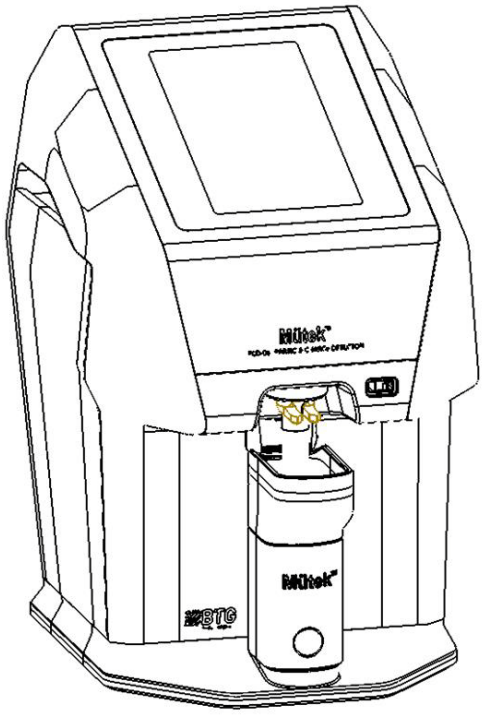
Operation Manual

Mütek™ PCD-06 (Light / Standard / Premium)

Particle Charge Detector



BTG
A Voith company

Original Instructions

BTG Instruments AB, 2021

List of contents

I. Basic safety instructions

1.1 Manufacturer's liability

Instructions in the operation manual

- All users of the Mütek™ PCD-06 have to observe this operation manual and to strictly adhere to the basic safety instructions and safety rules in order to ensure safe handling and reliable operation of the Mütek™ PCD-06
- This operation manual contains the most important safety directions to ensure the operational safety of the Mütek™ PCD-06
- In addition, all applicable accident prevention regulations have to be met for the specific location where the device is set up

Obligations of the user

The warranty and the service quality guarantee shall only be valid if the following conditions are met:

- The Mütek™ PCD-06 may only be operated by persons who are proven to have been thoroughly trained and authorized by BTG Instruments AB
- The user has to observe all instructions by BTG Instruments AB regarding operation and maintenance of the Mütek™ PCD-06
- Storage and maintenance of the instrument shall be in accordance with the instructions given by BTG Instruments AB
- Use of any parts other than the original spare parts and expendables of BTG Instruments AB will cause instant termination of warranty, availability and guarantee claims
- Any faults which may affect operational safety of the system have to be eliminated immediately
- When the system is obviously damaged or parts inside the system are loose or the system does not work, the Mütek™ PCD-06 has to be shut down.

Hazards involved in handling the Mütek™ PCD-06 Particle Charge Detector

The Mütek™ PCD-06 has been built to reflect state-of-the-art techniques and to meet recognized safety rules. However, its operation may entail the risk of accidents for users or third parties or of damage to the Mütek™ PCD-06 or to other property. The Mütek™ PCD-06 shall only be used:

- For the intended applications outlined in this operation manual
- When it is in a condition meeting all safety rules

Users are obliged to immediately eliminate any faults that may affect operational safety.

Warranty and liability

This instrument was built and tested in accordance with EU Guidelines 2014/30/EC and 2014/35/EC. It complied with all safety regulations at the time it left the factory. To maintain this condition and ensure safe operation, the user must observe the safety instructions and warning notes contained in this op-eration manual. It is the responsibility of the owner and user to make sure that all safety devices and requirements concerning the operating environment of the instrument are adequately provided for.

Basically, the General Terms and Conditions of BTG Instruments AB will apply. These will be made available to the operator of the Mütek™ PCD-06 at the latest on the day the sales contract is concluded.

BTG Instruments AB shall not be liable for any warranty or liability claims for personal injury or property damage that may be consequential to one or several of the following causes:

- Non-legitimated use of the Mütek™ PCD-06
- Opening of the instrument on the rear or at the top plate or at the bottom plate of the system
- Inexpert installation/ assembly, commissioning, operation and servicing of the Mütek™ PCD-06
- Operation of the Mütek™ PCD-06 although its safety devices and protection facilities are defective, inexpertly mounted or malfunctioning
- Failure of the user/ operator to adhere to the instructions given in this operation manual regarding transport, storage, assembly, commissioning, operation and maintenance of the Mütek™ PCD-06
- Modifications made to the Mütek™ PCD-06 without prior approval of BTG Instruments AB
- Inadequate routine checks of parts/assemblies of the Mütek™ PCD-06 which are subject to wear
- Inexpert repairs
- Catastrophes caused by the effects of foreign objects or by Acts of God

Trouble-free and safe operation of the instrument is only possible subject to correct transport, storage, installation and assembly and to careful handling and maintenance.

