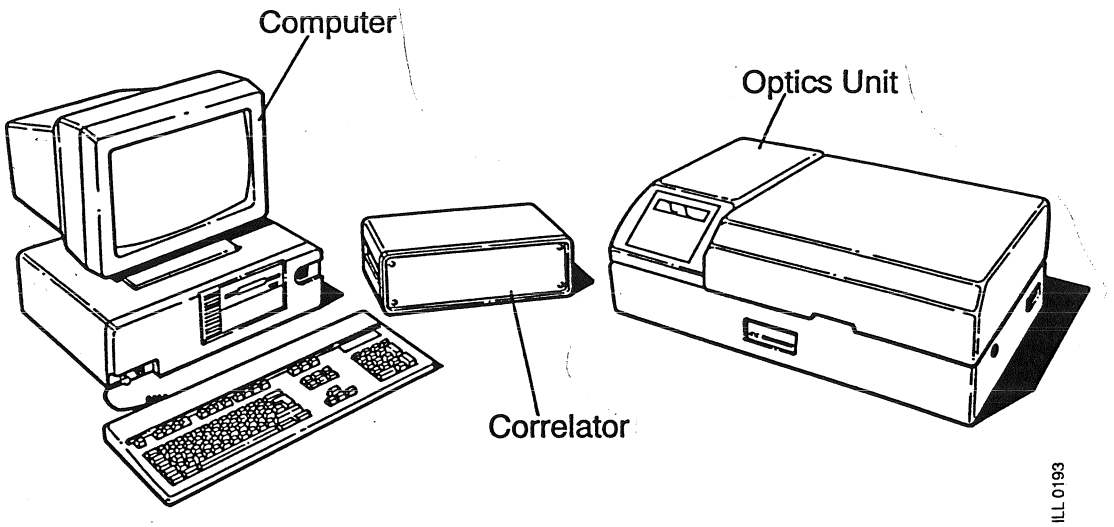


Fig. 1.2 ZetaSizer 4 System Components

A ZetaSizer 4 system comprises an optics unit, a digital correlator, and a personal computer that controls the whole system. The computer may also be connected to additional external peripherals, such as a printer. Each of the units is described in some detail below. Fig 1.2 is an overall view of the system and Fig 1.3 is a block diagram.

1.2.1 The optics unit

The optics unit contains a 5 mW Helium Neon laser that illuminates a sample of particles as a dilute suspension in a suitable liquid. "Dilute" means that the bulk of the incident light passes through the sample, the proportion that is scattered or absorbed being small. The sample is

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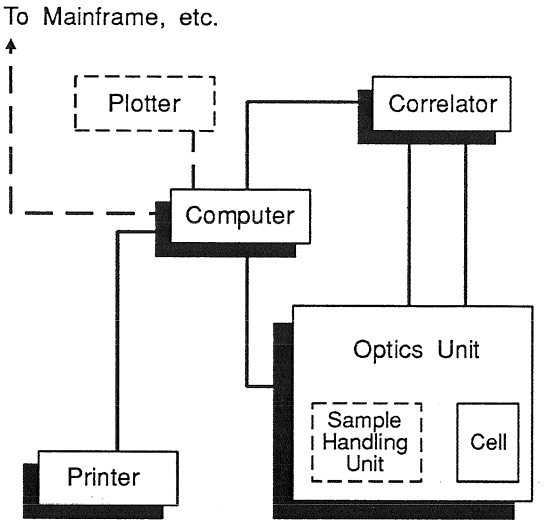


Fig. 1.3 Block Diagram

placed in one of a range of cells, described below, some intended solely for electrophoresis measurements, one solely for particle size analysis and one for either type of measurement. All are temperature controlled (to 0.1°C nominally), since both types of measurement require the temperature to be stable and known. The temperature is controlled by a "Joule-Peltier" device and a precision temperature sensor in the cell table. The former is a solid state "heat-pump" that can both heat and cool material in contact with it: the waste heat is dumped into the table mounting plate which can be circulated with cooling (or heating) liquid to improve the temperature regulation and to extend the range of temperatures accessible. A separate temperature sensor is placed

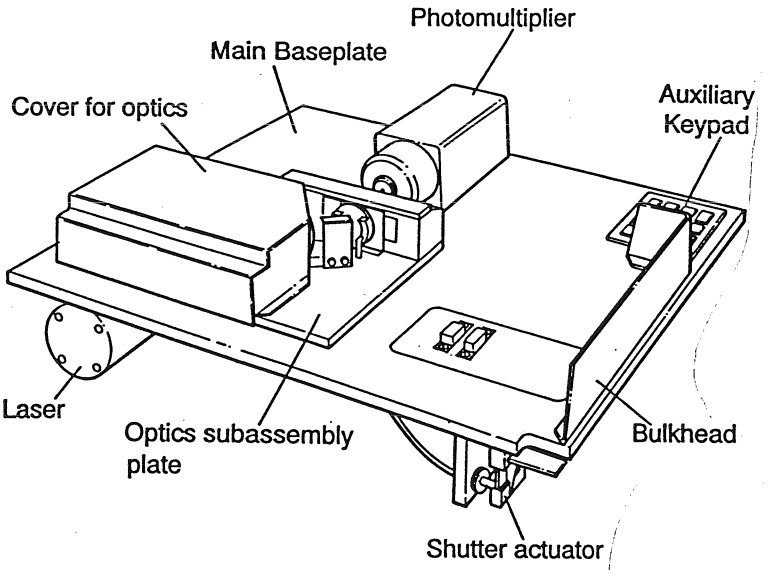


Fig. 1.4 Overview of the Optics

in the cell to measure the sample temperature more accurately

The cell table is automatically positioned by a stepping motor to a predetermined position for each type of cell.

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Scattered light from the sample is collected by a "photon-counting" photomultiplier with a minimum response time of 50 nanoseconds. The scattered light is picked up from a defined "probe volume" over a narrow range of angles defined by an aperture of either 200 or 400µm diameter, at the front of the photo-multiplier. Changing the size of the aperture is easily done by the user.

The photomultiplier angle is selected by a stepping motor drive, as is the position of its axis of rotation. The latter movement allows the position of the photomultiplier focus to be automatically selected for the type of cell to be used, at the scattering angle required.

The type of cell fitted is automatically sensed by the optical unit. In certain cells, the proportion of the laser light passing through the sample unaffected is measured by a laser monitor diode.

The scattering angle is defined not only by the position of the detector, but also by a laser beam selection system. For electrophoresis measurements the scattering angle is chosen automatically, but for sizing, the user can choose a range of scattering angles. The Malvern multi-angle analysis method, (available separately), combines data taken at several angles to maximise the information that can be extracted from the data.

The signal from the photomultiplier is passed to a digital correlator that is used to measure both the intensity (counts/second), and the autocorrelation function of the scattered light. This function can then be passed to the computer for further analysis to extract the spectrum of particle motion. The motion can be either random diffusion leading to a size determination, or forced motion in an electric field (electrophoresis) leading to a mobility and then a zeta potential. In the latter measurement the sample is illuminated by two laser beams, scattered light from both being detected simultaneously. During this measurement a voltage (up to 400V) is applied to the cell. The voltage and the current flowing are sensed by the optical unit. If the current demanded is too high the duty cycle (proportion of time for which the voltage is applied) can be reduced, as can the demanded voltage itself. Under the Easy Mode of operation which is described fully in chapter 3, the duty cycle is controlled automatically if the current sensed is excessive.

The various functions outlined above (cell positioning, temperature control, voltage set etc.) are controlled by a microcomputer built into the optics unit. The program which it uses is permanently stored in the optics unit. This unit "talks" to the data processing computer via an RS232C communications link. Some functions of the microcomputer in the optics unit can be controlled using the external keypad. An extension to this keypad inside the unit is used during optical alignment to set and record the positions of the cell walls. These are subsequently used in automatic operation.

The optics unit has a small visual display. This is used to show what operation the unit is currently carrying out, such as "SET RIGHT CELL WALL" or "SIZING". The display also shows the result of the last experiment, either mean size and polydispersity, or zeta potential and mobility. Finally, values set by the optics unit, and measured during the last experiment can be checked, by pressing the **STEP** + and **STEP** - keys. Parameters such as the cell type, pH and the cell temperature can be easily verified in this way.

1.2.2 The standard electrophoresis cell - ZET5104

The ZET5104 is the normal cell for electrophoresis, it can also be used for particle sizing at angles close to 90°. This cell uses a 4mm diameter quartz capillary. The platinum electrodes are in separate holders which are mounted so as to be coaxial with the measurement capillary and in contact with the sample.

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When a voltage is applied across the electrodes, the liquid flows in the cell, due to the electro-osmotic effect. The flow occurs in one direction close to the wall and in the opposite direction in the centre of the tube. In order to measure the true mobility of the particles, measurements must be carried out in the stationary layer, which lies where the two directions of flow exactly balance out. This is at a point exactly 14.62% of the radius of the cell from the cell wall. However, it is not necessary to position the cell, as the ZetaSizer 4 does this automatically. When the cell is aligned initially, the optics unit stores the position of the two cell walls. Thereafter, whenever an electrophoresis experiment is requested, the position of the crossing point of the two laser beams is set to the correct point in the cell.

The sample handling unit can be used to flush and inject samples into the ZET5104 cell.

1.2.3 The small bore cell - ZET5103

The ZET5103 is specially adapted for measuring biological samples suspended in physiological saline, or other highly conducting solutions. The ZET5104 cell cannot easily be used for such samples, as the high current flowing in the cell causes the sample to heat. The ZET5103 cell overcomes this problem by using a 0.7mm diameter glass tube, so that for a given voltage gradient, the current flowing is about 30 times lower, (the current is proportional to the cross-sectional area of the tube). The tube is also about half the length of the tube in the ZET5104, so that for a given applied voltage, the voltage gradient is doubled. In this way, the resolution is improved. In addition, the walls of the ZET5103 are coated with a neutral polymer, so that the liquid does not flow in the tube when a voltage is applied, as is the case with the ZET5104. Measurements of the true mobility can now be made anywhere across the tube. The measurement is actually made at the centre of the tube, to avoid any light scattered from the walls of the tube affecting the results.

Because the tube of the ZET5103 cell is so narrow, optical alignment is more critical than with the ZET5104, also the presence of the polymer layer increases scatter from the cell walls. The polymer layer is thick enough to prevent liquid flow in the cell at high salt concentrations, but does not allow measurements of samples in low salt concentrations, specifically for samples made up in distilled water.

The polymer which coats the wall has been shown to give reliable results when used with biological samples, however the use of the ZET5103 cell with other types of sample should be treated with caution. It is possible that the polymer may contaminate the particle surface, which will make the mobility measurements unreliable.

The sample handling unit is not recommended for use with the ZET5103.

1.2.4 The sizing cell - ZET5110

The ZET5110 cell is optimised for particle sizing. Unlike the ZET5103 and ZET5104 cells, the sample is placed in a vertical cylinder, so that no geometric corrections are needed to determine the true scattering angle. Sizing can be carried out at any angle between 0 and 135°. As for the ZET5103 and ZET5104 cells, the sample is surrounded by an index matching bath which also serves to transfer heat from the Peltier heating element to the sample.

A simple manual mode command allows scanning of the scattering angle, so that plots of light intensity against angle can be made. These are useful for choosing the best angle for sizing, sometimes sharp drops in intensity occur at certain angles. These minima should be avoided, as the statistics of the measurement are worse at such angles. The ZET5110 is the preferred

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cell for gathering data to be used in the Malvern multi-angle analysis programme, (available separately). This programme gives high resolution of complex size distributions by combining data gathered at different scattering angles

At present, the sample handling unit cannot be used with the ZET5110 cell.

1.2.5 The low conductivity cell - ZET5126

When particles are suspended in liquids of low conductivity, very large field gradients, of the order of 100's of volts per cm., must be applied to the cell, in order to obtain accurate electrophoresis data. Malvern has designed, and patented, a cell for making measurements in such liquids. In the ZET5126 cell the electrodes are only 1mm apart, so that very high voltage gradients can be obtained, without needing to apply higher voltages than for the ZET5103 and ZET5104 cells. The geometry of the ZET5126 cell avoids the problems of electro-osmosis.

The sample handling unit cannot be used with the ZET5126 cell.

1.2.6 The sample handling unit ZET5115

The sample handling unit is an optional addition to the ZetaSizer 4 which completely automates the injection of samples into the ZET5104 cell. The unit consists of a stepper motor driven syringe and two pinch valves which control the flow of liquid. The ZET5115 is supplied fitted with a $10cm^3$ plastic syringe which is easily replaced and also with a $10cm^3$ glass syringe which may be replaced by the user if required. Tubes from the front of the instrument are routed to brackets containing sample and electrolyte or water.

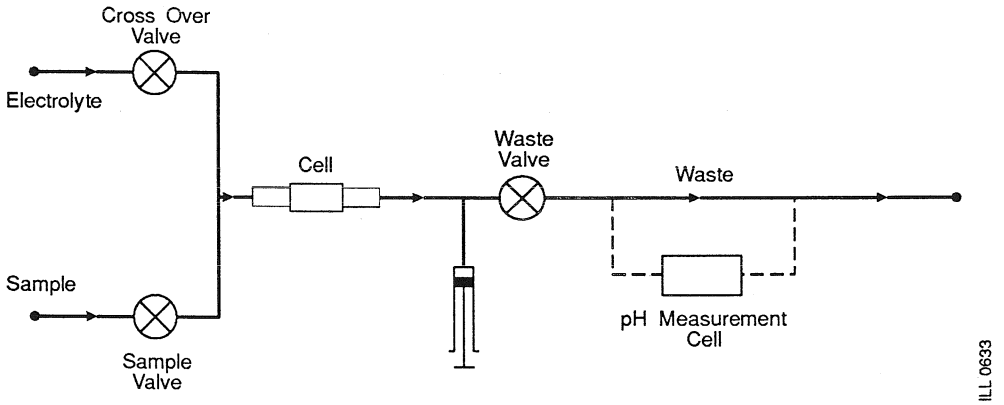


Fig. 1.5 Sample Handling Unit

The unit is controlled either from a programme or using the keypad on the ZetaSizer 4.

