Instruction Manual

for

ZetaPALS, Zeta Potential Analyzer

Brookhaven Instruments Corporation
Brookhaven Corporate Park
750 Blue Point Road
Holtsville, New York 11742 USA

Telephone: +1(631)758-3200
Fax: +1(631)758-3255
e-mail: tech@bic.com
web: www.bic.com

Please Read

This is your instruction manual for the Brookhaven *ZetaPALS*. Please read it carefully before making measurements. You should also read the instruction manual for the *ZetaPlus* instrument since the basic theory underlying the measurement of zeta potential using electrophoretic mobility is common to both variants and is not reproduced here. Sections I (Introduction), IV (Theory), VII (Sample Preparation) and the Appendices are especially relevant.

The *ZetaPALS* option consists of hardware and software that extends the capabilities of the Brookhaven Instruments *ZetaPlus* zeta potential analyser, or the *90Plus* particle size analyzer to provide another method of measuring electrophoretic mobility, and hence determining zeta potential. This method is referred to as PALS or Phase Analysis Light Scattering and is a far more sensitive technique than the laser Doppler method used in the basic *ZetaPlus*. This extra sensitivity is particularly useful, though not limited to, the measurement of low electrophoretic mobilities. Such mobilities can arise in a variety of situations but particularly when a dispersion of particles in media of low dielectric constant or high viscosity must be characterized. Another case is that of a dispersion in a highly conductive medium when the high concentration of ions leads to strong electrostatic shielding of the particles and limits the electric field that may be applied. Yet another case is when the mobility is low simply because the zeta potential itself is very low, close to an isoelectric point, for example.

The additional components making up a *ZetaPALS* unit are ...

1. PALS plug-in electronic card. This plugs into the CPU bus next to the standard *ZetaPlus* card and connects to it via a 40 way ribbon cable, which also extends to the power distribution board.

2. PALS HV high voltage module. Mounted beneath the optics table.

3. Cables.

4. PALS Zeta Potential Analyzer Software. This requires Windows 3.X or higher and 8 Mb of RAM.

5. This manual.

Optionally you may have purchased the ceramic- or Kevlar-supported, solvent-resistant electrodes.

The units above will have been factory fitted unless an in-field upgrade has been agreed upon. The parts should only be fitted by Brookhaven personnel or an appointed representative.

Copyright Notice

WARRANTY

Brookhaven Instruments Corporation (hereinafter known as BIC) warrants that the product is free from defective material and workmanship. Under the terms of the warranty, BIC agrees to correct by repair or, at BIC's election, by replacement, any parts which prove to be defective through no fault of the user.

This warranty is limited to the original purchaser of the product.

The product shall be shipped, freight prepaid and insured in full, or delivered to a facility authorized by BIC to render the service provided thereunder, in either the original package or in a similar package affording an equal degree of protection. The purchaser must contact BIC for instruction prior to returning the product.

The product shall not have been previously altered, repaired or serviced by anyone other than a service facility authorized by BIC. The product shall not have been subjected to accident, misuse or abuse, or operated contrary to the instructions contained in the instruction manual or manuals.

BIC shall not be liable for direct, indirect, incidental, consequential, or other type of damages resulting from use of this product other than the liability stated above. These warranties are in lieu of all other warranties, expressed or implied, including, but not limited to, the implied warranties of merchantability or fitness for a particular purpose.

The BIC warranty extends for a period of 90 days. This period from the date of receipt of the equipment, and it applies only to the original purchaser. The warranty period is automatically extended to 1 year (except as noted below) from the date of receipt of the equipment provided all invoices for said equipment, including transportation, if applicable, are paid within 30 days after receipt of invoice.

The BIC warranty extends for a period not exceeding the warranty period of the Original Equipment Manufacturer where applicable. The typical warranty period on printers and computer peripherals is 90 days. Please contact BIC for copies of applicable OEM warranties.

This warranty does not cover replacement of fiber optic probes or connectors damaged by dropping or from corrosion caused by lack of cleaning. Please refer to the relevant section for information on caring for your ZetaPALS and its accessories.

Software License Agreement

Carefully read the following terms before using the software provided with this system. Use of the software indicates your acceptance of these terms. If you do not agree with the terms, promptly return the software and system. BIC refers to Brookhaven Instruments Corporation.

Terms:

1. In purchasing this software you are granted a nonexclusive license to use the software product on one computer.

2. BIC retains title to, and ownership of, the software product. The software product may not be modified without the written consent of BIC.

3. Duplication of the software product for any purpose other than backup protection, including duplication for any commercial use, is a violation of the copyright laws of the United States of America and of other countries.

Information produced by using BIC software and it's manual, including the resulting displays, reports, and plots are believed to be accurate and reliable. However, no responsibility is assumed by Brookhaven Instruments Corporation for any changes, errors, or omissions.

