

Charge Analyser II

Operating Manual

Software Versions: 2.00 - 4.10

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FCC Notice

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy, and if not installed and used in accordance with the instructions manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Caution: any changes or modifications to the equipment not expressly approved in this manual may void the user's authority to operate the equipment

European Community Compliance

The Charge Analyser conforms to the following product specifications:

EMC	EN50082-1 (Immunity)
	EN55014 (Emissions)
LVD	BSEN61010

1. Getting Started

Thank you for purchasing Rank Brothers equipment. Before use, please ensure that you have read and understood both this operating manual and that for the Dosimat. Store these manuals in a safe place for future reference.

1.1 Do Not

- Do not plug into your local mains supply until you have checked that your local supply voltage matches that stated on the label at the rear of the instrument (adjacent to the mains inlet connector).
- Do not change the fuse or remove any covers with the mains inlet lead connected.

1.2 Do

- Do ensure that if the moulded plug is removed from the mains inlet lead it is disposed of safely.
- Do read and understand this manual before use.

1.3 Connection to your Mains Supply

Important: This unit must be earthed to ensure operator safety. The mains inlet lead may have a moulded plug fitted that is not suitable for connection to your local supply. If it is necessary to remove this plug and fit a suitable one, the removed plug must be safely disposed. The removed plug would present a serious shock hazard if plugged into a suitable supply with the bare wires exposed.

The wires of the mains inlet lead are coloured as follows:

GREEN and YELLOW	EARTH
BLUE	NEUTRAL
BROWN	LIVE

As the colours of the wires in the mains lead may not correspond with the coloured markings identifying the connections in your plug, proceed as follows:

- The wire coloured **GREEN and YELLOW** must be connected to the terminal in the plug marked with the letter **E** or the earth symbol or coloured **GREEN** or coloured **GREEN and YELLOW**.
- The **BLUE** wire must be connected to the terminal marked **N** or coloured **BLACK**.
- The **BROWN** wire must be connected to the terminal marked **L** or coloured **RED**.

Before connecting the unit to the local power supply it is important to ensure that the voltage selector switch on the rear panel is set to suit your local supply voltage.

If it is necessary to adjust the voltage selector switch, the power lead must first be removed from the control unit, and the fuse must be replaced by one of the correct type

and value. Failure to use the correct fuse may jeopardize operator safety, and may cause the control unit to fail to operate. The correct fuse is as follows:

220V/240V	T160mA 20mm
110V/120V	T250mA 20mm

If the power plug is a fused type, it should be fitted with a 3A fuse.

Ensure that the Metrohm Dosimat 665 is also set for your local supply before connection. Refer to the supplied Metrohm manual for further information.

The unit contains no user serviceable parts. The cover should only be removed by competent personnel, after first switching off the power supply and disconnecting the mains inlet lead.

For any servicing or repairs, the unit should be returned to the manufacturer with a covering letter. Please ensure it is carefully packaged to avoid damage during shipment.

2.Setting up the Instrument

Before setting up the instrument for the first time, please refer to the parts list in Appendix V to ensure that no parts are missing. The main components of the Charge Analyser II (CAII) are as follows:

- The CAII control unit. This is the main box containing the electronics and the power supplies, display, keys, etc. for the instrument.
- The Streaming Current Detector (SCD). This is the blue unit, which incorporates the SCD cell, motor and rotational sensors into the stand (optional).
- The Photometer Cell (optional).
- The Metrohm Dosimat 665. This is a precision dosing system and comes complete with a magnetic stirrer and two 10ml exchange units. (The Metrohm instruction manual gives a full description of the unit and its operation.)

Important: All the appropriate connections to both the CAII control unit and the Dosimat should be made before switching on either unit.

2.1Connections to the CAII Control Unit

The CAII control unit controls the whole instrument and thus has the majority of connections. All of the connections, except one, are made via connectors on the rear panel. The numbers below refer to the labels in Figure 1:

1. **The power supply inlet connector** should have the local power supply lead plugged into the bottom.
2. **The power on/off switch** is located in the middle of the power supply inlet connector.
3. **The fuse holder** is located in the top of the power supply inlet connector and should always have the correct fuse fitted.

4. **The supply voltage selector switch** should be set to match your local supply voltage (see Section 1 for further information).
5. **The printer output connector** enables the control unit to be connected to any printer with a standard parallel interface. Any IBM compatible parallel printer lead with a 25-way plug for the computer port should be suitable to connect your printer to the control unit.
6. **The Dosimat interface** is connected, via the lead supplied, to socket A at the rear of the Dosimat. N.B. should the CAII control unit appear to lock up while displaying *Dosimat filling* it may be caused by this lead not being correctly connected (see Appendix IV).

2.2 Additional Connections for the Streaming Current Detector Cell

The Cell Motor Output socket connects to the cell motor via the cell motor lead (7-pin DIN plug to 7-pin DIN plug), the other end is plugged into the cell on the underside of the blue cover. To connect the plug, push it into the socket by the black plastic part at the rear of the plug. To disconnect the plug it is necessary to remove it by pulling the silver metal part only. This prevents excess strain on the connections to the pins. The cell socket on the front panel connects the control unit to the removable part of the Streaming Current Detector cell using the supplied BNC-to-BNC lead.

2.3 Additional Connections for the Photometer Cell

The Photometer cell connects to the rear of the instrument in the **Cell Motor Output socket**. The front panel socket used with the SCD cell is not required.

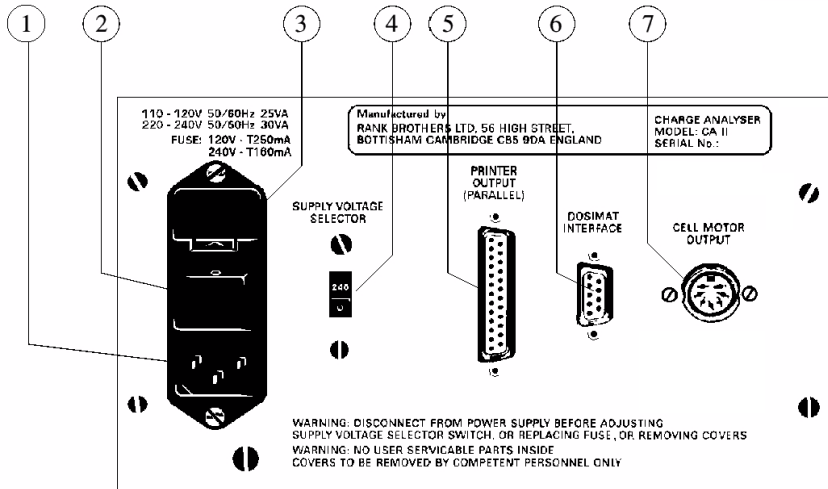


Figure 1 - The rear panel of the CAII control

2.4 Connections to the Dosimat 665

The Dosimat has four connections to be made from its rear panel as follows:

- **Socket A** connects to the Dosimat interface socket at the rear of the control unit via the cable supplied.
- **Socket B** connects to the RS 232 interface; it does not require connection to the control unit.
- **Socket C** connects the remote keyboard to the Dosimat.
- **Socket D** connects to the dosing button accessory. This does not need to be connected, as the keyboard provides a full function remote control for the Dosimat.
- **Socket E** connects to the stirrer unit via the appropriate lead (3-pin DIN plug to 5-pin DIN socket).
- **The power inlet connector** connects the power lead to your local supply.