1.2.7 The pH measurement cell

The sample may be routed via the pH measurement cell mounted in front of the cell connector. A standard combination electrode is supplied with the system which should be carefully pushed into the aperture in the top of the pH measurement cell. The outlet from the electrophoresis cell may be routed via the pH measurement cell to enable the sample pH to be measured in line. The pH reading is relayed to the computer at the end of every measurement.

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1.2.8 The Correlator

The correlator used is the "multi-8" multibit with 72 data channels and 7 monitor channels with Variable Time Expansion (VTE). VTE enables the correlator to be set up as four subcorrelators, each analysing the same data but at different sampling times. Sample times can range from 50ns. to 1 second. This extends the dynamic range of the instrument so that the 72 channel correlator, set up as four 16 channel sub-systems, has a range from the smallest to the largest sample time of 50,000:1.

An expandable version of the 7032 correlator series is available for this system enabling the unit to be upgraded to 136 or 264 channels (plus 11 and 19 monitor channels respectively). This in effect extends the range of the equipment for polydispersity analysis to some 106:1 (one million to one)

Switching between linear (serial) mode and VTE (parallel) mode operation is achieved by a simple software command. A Malvern interface is fitted to the computer, with all the normal computer facilities retained. The multi-8 correlator operates totally in real time, ensuring the maximum data precision in the shortest possible time.

1.2.9 The Computer System

The NEC 386/SX normally supplied for this system is a 16 bit IBM compatible with 4MB RAM and a 45MB hard disk. A 3 $^1/_2$" 1.4MB floppy disk drive is also incorporated to allow for the storage of data on floppy disks.

If other computers are to be used, then as a guide, a 40 MB hard disk with a 720kB floppy disc will be required. Also it is recommended that your system be equipped with 4MB RAM to maintain acceptable performance.

The computer can be supplied in a choice of configurations. A second floppy disk drive can be added. A 20MB tape streaming facility is also available to enable the complete contents of the hard disk to be "backed-up". This manual covers the use of the basic model.

The graphics are compatible with IBM's VGA standard. If a monochrome monitor is fitted, the software will generate a monochrome greyscale, allowing some contrast in overlaid graphics. The user can override this effect using a single command in the Malvern software to obtain simple monochrome graphics.

The keyboard is a standard 102 key IBM-AT layout with 12 function keys. A printer compatible with the IBM Graphics Printer is also required to obtain hard copy of results and data.

The Malvern ZetaSizer 4 is designed to run under Microsoft Windows ™ version 3 or later. The code is written in C++ and compiled. Besides facilities normal to Windows, it includes a user programming facility based on a BASIC like interpreter so that custom displays, printouts and measurement sequences can be set up.

1.2.10 Interfacing the ZetaSizer 4 to a titrator

The ZetaSizer 4 software is able to control an Autosampler via a second RS232 serial interface to enable measurements such as mobility vs pH, or a series of samples, to be automated. A suitable autosampler is available from Malvern as an option.

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1.3 Environmental Conditions

The ZetaSizer 4 needs to be mounted on a solid, vibration-free base for optimum operation. The selection and preparation of a suitable site prior to unpacking will ensure trouble free operation and will be reflected in the quality of the results. The equipment is designed and intended for use within a normal laboratory environment with the following conditions recommended;

Humidity	<70%.
Temperature	between 10° and 30° C.
Power requirements	110/240V AC; 50/60 Hz; 500VA.

1.4 Technical Specifications

1.4.1 General

Model	ZetaSizer 4 Particle Electrophoresis and Multi Angle Particle Size Analyser.
Laser	5mW He-Ne; 633nm.
Signal Processor	Malvern 7032N/72C Multi-8 bit correlator with 72 channels, Variable Time Expansion (VTE) and 16 bit IBM compatible computer with 4MB RAM, 45MB hard disk and single flexible disk drive.
Data	Graphical and tabular formats for particle size, electrophoretic mobility and zeta potential distributions; graphical plots of any two parameters e.g. mobility vs. pH, particle size vs. zeta potential.
Communica-tions	Full bi-directional RS232C standard.
Printer	Compatible with IBM Graphics Printer.
Plotter	Optional Extra Compatible with Hewlett Packard.
Power requirements	110/240 V; 50/60 Hertz.

1.4.2 Particle size analysis

Size range	5nm - 5µm approx.
Angular range	0 - 140° overall (for ZET 5104 or ZET 5110 cells).
Cells	Standard electrophoresis cells (see below) or ZET 5110 cylindrical particle sizing cell.
Operating modes	Focused beam; parallel beam.

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1.4.3 Electrophoresis

Mobility range	–10 to +10μms-1V/cm-1, typical, no maximum exists.
Operating modes	Crossed beam; reference beam.
Applied voltage	400V max.
Field strength (max)	160 V/cm (ZET5103 cell). 80 V/cm (ZET5104). 400 V/cm (ZET5126 cell). Duty cycle operation limits heating in high conductivity media.
Cells	ZET5103 Small bore (0.7mm diameter). ZET5104 Large bore (4mm diameter). ZET5126 Low conductivity cell. ZET5110 Particle sizing cell.
Automation	Automatic setting and sensing of applied voltage; automatic location of stationary level or other selected position in cell.
Optical modulator	0, 125, 250, 500 or 1000Hz frequency.

1.4.4 Other parameters

Manual syringe system	Standard
Sample handling system	Optional: Comprises: Automatic precision metering system; cell can be flushed and sample injected automatically; Can be interfaced to proprietary autosamplers. Measurement of conductivity, pH (and certain other specific ions).

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CHAPTER 2

Installation and System Check-out

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2.1 Unpacking the optics unit

To protect the mechanisms of the optics unit during transport, various parts of the instrument are held in position by transit bolts and other means. The transit arrangements **must** be removed **before** applying power to the unit. Failure to remove **all** transit arrangements could cause damage to the unit.

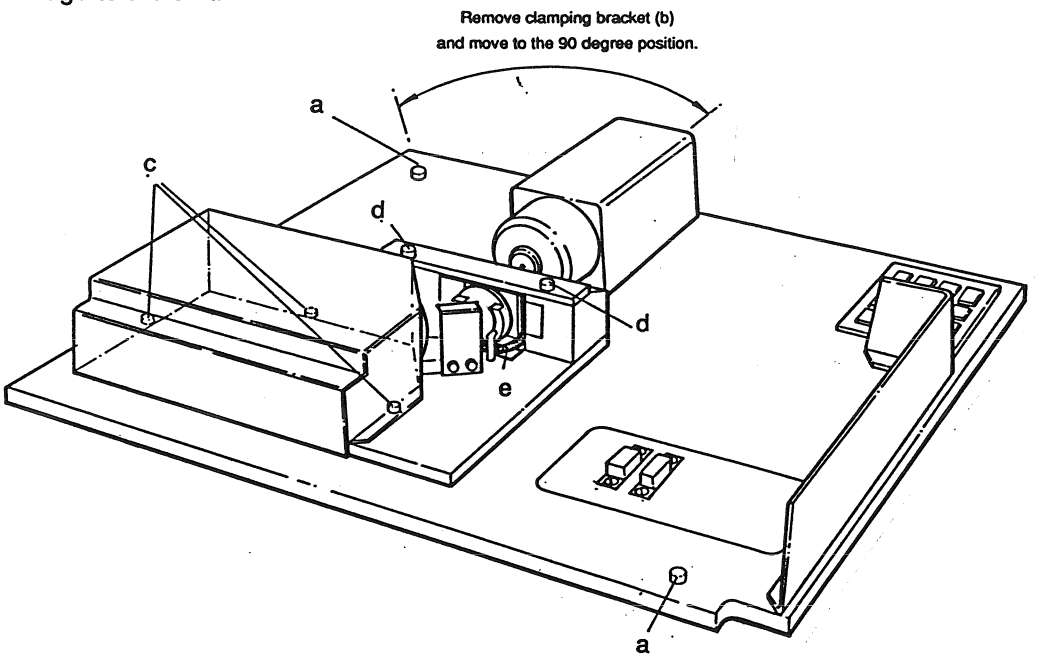


Fig. 2.1 Transit Arrangements

Fig 2.1 shows the position of the transit arrangements. The two locking screws (a) should be removed and the photomultiplier arm should be pulled to its 90° position. This will allow access to the 3mm hex headed screws holding the clamp bracket (b) to the rear of the photomultiplier arm. Remove the clamp bracket.

Remove the optics cover by unfastening the three retaining screws (c) and remove the tape holding the beam select mirror in position.

Replace the optics cover.

Remove the attenuator motor assembly by unscrewing the two screws (d). N.B. It is not necessary to disconnect the attenuator motor connector. Remove the rubber ring (e) and replace the attenuator motor assembly.

The ZetaSizer 4 optics unit is now ready for use. However before a measurement can be made a cell must be fitted. The procedure for this is described in section 2.9.

2.2 Check list of items delivered

On completion of unpacking and prior to connecting up, a complete check of all items delivered against the packing list should be made. Notify Malvern of any discrepancies, in writing, immediately. All items are listed in Table 2.1. The item numbers provide a useful method of referring to items in any communication with Malvern, but have no other significance.

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Table 2.1 Major Items

Item No	Description	Qty	Details
1	NEC 386/SX.	1	Complete with VDU and keyboard.
2	Correlator 7032.	1	
3	Printer.	1	Dot matrix hard copy printer.
4	Optics unit.	1	See also Table 2.2 for ancillaries.
5	Large bore Cell.	1	ZET 5104 see also Table 2.3 for ancillaries.
6	Sizing Cell.	1	ZET 5110.
7	Electrophoresis Standard.	1	AZ55.
8	Pack of 100 Sample Cuvettes plus 10 lids.	1	PCS12.

See also Table 2.4 for Other Ancillaries:

Table 2.2 Optics Unit Ancillaries

Item No	Description	Qty	Details
1	Plastic Accessory Case.	1	
2	Eyepiece Assembly.	1	including clamping screw.
3	200 micron pin-hole.	1	
4	400 micron pin-hole.	1	
5	Wire Target.	1	
6	Allen Keys.	6	sizes 1.5, 2, 3, 4, 5 & 6mm.
7	Fuses.	2	dependent on model and voltage of country.
8	Alignment target.	1	
9	Hexagon drivers.	3	3mm, 2.5mm, 1.5mm.
10	Laser override tool.	1	
11	Screwdrivers.	2	Flathead, 6" & 4".
12	pH electrode.	1	
13	Lens tissues.	1	Pack of.
14	Silicone tube.	1	2 metre length.
15	Heat sink compound.	1	

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Table 2.3 ZET 5104 - Large Bore Cell Ancillaries

Item No	Description	Qty	Details
1	Plastic Accessory Case	1	
2	Specimen Tube	2	large bore
3	Plastic Syringe	2	10ml SYR10
4	O Ring Type 010	4	ORV010
5	O Ring Type 017	4	ORV0017
6	O Ring Type 018	8	ORV0018
7	O Ring Type 019	4	ORV0019
8	End fittings	Supplied with tube assy.	
9	End plugs		
10	'T' pieces		

Table 2.4 Other Ancillaries

Item No	Description	Qty	Details
1	Interface Card	1	7032-3010 (installed in computer)
2	ZetaSizer 4 Software	1	On $3^1/_2$" discs
3	Blank Discs	2	$3^1/_2$" 720kB unformatted discs
4	Microsoft Windows Software	1	
5	Processor Mains Lead	1	
6	Correlator Mains Lead	1	
7	Ribbon Cable	1	37 way 2 metres long
8	Co-axial Lead	2	BNC connectors 2 metres long
9	RS232C Lead	1	
10	AZ5110 accessory case	1	
11	Alignment Tool	1	AZ60A
12	Round cell	2	RR84
13	Round cell stopper	2	S667
14	Pack 100 round cells	1	PCS12

2.3 Connecting the units

After checking that all the parts of the ZetaSizer 4 have been correctly delivered, the different units can be connected together. A total of four power sockets will be needed, for the optics unit, correlator, computer and printer. The most convenient way to position the units is to have the computer in the centre, with the optics unit to its right and the correlator to its left. The printer can be conveniently placed on top of the correlator. Figure 2.2 shows a schematic view of all the units of the ZetaSizer 4, with all the connecting cables fitted. Apart from the four power cables, eight cables need to be fitted in all:

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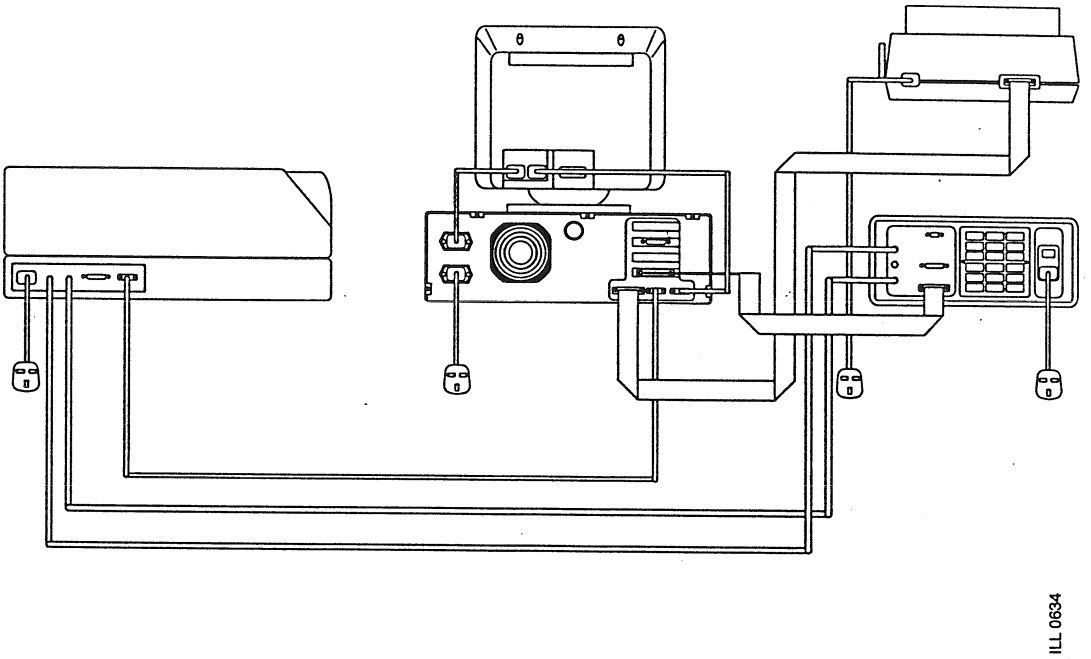


Fig. 2.2 ZetaSizer Interconnection Diagram

- The correlator cable connecting the correlator to the computer. This is a 37 way cable, which is blue. One end fits into the back of the correlator into the socket marked "COMPUTER" and the other end into a vertical socket at the back of the computer.