Table of Contents

I. INTRODUCTION

This document describes the Brookhaven Instruments Corporation *ZetaPALS*. It is intended to supplement the user manual supplied with the *BIC ZetaPlus* covering those aspects that are specific to PALS operation.

PALS is an acronym for Phase Analysis Light Scattering - this is an extension of laser, electrophoretic light scattering (ELS). ELS is used to the measurement of the velocity of moving particles that scatter laser light. PALS is interesting because it is much more sensitive than conventional ELS. It has been shown that using PALS one can measure velocities that are as little as 1000 times smaller.

In the BIC *ZetaPALS* the benefits of PALS are applied to the measurement of electrophoretic mobility (EPM) and the calculation of zeta potential. The velocity that is measured here is the product of the EPM and the electric field. The ordinary *ZetaPlus* measures the EPM by conventional ELS. The ELS technique can measure samples of EPM greater than about 0.2 (microns/second)/(volt/cm) and greater and salt concentrations as high as about 20 mM of a 1:1 electrolyte like $NaNO_3$. Lower EPMs are found in media of low dielectric constant, high viscosity or higher salt molarity, or where the zeta potential itself is low (close to the IEP for example). The *ZetaPALS* is useful in such cases. It too has the ability to perform measurements on samples that are within the capability of the *ZetaPlus*, although, the greater sensitivity leads to more accurate measurements in a given experiment duration.

Externally the *ZetaPALS* is identical to the *ZetaPlus*. Internally extra electronic modules are added that provide the phase analysis and field drive functions. In addition, an interlock is provided that prevents the application of high voltage fields when the sample door is open. The instruments range is as follows:

... low range: 0 - 28 V/cm, 1 to 250 Hz, sine or square wave.

... high range: 29 - 555 V/cm, same timing characteristics.

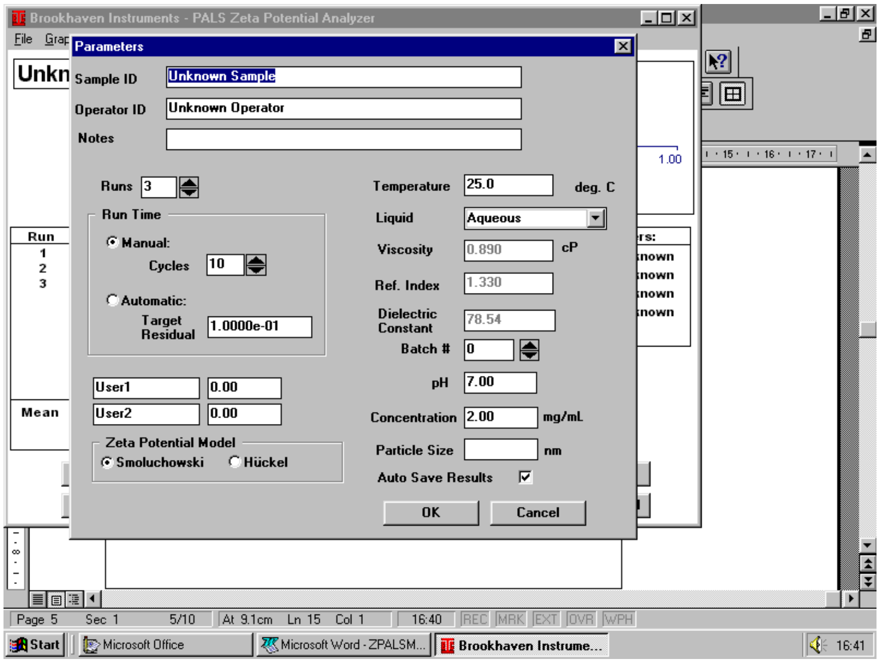
Measurements using many nonaqueous media require the use of glass or quartz cells and the Kevlar supported electrodes. The cells are 1cm square, however be careful when using non-BIC supplied cuvettes as the exact dimensions vary between suppliers**.** For most aqueous based samples disposable acrylic cells and Plexiglas (also known as Perspex or Lucite) based electrodes may be used.

II. Making A Measurement

Sample preparation is undoubtedly the most important consideration in making use of the *ZetaPALS*. A good dispersion at an appropriate concentration in the chosen medium must be presented to the instrument. The cell and electrodes must be clean and appropriate for the chosen medium. Guidance on sample preparation is given in section VII of the *ZetaPlus* manual. Exactly the same considerations apply to the *ZetaPALS*.