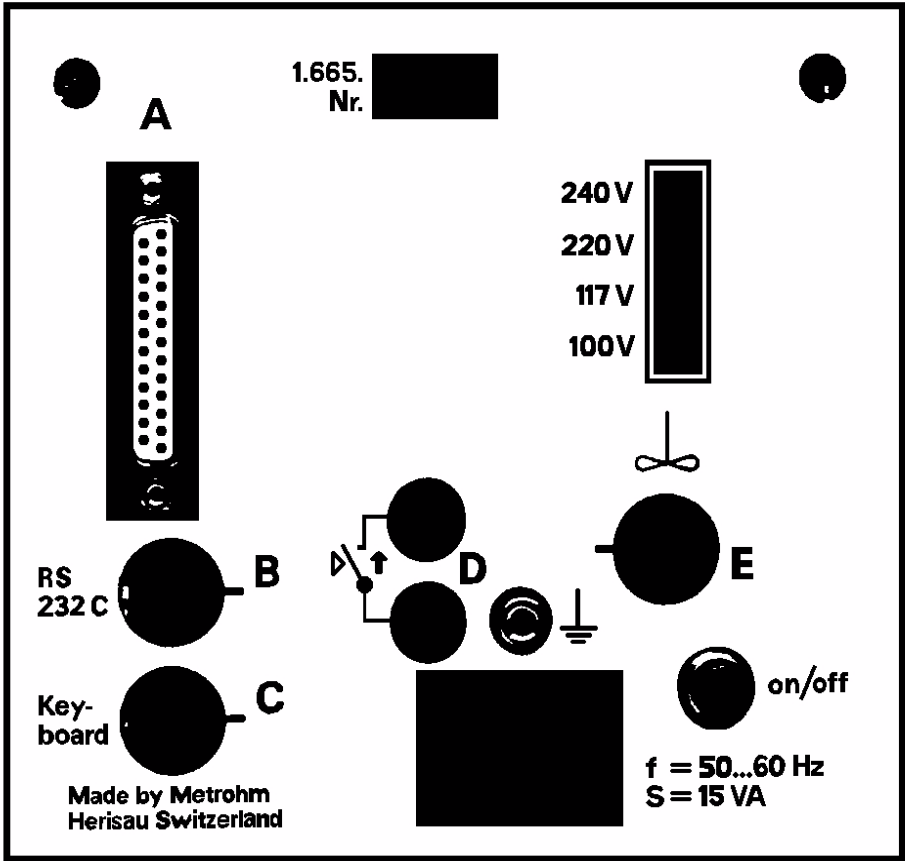


Figure 2 - The Dosimat 665 rear panel

3.The Controls and Displays

3.1The Control Unit (when using the Streaming Current Detector Cell)

See Section 6 for the relevant information when using the Photometer Cell.

The CAII is operated by four keys on the front panel, with information displayed on the two line Liquid Crystal Display (LCD). In addition there are two LEDs and a Buzzer. The keys have no repeat or roll over action, if a key is pressed and held, no other key will have any effect until the first key is released. If the control unit appears not to respond to any key presses check that one of the keys has not become jammed down.

The <MOTOR> key is used to start the cell motor. When the control responds to the key, it illuminates the LED in the switch to show that the motor should be running. Note that the motor switches off automatically after approximately 4 minutes if the titration has not been started. This is to ensure that the motor cannot inadvertently be left running for long periods.

The <TITRATE> key will start an automatic titration of the sample. The key is only active when the cell motor is running. To activate a titration hold down the key until its LED is illuminated, showing that the titration is in progress.

When the <PRINT> key is pressed, titration data will be sent to any printer connected to the parallel printer output at the rear of the unit. While printing is enabled, the <PRINT> key LED will be illuminated. Press the <PRINT> key a second time to stop printing, the LED will be extinguished. N.B. if the CAII is unable to send data to the printer, it will beep twice, an error message will be displayed, the <PRINT> key LED will automatically be extinguished and the titration will continue. The following information is printed after each reading:

- The charge value.
- The total amount of titrant dosed into the sample.
- The phase of the signal (between ± 180 degrees).

N.B. the <PRINT> key is only active while the cell motor is on and printing only occurs during a titration.

The <STOP> key can be used at any time and will switch off any of the other three keys that are on. It may be necessary to hold the key for a second or so until the LEDs are extinguished indicating that the control unit has responded. The motor may not stop immediately after the LEDs are extinguished but should stop within a second or so, this is normal. The action of the <STOP> key depends on which point the control unit has reached in its program sequence when the key is pressed:

1. If the cell motor is on but titration has not commenced then the key will simply stop the motor.

2. If the CAII is titrating then all three LEDs (if illuminated) will be extinguished and an error message, showing that the titration has been aborted, will be displayed.

The **DOSING LED** is a green LED and is illuminated whilst the Dosimat is actually dosing standard polymer into the sample.

The **TITRATION END LED** is a red LED and will flash during the following circumstances:

- An automatic titration is successfully completed. The LCD alternately displays the end point dose and charge, and the last charge and dose of the titration.
- The <STOP> key was pressed during a titration. The LCD displays:

```
** Titration aborted **
```

- More than 9.7ml of titrant was dosed into the sample without finding an end point. The LCD displays:

```
** Endpoint not found **
```

At this point pressing any key causes the Dosimat to refill and the CAII is then ready to perform the next titration (once the cell has been cleaned). N.B. the motor is automatically stopped just before the LED starts to flash.

The **LIQUID CRYSTAL DISPLAY** has 2 lines of 24 characters each and is used as follows:

- When available the top line always displays the charge and volume of standard polymer dosed into the sample, otherwise n/a is displayed.
- The second line shows help information or error messages depending on what is required.

The **BUZZER** is used to give warnings as follows:

- One beep indicates that the key being pressed is not active e.g. the <TITRATE> key whilst the cell motor is stopped.
- Two beeps indicate a problem with the printer.
- Three beeps indicate that an automatic titration has finished, or has been aborted.

3.2The Dosimat

It is suggested that the following instructions be read in conjunction with the Dosimat instruction manual. Programming of the Dosimat is carried out from the remote keyboard. The unit is shipped set in the repetitive dose mode, thus when powered up should display:

```
DIS R 0.000ml
```

In this mode the unit will dose a fixed volume when <GO> is pressed, then will refill automatically and be ready to dose another fixed volume the next time the <GO> button is

pressed. The unit has been programmed to dose at a speed set by the $\langle dV/dt \rangle$ knob on the front of the unit, with a default dose of 2.0ml, which can easily be altered as follows:

1. Press the $\langle \text{DELTA VOLUME} \rangle$ key (top row, middle key). The display will show the current programmed value. To accept this value press $\langle \text{ENTER} \rangle$, otherwise:
2. Key in the new volume required, then press $\langle \text{ENTER} \rangle$ to confirm. N.B. the exchange units supplied can only dose 10ml maximum. Entering a higher value causes the unit to default to 10ml.
3. The new volume is now programmed in and will be remembered even after the Dosimat has been switched off.

Should the Dosimat not be in the Repetitive Dose mode, or should you require a different mode, press the $\langle \text{MODE} \rangle$ key repeatedly until the display shows the required mode, then press $\langle \text{ENTER} \rangle$ to confirm. The mode will be remembered after switching off.

Whilst the CAII is titrating, the Dosimat is being controlled exclusively by the CAII, this being shown on the Dosimat by the display changing to *PULSE*. The CAII then has complete control of the Dosimat, it will no longer respond to any key presses on the remote keyboard or front panel. At the end of the titration the Titration End LED will flash on the CAII. Pressing any key on the CAII causes the Dosimat to be refilled and then revert to the repetitive dose mode (or whatever mode it was in prior to the titration).