- The signal cable connecting the optics unit to the correlator. This is a thin cable with a cylindrical plug. One end fits into the plug marked "SIGNAL" on the side of the optics unit, and the other into the plug marked "SIGNAL A" at the back of the correlator.

- The strobe cable. This is identical to the signal cable. It connects the socket marked "STROBE" on the side of the optics unit to the socket marked "STROBE" on the back of the correlator.

- The serial cable. This is a round cable with 25 pins in one plug and 9 pins in the other. It is connected between the socket on the side of the optics unit marked "RS232" and the serial socket at the back of the computer which is also marked "RS 232".

- The video cable. Is the cable attached to the monitor with a rectangular plug is plugged into the back of the computer. The plug on the computer is at the bottom right of the rear panel.

- The monitor power cable. The cable attached to the monitor with a three pin plug is plugged into the back of the computer, at the top left, when facing the back.

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- The printer cable. This cable is rectangular, slightly wider than the serial cable, but one plug has a central core instead of pins. The plug with the central core plugs into the back of the printer, and the other into the socket at the back of the computer that is labelled with a printer symbol.

- The keyboard cable. This is a coiled cable attached to the keyboard. The plug fits into the front of the computer

2.4 Checking out the Computer

The computer is usually delivered with all the necessary Windows and Malvern system files already loaded onto the hard disk and ready for use. If this is not the case, see the installation section in the PCS Software Reference manual. Before using the software, be sure that you have read and understood the Software Licence agreement, which can be found at the front of this manual. After the computer has been switched on, it runs a programme that checks various hardware connections, memory (RAM) and disk drives (this process is sometimes called "Booting-up" the computer). During this procedure, a list of diagnostic messages are displayed which should indicate that all the tests have been passed.

A Malvern group window will be displayed. Selecting the ZetaSizer icon with the mouse and clicking with the left button will start the program.



2.5 Checking Out the Printer

The printer supplied is compatible with the IBM Graphics Printer. It is fitted with a parallel (or Centronics standard) interface. The printer manual supplied should be consulted for the setting up procedures. Before switching on the printer ensure that it is set to the required voltage of the country. The printer has a self-test programme that can be carried out independently of the computer.

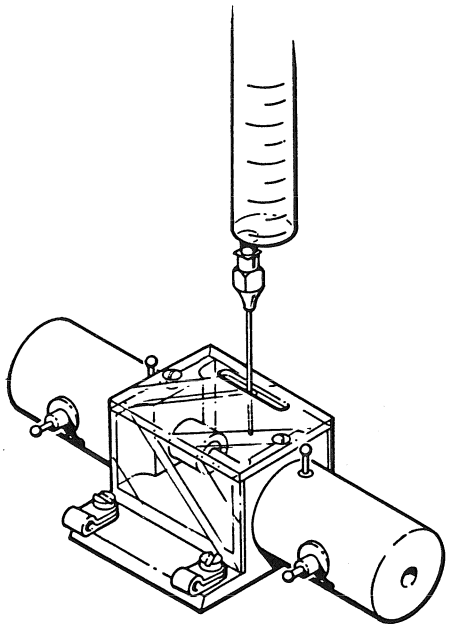
Set any options the printer prompts for, either through a menu or by switch settings, so that the paper length corresponds to the paper intended for use. The ZetaSizer 4 software controls the print size and line spacing of the printer and gives the best results when the paper size is 9.5" x 11" (241mm x 279.5mm). To ensure printing skips over the page perforations always set "Top-of-form" when turning on the computer system.

2.6 Filling the baths of ZET 5103 and ZET 5104 cells

Before fitting the cells into the optics unit, the index matching bath must be filled. This procedure must be followed whenever the glass capillary in the ZET5104 cell is removed for cleaning. (see chapter 4 for more details)

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Fill the bath with filtered deionised water or alcohol using a syringe and hypodermic needle. If a source of well filtered water is not available ordinary deionised water may be used if a syringe filter (preferably 0.2µm pore size) is inserted between the syringe and the needle. (see fig 2.3) . If the water is de-gassed then the formation of bubbles in the index matching bath will be minimised.



ILL 0671

Fig. 2.3 Filling the baths

2.7 Fitting the cells

Fitting any of the cells to the ZetaSizer 4 is very simple. Before fitting ensure that a thin layer of heat transfer compound is on the cell base. A tube is supplied in the instrument case. Slacken the locking screw on the cell table so that the clamping plate is loose. Slide the cell onto the cell table so that the cell base is in close contact with the cell table and the bevel on the edge of the cell passes under the clamping plate. In the case of the ZET 5103 and ZET 5104 adjust the position of the cell so that it is approximately central on the cell table. In the case of the ZET 5110 and ZET 5126 push the cell against its end stop. Tighten the locking screw on the cell table. With the exception of the ZET 5110 plug the cell connector into the 9 pin "D" type connector just to the rear of the pH electrode. Place the cell temperature sensor in the position in the cell.

Press 07 on the keypad inside the optics unit and the current cell type will be displayed.

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CHAPTER 3

Normal Operation

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3.1 The optics unit

When the optics unit is switched on the message "INITIALISING SYSTEM VERSION *Version number*" is displayed. The unit then references all the stepper motors, and checks the photomultiplier and modulator.

3.1.1 The optics unit display

The optics unit display can be used to monitor the current state of the ZetaSizer. At the end of the calculation following an experiment, a summary of the results is downloaded so that a quick check can be made. Ten experimental parameters can also be checked by pressing the **STEP +** and **STEP –** keys below the display.

The parameters are:

- **DELTA V** - For electrophoresis shows the difference in applied voltage between the voltages sensed in each direction. **DELTA V** must be low if good resolution is to be obtained.
- **DUTY CYCLE** - For electrophoresis, shows the percentage of the time that voltage is being applied to the cell. See the section on duty cycling later in this chapter.
- **LASER POWER** - In the ZET 5104 and ZET 5110 cells shows the percentage of the laser power which is not being scattered or absorbed by the sample.

  Note that the laser power monitor only works with the light baffle in the ZET 5110 cell removed. This can cause problems at angles other than 90°
- **ANGLE SET** - Shows the current scattering angle.
- **CELL TEMPERATURE** - Shows the current cell temperature.
- **CELL TYPE** - Shows the cell type currently stored in the optics unit memory. If incorrect, it can be reset using the sequence **0 7** on the internal keypad.
- **SAMPLE pH** - Shows the pH currently sensed by the pH probe.
- **SENSED VOLTAGE** - For electrophoresis in the electrophoresis cells, shows the voltage sensed by the electrodes close to the end of the capillary. See chapter 4 for more details on the construction of the cells.
- **APPLIED VOLTAGE** - For electrophoresis in the electrophoresis cells, shows the voltage applied across the electrodes in the electrode chambers.
- **CELL CURRENT** - For electrophoresis, shows the current flowing during the last experiment.

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3.1.2 Operation of the keypad and the external keys

Many of the operations of the ZetaSizer 4 can be controlled using the keypad just inside the cover of the optics unit and the external keys on the left of the optics unit. The functions of the keypad keys are listed inside the lid. In this section their use is discussed in more depth. Many of the operations involve running the laser with the lid open, these are maintenance procedures and should only be carried out by properly trained personnel.

The external keys below the visual display reproduce exactly the functions of the internal keypad keys 0-5. Their normal functions can be carried out with the lid shut. No special training is required for their use.

When any of the operations described below are being carried out, the computer will not be able to communicate with the optics unit. All program operations which do not need to talk to the optics will work as normal. It may be necessary to disconnect and reconnect the RS232 (under Setup-RS232) before carrying out some operations later. Communications between the computer and the optics unit can only be re-established when the unit is not busy. In this case either the message "WAITING", or the values of one of the experimental parameters, is displayed on the optics visual display.

3.1.3 Normal key functions

- **Keypad key "0" or external key "ALT" (Alternative).**
  This key does not have any function of its own, but when pressed before another key, the function listed on the right hand side of the list inside the lid of the optics will be performed. Do not press the second key until the message "ALT FUNCTION" appears on the visual display of the optics unit.

- **Keypad key "1" or external key "RUN".**
  Initiate measurement. Pressing this key starts an experiment using the parameters stored in the optics unit.

- **Keypad key "2" or external key "INJECT".**
  Inject sample. If a sample handling unit is fitted, it will inject 3ml of sample from the port at the front of the optics unit. This command is equivalent to the MEASURE-INJECT sample software command. If no sample handling unit is fitted, this command will time out after about 1 minute and the message "WAITING" will reappear. Subsequently, it will work exactly like **ALT INJECT** or **0 2**, (see below). See the section in this chapter on the sample handling unit for more details.

- **Keypad key "3" or external key "FLUSH".**
  Flush system. If fitted, the sample handling unit flushes 10ml of electrolyte through the cell. This command is equivalent to the software command MEASURE-FLUSH. If no sample handling unit is fitted, this command will time out, in the same way as **INJECT**. It will subsequently work in the same way as **ALT FLUSH** or **0 3**, which is explained below. See the section of this chapter on the sample handling unit.

- **Keypad key "4" or external key "STEP +"**
  Display select. Alters the parameter displayed on the optics visual display. The parameters are arranged in a circle, so that any parameter can be reached by pressing either **4** or **5** (**STEP +** or **STEP -**) a sufficient number

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of times.

These keys are also used, together with **5** and **STEP-**, to adjust the position of the cell, so that the cell walls are correctly memorised by the optics unit. This is a maintenance procedure, for more details see chapter 4.

- **Keypad key "5" or external key "STEP -"**
  Display select. Selects parameters for display in the opposite direction to **4** or **STEP +**. This command is also used to move the cell during optical alignment.

- **Keypad key "6"**
  Save array. Pressing this key after carrying out optical alignment saves the positions of the cell walls and other parameters, such as the voltage used during electrophoresis experiments. The use of this key is described in full in chapter 4 which deals with maintenance procedures.

- **Keypad key "A"**
  Switch on and off photomultiplier. Used in maintenance procedures.

- **Keypad key "C"**
  Start alignment procedure. The use of this key is fully described in chapter 4 under maintenance procedures.

- **Keypad key "D"**
  Move beam select mirror. The beam select mirror is moved to toggle between parallel beam and focused beam mode. Note that the cell and PM are not moved to the correct positions by using this key alone. **D** can be used to move from cross beam mode to parallel beam mode, using the key sequence **D E 8 E**. To move from parallel beam mode to cross beam mode press **D 0 4 4**.

- **Keypad key "E"**
  Reference motors. Of the four stepper motors controlling the optics unit three have sensors to check their zero points. Pressing **E** and then **7**,**8**, **9** and **A** will re-zero the PM angle, cell, PM axis and attenuator motors respectively. . After completion of the operation, **E** must be pressed again to bring back the message "WAITING" on the optics unit visual display.

- **Keypad key "F"**
  Stop measurement. This key is useful to stop experiments which have been started using keypad key **1** or the external key **RUN** when no length of experiment is defined. It is equivalent to the Measure-Stop zetasizer.

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3.1.4 Alternative key functions

After pressing either the keypad key **0** or the external key **ALT** some of the keys alter their function.

- **"0 2" or "ALT. INJECT"**
  Open valves for sample injection. If a sample handling unit is fitted this function can be used to carry out manual injection of a sample from the port on the front of the optics unit. The correct valves are opened to allow injection, but the syringe does not operate. To close the valves after injection, **2** or **INJECT** must be pressed again. This procedure is useful to dislodge bubbles from the system, as flowrates higher than those applied by the internal syringes can be obtained.

- **"0 3" or "ALT FLUSH"**
  Open valves for system flush. This allows manual flushing in the same way as **ALT INJECT** allows manual injection. To close the valves after flushing, **3** or **FLUSH** must be pressed again. See the section in this chapter on cell connections.