Make sure that you are aware of, and adopt the proper handling precautions necessary for the sample and medium you are using. Many nonaqueous media should be handled in a fume hood; solvents such as concentrated acids and alkalis can be corrosive. Disperse your sample in the chosen media and select a cell and electrode combination that is suitable for the medium. A typical concentration results in a slightly turbid sample. The instrument automatically adjusts the light intensity to cope with a wide range of concentrations, so precision is not required. Place about 1.5 ml in the cuvette, carefully insert the electrodes, catching any spillage. Open the trap door, connect the lead to the top of the electrode, and place the cuvette in the holder with the lead going to the right (as you face the front of the instrument).

II.1 The Parameters Dialogue



Sample ID: Unknown Sample

Operator ID: Unknown Operator

Notes:

Runs: 3

Run Time

● Manual:

Cycles: 10

○ Automatic:

Target Residual: 1.0000e-01

User1: 0.00

User2: 0.00

Zeta Potential Model

● Smoluchowski ○ Hückel

Temperature: 25.0 deg. C

Liquid: Aqueous

Viscosity: 0.890 cP

Ref. Index: 1.330

Dielectric Constant: 78.54

Batch #: 0

pH: 7.00

Concentration: 2.00 mg/mL

Particle Size: nm

Auto Save Results ☑

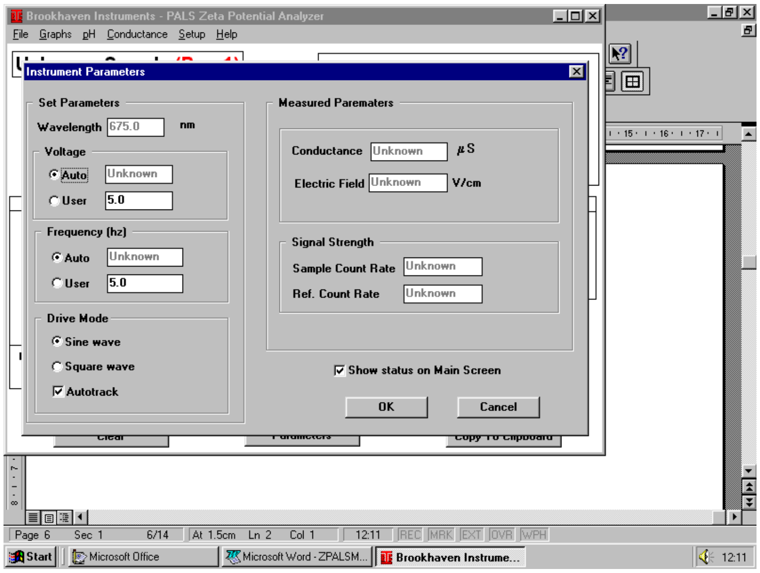
Load the software by clicking on the ***ZetaPALS*** icon. Before starting a measurement click *‘Parameters’* to bring up the dialogue in which these values can be viewed and altered. Enter a description or sample title in the text boxes provided. Particularly, check that the sample’s suspending liquid has been correctly identified under ‘*Liquid*’. Clicking the down-arrow to the right of the ‘*Liquid*’ text entry box will display a list of common liquids for which information has been stored in the database. Selecting the liquid you are using will automatically set the parameter values for viscosity, dielectric constant and refractive index. *Note that these values are used in the calculation of zeta-potential from mobility, and they are also used by the ‘automatic’ mode’s field setting to select an appropriate applied electric field. If you are simply interested in the electrophoretic mobility, the correct values are not necessary*. If the liquid you wish to use is not present in the database, you may manually enter the viscosity and dielectric constant values into the correct fields by selecting “*Unspecified*”. You may add other values to the database yourself by editing the file "PALSDB.TXT" using Notepad. (If you plan to do particle sizing, also get the value for refractive index). Use the ID and notes’ fields so that the sample run can be correctly identified later.

Take care to also select either Smoluchowsky or Hückel for the calculation method. Most nonaqueous media (with the exception of alcohol) require Hückel. If in doubt contact Brookhaven for advice. Note that you also set the temperature for the measurement on this screen. As the viscosity (and maybe the dielectric constant) are quoted at specific values of temperature, be careful that you use an appropriate one. The database is set up for automatic calculation as a function of temperature for the aqueous case and it is suggested that this be used when possible. Note that the suspensions used are dilute and the pure liquid values generally apply. For dispersions in 'ordinary' acids and alkalis and inorganic salts, use the pure liquid (water) value automatically calculated when “Aqueous” is selected.

Note that under ‘Run Time’, the number of cycles can be set by selecting ‘*Manual*’ or ‘*Automatic*’. In the former case, simply enter the number required (typically 10-20). The number of cycles and runs is not critical, but at least 3 runs of 10 cycles should be performed so that an assessment of reproducibility can be made. In the ‘Automatic’ mode, the measurement will continue until the relative residual calculated for the data is less than the value entered here. A typical value would be 0.01.