N.B. V_{lim} must be off for the CAII to titrate correctly, because the CAII is unable to detect the value of V_{lim} . If V_{lim} is set to say, 4.00ml, the Dosimat will stop dosing at 4.00ml although the CAII, being unaware of this, will continue titrating and give an incorrect endpoint dose. The CAII is able to control the Dosimat regardless of the mode, dose speed, dose volume, etc. that the user has selected

The Dosimat is supplied with two Exchange Units complete with reagent bottles and dosing pipettes. Normally these would contain your standard anionic and cationic polymers. This enables you to easily titrate a sample with either polymer or to dose a known volume of one polymer then 'back titrate' with the other polymer. The exchange units can only be removed from the Dosimat when they are full (it may be necessary to operate the $\langle \text{FILL} \rangle$ button). The exchange unit will then slide forwards and lift off, and the second exchange unit can be slid on until it latches in place and the Dosimat display then reverts to the normal mode.

To ensure correct operation the Dosimat should be powered up *after* the CAII control unit. If the Dosimat appears to initialise incorrectly, check that the CAII control unit has been switched on first. If problems still occur then as a further check, the CAII control unit should be disconnected before switching on the Dosimat.

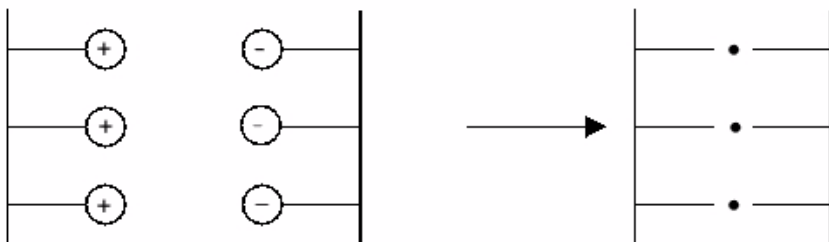
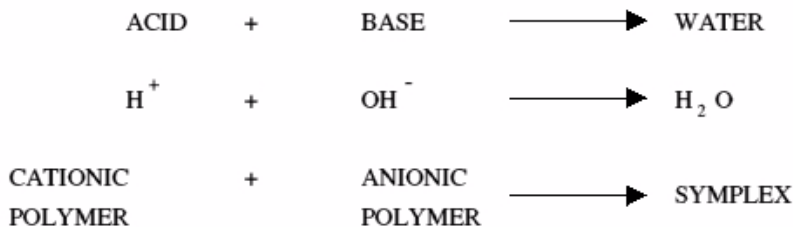
N.B. the Dosimat has several versatile modes and can be used separately from the CAII for other laboratory functions. If it is not being used with the CAII then it should be disconnected before being used independently to ensure the unit powers up correctly. The Dosimat manual gives additional information on independent use.

4. Principles of Operation

The Charge Analyser II (CAII) is able to carry out a fully automatic polyelectrolytic titration of a sample and is therefore able to determine the concentration of ionic charges bound on polymers in solution. N.B. the instrument is designed to test liquids only and samples containing particles will need to be filtered, to remove particles larger than 1 micron, prior to carrying out a titration.

4.1 Polyelectrolyte Titration

The principle of polyelectrolyte titration is based on the fact that the standard polymers chosen form 1:1 compounds with each other in relation to their charge, therefore corresponding to an acid/base neutralisation thus:

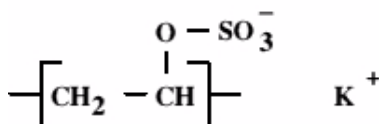


In acid/base titration the acid or base content of an unknown sample is determined by consumption of a standard base or acid, as appropriate, until the point of neutrality is reached. In polyelectrolyte titration a standard anionic or cationic polymer is titrated with an unknown sample until a point of zero charge is reached.

4.2 The Standard Polymers

The standard polymers recommended for use in the titration are Potassium Polyvinyl Sulphate and Hexamethrine Bromide:

- Potassium Polyvinyl Sulphate (PVS_K) is used as the standard anionic polymer and the molecular weight of the monomer is 162.



- Hexamethrine Bromide (Polybrene) is used as the standard cationic polymer and the molecular weight of the monomer is 374.

4.3 The Streaming Current Detector Cell

The Streaming Current Detector cell (SCD cell) determines the charge of the sample and hence the end point of the titration. The cell (see Figure 3) consists of a precision bore cylinder closed at the bottom end and containing two electrode rings, one at the bottom, and one approximately a third of the way up. The electrodes are connected to the socket near the top of the cell and measure the streaming current induced. Slightly above the upper electrode there are a set of holes to allow the sample in and out of the cell, with a single hole above these acting as a vent hole to prevent sample being pumped into the motor/cover area.

A precision piston oscillates up and down in the cylinder with a frequency of approximately 4hz. Polymers, having a tendency to adsorb onto interfaces, become attached to the piston and cylinder walls. The mobile counterions of the fixed electrolyte are carried away in the liquid stream creating an electric current due to the partial charge distribution measured between the two electrodes. This electric current is measured by the electronics in the control unit and used to control the titration.

N.B. because the polymers adhere to both the piston and cylinder walls during a titration the accuracy of the titration is dependent on the cleanliness of the cell and the operator's ability to remove all traces of the previous sample between titrations (see Section 5).

4.4 The Titration with the Streaming Current Detector Cell

The control unit is microprocessor controlled and linked to both the SCD cell and a Metrohm Dosimat 665 precision dosing system, giving complete control of the titration.

The sample to be analysed is first diluted in deionised water and would normally be given a positive charge by adding known volumes of standard cationic polymer if not already positive (back titration). The sample is now titrated, with the control unit monitoring charge readings from the cell and dosing a volume of standard polymer into the sample, based on their magnitude. The charge value is allowed to stabilise and then the control unit doses another volume of polymer based on the new charge reading.

This process continues until the phase of the charge signal has passed through zero (i.e. the phase has changed sign from the value at the start of the titration). The control unit will then take about 16 more readings before turning off the cell motor and displaying the end point dose and charge value of the titration. This end point dose is calculated by the dose at the minimum charge value during the titration.

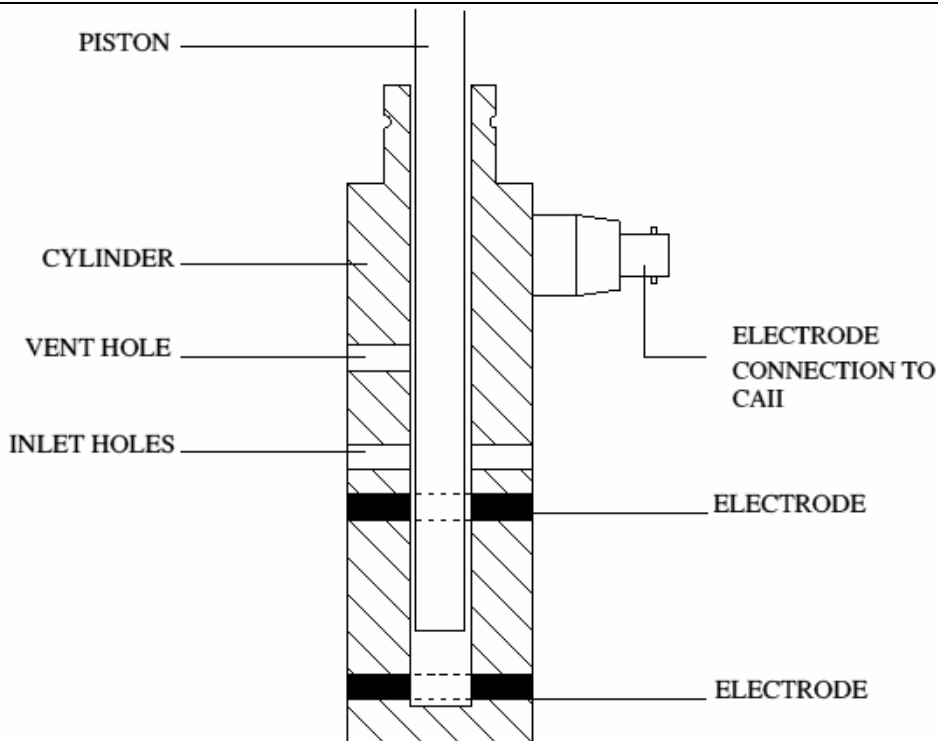


Figure 3 - The streaming current detector cell

5. Performing a Titration with the Streaming Current Cell

5.1 Cleaning the Streaming Current Detector Cell

Before starting a titration it is extremely important that the cell is completely clean to ensure that any sample from a previous titration is removed. Cleanliness of the cell will ensure good repeatability and accuracy of titrations and should be carried out after every titration. The following cleaning procedure may be found helpful:

1. Disconnect the lead from the cell then remove the cell from the stand by lifting the knurled locking ring and sliding the cell downwards off the piston (see Figure 4).