- **"0 4" or "ALT STEP -"**
  Move to the right cell wall. This command is used to check whether the optics unit has stored the correct position of the right cell wall in memory. This is a maintenance procedure, which is fully described in chapter 4. After checking, either the **4** or **STEP** - key must be pressed again to return to the message "WAITING". Note that the cell will not move back to the correct position for electrophoresis until the beginning of the next experiment. 0 4 can also be used to change from parallel beam to cross beam mode, during maintenance.

- **"0 5" or "ALT STEP +"**
  Move to the left hand cell wall. This command sequence acts exactly like **0 4** or **ALT STEP -**. After checking, press **5** or **STEP +** to return to the message "WAITING".

- **"0 7"**
  Check cell type. This command sequence is used after changing the type of cell fitted, so that the optics unit updates its stored data. The optics unit also automatically detects the type of cell fitted when it is switched on. The cell type currently stored can be checked using the optics visual display, by pressing the **STEP +** and **STEP -** keys until the cell type is displayed.

3.2 Choice of cell

The ZetaSizer 4 can be fitted with four different types of cell. Changing from one type to another is a very quick and simple operation. The optics unit automatically senses which type of cell is fitted and sets the correct parameters for measuring samples and calculating results. The four cells are:

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The ZET 5104 wide capillary cell.
The ZET 5103 narrow capillary cell.
The ZET 5110 sizing cell.
The ZET 5126 low conductivity cell.

The following list of questions to ask about the nature of the sample and the kind of measurement to be made should make clear the correct cell or cells for any application:

If electrophoresis measurements are required on samples suspended in liquids other than water, or some polar organic solvents, Then use the ZET 5126 cell.

If high resolution electrophoresis measurements are required on biological samples in physiological saline, Then use the ZET 5103 cell.

If size measurements are required at multiple angles, or at angles far from 90° , Then use the ZET 5110 cell.

If both sizing and electrophoresis measurements are required on the same sample, Then use the ZET 5104 cell.

The ZET 5104 cell is the general purpose cell. It should be used for all electrophoresis measurements, except when the sample is in highly saline aqueous solutions or is suspended in an organic liquid of low dielectric constant. It can also be used for many size measurement applications, if the size distribution is narrow, e.g. polymer latex standards, or if a detailed size distribution is not needed.

3.3 The sample handling unit

A schematic diagram of the unit is shown in figure 1.1. The syringe sucks sample or electrolyte straight from the ports at the front of the optics unit into the cell. In this way the dead volume is minimised and there is little chance of cross contamination between samples. Two commands are used for handling the liquids, **Inject** and **Flush**.

- **Inject** - is used to fill the cell with sample. The sample is placed in a container in front of the optics and a tube connected to the sample port dips into the container. When an injection starts, the sample valve opens, so that sample is sucked into the cell by the syringe. When the required volume has been sucked in, the sample valve closes and the waste valve opens. The syringe then empties to waste, ready for the next command.

- **Flush** - is used to rinse the cell with electrolyte between samples. The electrolyte is placed in a container into which a tube connected to the electrolyte port at the front of the optics is dipped. At the beginning of a flush, the crossover valve opens, but the sample valve remains closed. Electrolyte is drawn through the cell. When the requested volume has been drawn in, the crossover valve closes and the waste valve opens. The syringe is then emptied to waste.

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3.4 Cell connections for the ZET 5104 cell

In this section the connection of the sample handling system to the ZET 5104 cell is described. This system is used to fill the cell with sample and to flush it between samples.

3.4.1 Without the Sample Handling Unit

If the optional sample handling unit (SHU) has not been fitted, the tubing connecting the cell, and entry and exit ports is as shown in figure 3.1. Two tubes connect the cell to the base.

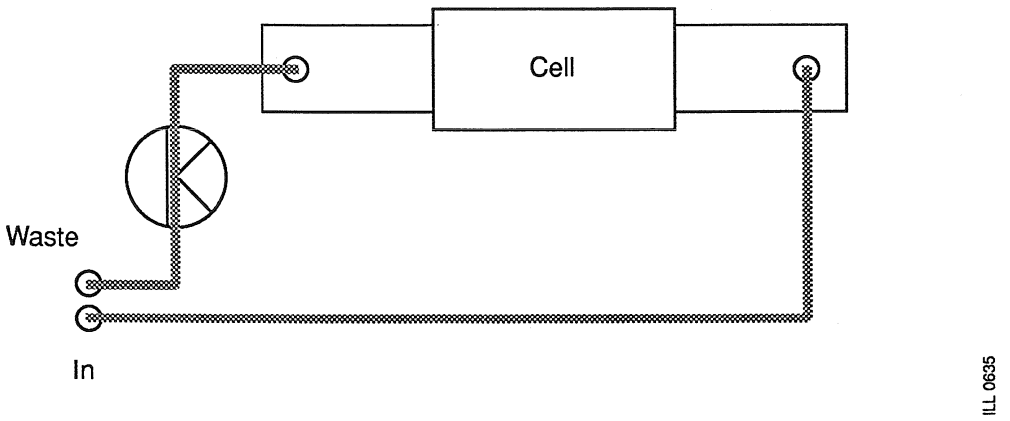


Fig. 3.1 ZET 5104 Cell Connections (without Sample Handling Unit)

- Connect a tube from the port marked "**IN**" in front of the cell base to the sample port at the far end of the top of the cell. Both ends of this tube will already have plugs attached to them. The tube is attached to the far end of the cell so that air bubbles are efficiently dislodged from the cell window where the laser enters.

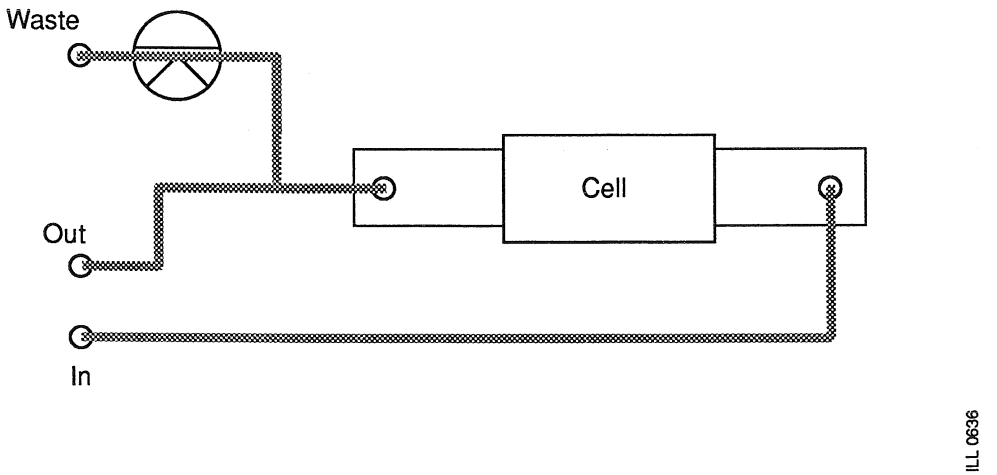


Fig. 3.2 ZET 5104 Cell Connections (with Sample Handling Unit)

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- Connect a tube from the port on top of the near end of the cell through the valve in front of the cell and then to the left of the cell to connect to the port marked "**WASTE**", which goes to the sample waste port on the right hand side of the optics unit. Both ends of this tube will have plugs attached to them. When changing from one cell to another, this tube can be left in place.

3.4.2 With the sample handling unit

The sample handling unit is an optional addition to the ZetaSizer 4 which completely automates the injection of samples into the ZET 5104 electrophoresis cell. The connection of the tubing is shown in Fig 3.2. The organisation is basically the same as that without the sample handling unit, except that a T-piece is inserted just before the valve in front of the cell. One arm of this T-piece is connected by a short tube to the syringe port, which is marked "**OUT**".

3.5 Cell connections for the ZET 5103 cell

The connection of the tubing for the ZET 5103 cell is the same, whether or not a Sample Handling Unit is fitted. In fact, the high back pressure produced by the ZET 5103 cell means that manual injection must be used.

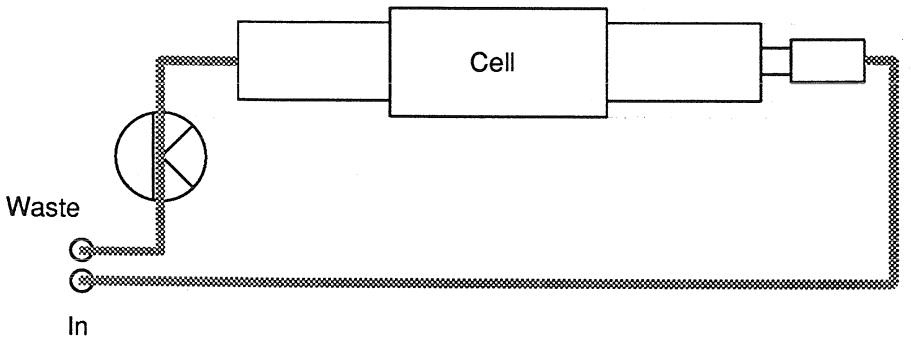


Fig. 3.3 ZET 5103 Cell Connections

The narrow capillary used in the ZET 5103 makes it susceptible to very small flows of liquid, so the tubes at the entry and exit of the cell must be closed near to where they leave the cell. At the entry, (the far end of the cell once it is mounted), there is a non-return valve. This is a black cylinder about 6mm in diameter and 20mm long which is stored in the ZET 5103 cell accessory box. It fits directly into the hole in the end of the cell block. At the exit, the front of the cell once it is mounted, the tubing goes straight to the waste valve.

Injection of the sample can either be from the port at the front of the optics unit, or directly into the cell, using a 1ml syringe. Direct injection is particularly useful if only small volumes of sample are available.

For injection from the front port, connect the cell as in Fig 3.3.

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Connect a tube from the port marked "**IN**" in front of the cell base to the non-return valve at the far end of the cell. This tube will have luer fittings at both ends.

Connect a tube from the port in the near end of the cell through the valve in front of the cell and then connect to the port marked "**WASTE**", which goes to the sample waste port on the right hand side of the optics unit. Both ends of this tube will have luer fittings attached to it. When changing from one cell to another, this tube can be left in place.

Injection of samples and flushing the cell is exactly the same as for the ZET 5104 cell without a Sample Handling Unit.

Direct injection into the cell is illustrated in Fig. 2.3. The fitting of the tubing is exactly the same as described above, except that the first step is omitted. Injection is just the same as for injection from the front port. However, the **FLUSH** key can no longer be used to clean the cell.

3.6 Cell connections for the ZET 5126 cell

The cell connections for the ZET 5126 cell are very simple. A syringe is placed on the waste valve in front of the cell, so that injection is directly into the hole at the near end of the cell. A tube is connected from the far end of the cell, through the waste valve and out to waste. This tube has a luer fitting at both ends.

3.7 Temperature control

Correct temperature control is essential to the proper operation of the ZetaSizer 4. The conversion of the raw data from the correlator into sizes and electrophoretic mobilities depends on the correct viscosity of the suspending liquid being used in the calculations. The viscosities of aqueous samples are calculated from the temperature measured by the temperature sensor. Always allow some time for temperature equilibration before making measurements, particularly if the temperature of the cell is very different from that of the sample before injection.

ENSURE THAT THERE IS ALWAYS A LAYER OF HEATSINK COMPOUND BETWEEN THE CELL AND THE CELL TABLE

If size standards, such as monodisperse latex spheres, give incorrect results, even after allowing a few minutes for the temperature to equilibrate, first check that sufficient heatsink compound has been applied to give a light covering to the whole surface of the cell table, (fill all the spaces between the cell and the cell table.)

If the ZetaSizer 4 is to run at temperatures more than 10 degrees below ambient, then water should be circulated through the cell table, using the ports which emerge beside the waste outlet.

THIS CIRCUIT IS NOT DESIGNED TO BE USED WITH HIGH PRESSURE WATER FROM TAPS

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Water should be circulated from a temperature controlled bath, or a reservoir using a small peristaltic or centrifugal pump. The arrangement of the tubing for each sort of pump is shown in figure 3.4.

Circulating water through the cell table gives better temperature control at all temperatures, so it is recommended that this is done.

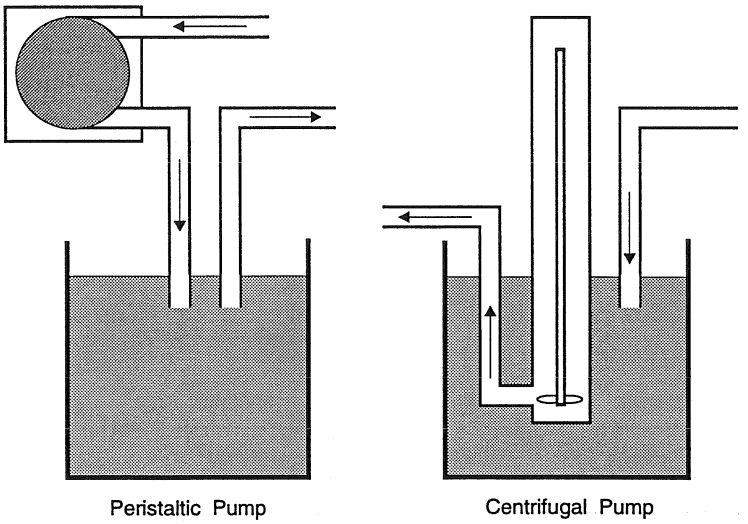


Fig. 3.4 Water Circulation Plumbing Diagram

The maximum temperature which can be used is 60°C. The Peltier element and heat sensor will control at this temperature, if heated liquid is circulated through the cell table.