Close the dialogue box. The values selected will be stored and used until another entry is made and saved.

II.2 The Instrument Parameters Dialogue



Set Parameters

Wavelength 675.0 nm

Voltage
- ● Auto Unknown
- ○ User 5.0

Frequency (hz)
- ● Auto Unknown
- ○ User 5.0

Drive Mode
- ● Sine wave
- ○ Square wave
- ☑ Autotrack

Measured Parematers

Conductance Unknown µS

Electric Field Unknown V/cm

Signal Strength

Sample Count Rate Unknown

Ref. Count Rate Unknown

☑ Show status on Main Screen

If you are not an experienced user go to **‘Setup’/’Instrument Parameters’**and check that ‘Auto’ has been selected for voltage and frequency. Normally leave the default settings of sine wave and ‘*Autotracking*’ unchanged.

You may now begin the measurement by clicking the **‘Start’** *button*.

The instrument will first measure the conductance, then adjust the laser beam intensity passing through the sample to optimize the sample’s count rate (also referred to as the scattered light signal). When data collection begins, you will see the cycles counter increasing and mobility values will appear in the upper portion of the main screen. When the run is complete (number of cycles completed or relative residual achieved) the results will appear in the tabulation chart. The next run then starts. When the designated set is complete, the mean values for the measured quantities are calculated and displayed. Also, the combined value of the total data recorded is summed together. In most instances, if the mean and combined values for mobility and zeta potential agree to about the standard error of the measurement, it is taken to be satisfactory. The residual of the combined value will usually be less than any of the individual runs. These criteria can be taken to indicate a reliable measurement. If a trend of increasing or decreasing mobility is seen, it is probable that thermal equilibrium has not yet been reached and the measurement should be repeated. Ordinarily, we expect to obtain reproducibility of ~5% or better on a well dispersed sample. If a large number of runs (>5) have been performed, you may wish to exclude the highest and lowest values and recalculate the mean and combined results. This can be done by clicking on the run or runs in the tabulated list. The runs removed are *greyed out.* Clicking on a *greyed-out* run will restore it.

Detailed reports are saved in the database folder selected by the user (automatically done if ‘Auto Save’ is checked in ‘**Parameters**’). If you wish to save it somewhere else as well, **‘File - Save As’** gives you the opportunity to select a different folder and also amend the sample ID. Entering a file name ending in .pal (eg. mysample.pal, run1.pal). This is the standard file extension for measurements analyzed by PALS.

Data can later be reloaded by clicking on **‘File/Database’**, and selecting the folder and file name required. If the data is to be re-analyzed, (this would usually be required if the values for dielectric constant or viscosity had been wrongly set), go to the **‘Parameter**’ dialog and alter the values (and title, if required). Click **‘OK’** and the data records in memory will be re-analyzed with the new values. If the new results are to be saved they should be **‘Saved’** into a new file.

Results can be printed using **‘File/Print’** (possibly using **‘File/Printer Setup’** first to establish a printer selection). A standard page format can hold the information seen on the screen after a measurement.

Before making another run, clear the previous one by clicking on **‘Clear’**. If the data has not yet been saved, the user will be warned at this point.

II.3 More Advanced Measurements

The '*Automatic*' mode settings for applied voltage, frequency, and runtime are intended to deal with most samples, but you may need to consider changing these parameters for some situations. The instrument utilizes the values for the dielectric constant and the measured conductance in deciding if it is appropriate to apply a high or low voltage in order to establish a high or low electric field. A high field is appropriate when the ratio of dielectric constant to viscosity for the suspending medium is <20 (see Table 1 in the *Theory Section* of this manual). This ratio predicts if the mobility is low. A high field is only applied if the sample's conductance is less than 5 µS. (This threshold can be adjusted if necessary, but seek advice before attempting to do this). If the user has selected a value for the dielectric constant and viscosity that indicate expectations of low mobility, the instrument will select the high voltage range (defaulting to 100 V, yielding a field of 278 V/cm). Having measured such a sample, if the mobility is very low in absolute magnitude (<.05), you may wish to establish the highest field possible which corresponds to an applied voltage of 200 V across the electrode gap of 3.6 mm (~600 V/cm). To do this go to the **'Setup/Instrument Parameters'** dialogue, click the voltage selection to '*User'* and enter 200. You may also reduce the frequency so the field is established for a longer time period (this will also assist in determining very low mobilities). Click the frequency button to '*User'* and enter 2 Hz, the lowest recommended operating frequency.