If the Mütek™ PCD-06 is sent in for repair, the whole system has to be returned, including measuring cell and displacement piston.

The guarantee for the Mütek™ PCD-06 is one year starting as of the date of delivery.

Regarding defects covered by the guarantee, BTG Instruments AB shall not be liable for any dam-ages exceeding the cost price.

The guarantee shall only extend to parts found defective by BTG Instruments AB. The company is entitled to decide whether such parts are to be exchanged or repaired provided the parts have been returned in time to the nearest authorized retailer of BTG Instruments AB.

This guarantee does not extend to parts that have been repaired or modified by the user/ operator without the prior written approval of an authorized retailer of BTG Instruments AB.

BTG Instruments AB neither assumes any obligation in connection with the sale of the instrument nor has it instructed any other person to undertake such obligation.

⚠ Caution

Any changes or modifications not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment.

Disclaimer of liability

BTG Instruments AB has checked the contents of the printed manual for consistency with the hardware & software described. However, since deviations cannot be entirely excluded, BTG Instruments AB does not accept responsibility for absolute consistency. The contents of this manual are regularly revised and any corrections that may be necessary will be included in later issues.

BTG Instruments AB reserves the right to make technical changes to this document.

1.2 Intended applications of the PCD-06

The Mütek™ PCD-06 shall be used exclusively for the purposes defined in this operation manual and in the technical description and shall only be operated in conjunction with external instruments and components recommended or approved by BTG Instruments AB.

The Mütek™ PCD-06 Particle Charge Detector shall solely be used for charge identification of aqueous samples and for determining their isoelectric point. The instrument is exclusively intended for indoor use.

Since any other or further-reaching application is considered illegitimate by BTG Instruments AB, the company shall not be held liable for damages resulting from such use.

To comply with the intended applications as defined above, the user/operator is required

- To follow all the instructions in this operation manual and
- To perform all inspection and maintenance tasks as specified

1.3 Non-legitimate use

Illegitimate use (misuse) is defined as follows:

- Any usage other than the allowed usage defined above or extending beyond the defined usage
- Failure to follow the instructions given in this operation manual
- Failure to observe the safety directions
- Failure to remedy immediately - i.e. before continuing operation of the instrument - any defect which might endanger its operational safety and reliability.
- Any unauthorized modification to the instrument

1.4 Qualification of personnel

The instrument may be commissioned and operated only by qualified personnel. Qualified personnel as defined in the safety instructions of this operation manual are persons trained and instructed by BTG Instruments AB.

1.5 General safety directions

For all installation, operation, maintenance and repair work performed on the Mütek™ PCD-06 Particle Charge Detector, it is essential that the directions given below be followed:

- Installation and operation have to be performed by trained personnel
- Maintenance work described in this manual shall exclusively be carried out by trained staff
- Maintenance and repair work not described in this manual shall only be performed by BTG In-struments or by personnel trained by an authorized service agent of BTG Instruments AB
- When working on electrical and electronic parts, be sure to power off the instrument at the main switch and to disconnect the power plug
- When replacing defective parts, make sure to use only original spare parts and expendables
- When handling hazardous materials, please observe the directions for protective equipment, industrial hygiene and proper disposal
- Hazardoous chemicals should be handled by trained personnel according to regulation
- MSDS for any hazardous chemicals sold from BTG is available on https://my.btg.com/en/

1.6 Informal safety directions

Take care to keep this operation manual handy at all times on the Mütek™ PCD-06. In addition, make sure that documentation on general and local accident prevention and environmental protection standards is near at hand and strictly adhered to.

Keep all safety instructions and warnings on the Mütek™ PCD-06 in a legible condition and replace, if necessary.