3.8 The pH probe

The pH probe is a normal laboratory glass combination electrode. Before use, remove the rubber teat from the end.

Clean the probe with a soft tissue, and store the probe in a buffer solution.

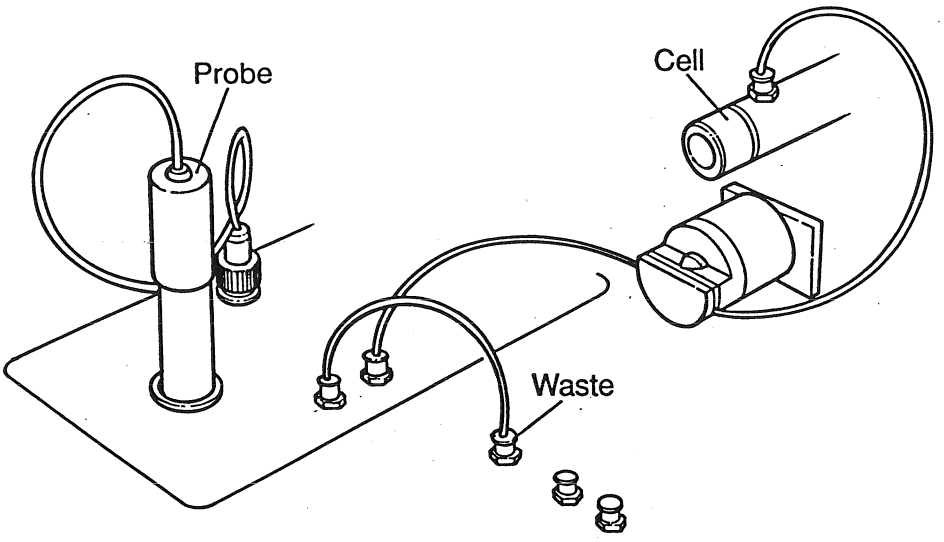
Top up the liquid inside the probe **only** with 3.8M KCl saturated with AgCl.

The probe can be recalibrated using Setup-Instrument-Calibrate pH.

3.8.1 Using the pH probe

The pH probe should be inserted in the sample stream as shown in figure 3.5. This method can be used with or without the ZET5115 sample handling unit option.

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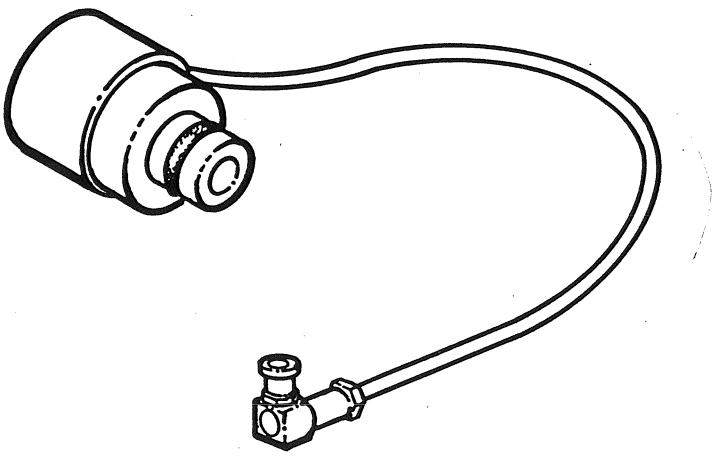


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Fig. 3.5 Using The pH Probe

3.9 Fitting the laser power monitor to the ZET5104

The laser power monitor (Fig. 3.6) is supplied in the ZetaSizer accessory case. It is fitted to the end of the ZET 5104 cell nearest the operator. Fixing is by pushing the snout of the laser power monitor into the hole in the end of the cell. The light detecting element fits over the cell window. The lead fits into a plug in front of the cell connector.



ILL 0217

Fig. 3.6 Laser Power Monitor

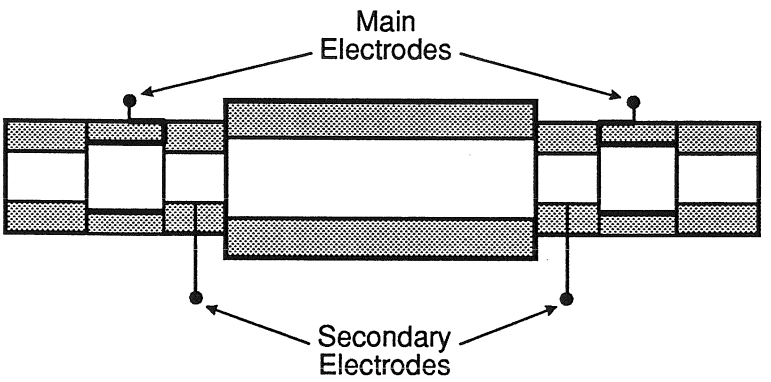
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3.10 Electrophoresis in the capillary cells

In this section procedures are described for carrying out electrophoresis in the ZET 5104 and ZET 5103 cells. The construction of these two cells is very similar, apart from the glass capillary, so they have the same operating procedures and symptoms/cures, when not working correctly.

3.10.1 Structure of the cells

A schematic diagram of the ZET 5104 and ZET 5103 cells is shown in Fig. 3.7.



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Fig. 3.7 Capillary Cell Schematic

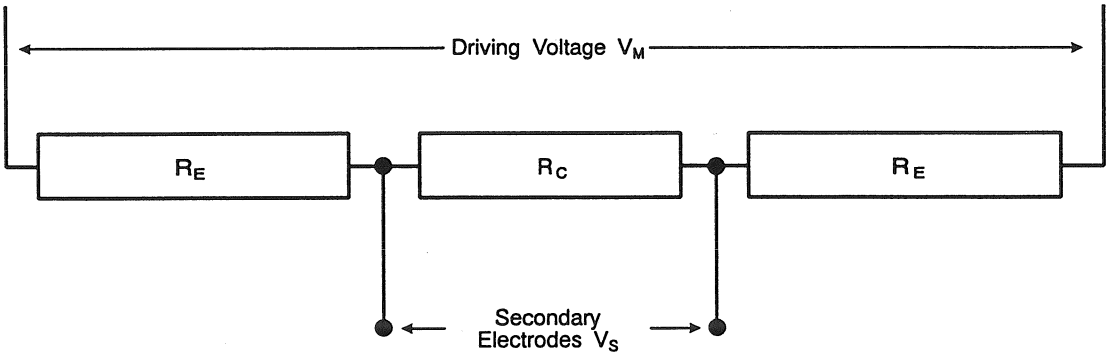
The main (driving) electrodes are made of platinum covered in platinum black. They are fitted inside the electrode plugs, which can be removed from the cell.

The internal diameter of the cell within the electrode is larger than the diameter of the glass capillary, to minimise voltage losses between the driving electrode and the end of the capillary. This maximises the voltage gradient across the measuring volume, however some drop is inevitable, so secondary (sensing) electrodes are located in the end blocks close to each end of the capillary.

The diameter of the capillary in the ZET 5104 is governed by the need that it should be large enough to locate the "stationary level", or "stationary layer", accurately, whilst not raising the current required to support a voltage gradient high enough to give reasonable resolution. The electrical resistance of the capillary varies directly with length and inversely with cross sectional area. To achieve high accuracy, a high voltage is desirable, but Joule heating of the cell must be avoided as it causes inaccurate results.

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The electrical "equivalent circuit" of the cell is shown in Fig. 3.8. The electrode and assemblies each form a resistor, as does the capillary itself. As the voltage drop across any element is proportional to its resistance, we try to ensure that the resistance of the electrodes is much lower than that of the capillary. If bubbles are trapped next to the electrodes, the corresponding resistance will be very high, and practically no voltage will be applied across the capillary.



$$\text{Current} = \frac{V_M}{R_C + 2R_E} = \frac{V_S}{R_C}$$

ILL 0640

Fig. 3.8 Electrical Equivalent of a Cell

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CHAPTER 4

Maintenance

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4.1 Introduction

Maintenance procedures for the cells are given. They include cleaning the cells and checking their optical alignment. These procedures do not form part of the normal operation of the ZetaSizer 4. The fundamental re-alignment of the optical components, which are protected by baffles is not described here, this is covered in the service manual. If service is needed, contact Malvern or their local representatives.

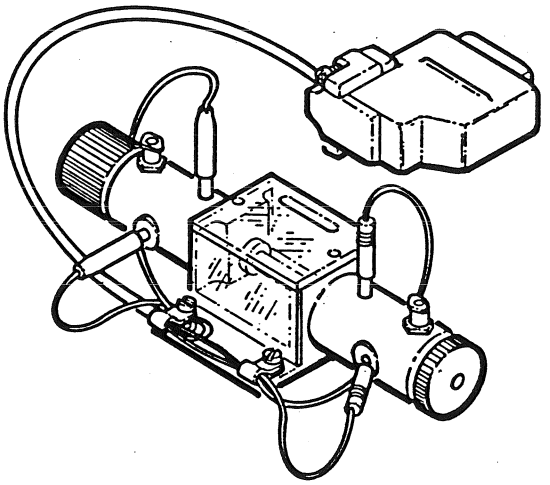
During some stages of the cell alignment, there may be direct access to laser radiation, since the interlock shutter must be overridden. The interlock shutter is the switch found under the cover of the optics unit to the left of the front. During normal operation, the laser beam is only switched on when this switch is depressed by closing the lid.

The operations described only require the viewing of scattered laser light, and so can be safely performed by personnel who have been suitably instructed and made familiar with the possible hazards involved.

Before performing any of the procedures requiring override of the interlock shutter, users must be sure that they are familiar with the laser safety information contained in this manual.

4.2 Maintenance of the ZET 5104 cell

The ZET 5104 is the normal electrophoresis cell. It should be kept scrupulously clean, if accurate results are to be obtained. The ZET 5104 is shown in Fig. 4.1.



ILL 0221

Fig. 4.1 The ZET 5104 cell.

4.2.1 Removing the ZET 5104 cell from the ZetaSizer

Slacken the locking screw on the cell table and draw the cell towards you, and clear of the cell table.

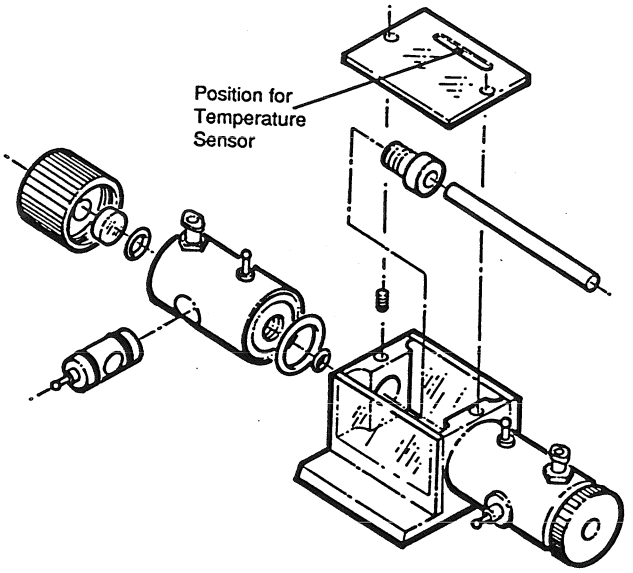
Unplug the two tubes from the cell, unplug the cell connector and remove the cell.

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4.2.2 Dis-assembly of the ZET 5104 cell

Figure 4.2 shows the parts of a ZET 5104 cell. To dismantle a ZET 5104 cell for cleaning carry out the following steps:

- Disconnect the main electrode leads from the electrode holders at each end of the cell by turning the plastic ends to one side, (do not disconnect by pulling). Disconnect all the tubing from the cell. Each electrode can now be removed using a small probe such as the smallest allen key provided with the system by pushing the probe through the small hole opposite the main electrode and pushing the electrode holder out.



ILL 0222

Fig 4.2 ZET 5104 Cell Assy.

- Check that the cylindrical platinum electrodes inside the electrode holders are in good condition, they should be a uniform matt black. If they appear shiny, or if the black coating is uneven, they will need to be replatinised, see section 4.2.4.

- The cell windows can now be removed for cleaning. Unscrew the knurled nuts at each end of the cell and remove the window. The windows should be cleaned with an optical lens cleaning tissue, and a little propan-2-ol (isopropanol) if necessary.

- Drain the index matching bath using a syringe and needle, to avoid spills later. Remove the electrode block from each end of the cell by unscrewing it. The capillary will probably come out with one of the electrode blocks. If not it may be withdrawn once both blocks have been removed.

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- The capillary tube can now be removed for cleaning. Normal glassware cleaning techniques can be used, such as detergent, scrubbing, ultrasound, but always be sure to rinse the tube thoroughly with clean water before replacing it. For some research applications, more vigorous chemical cleaning may be needed, using concentrated acids. After cleaning, the tube should only be handled near to its ends, to avoid contamination of the area where scattering occurs.

4.2.3 Assembly of the ZET 5104 cell

- Replace the windows in the electrode blocks. The 'O' ring must be carefully pushed into the retaining groove in the electrode block, using a blunt rod, to ensure a good seal. The screws retaining the windows should be well tightened to ensure a good seal.

- Replace the capillary tube in the index matching bath. Ensure that the tube protrudes equally from both ends of the bath.

- Slide the electrode blocks back into place over the ends of the capillary. Screw the electrode blocks firmly into place.

- Refill the index matching bath, as described in section 2.6.

- Reconnect the tubing to the cell.
  The ZET 5104 cell is now ready to remount, as described in section 2.7.