You may also increase the number of runs by setting runtime to '*Manual'*. Accumulation time is determined by the number of cycles the voltage is applied. A cycle consists of a burst of two field periods. If the field frequency is 10 Hz this takes 0.2 seconds. Typically about 10 cycles are enough for a single measurement of a 'good' sample. Very low mobilities (or unnedessarily low fields and high frequencies) can lead to small signals require more cycles. The limit is 500 cycles, though in '*Automatic*' mode, the limit is 100 cycles. Once in the '*Automatic*' mode, after performing 5 cycles, additional ones are added until the *'Relative Residual'* (a measure of data fit quality) falls to less than a target value, typically 0.1. A poor fit will generally have a residual >1, <0.1 for a good fit. For *ZetaPALS* ver.1.22 and higher the default settings are 5 Volts and 5 Hz, default settings for unknown samples.

The manual settings may assist in achieving better reproducibility for samples such as those in high salt of conductance > 10,000 µS. The automatic field settings deal with such cases by reducing the voltage and frequency in order to compensate. With such samples, it is a good idea to inspect the electrodes frequently - perhaps making 3 single runs of 10 cycles then changing the sample and cleaning the electrodes as the passage of high current can lead to electrode blackening and the production of bubbles. If this happens excessively, it may be worth using manual settings of 3 volts and 20 Hz to reduce polarization effects. Also, extending the cycles to 20 or even 30 will assist, but be prepared to change the sample after each run, if necessary.

II.4 RESULTS INTERPRETATION

Listed below are important quantities determined by the PALS. Although the following terms are defined in depth in the glossary portion of the manual, a basic knowledge of these quantities will assist the user in data interpretation. The quantities are:

- Electrophoretic mobility - (microns/second) / (volt/cm)
- Zeta potential - millivolts (mV)
- Relative residual - a dimensionless number characterizing the fitted data

II.4a MORE ADVANCED INTERPRETATION

When performing PALS analysis, there are a number of indicators that reveal valueable information regarding the nature of a sample (especially if the sample is unknown). Two of these are the width of the phase graph and the sample's measured conductance.

Width is determined directly from a sample's mobility. The mobility is measured from the amplitude weighted phase difference function (AWPD). The square of the mobility is measured by the amplitude-weighted phase-structure function (AWPS). If the population of particles studied has a spread in mobilities, the square root of the AWPS' mobility will be larger and the difference measures the standard deviation of that spread. The width quoted here is defined as:

[Mobility (AWPS)/Mobility (AWPD)]

A width less than ~0.1 indicates an essentially narrow distribution of mobility. The AWPS is more sensitive to outliers and measurement noise. Therefore, the value for width is only calculated for the combined data set rather than each run.

The conductance of the sample is displayed in the '*Measurement Parameters'* table. In the case of aqueous samples and other polar liquids, conductance is a useful measure of the concentration of the free salt ions (if their concentration is not known in advance.) With the current electrode geometry a 1mM, 1:1 electrolyte yields a conductance in the range of 300 to 330 μS. The relationship between concentration and conductance is very roughly linear up to 10 to 20 mM, but then the conductance rises less rapidly.

III. Data Handling

In this section we outline the facilities for data handling. This will give the user an idea of what is possible, and where to find it. The controls used are standard Windows$^{(TM)}$ features so detailed instructions on how to use them are not given.The most important data handling functions are collected under the File menu.

Under *'Database'* are the functions you will use to save, delete, recall, print, or archive your data from a specified folder. These folders can be created or deleted. It is suggested that each user/experiment have their own folder. This will assist in keeping data in an orderly manner. Data can also be archived and restored from backup disks. Report files containing summaries of measured and calculated quantities can also be created and saved as text.

Each file contains a set of runs. Multiple files can be selected and compared using the Graph menu (main menu bar), to plot an SPC (statistical process control) chart, or to make two-variable graphs can be made. These tools are often useful, for example, if you make runs at a number of different pH values and wish to plot mobility or zeta potential against pH. The isoelectric point (IEP) is defined as the pH at which the zeta potential (mobility) I zero. Not all systems has an IEP even in water, and certainly the IEP has no meaning in any liquid where pH is not well defined.

IV. Theory

The reason for performing a phase analysis measurement may be understood by examining the relationship shown as equation 1. This is the relationship between mobility and zeta potential as a function of the parameters of the suspending liquid: the dielectric constant ε, and the viscosity η. The factor f(ka) is of the order unity. (For a discussion of f(ka) see the appendices to the *ZetaPlus* instruction manual).

$$\mu = \zeta \frac{\varepsilon}{\eta} f(ka) \quad [1]$$

μ is electrophoretic mobility. It is often expressed in practical units of (micron/second)/(Volt/cm), which for typical colloids in polar liquids like water, is typically in the range of 1 to 5. The correct SI formulation of meters^2/(Volt-second) gives these values scaled by a factor of 10^{-8}.

ζ is zeta potential, usually expressed in millivolts (mV).