Figure 4 - Operation of cell locking ring

2. The cell should then be brushed vigorously under running water using a stiff test tube cleaning brush. N.B. ensure that no water enters the BNC socket by holding a finger over it during washing, and do not completely immerse the cell in water.
3. The cell should then be brushed out with a dilute solution of household bleach (about 10% bleach in water) for about 20 seconds.

4. Finally flush out with distilled water. N.B. do not leave the cell immersed in bleach as this may affect the stainless steel top.
5. The cell can now be refitted to the stand by lifting the knurled ring and sliding the cell over the piston until it is fully pushed into the stand. The knurled ring can now be lowered, locking the cell in place.

5.2 The Titration

1. Connect the control unit to the Dosimat and cell (see Section 2) and connect to your local supply.
2. Switch on the CAII control unit followed, after a couple of seconds, by the Dosimat 665. The CAII will turn on all the LEDs and its buzzer to ensure they are all functioning correctly and the display will show a start up message with the software version number and the date released (e.g. Version 3.00 1991).

N.B. due to the random nature of the electronics at power up it is possible that the motor may rotate for a couple of revolutions immediately before the start up message appears. This is part of the normal operation and should not cause concern.

3. At the end of the initialisation period the LEDs and buzzer will switch off and the following is displayed:

```
** Checking cell type **  
**** Please wait ****
```

Then after a few seconds the following display will appear:

```
Chge: n/a Dose: n/a ml  
Press <MOTOR> when ready
```

The `Chge` refers to the value of the charge. The `Dose` refers to the volume of titrant added to the sample (this corresponds to the volume displayed on the Dosimat). `n/a` indicates that a value is not available for display.

Unless it has been reprogrammed to a new mode from the keyboard or has required an initialisation, the Dosimat should be displaying:

```
DIS R 0.000ml
```

Refer to the Dosimat manual for further information.

4. Holding a plastic cup over the cell, the stirrer unit of the Dosimat can be swung underneath and the cup then placed on the stirrer unit. The cup should be filled with distilled water so that the ring of inlet holes on the cell is covered by about 3mm of water (approx. 140ml). The magnetic stirrer bar should be added to the cup, the stirrer

unit switched on and the speed set to approximately 5, ensuring the sample is kept adequately mixed during the titration.

5. Press the <**MOTOR**> key to start the cell motor and the display will now show:

```
Chge: XXXX.X Dose: n/a ml  
Press <TITRATE> to start
```

XXXX.X is the value of the charge from the cell, with negative values indicating the sample is anionic and positive values indicating the sample is cationic. The Charge Value displayed is arbitrary and has no units. Note that pressing any of the other keys will cause the control unit to beep and ignore that particular key.

If the cell is completely clean the charge value will be somewhere between -250 and -350 approximately. Indications of a dirty cell will be a low reading, below about -250, or a high reading that slowly drops to below -250. Various factors determine this initial charge value e.g. quality of the water, motor speed, cell wear etc., thus it may be that a particular instrument always has an initial value of say -400, whereas another instrument will have an initial value of -200. Providing that the value is stable and does not start at say -400 then drop to -200 then it can be assumed that the cell is clean and a titration can then be started.

If there is any doubt then it would be wise to re-clean the cell and start with a completely fresh cup of distilled water and compare the charge values. To do this, press the <**STOP**> key to stop the motor. The CAII will then revert to the state at step 3 above, and the cell can be cleaned. The titration should then be continued from step 3.

6. A known volume of sample is now added to the distilled water in the cup. This would normally be between 10ml and 50ml depending on the sample. It should be noted that whilst larger volumes of sample will theoretically give a more accurate titration, higher concentrations of sample may not give a satisfactory titration. Providing the titration requires at least 1ml of standard polymer to reach an end point then reasonable accuracy should result.
7. The pH of the sample should now be noted in case a correction factor is required for the charge calculation (see Section 8), and also the charge value. If the sample is anionic (the charge reading is negative) a precise volume of standard cationic polymer should be added (normally 2 - 5ml of Polybrene) so that the charge reading will now be at least +100. If the sample is cationic (the charge reading is positive) a precise volume of standard anionic polymer should be added (normally 2 - 5ml of PVSK) so that the charge reading will be at least -100. The addition of a precise volume of standard polymer is easily carried out by loading the appropriate exchange unit onto the Dosimat and then, using the DIS R mode to dose into the sample a fixed volume. The volume dosed can be adjusted with the <**DELTA VOLUME**> key on the remote keyboard. Setting this to a volume such as 1ml allows dosing of 1ml amounts of
-

standard polymer by repeatedly pressing the Dosimat <GO> key until the charge has completely reversed.

8. The exchange unit containing the oppositely charged standard polymer should now be loaded onto the Dosimat ready to start a titration. Ensure that the pipette tip has been positioned in the plastic cup. If a print out of the full titration is required the <PRINT> key should now be depressed until its LED is illuminated. The titration is now started by pressing the <TITRATE> key until its LED is illuminated. The bottom line of the LCD now displays:

```
*** Titrating ***
```

This shows the titration is in progress and after dosing some standard polymer the top line will show the current amount dosed, with the green dosing LED illuminated whilst the Dosimat is actually dosing.

The Dosimat display will change to:

```
PULSE X.XXX ml
```

X.XXX being the amount so far dosed. N.B. whilst in pulse mode the Dosimat will not respond to any key presses on the remote keyboard.

9. The titration will now continue with the charge value dropping towards zero. The charge should then reverse (when the absolute charge value has dropped below about 20) and the titration will continue for another sixteen readings. Whether the titration was successful or not, the CAII will switch off the cell motor, beep three times, all the key LEDs will be extinguished and the red titration end LED will flash. There are four possible situations when the above will occur:
 - The end point has been found (a successful titration).
 - The stop key has been pressed during the titration.
 - More than 9.7ml of titrant has been dosed into the sample (the exchange units supplied will only dose a maximum of 10ml thus the control unit stops before this happens). This error normally occurs when either the titration has been carried out using the wrong standard polymer, the sample was too concentrated (repeat the titration adding less sample at step 6 above) or the pipette tip was not in the plastic cup.
 - A fault occurs with the cell.

If the titration was successful the LCD will alternate between the following two displays:

```
Chge: XXXX.X Dose: Y.YYY ml  
Press any key to continue
```

and

```
*** End point values ***
```

Chge: AAAA.A Dose: B.BBB ml

Where XXXX.X and Y.YYY are the charge value and amount of titrant dosed when the titration was stopped, and AAAA.A and B.BBB are the charge value and amount of titrant dosed at the end point of the titration.