4.2.4 Re-platinising the ZET 5104 and ZET 5103 electrodes

The main electrodes in the ZET 5104 and ZET 5103 are identical. They are made from cylindrical sheets of platinum. The platinum surface is "blacked", a process which greatly increases the effective surface of the electrodes. Blacking reduces problems of gas production at the electrodes during electrophoresis.

During use, the black coating will eventually become damaged and worn, it will look uneven, or shiny. At this stage, it will no longer be effective for the prevention of outgassing. The following procedure can be used to recoat the electrodes, or they can be returned to Malvern for treatment.

The treatment uses a bench power supply capable of delivering 0.1A at 24V with a constant current mode. Chlorine is evolved during the process, so it should be carried out in a fume cupboard.

The solution used to recoat the electrodes is made up by dissolving 1g of platinic acid and 0.02g of lead acetate in 50ml of distilled or deionised water.

The electrodes should be treated as a pair. They are connected to the power supply and placed in a Petri dish filled with treatment solution so that the active area of the electrodes is just covered. The supply is set to deliver 50mA for 5 minutes. After this treatment, the electrodes are removed from the solution, shaken dry, rinsed in distilled water and replaced in the treatment solution. The treatment is repeated for another 5 minutes, this time with the connections to the power supply reversed. This process is continued until the coating of the electrodes appears matt black and even. This will normally take less than 30 minutes total coating time.

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4.2.5 Cleaning the capillary without disassembly of the ZET5104 cell

If the inner cell wall is very dirty, it will scatter a lot of light ("flare"). Apart from problems arising from contamination of samples, this flare makes proper determination of the position of the cell walls difficult. The capillary can be simply cleaned by firstly removing the window at one end of the cell, using the knurled screw and the electrode holders. This is best done with the cell removed from the cell table and held vertically with the far end of the cell at the bottom. The capillary can be cleaned with either a cotton bud, from which most of the cotton has been removed, or a pipe cleaner with its end doubled over to avoid scratching the cell wall. After cleaning, the cell window must be carefully replaced to avoid leaks.

On no account must the capillary be cleaned in situ with the electrode holders in place. This will certainly remove the "blacking" from the electrodes.

4.3 Checking the beam heights

This procedure can be quickly carried out when changing from one cell to another. It is done with no cell in place. Open the cover of the optics unit. Set the photomultiplier to 90° and switch it off by pressing the internal keypad key **A**. Locate the two white lines marking the exit lines of the laser beams coming from the two mirrors, at 45° to the axis of the ZET 5104 cell. These are the right hand lines of the two pairs of parallel lines to the right of the optics unit base. Place the target (supplied in the accessory case) in a vertical position on each of the alignment lines in turn, close to the photomultiplier. The cross should be towards the top of the target. When the interlock is overridden, by pressing the switch inside the lid, the laser beam should hit the cross on the target. If this is not the case, then the mirrors need re-aligning, this is a service operation, contact Malvern, or their local representative. Assuming that the beams are at the right height, as is usually the case, the next stage of alignment can be carried out. The PM can be switched on again by pressing key **A** once more.

4.4 Alignment of the ZET 5104

Before aligning the ZET 5104 cell, the user should understand the aim of the exercise, and the appearance of the beams when viewed using the viewer fitted to the PM.

For electrophoresis to be carried out, the two laser beams must both pass through the diameter of the glass cylinder. They must be in the same plane and they must cross at a point where the PM lens will focus on them. The crossing point must be in the stationary layer of the cell, otherwise an incorrect mobility will be measured for the particles. If these conditions are not all fulfilled, it will be impossible to obtain accurate electrophoresis results.

To view the laser beams from the PM, first switch off the PM, using internal keypad key **A**, remove the plug from the top of the PM and replace it with the viewer from the accessory box. Finally remove the pin-hole from the front of the PM. After viewing is completed, remove the viewer, replace the top plug and pin-hole and switch on the PM by pressing key **A** again.

When the laser beams are correctly aligned, they will appear in the viewer as a single vertical red band down the centre of the field of view. The pin-hole appears as an ellipse, with the left hand edge brightly lit. Its position is adjusted using the two knurled screws on the top and the front of the PM. If the pin-hole cannot be seen, it can be illuminated by pressing the button behind the PM, which makes it glow green.

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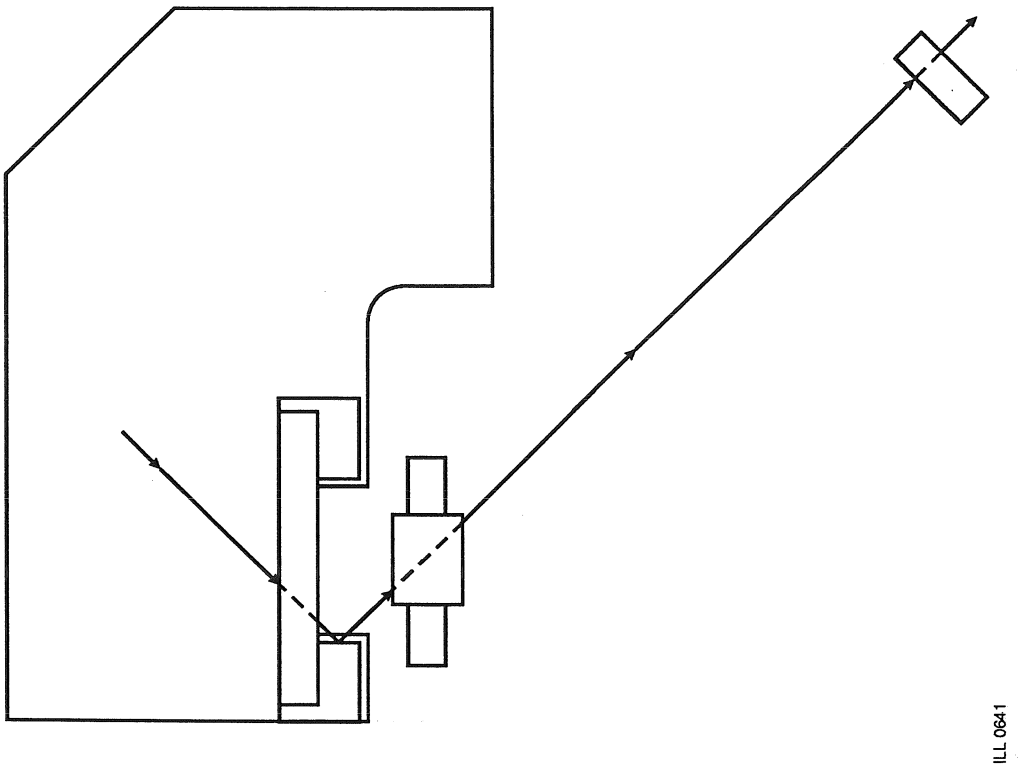


Fig. 4.3 Cell Level Alignment

4.4.1 Setting the ZET 5104 cell height

- Fit the cell, as described in section 2.7, and fill it with a moderately scattering sample. Position the cell so that the laser beams can be seen crossing inside the cell, when the locking screw on the cell table is fully tightened.

- Set the target as in Fig 4.3, and adjust the height of the cell by using the adjusting screw (see Fig 4.4) to set the beam exiting at the rear onto the cross wire of the target, exactly as in the previous section. The cell acts as a cylindrical lens and when it is not at the correct height, it will divert the previously aligned beam. The beams are now passing through the diameter of the glass cylinder.

4.4.2 Setting the ZET 5104 cell walls

For accurate electrophoresis, the laser beams must cross in the stationary layer of the cell. This can only be done if the positions of the cell walls have been correctly stored in the optics unit memory. To set the cell wall positions carry out the following steps:

- Fit the viewer to the bracket attached to the attenuator motor bracket and screw the interlock override device to the interlock switch inside the optics unit cover.

N.B. This will allow access to laser radiation.

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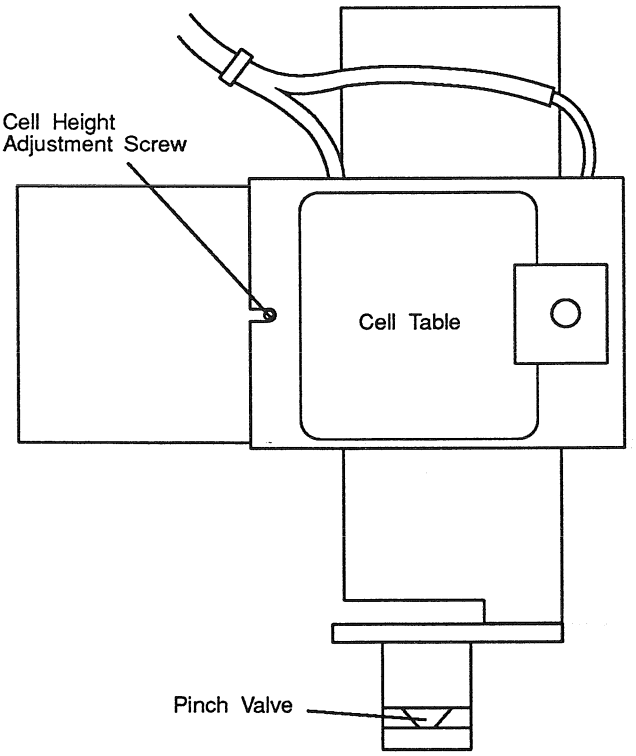


Fig 4.4 Cell Height Adjustment

- Fill the cell with a scattering sample, a 100nm to 300nm latex for example, and focus the viewer on the beam crossover point. The AZ55 is very suitable.

- Press **C** on the internal keypad. The message "SET RIGHT CELL WALL" will appear on the optics unit display. Use keys **4** and **5** to step the cell motor until the two laser beams form a single spot on the inside of the capillary. The two beams will form a V on the wall, with no sign of the beams crossing, or sitting side by side.

- Press **8** to set the beams crossing on left cell wall, the message "SET LEFT CELL WALL" will appear on the optics unit display. Use keys **4** and **5** to step the cell motor to align the beams as for the right wall.

- Press **8** to exit alignment mode, the optics unit display will return to the message "WAITING", or show the value of a parameter. Press **6** to save the settings, the message "SAVING ARRAY" will appear.

To check the position of the right cell wall press keys **0,4**. Press **4** again to exit. To check the position of the left cell wall, press keys **0,5**. Press **5** again to exit. In both cases the stepper motor settings are displayed on the optics unit display.

The ZetaSizer automatically calculates the position of the stationary layer from the stored positions of the cell walls and moves to the correct position just before beginning an electrophoresis experiment.

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4.4.3 Aligning the photomultiplier

- Set the laser beams crossing on the right cell wall by pressing **0,4** on the keypad.

- Fit the viewer to the PM, as described above.

- Looking down the viewer, check that the image of the two beams is in focus. First, adjust the length of the eyepiece to obtain the clearest possible image. If the red line is still not clear, then the PM is not in focus. Adjust this by unscrewing the two screws visible underneath the PM and sliding it back and forth along its axis until the clearest possible image is obtained. Finally, retighten the retaining screws. Sometimes a blurred image is caused by dirt on the viewer, this should be carefully cleaned with a lens tissue.

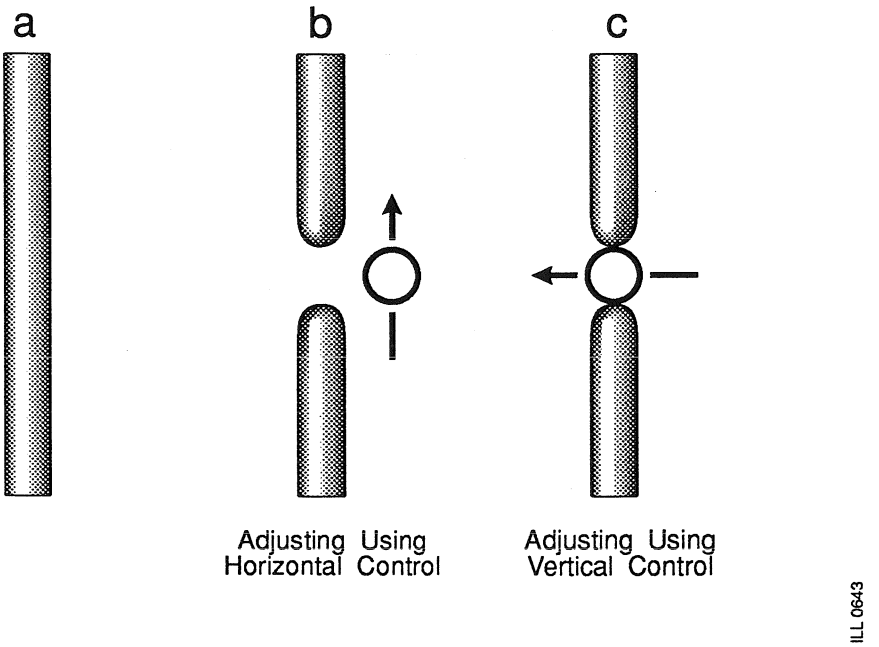


Fig. 4.5 Pinhole Adjustment

- Adjust the position of the PM, using the screws on its top and side, so that the pin-hole is just to one side of the laser beams. If the pin-hole is not readily visible the illuminator can be activated using the button on the far side of the PM. The two beams should meet to form a single band, with no dislocation at their meeting point. If there is a dislocation, this can be adjusted using the screw to the bottom left of the tower holding the front mirror. There is a hole in the baffle to gain access to this screw. If the beams meet, but do not form a straight line, then the optical alignment is wrong. Correction of this fault is a service operation.