1.7 Spare parts and expendables

Be sure to use exclusively original spare parts and expendables to ensure operational safety of the Mütek™ PCD-06 and to safeguard your guarantee claims. Please order these items from your local authorized representative of BTG Instruments AB.

1.8 Symbols

The following symbols and notices are used in this operation manual to designate hazards or to provide special information:

Information

"Information" includes tips for operators and provides useful information.

It helps you make optimum use of all functions of the Mütek™ PCD-06.

Hint

"Hint" refers to expert handling and operation of the Mütek™ PCD-06.

Non-adherence to these hints may lead to operational failures of the Mütek™ PCD-06 or to trouble in its environment.

Caution

"Caution" refers to a potentially dangerous situation. Failure to observe this information may result in slight personal injury or in damage to equipment.

Warning

"Warning" refers to the risk of accidents.

Failure to observe this information may result in severe damage to health or even in life-threatening injuries.

1.9 Packaging & transport

The Mütek™ PCD-06 Particle Charge Detector is a compact and sturdy unit for which a plastic transportation box is available to enable measurements on site.

Information

It is recommended to ordering the plastic transportation box to avoid transport damage.

Hint

The plastic transportation box is unsuitable for rail forwarding or postal delivery of the instrument.

- To exclude transport damage, never put any additional items in the trolley.
- To avoid transport damage to the precision contact pins, do not keep any small components in the measuring chamber.
- At outdoor temperatures below +15 °C [59°F], let the unit adjust to ambient temperature after transport before putting it into operation.

Hint

If the Mütek™ PCD-06 is exposed to temperatures below 0°C, the titrants might freezing causing damage to the bottles and tubes. Therefore do not leave the instrument in the trunk of your car at cold weather.

II. Product description

2.1 Theoretical background

Whether a suspension of particles or a true solution of large molecules, most materials dispersed in water carry an electric charge.

In such an aqueous system charges at solid/ liquid or liquid/ liquid interfaces play a crucial role.

They cause reaction mechanism of the type found in dispersions, suspensions or emulsions.

Measuring the streaming potential gives information about interactions of the components in water and is used to assess the stability of a system.

Double layer

Once a particle is dispersed in water it develops a charge at the particle surface which affects the distribution of the dissolved ions in the water. This results in an increased concentration of ions of opposite charge to that of the particle close to its surface. Closest to the surface a stationary layer of counter-ions forms; further away a diffuse layer surrounds the particle. Thus each particle carries an electrical double layer. Such a double layer can only be formed by particles at least 1 nm in size, the so-called colloids.

Electrokinetic effect

A relative movement of a liquid to a dispersed material generates an electrokinetic effect. If an electric field or a pressure is applied, the solid particles are caused to move relative to the liquid electrolyte or vice versa. Depending on which of the phases is moving, the electrokinetic effect can be identified via Streaming potential.

Streaming potential

If a liquid is forced along a particle surface under pressure, it will shear off the counter-ions of the diffuse layer and an electric field is built up. The resulting electric current is picked up by two electrodes and is either indicated by a streaming current signal or converted to a potential. The Mütek™ PCD-06 Particle Charge Detector displays the potential in ±mV. The sign of the measured value indicates whether the sample carries a positive (cationic) or negative (anionic) charge. The streaming potential is a relative value and depends on various influential factors, such as

- Dimensions of the measuring cell
- Cleanliness of the cell
- Conductivity
- Chemical properties of the sample
- Molecular weight
- Particle size
- pH
- Viscosity
- Temperature

To quantify the charges of a sample, a titration with an oppositely charged titrant (polyelectrolyte) has to be performed up to the point where a streaming potential of 0 mV is reached.

There is a correlation between streaming potential and zeta potential, which latter has been identified by electrophoresis. In actual practice, however, a comparison of the two potentials is seldom possible and if at all, only under defined ideal conditions.

2.1.1 Measurement

Charged colloids and particles of a size > 1nm can attract oppositely charged ions from the surrounding water. Separating these so called counterions from the particle creates a measurable potential difference. If such ion separation is achieved through a liquid flow, the potential is called streaming potential with the unit mV.

After a sample has been filled into the Mütek™ PCD-06 measuring cell, sample colloids (1) adsorb to the inner surfaces (2) of the measuring cell, while its attracted counterions (3) remain comparatively mobile. Driven by a motor, the displacement piston (4) creates an intensive liquid flow (5), which separates the counterions from the adsorbed sample material.