If the titration was unsuccessful the top line of the LCD will display the charge value and the amount of titrant dosed when the titration was stopped and the second line of the LCD will alternate between:

Press any key to continue

and one of the following three messages which show why the titration has ended:

Titration aborted - if the <STOP> key was pressed during the titration
or

End point not found - if more than 9.7ml of titrant was added
or

Cell fault - if a problem occurred with the cell

N.B. the last amount dosed will be different to the end point dose as the control unit always titrates past the end point. The Dosimat will be frozen at the total amount of titrant dosed, remaining in pulse mode until a key is pressed on the CAII.

10. The end point dose (value B.BBB above) should be noted along with the pH value, these are used in the calculation of the quantitative value of the charge of the sample (see Section 8).
11. Pressing any CAII key will now cause the Dosimat to be refilled. When complete, both the CAII and the Dosimat will revert back to their displays as at step 3. The cell can now be cleaned and a further titration carried out if required.

6.Performing a Titration with the Photometer Cell

6.1Finding the End Point with the Photometer Cell

6.1.1Principle

The photometer cell attachment for the Charge Analyser II allows a colourimetric titration, employing the same polymers as with the streaming current detector and using a coloured indicator.

The photometer detects a change in transmission of a constant wavelength light source during the titration. The wavelength of the light is such that blue coloured materials (such as a blue dye in solution) absorb it.

The indicator used (Toluidine Blue O) is a cationic blue dye and changes colour from blue to pink as the titration progresses from cationic to anionic. At the start of a titration, when the sample liquor is blue, the liquor will absorb more light and hence the transmitted signal will be low. Conversely, when the indicator turns pink as the titration progresses, less light is absorbed and more transmitted and hence the signal from the detector increases. It is from this increase in transmitted (detected) light that the photometer is able to find the end point of the titration.

6.2 Description of the Equipment

The Charge Analyser with photometer cell consists of three parts: the photometer cell, the control unit and the metering unit. The photometer cell and the metering unit are connected to the control unit by a lead.

6.2.1 Photometer Cell

The photometer cell contains a light source that emits yellow light (this wavelength of light is absorbed by blue colours). The light passes into the sample liquor and returns via a mirror to a detector. (See Figure 5.) The detector measures the amount of light it receives (the amount of light transmitted) and converts this into an electrical signal. This signal is sent to the control unit via a cable for evaluation.

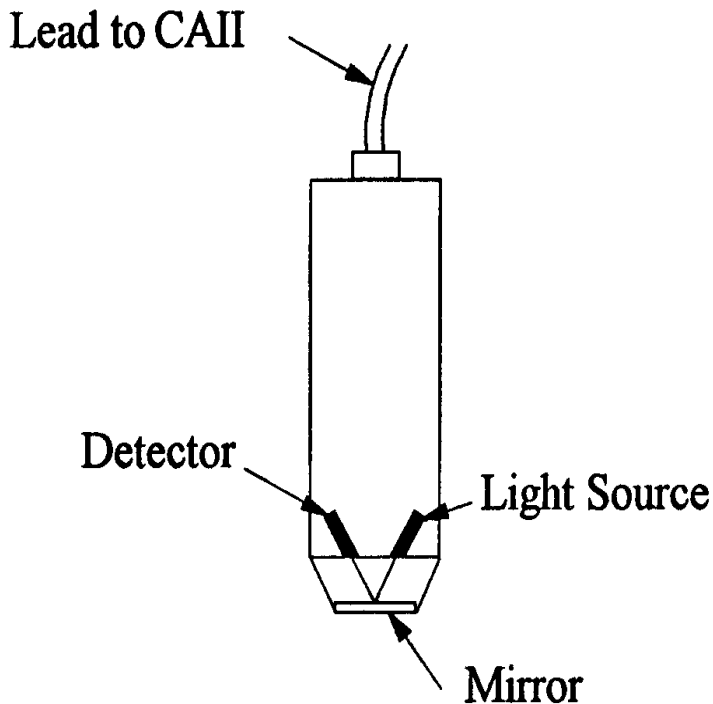


Figure 5 - The photometer cell

6.2.2 Metering Unit

The metering unit is described in detail in the enclosed documentation from Metrohm. It is connected to the control unit via the RS 232 interface. During titration, the CAII changes the Dosimat to pulse mode, thereby having total control over polymer dosing.

6.3 Operation of the CAII with the Photometer Cell

See sections 2.1, 2.3 and 3 for the connections and controls for the CAII and Dosimat 665. The unit should be switched on for 5 minutes, with the cell immersed in a sample, prior to a titration to allow the cell electronics to stabilise.

1. Switch on the CAII, followed by the Dosimat 665. The CAII will turn on all the LEDs and its buzzer and display a start-up message, CHARGE ANALYSER, with the software version number and the date released. At the end of the initialisation period, the LEDs and buzzer switch off and the following is displayed:

```
** Checking cell type **  
**** Please wait ****
```

If the photometer cell is plugged into the back of the CAII (and after the CAII has checked which cell type is being used), the display will show:

```
Trns: n/a Dose: n/a ml  
Press <MOTOR> when ready
```

On the top line of the display, `Trns` is the transmitted light signal coming from the cell and is an arbitrary value with no units. `Dose` refers to the amount of titrant added during the titration and corresponds to the display on the Dosimat during titration. The bottom line of the LCD gives the instructions telling the operator which key to press. When the above values are not available, the letters `n/a` are displayed.

The Dosimat should display:

```
DIS R 0.000ml
```

This display is shown if the Dosimat has not been reprogrammed from the keyboard or has not required an initialisation. (If in doubt, consult the Dosimat manual.)

2. A plastic cup should be filled with distilled or deionised water (fill it to the same level each time, about 3/4 full is adequate). The cup should then be raised up over the photometer, which is at an angle, and placed on the stirrer unit of the Dosimat. The magnetic stirrer bar should be added and the Dosimat stirrer switched on. It is important that no air bubbles are caught in the photometer window as this prevents the detector from picking up the signal properly. This can usually be avoided by slowly raising the cup over the photometer, and if necessary giving the cell a shake.

- Pressing the <MOTOR> key will display the signal the detector receives. If air bubbles are caught in the photometer window, the unit will beep and the bottom line of the display will show:

```
*** Reading unstable ***
```

If present, any air bubbles should be removed from the window by gently shaking the photometer cell from side to side and/or tilting it at an angle.

The display should show something similar to the following:

```
Trns: -595.5 Dose: n/a ml  
Press <TITRATE> to start
```

With clean water the reading should be approximately -600. If the reading is below -500 then the cell should be cleaned. The accuracy of the titration is only slightly affected by a dirty cell, providing the readings remain above -400 with clean water. Pressing any of the other keys will cause the unit to beep, and it will ignore the key-press.

6.4 Cleaning the Photometer Cell

The photometer cell can be cleaned by rinsing it with distilled or deionised water. If the contamination of the cell is considered to be severe, the cell may be cleaned with water and a bottle brush or wiped with household tissue soaked in acetone.

6.5 Titrations

The method by which a titration is carried out usually follows this sequence:

- Addition of a known quantity of test sample to the distilled/deionised water in the plastic cup.
- Addition of an accurate amount of standard Polybrene solution (normally 2 to 5ml).
- Addition of an accurate amount of Toluidine Blue O (normally 0.5ml of a 132mg/l solution).
- The titration is started by pressing the <TITRATE> key, and the solution is titrated using the standard PVSK solution.
- From the value given for the end point dose, the charge of the sample is calculated.

6.6 Performing Blank Titrations

Two blank titrations must be carried out before any calculations can be made:

6.6.1 Titration of the Indicator

The indicator requires a small amount of PVSK before colour change occurs (since Toluidine Blue O is cationic).