- The pinhole now needs to be centred at the crossing point of the laser beams.
  The best way to do this is to cause the beams to cross just outside the capillary and adjust the pinhole to the centre of the apparent "gap" between the beams. This procedure should be done as the next operation after

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aligning the cell walls.
Looking into the viewer fitted into the photomultiplier, adjust the knurled knob (Vertical adjust, see Fig 4.6) in front of the viewer so that the red scattering line is just to one side of the pinhole. This is easily seen by pressing the illuminator button on the side of the photomultiplier.(Fig. 4.5a)

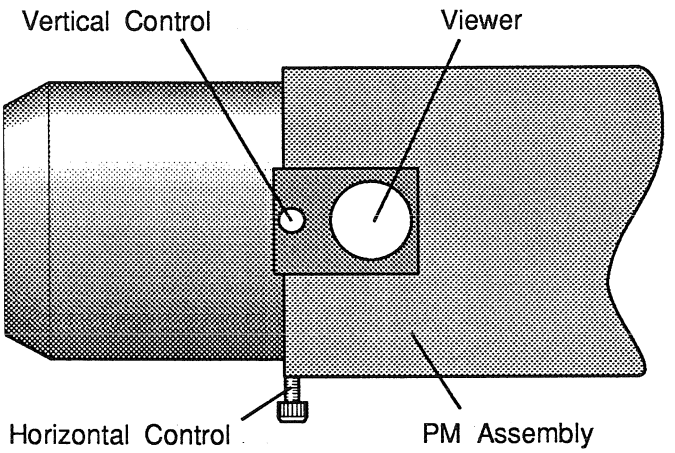
Press **8** on the keypad. "Cell step" will appear on the LED display.

Press **5** until a "gap" appears between the two scattering beams (Fig 4.5b)

Adjust the two knurled Knobs at the top and the side of the photomultiplier to bring the pinhole perfectly aligned between the beams. (Fig. 4.5c)

Press **8** on the Keypad.

This completes the PM alignment.



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- Remove the viewer from the PM and replace the top plug and pin-hole. Press **A** to switch on the PM.

4.5 Alignment for reference beam mode

Reference beam mode is used for high resolution electrophoresis measurements with the ZET 5104 and ZET 5103 cells. The PM is automatically placed so that light from the beam shining backwards goes directly into it. A filter is automatically placed in the beam, so that its intensity is low. Alignment is even more important in reference beam mode than in normal electrophoresis measurements.

Before starting a measurement in reference beam mode, make sure that the cell wall positions are aligned, as this cannot be done in reference beam mode.

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To select Reference Beam mode run the Macro program **Full Menu** which can be found under the "**Control**" main menu item. This will enable you to check the "**Reference Beam**" selection box on the "**Zeta Potential Setup Measurement**" menu item, under the main menu "Setup" item. Now click OK. on the setup dialogue box. (If the **Full Menu** Macro program is not installed on your system then go to the "**Control - Edit Program**" command, and enter "**enable refbeam 1**", then run this simple program.

The procedure for setting up reference beam (heterodyne) mode on the ZetaSizer 4 is as follows.

- First ensure that the system is correctly aligned in homodyne mode, preferably by obtaining a good and correct result with a known sample (AZ55)
- Switch off the photomultiplier high voltage supply by pressing A on the keypad. The count rate should drop to zero.
- Use Setup Measure to select reference beam operation as described above.
- The photomultiplier arm should go to the correct position for heterodyne mode and the attenuator should be in position so that the beam from the modulator mirror passes through the thickest of the attenuator plates.
- Check that the scattering beam (the beam that does not pass through the attenuator) is not clipping.
- Remove the front pinhole from the photomultiplier assembly (400µm or 200µm) and mount the viewing telescope onto the photomultiplier assembly.
- Press C on the optics keypad - the message "alignment mode" should appear and the attenuator should move so that the reference beam passes through the less dense attenuator and is visible on the pinhole of the photomultiplier.
- Check the focus of the photomultiplier and adjust as required. The image of the light from the cell should be set as below.
- Adjust the screw on TOP of the photomultiplier assembly so that the strip of light crosses the pinhole. On no account touch the adjustments on the side of the photomultiplier assembly.
- Use keys **4** and **5** on the keypad to move the bright spot onto the pinhole. When the spot is properly aligned it will nearly disappear down the pinhole.
- Refit the front pinhole and use the allen screws to adjust its position so that the pinhole is centred on the bright spot from the reference beam.
- Press **8** on the keypad. The attenuator will return to its operating position and the reference beam will no longer be visible.
- Temporarily block the scattering beam using a piece of card.

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- Go to **Measure - Inspect - Display - Ratemeter** to see the count rate from the detector.

- Press "A" on the keypad on the instrument and check that the counts are displayed on the rate meter.

- Adjust the position of the reference beam by using the horizontal and vertical adjustments on the modulator mirror mount. Use these two adjustments to maximise the count rate which should rise to about 2000 in the count rate box when correctly adjusted.

- Remove the card blocking the scattering beam.

- Make measurements.

A few pointers may be useful:

- The sample should be very dilute so that the ratio of the scattered intensity to the reference is at least 1:100 ie reference beam is at least 100 times the intensity of the scattered light.

- Care must be taken at all times to avoid disturbing the alignment of the cell.

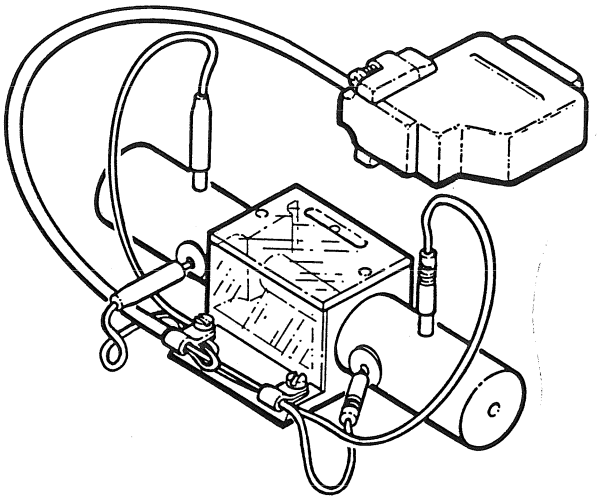
Use the Ratemeter to check the intensity of light from each beam, by blocking each one in turn at the point that they enter the cell. Take great care at this stage not to disturb the position of either the cell or the attenuator disc, which is close to the cell window. The intensity from the reference beam, i.e. that shining forwards onto the PM, should be at least 1000 times more than that from the scattering beam. If the ratio is much less than 500:1, then resolution may be lost. The ratio can be adjusted by altering the concentration of the sample.

Electrophoresis measurements can now be made in the usual way. The system will not move away from its reference beam position until the Setup Measurement dialogue is used to de-select **Reference Beam Mode**, by unchecking the **Reference Beam** check box

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4.6 Maintenance of the ZET 5103 cell

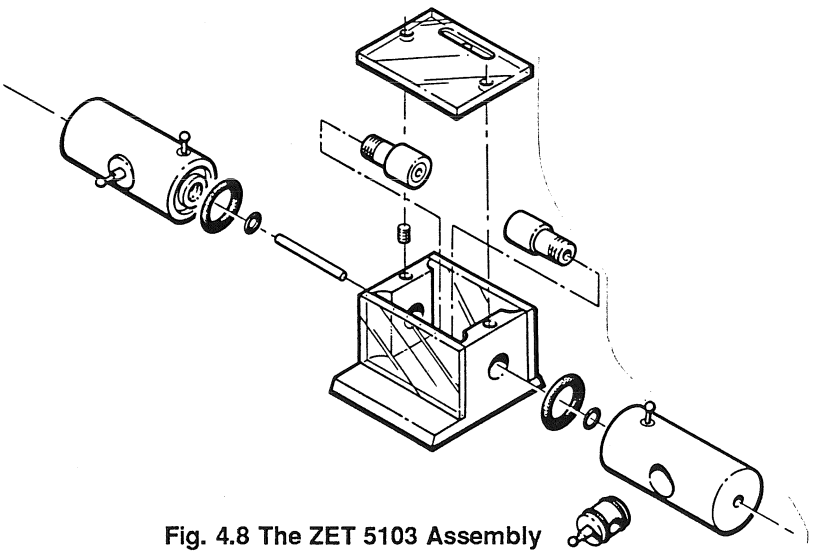
The ZET 5103 cell is very similar to the ZET 5104 cell. Fig.4. 7 shows the cell while Fig.4.8 shows its assembly. The only difference is in the size of the capillary. Instead of a 4mm diameter glass tube, a 0.7mm tube is used. This tube has been coated with an uncharged methyl cellulose coating, which means that there is no electro-osmosis. Since there is no flow in the cell, measurements can be made anywhere across the capillary, in fact they are made in the centre.



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Fig 4.7 ZET 5103 Cell

As the capillary is polymer coated, it cannot be cleaned internally. If inaccurate results are found with standard samples, the capillary must be replaced. However, the outside of the capillary can be cleaned, using an optical tissue and a little "propan-2-ol", if necessary.



ILL 0670

Fig. 4.8 The ZET 5103 Assembly

4.6.1 Replacing the ZET 5103 capillary without dis-assembly

The capillary is held in place by a silicone rubber seal at each end. Two specially shaped rods are supplied in the cell accessory case, so that the capillary can be removed from the cell, whilst keeping the seals open with a rod. The capillaries are quite delicate, the ends can easily be chipped by the metal tip of the rod, so this operation should be carried out with great care.

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- Remove the cell from the ZetaSizer. Empty the water from the thermal bath with a syringe.

- Disconnect the main and auxiliary electrodes and unscrew the electrode blocks.

- Push one of the rods supplied into each end of the cell, until the pointed end of the rod touches the end of the capillary.

- Gently push the capillary out of the cell, making sure that the end of the capillary remains in contact with the end of the rod. Push the rod which is still inside the cell untill the end protrudes from the cell.

- Place the new capillary between the end of the rod in the end block and the other rod. Carefully pull the rod inside the cell backwards so that the capillary is gently moved into position. Again, make sure that the capillary is always in contact with the ends of the rods. When the capillary can be seen to be centrally positioned, remove both rods.

- Refill the thermal bath with filtered water, as described in section 2.6.

4.7 Alignment of the ZET 5103 cell

Aligning the ZET 5103 cell is the same as aligning the ZET 5104, apart from some differences due to the smaller capillary. The procedures given for the ZET 5104 can be followed, as long as these points are observed:

- The light spots of the exit beams are never as well defined as for the ZET 5104, after passing through the ZET 5103's smaller capillary, but they can still be used for alignment.

- Setting the cell height can be harder, as the beam may miss the capillary completely. Ensure that the beam is in fact crossing the capillary and not simply passing straight through the thermal bath.

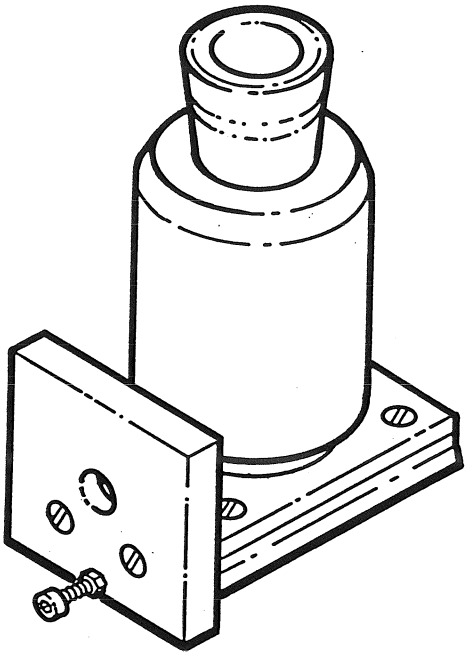
4.8 Alignment of the ZET 5110 cell

Fig 4.9 shows the ZET 5110 cell , while Fig 4.10 shows the same cell dis-assembled.

- First, the cell table must be aligned so that, in parallel beam mode, the beam passes through the diameter of the cell. Fit the cell to the table and lock it into place with the locking screw. The light baffle, (the piece of black metal which fits inside the cell's bath), should be removed. Alignment is easier if the water in the bath is scattering some light, so that the beams can be clearly seen.
  The laser beam passing through the cell should form a bright spot on the front baffle, if it does not, rotate the outer jacket into the correct position. Place the alignment wire, from the cell accessory case, in the cell so that the beam is not blocked by the wire's supports. Press keypad key **C**, the message"SET RIGHT CELL WALL" will appear on the optics unit display.

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Use keypad keys **4** and **5** to adjust the position of the cell until the bright spot changes to become the longest possible horizontal line, due to the laser



ILL 0229

Fig 4.9 The ZET 5110 Cell

beam illuminating the alignment wire. By gently moving the top of the alignment wire, its shadow can be seen in the bright spot. You may find that an easier method of alignment is to make sure that the shadow is in the centre of the spot.
Press keypad key **8**, the message "SET LEFT CELL WALL" will appear. Use keypad keys **4** and **5** to adjust the cell as before. Press keypad key **8** again, the message "WAITING" should appear. Press key **6** to save the cell positions. The cell is now in the correct position on the axis perpendicular to the beam.

- Remove the alignment wire and the outer jacket from the bath.

- Now the adjustment screw at the front of the cell must be set so that the cell is in the correct position along the axis of cell table. This is done by checking the position of the beams leaving the cell. The cell is correctly aligned when the beams are not deflected by the cells.