At the electrodes built in the measuring cell, the counterions induce a current which is rectified and amplified electronically. A streaming potential with the appropriate sign is shown on the display.

The integrated titrator recognizes the sign and automatically adds a polyelectrolyte of opposite charge and known concentration. The poly-electrolyte addition keeps going, until the end point (EP) of the titration is reached. This is the point of neutral charge (= 0mV), where all existing charges in a sample are neutralized. Out of the known titrant consumption required to reach the point of neutral charge, the charge content of the sample is calculated and displayed as measurement result.

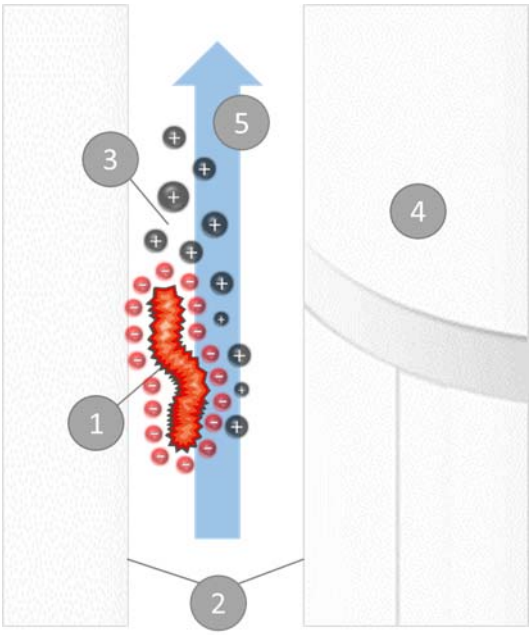


Fig. 2-1 Inside the measuring cell of a Mütek™ PCD-06

Designations

1	Sample colloids	4	Displacement piston
2	Inner surfaces	5	Liquid flow
3	Counterions		

2.1.2 Calculation of charges

Polyelectrolyte titration

For quantitative charge measurements of the sample, a polyelectrolyte titration has to be conducted which uses the streaming potential to identify the point of zero charge (0 mV).
To this end, add an oppositely charged polyelectrolyte of known charge level to the sample as a titrant.

The titrant charges neutralize existing charges of the sample. Titration is stopped as soon as the point of zero charge (0 mV) is reached.

The titrant consumption obtained in ml forms the basis for further calculations.

Provided equal samples are titrated under identical conditions, the consumed titrant volumes can be directly compared. Normally, however, the charge level present in aqueous samples is expressed in terms of the anionic or cationic demand in eq/l.

Calculation of cationic/ anionic demand

The cationic or anionic demand [eq/l] of a sample is calculated by the following formula:

$$C2 = \frac{C1 * V1}{V2}$$

C1: Concentration of titrant in [N] or [eq/l]
C2: Concentration of the titrated sample in [eq/l]
V1: Titrant consumption in [l]
V2: Sample volume to be titrated in [l]

Example:
10 ml filtrate of an anionic fiber suspension is pipetted into the measuring cell of the Mütek™ PCD-06 and titrated with a 0.001 N cationic titrant solution until the point of zero charge is reached. In the example, 5 ml cationic polyelectrolyte (cationic titrant) is required.

The cationic demand of the sample is the following:

$$C2 = \frac{0.001\ \text{eq/l} * 0.005\ \text{l}}{0.01\ \text{l}} = 0.0005\ \text{eq/l}$$

0.0005 eq/l or 0.5 meq/l or 500 µeq/l
If the charge level is to be identified for an active substance (starch, wet strength agent) or for solids (pigment slurry), the mass of the product should additionally be included in the calculation to obtain the so-called specific charge density.

Calculation of the specific charge density

The specific charge density q [eq/g] is calculated by the following formula:

$$q = \frac{V * c}{m}$$

V: Consumed titrant volume [l]
c: Titrant concentration [eq/l]
m: Solids of sample or its active substance [g]
q: Specific charge density [eq/g]

Example:
A 10 ml sample of a 0.1% $Ti0_2$ suspension contains 0.01 g solids at m = 0.01 g.
The consumption of cationic polyelectrolyte up to the point of zero charge is 2.5 ml for a titrant concentration of 0.001 N.