Twice the amount of indicator (i.e. 1 ml) should be added to the clean distilled or deionised water. This should then be titrated with the standard 0.0005N PVSK solution. The resultant titre should then be divided by 2 to give the true titre for 0.5ml indicator.

E.g. 1ml indicator gave an end point of 0.642ml. The value for 0.5ml indicator is thus 0.321ml, and this value should be used in all calculations.

6.6.2 Finding the Ratio Factor of the Polymers

Both standard polymer solutions should have been accurately made up to 0.0005N. (PVSK is taken to be the main standard and the Polybrene concentration is adjusted to equate to the PVSK, at pH 7.)

It is unlikely that the PVSK and Polybrene are exactly the same strength and so, the PVSK : Polybrene ratio will not be 1 : 1. Under certain circumstances, such as different pH or water conditions this difference will be exaggerated. We therefore need to include a factor in the calculation to allow for this deviation.

With each series of tests, a blank titration must be included, i.e. Titrate 5ml Polybrene with PVSK and record the result.

E.g. assume 5ml Polybrene is titrated with 4.850ml PVSK.
Here the Polybrene is weaker than the PVSK and would need to be multiplied by a factor of:

$$\frac{4.850}{5.000} = 0.970$$

All Polybrene amounts should be multiplied by the factor obtained from the blank titration.

6.7 Quantitative Evaluation of an Anionic Sample

This category includes most paper mill samples and anionic polymers. (Note: it is important that a blank titration is first carried out, see Section 6.6.)

A known amount of sample, prefiltered in the case of those taken from paper mills, is added to the plastic cup containing the distilled/deionised water. When evaluating paper stock filtrates, one would normally use between 10ml and 50ml of sample (accurately pipetted). Under certain circumstances, such as a heavily contaminated sample, it may be necessary to use only 1ml to 5ml. The larger the sample, the more accurate the result of the titration; but heavy contamination may interfere with the test and therefore a reduction in sample size is necessary to achieve a meaningful result.

The addition of an accurate amount of the standard cationic polymer, 0.0005N Polybrene, should be made. The Polybrene can be dosed from the exchange unit, with the Dosimat set to the DIS R mode. The **DELTA VOLUME** facility is set to dose the required quantity, which would normally be between 2ml and 5ml. 0.5ml of the standard 132mg/l Toluidine Blue O solution is accurately pipetted into the test solution. Replace the Polybrene

exchange unit with that of the PVSK and fit the dosing tube into the holder so that it feeds into the test solution.

Press the <TITRATE> key, holding it down until the small red LED is lit. The titration has now begun. The LCD display will show changes in Trns and Dose and the bottom line will indicate *** TITRATING ***. The small green LED labelled Dosing will light each time polymer is being dosed into the test solution.

As the titration progresses, the indicator will slowly change from blue to pink. Dosing continues until the computer is satisfied the end point has been passed. At this point, the CAII will beep three times to indicate the end of the titration and the titration end light will flash. The bottom line of the display will show

```
Processing please wait.
```

Once the machine has calculated the end point it will beep a further three times. The display will now alternate between the following two displays:

```
End point dose: X.XXXml  
Press <PRINT> for chart
```

```
Dev from 90% pt: X.XXXml  
Press any key to restart
```

It is the end point dose, the value that the computer has calculated to be the end point that is used in the calculation.

The Dev from 90% pt means the deviation from the 90% point in the titration. It is the deviation in titre between a point that is 90% of the end point, and that of the end point itself. This value gives an indication of the gradient of the titration curve. The steeper the curve, the lower the value of the deviation from the 90% point, and the more accurate the end point value. If the <STOP> key is pressed during the titration, the CAII will beep three times and display the current transmission and dose. The bottom line of the display will alternate between:

```
** Titration aborted **
```

and

```
Press any key to restart
```

Pressing any key resets the instrument. The bottom line of the display then shows Dosimat filling. When this operation has finished, the control box and Dosimat revert to the start up display.

6.8 Calculation of Charge (in microequivalents/litre)

The equation used to calculate the charge of an anionic sample is as follows:

$$\text{Charge } (\mu\text{equiv/l}) = \frac{(\text{AP} - \text{ind}) - (\text{CP} \times \text{F}) \times 1000}{2\text{V}}$$

Where:

AP = volume of anionic PVSK (titre value)

ind = titre due to water and indicator (see Section 6.6.1)

CP = volume of cationic Polybrene

F = Factor (see Section 6.6.2)

V = volume of test sample

6.8.1 Example 1

20ml of sample and 5ml of Polybrene (factor 0.97) were titrated with 2.963ml PVSK. The titre due to 0.5ml indicator and water was 0.321ml.

$$\begin{aligned} \text{Charge} &= \frac{(2.963 - 0.321) - (5 \times 0.97) \times 1000}{2 \times 20} \\ &= -55.2 \mu\text{equiv/l} \end{aligned}$$

N.B. the calculated value must be negative for an anionic sample.

Regarding the sample size for an anionic product (chemical), the recommended amount would be 1ml of a 1g/l solution. For the calculation, the volume of the test sample would be taken as 1.00 and the answer converted to milliequiv/g (not $\mu\text{equiv/l}$).

6.8.2 Example 2

1.00g of a 1g/l solution of unknown anionic polymer and 5ml of 0.0005N Polybrene were titrated with 4.429ml 0.0005N PVSK, at pH 7. The titre due to 0.5ml indicator and water was 0.313ml.

Using the same equation:

$$\begin{aligned} \text{Charge} &= \frac{(4.429 - 0.313) - (5 \times 1.00) \times 1000}{2 \times 1.00} \\ &= -442 \mu\text{equiv/g} \\ &= \frac{-442}{1000} = 0.442 \text{ milliequiv/g} \end{aligned}$$

This value would be called the charge density of the product at pH 7. It must be remembered that this value changes with respect to pH. When comparing chemicals, they must all be assessed at the same pH.

6.9 Quantitative Evaluation of a Cationic Sample

This category includes cationic chemicals and some paper mill samples (especially those using alum). The principle here is exactly the same as previously mentioned in Section 6.7.

1. Add the sample to the distilled/deionised water (prefilter in the case of mill samples).
2. Dispense 0.5ml of indicator.
3. Titrate using PVSK.

The same mathematical formula as before is used to calculate the charge.

7. Helpful Information

7.1 The End Point

The CAII will titrate the sample until the phase of the charge has changed sign (and thus the charge value of the sample has changed sign). The endpoint dose is then displayed; this value is the dose when the charge was at a minimum value. By connecting a printer to the parallel printer output, the CAII will print the charge, the dosed volume and the phase of the signal for every reading taken throughout the titration. This printout gives information that could be useful when you are having difficulty with a titration (see below).

7.2 Limitations of the CAII

It is important to realise that the CAII may not be able to titrate all samples, especially those with conductivity over a few milliseimens and those that the standard polymers are not able to neutralise the charge of. If the conductivity is too high it may be possible to dilute the sample into a larger quantity of deionised water, using for example a 500ml plastic beaker, instead of using the 140ml plastic cups supplied.

Some samples may appear to titrate successfully but if a print out is studied it can be seen that the end point occurs when the charge value is still quite high (normally the absolute charge value will be less than 20). This raises two points:

- The titration algorithm reduces the amount of standard polymer dosed as the charge value reduces towards the end point. For example, if the charge is 80 at the end point then the last dose may have been about 25 microlitres, reducing the end point accuracy considerably.
- The sample contained a chemical that the standard polymer was unable to neutralise.

Care is thus required in interpreting the titration data.

7.3 The Titration Algorithm about the End Point

The control unit will decide to dose if the absolute value (ignoring the sign) of the charge has either:

- Stabilised to within a fixed amount from the previous charge value.
- Increased from the previous charge value.