Table 1 Ratios of ε/η For A Few Common Liquids

Liquid	*ε/η*	***Ratio To Water***
Water	87.55	1
Methanol	61.11	0.7
Toluene	4.25	0.05
Ethylene Glycol	2.35	0.03
Oleic Acid	0.095	0.001

If we now consider a particle system in which the zeta potential is constant, despite the fact that very different liquids (see Table 1) are used to make the dispersion, we see that the mobility that will arise can be much smaller than the value when water is used. In general, dispersions in organic liquids may have mobilities lower by several factors of 10 than aqueous based systems. Low mobilities can also occur in dispersions of high salt content, in higher viscosity liquids, or close to an isoelectric point. Conventional laser Doppler electrophoresis is limited to mobilities of the order of 0.5×10^{-8} m^2/(V·s) and cannot readily be used to measure such low mobilities due to limits of frequency resolution and background drifts in the sample due to thermal fluctuations. However, direct frequency analysis is not the only way of measuring the Doppler shift. If the Doppler signal is compared to a reference signal corresponding to stationary particles using phase quadrature, any small change in frequency eventually gives rise to a change in phase between the signals. This phase change Q(t) can be related to the Doppler shift and hence the electrophoretic mobility in the following way:

Measure a phase Q(t) in the scattered light field at scattering vector q which changes due the movement of particles with mobility μ in an electric field E(t). Given that the scattered light has an amplitude of A,

$$\langle Q(t)-Q(0)\rangle = \langle A\rangle q\mu\int_0^t E(t')dt' \quad . \qquad [2]$$

For the case of a sinusoidal field the integral gives the following result,

$$\langle Q(t)-Q(0)\rangle = \langle A\rangle q\mu\{\cos(\phi)-\cos(\omega t+\phi)\}/\omega , \qquad [3]$$

where ω is the frequency of the applied electric field.

The ZetaPALS hardware incorporates a digital signal processor which can extract Q(t) directly from the Doppler signal. The other quantities are known or controlled so the mobility can be determined and an estimate of zeta potential by applying equation [1] in either the Smoluchowsky (f(ka)=1.5) or Hückel (f(ka)=1) limits.

One of the problems with microelectrophoresis is the presence of small thermal drifts in the sample. These manifest themselves as a collective velocity component added to the electrophoretic motion. Over short periods of time this component may be taken to be constant, but unknown.

If we add the effect of a constant velocity V_c we obtain equation [4].

$$\langle Q(t)-Q(0)\rangle = \langle A\rangle q\mu\{\cos(\phi)-\cos(\omega t+\phi)\}/\omega + V_c t \qquad [4]$$

The effect of the constant velocity is to add a slope to the phase signal. This can be removed experimentally by adjusting the reference frequency appropriately. This is known as '*Autotracking*' and improves the precision of the system. *'Autotracking'* is a useful feature that minimizes convection effects due to sample heating and other parasitic flows by tuning the phase analysis to remove any collective velocity. This shows up as a slope superimposed on the oscillating phase (AWPD) function. It should be left selected (checked) in normal circumstances.

V. Glossary

The most important words and terms you will find when using the *ZetaPALS* are listed and briefly described in this section. These are terms that are specific to the *ZetaPALS* in most cases. Familiarity with the operation of Microsoft Windows$^{(TM)}$ is assumed so terms like 'command button', 'menu item', 'dialogue box' are used without further explanation.

Auto	In the **'Parameters'** dialogue this is associated with automatically setting the run time or number of cycles of each run. In '***Setup/ Instrument Parameters'.*** *'Auto'* buttons instruct the instrument to choose the appropriate values for voltage and frequency based on the sample characteristics.
Autosave	A Check box in the parameters dialogue. If checked results will automatically be saved in the current folder in the file database.
Autotracking	A method by which the *ZetaPALS* improves the precision by continually adjusting the reference signal to remove the effect of thermal drifts in the sample.
Cell	The measurement cell in which the sample dispersion is placed. Into this the electrodes are inserted. About 1.5 ml of dispersion is needed. Cells can be plastic or glass (quartz). The latter are needed for some aggressive solvents in which case Kevlar or ceramic electrode supports must also be used.
Cell Holder	Opening the sliding hatch reveals the square opening into which the cell is placed. The electrode connection lead is also just inside the hatch and should be attached when the cell is inserted. The cell is oriented in the holder with the connector facing the right side of the instrument.
Clear	Remove the current data from memory to allow a new measurement to start.
Combined	A measurement of several runs has all the data (unless specifically excluded by the user) added together to produce a single result. This is the 'combined run'. Individual or combined data can be graphed in the small or Zoomed graph.
Concentration	A field in the **'Parameters'** dialogue where the user can enter a value for concentration (in mg/ml) of the sample. This is not measured by the instrument or required in the calculation of results, but may be used as a dependant plotting variable in the two-variable plots.