The specific charge density is the following:

$$q = \frac{0.0025\ \text{[l]} * 0.001\ \text{[eq/l]}}{0.01\ \text{[g]}} = 250 * 10^{-6}\ \text{[eq/g]}$$

$250*10^{-6}$ eq/g or 0.250 meq/g or 250 µeq/g

By multiplying the specific charge density in eq/g by the Faraday's constant F = 96485 C/eq, the total charge in C/g will be obtained.

2.1.3 Determination of the isoelectric point

Acid/ base titration

Apart from charge measurements, the Mütek™ PCD-06 lends itself to identifying the isoelectric point (IEP) of dispersions.

The IEP is the pH-dependent point of zero charge of a particle.

By adding a titrant (acid/ base) drop by drop, the sample's pH is shifted until the isoelectric point (streaming potential = 0 mV) is reached.

Cationic samples are titrated with a base and anionic samples with an acid up to the point of 0 mV.

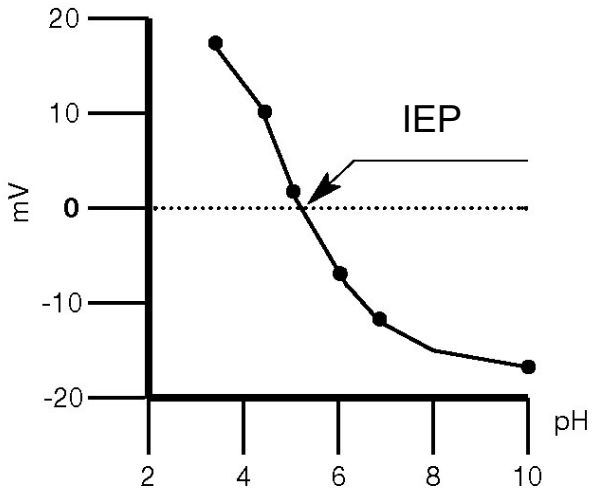


Fig. 2-2 Schematic of isoelectric point

2.1.4 Titrations curves

Information

The Mütek™ PCD-06 Standard or Premium continuously generates a titration curve while performing a sample titration.

Titration curves are basically difficult to interpret, because the curve progression does not exclusively reflect the sample material, but rather the adsorption behaviour of the sample at the surface of the measuring cell. This is why the curve progression should not be taken as a basis for the discussion of results. Instead, under certain conditions, the curves provide information about the nature of the sample under test. The titration curves shown in the following may roughly be associated with different stock systems:

2.1.4.1 Colloidally dissolved particles

A titration curve obtained for a colloidally dissolved sample is shown in Fig. 2-3.

At the onset of titration, the streaming potential remains essentially unchanged, since the non-adsorbed colloids are neutralized and do not therefore contribute toward the generation of a streaming potential.

Later on, the streaming potential undergoes a sharp drop, which is indicative of the fact that adsorbed charge carriers are neutralized at the cell wall and piston.

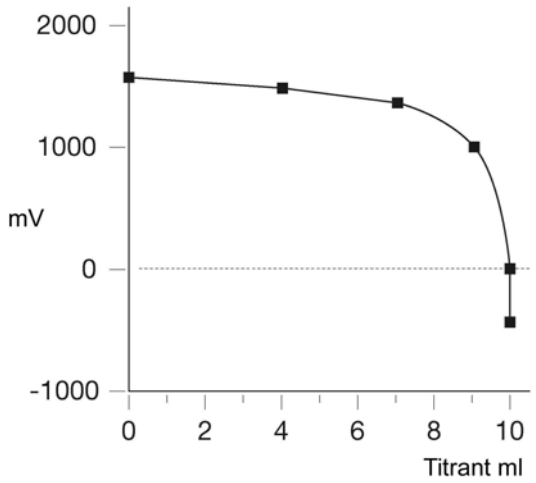


Fig. 2-3 Colloidally dissolved particles

2.1.4.2 Solid particles

A titration curve for an aqueous suspension of particles is shown in Fig. 2-4.

Here the titration curve is nearly linear, because all solid particles contribute toward the creation of a streaming potential.