If a print out is studied it will be seen that, while the sign of the charge is the same as at the start of the titration, the control unit will dose further polymer only when the charge stabilises. However when the sign of the charge reverses (around the end point of the titration) the control doses after every reading as the absolute value of charge is now increasing.

7.4 Effects of pH on the Titration and Calculation of the Correction Factor

The amount of PVSK required to neutralise a given amount of Polybrene varies with the pH of the sample and thus a correction factor for any pH needs to be determined. For any given volume of Polybrene, slightly more PVSK is required in the acid region and slightly less in the alkaline region.

The PVSK is accurately prepared to 0.0005N and is used as the reference standard polymer. It is unlikely that the two standard polymers have been mixed to exactly equal strengths (see Appendix I), and thus a titration would be required to determine a correction factor for the Polybrene. It is easy at this stage to produce a correction factor for a range of pHs and the correction factor used in the charge calculation. To calculate this correction factor simply titrate a sample of 5ml Polybrene in distilled water against PVSK. This titration should be carried out at a range of pH values, with the mean dose of two titrations for each pH being used to ensure greater accuracy. The correction factor is the ratio of the amount of PVSK dosed to the amount of Polybrene (in this case the end point dose divided by five).

A graph of pH (y-axis) against correction factor (x-axis, PVSK/ml of Polybrene) is plotted and this should virtually be a straight line. The correction factor for any pH can then be obtained. It should be noted that this graph would only be valid for the standard polymers used, and a new set of data would need to be plotted every time a fresh quantity of either polymer was prepared. The correction factor may also vary slightly from one sample of distilled water to another depending on the quality of water. The correction factor is for the Polybrene (the PVSK being the reference standard) and is used to correct the actual volume of Polybrene titrated.

7.5 Alternate Titration Method for Interfering Cationic Samples

There may be certain cationic samples that interfere with the CAII cell causing problems during a titration. If this does occur then it may be advantageous to modify the procedure as follows:

1. Prepare the CAII for a titration in the usual way checking the cleanliness of the cell with a sample of distilled water.
2. In a separate plastic cup add a known quantity of sample as usual then add 5ml of PVSK from the Dosimat. Mix the two liquids then add 140ml of distilled water.
3. Place the cup on the stirrer unit and then titrate as normal using Polybrene. (N.B. the charge value should start highly negative.)

This alternative method allows the PVSK to complex with the cationic sample before it can adsorb onto the cell walls, and may produce a more reliable titration.

8. Calculation of Charge (in microequivalents/litre)

The calculation of charge is based on using PVSK, made up exactly to 0.0005N, as the reference standard for the titration (see Appendix I for information about preparation of the standard polymers). If the Polybrene has also been accurately prepared then at pH 7 the two polymers will titrate in deionised water in a 1:1 ratio, i.e. 5ml of PVSK will exactly neutralise 5ml of Polybrene. However as pH affects the titration (see Section 7.4) it is not essential that the Polybrene is exactly 0.0005N as the calculation includes a correction factor to take into account the effects of pH, the inaccuracy of mixing the Polybrene and the quality of the distilled water. It is important that when the instrument is used in the field with unknown distilled water, a series of titrations should be carried out to determine a correction factor for the water used, or at least to check that a previous factor is correct with this particular sample of water.

By definition a sample has an equivalence of one per litre if one litre of the sample exactly neutralises the charge of one litre of 1N PVSK.

The formula for the calculation of the charge of an unknown sample is as follows:

$$\text{Charge (equiv/l)} = \frac{(\text{AP} - \text{CP})}{V} \times N$$

Where:

AP = volume of anionic PVSK (ml)

CP = volume of cationic Polybrene (ml) x correction factor

V = volume of test sample (ml)

N = normality of the PVSK

N.B. although AP, CP and V are in ml the charge is still in equiv/l as the scaling factors cancel in the above formula.

8.1 Example

20ml of sample and 5ml of Polybrene were titrated with 2.387ml of 0.0005N PVSK. At the pH of the sample the correction factor for the Polybrene was 0.967

$$\begin{aligned} \text{Charge} &= 2.387 - \frac{(5 \times 0.967)}{20} \times 0.0005 \\ &= -0.0000612 \text{ equiv/l} \\ &= -61.2 \text{ microequiv/l} \end{aligned}$$

N.B. the Charge value will be negative for an anionic sample and positive for a cationic sample.

9. Appendix I

9.1 Preparation of the Standard Polymers

The two standard polymers recommended are Potassium Polyvinyl Sulphate (PVSK) as the standard anionic polymer and Hexamethrine Bromide (Polybrene) as the standard cationic polymer. Both PVSK and Polybrene are available from Aldrich Chemicals (PVSK is also available from Eastman Kodak, USA). It is recommended that PVSK is used as the reference standard and is mixed to exactly 0.0005N. The Polybrene is then prepared to as near as possible 0.0005N, although any slight inaccuracy of the strength of the Polybrene can be offset using the correction factor determined in Section 7.4.

It is possible to purchase standard Hyamin 1622 (Benzethonium Chloride) solution precisely prepared to 0.004M, which is used as a standard cationic solution. The PVSK is then prepared; titrating it against the Hyamin to ensure it is exactly 0.0005N.

Unfortunately the Hyamin is of low molecular weight; it is not possible to use the CAII to perform the titration against the PVSK. However, it is possible to manually titrate the Hyamin against the PVSK using Toluidine Blue O as an indicator, which turns from blue to pink in the presence of anionic polymers. It is necessary to know the purity of the sample of PVSK, if this is known it should be possible to exactly mix a 0.0005N solution without performing a titration with Hyamin. However, for a sample of unknown purity or as a double check of the strength, a titration should be performed.

To prepare the PVSK and Polybrene solutions proceed as follows:

1. Weigh out $(1.62 \times 100 / \text{percentage purity})$ grams of PVSK solid (if the purity is unknown assume 95%, i.e. use $(1.62 \times 100 / 95)$ or 1.705g of PVSK).
2. Make up a solution of this PVSK in 1000ml of distilled water giving a concentration of 0.01N.
3. Take 50ml of this 0.01N solution and make it up to 1000ml with distilled water giving a concentration of 0.0005N.
4. Take 100ml of distilled water in a 250ml beaker and add 0.1ml of a 0.1% solution of Toluidine Blue O as the indicator.
5. Titrate this sample using the 0.0005N solution of PVSK until the indicator just turns pink and note the volume used. The indicator itself consumes about 0.8ml PVSK to be neutralised.
6. Repeat steps 4 and 5 this time adding 1ml of the 0.004M Hyamin to the titration sample and again note the volume of PVSK consumed.
N.B. the titrations in steps 4 and 5 are easily carried out with the Dosimat in **DOS** mode slowing the dose rate down towards the end point.
7. The difference between the volume of PVSK used in the two titrations in steps 4 and 5 is the volume of PVSK needed to complex the Hyamin and should be exactly 8.0ml if the PVSK is exactly 0.0005N.
8. If the volume of PVSK titrated is not 8ml then the strength of the PVSK should be adjusted to give a solution of exactly 0.0005N. If the solution is already too dilute it will be necessary to repeat step 3 but calculate the volume of distilled water required to give the correct dilution.

9. We now have a PVSK solution of exactly 0.0005N and this is used as the reference standard polymer for all titrations.
10. To make up the Polybrene solution, weigh out approximately 1.9g of Polybrene and make up a solution with 1000ml of distilled water to give a strength of about 0.01N.
11. Take 100ml of this solution and make up to 1000ml with further distilled water to give a solution of approximately 0.001N.
12. 2ml of this solution can now be titrated in distilled water against the reference standard PVSK, using the CAII. If the Polybrene is exactly 0.001N then 4ml of PVSK will be required.
13. From the titration, the volume of distilled water required to dilute the Polybrene to 0.0005N can be calculated, and this volume used to produce the standard Polybrene solution.
14. Any minor inaccuracies in the strength of the Polybrene are not important and a correction factor needs to be found to accommodate this error (along with the errors occurring due to pH and the quality of the distilled water used) (see Section 7.4).
15. These two standard polymers can now be used in the exchange units of the Dosimat.