Conductance	The conductance of the sample is determined at the start of a measurement to assist in optimising the instrument settings and in calculating the characteristics of electric field to use (by adjusting the voltage and frequency). It is reported in microSiemens (µS). Typical values for aqueous-based samples will range from 30 µS to 30,000 µS. Many solvents will show values of 1 or less. 1 µS is the maximum precision for this quantity in the *ZetaPALS*. Conductance can also be measured by the clicking Conductance on the main menu without then proceeding to a complete measurement of electrophoretic mobility. Conductance must not be confused with conductivity; conductance does not include a cell constant.
Continue	A measurement that has been stopped can be continued. If the original total of runs and cycles is complete, the number of ions can be increased and the measurement continued.
Cycle(s)	A cycle consists of the application of the voltage to the sample and the recording of the change. A 'run' may consist of any number of cycles (up to a max. of 500), though 10 - 50 are typical values for this. The reason that more than one cycle is needed, is that except for very large mobilities, a single application of the voltage does not produce a sufficient 'signal-to-noise' ratio, and hence a reliable value for mobility. The number of cycles in a run is set in the **'Parameters'** dialogue. If *'Auto'* is selected for *'Run Time'* on the parameters dialogue, cycles will be collected and added until the relative residual is less than the target.
Dielectric Constant	A value for the dielectric constant of the medium in which the sample is dispersed is needed in the calculation of zeta potential from electrophoretic mobility. This is entered in the **'Parameters'** dialogue. A table of values for common liquids is installed in the software. The value for this quantity depends slightly on temperature so you may need to operate the instrument at a temperature for which it is available. If not otherwise stated operate at 25 ºC.
Electric Field	A field is generated when a voltage is applied across the electrodes in the cell. This causes the particles in the dispersion to move, allowing the electrophoretic mobility to be measured. The value is computed allowing for the applied voltage, the gap between the electrodes and the characteristics of the drive circuitry. It is stated in Volts/cm and ranges from 0 - 600 V/cm nominally.

Electrode(s)	The electrodes consist of a noble metal (usually palladium) mounted on a machined support. The standard electrodes are mounted on an acrylic support and are compatible with hydrophilic liquids and some inert organic liquids such as paraffins. Aggressive solvent requires the use of Kevlar or ceramic supported electrodes. The support also contains a temperature sensor to measure the temperature near the sample. The electrodes connect to the instrument via a multi-way connector mounted on top of the assembly.
Electrophoretic Mobility	The basic quantity that is measured in microelectrophoresis. It is the average velocity that the particles of the sample dispersion would maintain in an electric field of 1V/cm. Also referred to simply as 'mobility'.
Frequency	The electric field is an AC waveform that switches direction at a rate set by the frequency control. Typical values are 2 - 20 Hz. The lower the frequency the further the particles travel before they reverse direction so the larger the phase signal becomes. Typically this will be selected automatically by the software if the *'Auto'* button on the **'Setup'/'Instrument Parameters'** dialogue is enabled.
Graphs	The Graphs menu item allows the user to produce two-variable plots (e.g. mobility versus pH) and the 'statistical process control' or SPC plot which is essentially mobility (or zeta potential) vs. time.
Hückel	One limit for calculating zeta potential from the measured mobility using the dielectric constant and viscosity of the suspending fluid, the Hückel equation applies to non-aqueous based systems or very small particles. See the discussion in section IV of the *ZetaPlus* manual.
Liquid	The parameters dialogue has a database of different liquids to provide parameters such as viscosity, dielectric constant, and refractive index.
Log File	The user can specify which quantities are stored in a simple text summary of all that the instrument has measured since the log file was reset. This file (normally c:\bicw\palsw\data\logfile.txt) can be printed, viewed or plotted using standard software or locally produced programs.
Manual	Setting for 'frequency' and 'voltage' in the **'Setup Instrument'** dialogue to allow the user to control the voltage applied to the cell during the measurements.