  The beams crossing inside the bath should be clearly visible. The screw at the bottom of the front of the cell should be completely unscrewed, although not removed. Place the cell on the table as near to the back as possible. Press keypad key **D** to change to cross beam mode. The locking screw (Fig. 4.8) should be almost completely locked, so that the cell is very close to the position that it would have normally, but still allow the cell to move. Adjust the position of the cell by screwing the adjustment screw in. Place the alignment target on the instrument so that the vertical white line on the target

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is coincident with the line on the instrument. The cell position is adjusted until the beam exiting the cell strikes the target on the line. Fig 4.11

When the best position has been found, fully lock the locking screw and check that the cell is still in the right position, a slight adjustment may be necessary to make the spots coincide when the cell is fully locked into place. Finally tighten the locking nut on the adjustment screw, so that its position is fixed.

- Replace the outer jacket, fill the cell bath with clean filtered water and remove the laser override tool. Insert the internal light baffle/beam stop into the cell. It should be located by a small recess on the left of the bottom of the bath.

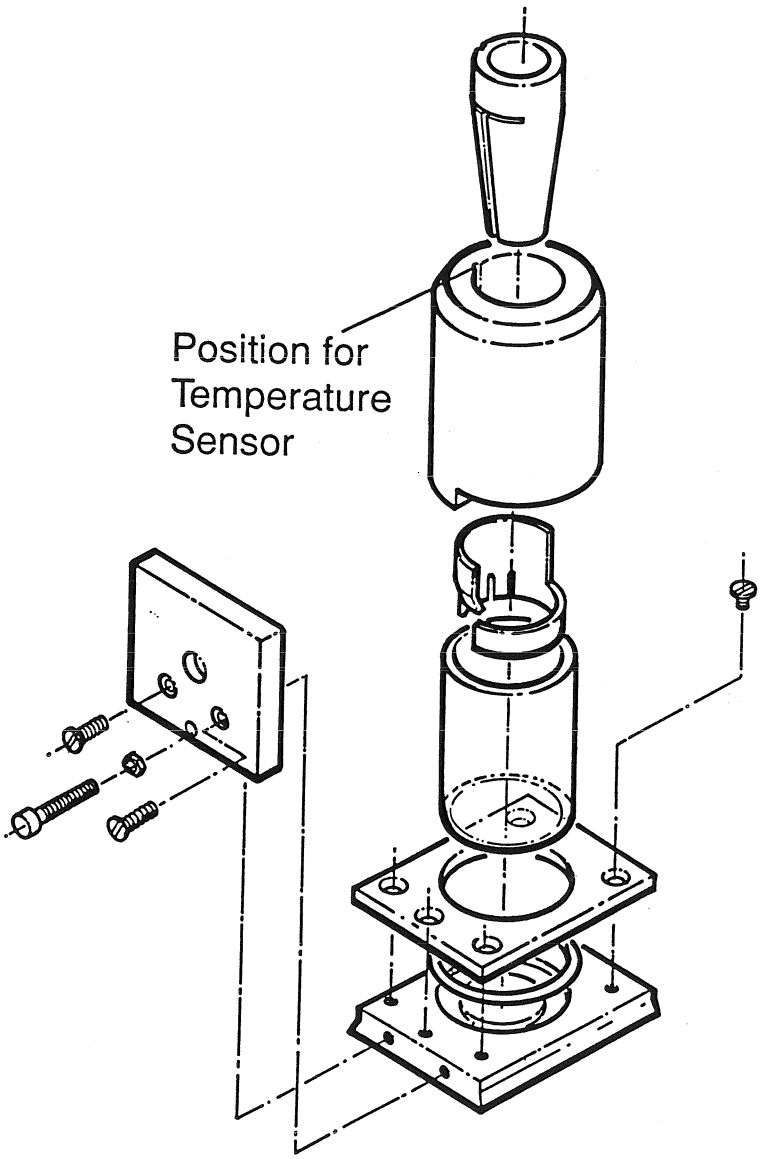
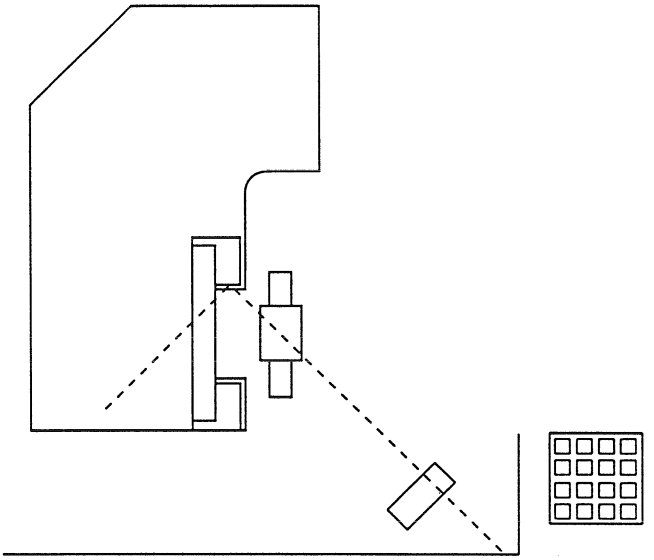


Fig. 4.10 The ZET 5110 Assembly

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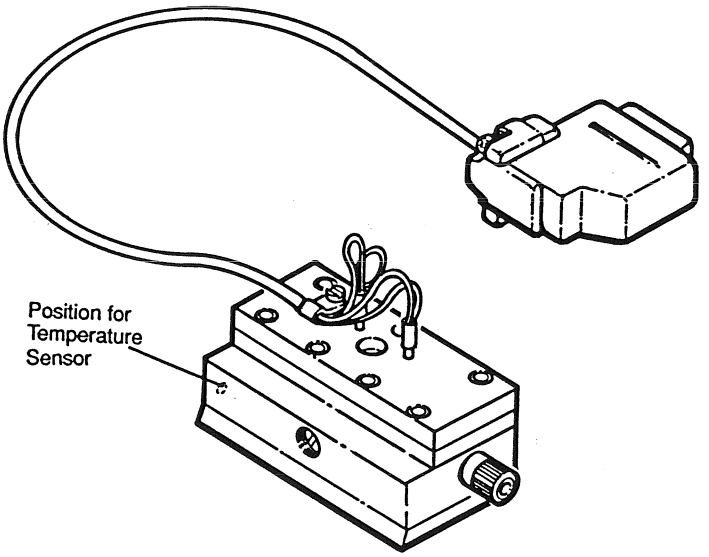


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Fig. 4.11 Alignment of the ZET 5110

4.9 Cleaning and Maintenance of the ZET 5126 cell

Fig 4.12 shows the ZET 5126 cell. The cell has a flow-through design, so for batches of similar samples dispersed in the same solvent the cell may be simply flushed through using clean solvent. However, for more thorough cleaning disassembly of the cell is recommended.



ILL 0232

Fig 4.12 ZET 5126 Cell

Remove the cell from the instrument and disconnect the electrode terminals by pushing sideways, not pulling. Great care is required in disconnecting the electrode terminals as excessive movement could cause damage to the electrode plating. Remove the seven screws retaining the top of the cell and loosen the knurled connector at the front of the cell in order to allow the internal parts of the cell to be removed for cleaning and inspection.

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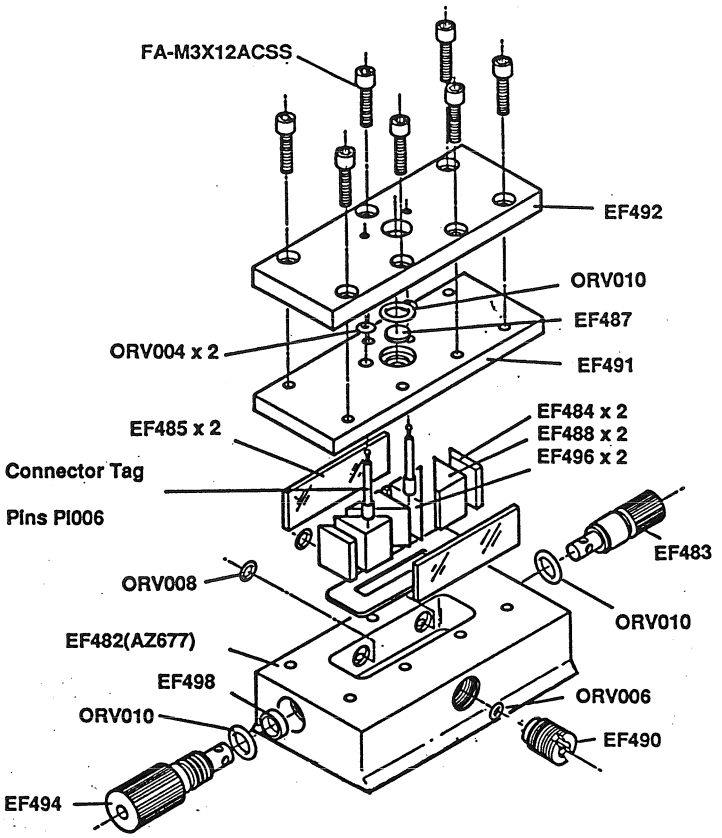


Fig. 4.13 The ZET 5126 Cell Assembly.

The removal of the internal parts of the ZET 5126 is best accomplished by inverting the cell onto a soft, clean surface such as lens tissue. Take care to ensure that no parts are mislaid as some of the glass parts are very small. Inspect the parts for any damage and clean them carefully using a suitable solvent such as propan-2-ol or ethanol. The cell parts, other than the plain glass parts, should not be cleaned with any aqueous solutions.

Reassembly is the reverse of disassembly. Check that the black plates are correctly oriented, as shown in Fig. 4.13. Care is also needed in the orientation of the prisms and the sealing of the windows. The prisms with the corners cut off are placed nearer the ends of the cell with the cut off corners on the side of the cell nearest the PM. The prisms with gold faces are placed nearer the centre of the cell with the mirrored part closer to the side of the cell where the beams enter.

The figures show that the inlet and outlet assemblies press on the two black glass plates which in turn press on the two pairs of prisms separated by a small glass spacer. It is the wedging action of the two prism pairs forcing the two rectangular windows against the "O" rings recessed into the cell walls which seals the system. The exhaust assembly has been designed so that it yields slightly under load to prevent the pressure from breaking the windows. However, overtightening of this component during assembly could cause permanent distortion or damage. Before final tightening of the exhaust assembly ensure that the glass spacer is properly positioned and that all the components are pressed down into the cell body.

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4.10 Alignment of the ZET 5126 cell

The electrodes of the ZET 5126 are large, parallel plates, which are only 1mm apart. This design means that electro-osmotic effects do not affect the measurement. Alignment ensures that measurements are carried out midway between the electrodes, to avoid flare, and not too close to their edges, to avoid edge effects. The gap between the electrodes can be viewed through the window on top of the cell. When the beams are correctly aligned, they form a symmetrical "X" in the gap. It is not necessary to use the microscope to align the ZET 5126.

- Fit the ZET 5126 cell to the cell table and screw its plug into the base of the optics unit. When the cell is first fitted to the cell table, undo the screw at the bottom of the front of the cell, so that it does not stop the free movement of the cell. After the initial alignment, the position of this screw is fixed, so that the cell can easily be placed in the correct position on the cell table. Press **0,7** on the internal keypad to update the cell type in the optics unit memory. If the optics unit is not already in cross beam mode, press **D** on the internal keypad. Fit the laser shutter override tool inside the left hand corner of the optics unit cover.

- Press **0,4** to move to the currently stored position of the "Right cell wall" for an ZET 5126 cell (as for the ZET 5110, the alignment is not actually at the cell walls). Press **4** again to return the optics unit to the waiting state. With the locking screw almost locked, move the cell forwards and backwards until the two light spots on the left hand side of the cell are symmetrically placed with respect to the beam entrance holes. If the cell is close to being aligned, the beams will pass through the holes and into the cell. Lock the cell into this position with the locking screw.

- Press **C** to set the "Right cell wall". Use the keypad keys **4** and **5** to adjust the position of the cell so that the beams cross in the gap between the electrodes. If the "X" formed by the beams is not symmetrical, then the position of the cell on the cell table must be adjusted a little. Unlock the locking screw slightly, move the cell to improve the appearance of the X and lock the screw again. Use the **4** and **5** keys to adjust the position of the cell so that the "X" is as close to the axis of the cell as possible, to avoid edge effects. It is not possible to align the axis of the cell with the beam crossover point, but this does not affect the performance of the cell.

- Press the keypad key **8** to set the "Left cell wall" position. Repeat the procedure given in iii). When the position and symmetry of the X is satisfactory the screw at the bottom of the front of the cell can be screwed in until it touches the cell table. It should be fixed into the correct position by tightening the nut against the end of the cell. Once this position is fixed, the cell can be placed in the correct position by sliding it onto the cell table from the front until the screw touches the cell table. Press **8** to exit the alignment procedure and **6** to save the "cell wall" positions.

- The position of the PM may need to be adjusted when the ZET 5126 cell is fitted. Switch off the PM by pressing **A** on the internal keypad. Remove the pin-hole from the front of the PM. Unscrew the plug from the top of the PM and replace it with the viewer. The laser beams crossing inside the cell should appear as a short bar of light (the image is much less bright than in the other cells). The pin-hole appears as a dark ellipse, lit on one edge. For correct alignment, the centre of the ellipse should coincide with the centre

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of the bar of light. The position of the pin-hole is adjusted using the screw on the side of the PM nearest the user and the screw on top of the PM next to the microscope. After alignment is complete, remove the viewer, replace the plug on top of the PM. Replace the pin-hole and finally switch the PM on again by pressing **A**. The ZET 5126 is now ready to make measurements, so the laser shutter override tool can be removed.

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