10. Appendix II

10.1 Example Calculation of the Charge Density of an Anionic Sample

The recommended procedure to calculate the charge density of an unknown anionic product would be to first mix up a solution of 1g per litre of product and then titrate 1ml of this solution.

10.1.1 Example

1.0ml of a 1g/l solution of unknown polymer is added to 5ml of 0.0005N Polybrene and titrated with 3.482ml of 0.0005N PVSK. The pH was 7 and the correction factor of the Polybrene 0.979. Using the formula for the calculation of charge (see Section 8) the volume of the test sample would be 1ml and the result would thus be in milliequiv./g.

$$\begin{aligned} \text{Charge} &= \frac{3.482 - (5 \times 0.979)}{1.00} \times 0.0005 \text{ equiv} \\ &= -0.707 \text{ milliequiv/g} \end{aligned}$$

This value would be called the charge density of the polymer at pH 7. N.B. it must be remembered that this value is dependant on the pH of the sample and thus when comparing different chemicals their charge densities must all be measured at the same value of pH.

11. Appendix III

11.1 Example Determination of the Charge Demand of Paper Stock

The charge analyser has been designed to measure liquid samples only (or ones with low concentrations of small particles) and thus a paper stock sample will need to be filtered prior to carrying out a titration. Filter paper should not be used, as cationic samples will absorb onto the paper giving erroneous results. Glass fibre filter pads are recommended as the best medium (for example a Whatman GF/C grade or similar) with a particle retention of 1.2 microns and above. It is advisable to discard the first few ml of filtrate. A titration on the filtrate can now be carried out in the normal way remembering to check the pH of the sample to obtain the correction factor of the Polybrene. Most paper stock samples show anionic activity and it is useful to know the cationic demand, this being defined as the amount of cationic charge required to reduce the sample to the isoelectric point. An effective way to find this cationic demand is as follows:

1. Take 5 x 200g samples of stock.
2. Stir in five different concentrations of a Cartafix product one concentration to each sample e.g. 0.1%, 0.2%, 0.3%, 0.4% and 0.5% of Cartafix by weight of dry stock.
3. After 2 minutes stirring, filter all five samples and titrate each in turn to calculate their charge.
4. Plotting a graph of Cartafix versus charge enables the extent of neutralisation to be readily examined.

This method gives a good guide as to the best substance to use for combating interfering substances and also the most suitable dosing level to use.

Note: Cartafix is the brand name of a range of cationic chemicals (manufactured by Sandoz Chemicals) used for the neutralisation of anionic interfering substances in paper making stock.

12. Appendix IV

12.1 Control Unit Error Messages

The software contained in the CAII has been written to aid and prompt the operator wherever possible. The buzzer always beeps when either the operator has pressed the wrong key, an error has occurred or the titration has ended (see Section 3.1). The second line of the LCD always shows either a prompt or an error message or both e.g. `Press <MOTOR> when ready` or `Titration aborted`. The error messages are listed below, together with possible causes and remedies:

**** Printer error ****

This is displayed when the CAII is unable to send data to the printer. Possible causes and the appropriate remedies are:

- No printer is connected. Press the <STOP> key to abort the titration, switch off the CAII, connect a printer of the correct type, switch on the CAII again and start a new titration.
- The printer is switched off-line. Switch the printer on-line then press the <PRINT> key until its LED is illuminated.
- The printer has run out of paper. Refill the printer with more paper, then ensure that the printer is on-line. Press the <PRINT> key to restart printing.
- The printer does not have a parallel interface and is thus incompatible with the CAII. Switch off the CAII and connect a suitable printer, then switch on, and start a new titration.
- The wrong printer cable is being used (any IBM compatible parallel port printer lead designed for your printer should be suitable). Switch off the CAII and printer, then ensure that the correct lead is plugged into the CAII and printer. Switch on the CAII and printer and start a new titration.

***** Cell fault *****

The CAII monitors that the cell motor is rotating and produces this error if not. Possible causes and their remedies are:

- The cell motor lead is not correctly plugged in to either the Cell or the Control unit. Switch off the CAII and check the motor lead is correctly plugged in at both ends, then switch on the CAII and retry.
- The cell motor lead is faulty. Check and obtain a replacement lead if necessary.
- The motor is running slowly. The motor should run at about four revolutions per second. If the speed is less than half this, the control will produce this error. Check that the cell is free to rotate and that the motor is receiving 24V (measured across the two terminals on the back of the motor inside the top cover of the cell stand). If the voltage is not within 5% of 24V then both the cell and control unit will need to be returned for checking/repair.
- The sensors inside the motor cover are faulty. Return the complete cell to us for repair/checking.

** End point not found **

The Dosimat 655 will automatically refill after dosing 10ml. This may cause problems with the control unit not knowing the exact amount of titrant dosed so to prevent this happening the CAII will abort from a titration when the dosed volume exceeds 9.7ml. Possible causes are:

- Too much sample has been used so that the standard polymer has been unable to titrate through the zero charge point. Repeat the titration using less sample. N.B. to maintain a reasonable accuracy it is sensible to ensure that at least 1ml of standard polymer is used during the titration.
- The polymer titrant used was of the wrong charge (e.g. an anionic sample was titrated using PVSK instead of Polybrene). Repeat the titration using the correct standard polymer.

** Titration aborted **

This occurs when the stop key has been pressed during a titration.

Dosimat filling

This message is displayed while the control unit is requesting the Dosimat to fill. Should the Dosimat be switched off, become unplugged from the CAII, or develop a fault, the CAII will sit waiting for a filled signal from the Dosimat. Thus if the CAII appears to lock up with this message being displayed ensure that the Dosimat is switched on and correctly connected to the CAII.

13. Appendix V

13.1 Parts List for Carrying Cases

The list below consists of all the parts that should be in the carrying case. This list may form a useful checklist for people using the instrument in different locations to ensure parts are not left behind.

13.1.1 Instrument Carrying Case

- 1 Charge Analyser II Control Unit
- 1 SCD Cell, motor and stand complete (optional)
- 1 Photometer Cell and stand clamp (optional)
- 1 Instruction manual for Charge Analyser II
- 1 Stiff cleaning brush for cleaning the cell
- 5 Plastic cups for titrating
- 2 Power leads (1 - Control Unit, 1 - Dosimat 665)
- 1 Cell motor lead (7 pin DIN plug to 7 pin DIN plug)
- 1 Cell lead (BNC to BNC)
- 1 Control Unit to Dosimat 665 lead (9 way 'D' plug to 25 way 'D' plug)
- 1 Stirrer lead for Dosimat 665 (3 pin DIN plug to 5 pin DIN socket) from Dosimat 665 to stirrer unit

13.1.2 Dosimat Carrying Case

- 1 Metrohm Dosimat 665 unit complete with magnetic stirrer
- 1 Keyboard for Dosimat 665
- 1 Dosing button for Dosimat 665
- 1 Instruction manual for Dosimat 665
- 1 Stand plus pipette clip for magnetic stirrer
- 2 10ml Exchange units for Dosimat 665 complete with reagent bottles, tops and pipes plus pipette tips
- 2 Spanners for exchange unit pipes
- 2 Tubes of grease for exchange unit pistons
- 2 Magnetic stirring bars for titration cups
- 1 Plastic cover for Dosimat 665