Mobility	See Electrophoretic Mobility.
Notes	See Sample ID and Operator ID.
Operator ID	In the parameters dialogue the user can enter text for this purpose.
Parameters	Values set by the user that describe the characteristics of the sample and the duration of the measurement process. The dialogue box where these values are supplied is brought up by clicking the **'Parameters'** command button. Include sample ID, operator ID, dielectric constant, viscosity, etc.
Particle Size	A field in the parameters dialogue where the particle size (in nanometers) can be entered. This could be the value measured with the PSDW software on the *ZetaPALS* itself. This is purely a label at present. Some methods of calculating the zeta potential can use this information so it is included for convenience in the database. It can be left set to 0 in many cases as it is not used with current Brookhaven software.
Phase/Phase Analysis	The phase signal measured by the *ZetaPALS* is plotted on the main screen. Phase is the integral of frequency so, the plot of phase against time is a track of the changing value of the Doppler frequency shift as the AC field changes.
pH	The *ZetaPlus* is fitted with a BNC connector to which a pH probe can be attached. The software allows the probe to be calibrated and measurements of pH performed under manual control. This pH value is then inserted into the parameter list and is saved in the database and optionally in the log and report files.
Print Report	The current measurement on the screen is sent to the system printer.
Ref. Count Rate	The intensity of the reference beam expressed in 1000's of photon detections/second. The reference beam is optically mixed with the scattered light from the sample to produce the total optical signal on which the phase analysis is performed. In effect the reference beam provides a signal corresponding to fixed- speed, virtual particles. Moving particles in the sample produce a relative change in optical phase that is synchronous with the driving electric field and this synchronous movement can be distinguished from random thermal and other movements by the PALS processing.

Refractive Index	A value for the suspending liquid can be entered in the parameters dialogue. It is also recalled from the 'Liquid' database. It is used in calculating the q vector and true scattering angle in the sample medium.
Relative Residual	The phase function consisting of discrete values of phase plotted at regular measured intervals along a time axis is fitted to the experimentally measured phase points in order to extract the mobility of the scattering sample. The sum of squares of the difference between measured and fitted data divided by the total amplitude of the fitted data represents the quality of the fit and is therefore defined here as the relative residual. A small relative residual (of the order of 0.01) represents a good fit and a reliable result. Large values of residual imply that the experimental data is noisy and longer measurements (more cycles) or a larger voltage or lower frequency may be necessary.
Report File	The user can select individual runs from the database and include a selection of the measured quantities and parameters in simple text file suitable for import into spreadsheets and word processor documents.
Runs	When **'Start'** is clicked, the instrument performs a measurement consisting of a number of runs, each divided into a number of cycles. The main reason for dividing a measurement into several runs is to demonstrate the reproducibility of the measurement. All the runs can be added together and analyzed as if they were a single run to obtain a single result. Individual runs can be excluded by clicking on the values given in the tabulation on the main display.
Sample ID	A text string to identify the sample currently in the instrument and to use as a useful tag for later identifying the data.
Smoluchowsky	One limit for calculating zeta potential from the measured mobility using the dielectric constant and viscosity of the suspending fluid, the Smoluchowsky equation usually applies to aqueous based systems with large particles and at least a moderate salt concentration. See the discussion in section IV of the *ZetaPlus* manual.
Standard Error	The tabulated quantities (mobility, zeta potential, relative residual) are analyzed statistically to calculate the mean and standard error of the mean using the normal definitions of these quantities. Standard error equals the standard deviation divided

	by the square root of the number of measurements.
Start	Start collecting data. Initially the sample conductance is checked then the strength of the scattering and reference signal. The intensity of the laser beam that produces the scattering signal is adjusted to an optimal value, if possible. (The reference beam should be between 2 and 20 times stronger than the scattering signal ideally.)
Stop	Stop collecting data.
Target Residual	During data collection as each cycle is collected it is added to the previous total and the data reanalysed. If *'Runtime'* has been set as *'Auto'*, then the relative residual must be less than the target value before the run stops. (The number of cycles collected must also be >5 and <100.)
Temperature	The set temperature that the instrument regulates. Since viscosity (and other parameters) vary with temperature it is important that the sample temperature is stabilized to better than 0.5 ºC. Changed under **'Parameters'** dialogue.
User	Buttons on the **'Setup'/'Instrument/Parameters'** dialogue, that enable the user to enter values for the voltage and frequency.
Voltage	The voltage across the electrodes that generates the electric field. Ranges from 0 to 200 volts. Typically 5 V (max. 10 V) for aqueous samples, 100 Vor more for non-polar liquids.
Viscosity	The value of viscosity (in centipoise, cP) of the suspending liquid at the set temperature. The database (under '*Liquid*' in the **'Parameters'** dialogue) has some useful values for a range of common liquids, including water. The viscosity is calculated for a modest range of temperature (5-70 ^{o}C).
Wavelength	The wavelength of the laser source in nanometers. Displayed in the **'Setup-Instrument'** dialogue.
Zeta Potential	A quantity (expressed in mV) calculated from the electrophoretic mobility. Depends on the viscosity and dielectric constant of the medium, and the choice of Hückel or Smoluchowsky models. See *ZetaPlus* Manual section IV and the Appendices for more details. This value is calculated

	because it can be used in theoretical predictions of dispersion stability.
Zoom	Expand the graph of the experimental and fitted phase data to fill the main form.