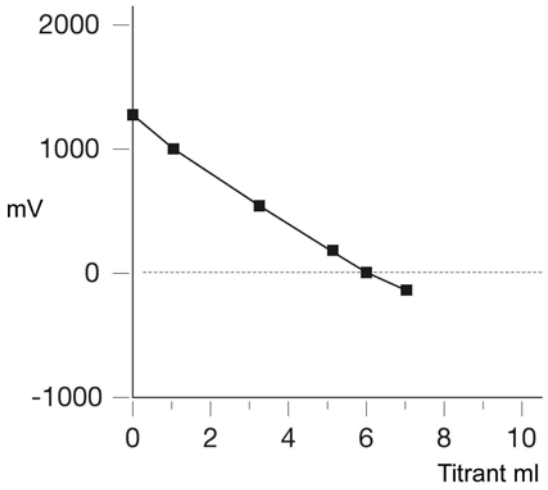


Fig. 2-4 Suspended solids

2.1.4.3 Heterogeneous stock systems

A titration curve of heterogeneous systems (colloids, dissolved material, solid particles, fibres etc.) is shown in Fig. 2-5.

After dropping sharply at the beginning, the curve flattens out toward the end of titration.

The buffer effect around the point of zero charge may be caused by the fact that titrant is included in the fibres.

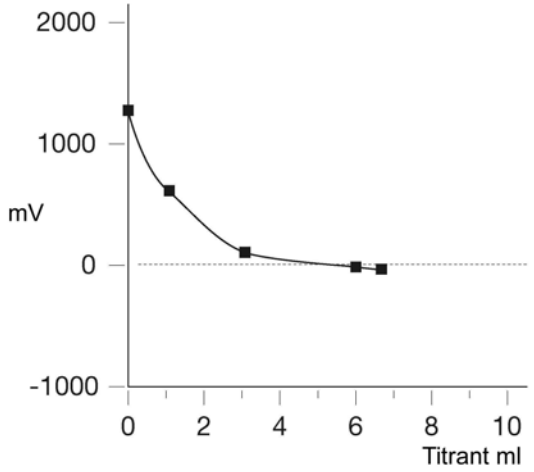


Fig. 2-5 Heterogeneous stock systems

2.2 Applications

Papermaking

- Improved machine running by controlling anionic trash
- Improved retention control
- Enhanced chemical performance
- Stable paper quality

Effluent treatment

- Optimized chemicals by controlled flocculant dosages
- Increased dry content of sludges through improved dewatering
- Exact identification of the demulsification point of emulsions and cooling lubricants
- Optimized selection of flocculants

Filler/ pigment processing

- Determination of the optimum dispersed condition and of dispersant dosages
- Selective flocculation of particles after identification of the isoelectric point

Other applications

An identification of charges is also very important for producers from the beverage and food industry and in the ceramics, dyes, cosmetics and pharmaceuticals sectors. In all these areas the Mütek™ PCD-06 is finding increasing application.

2.3 Designation of the PCD-06

Hint

Check if a legible type plate (below) is provided on the rear panel of the device. Please replace if il-legible or missing.

2.3.1 Type plate PCD-06

The following type plate is provided on the rear panel of the instrument:

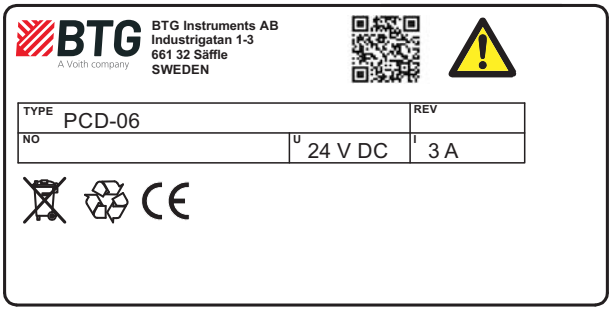


Fig. 2-6 Type plate PCD-06

Warning sign:

The device is designed for industrial use. Installation, handling and service must only be carried out by trained and authorized personnel and according to relevant standards. Read the manual for detailed information and pay special attention to the warning signs.

2.3.2 Service sticker PCD-06

You will find a service sticker on the wall of the instrument. It shows the date of the last functional inspection of the Mütek™ PCD-06 together with a recommended date for the next service.

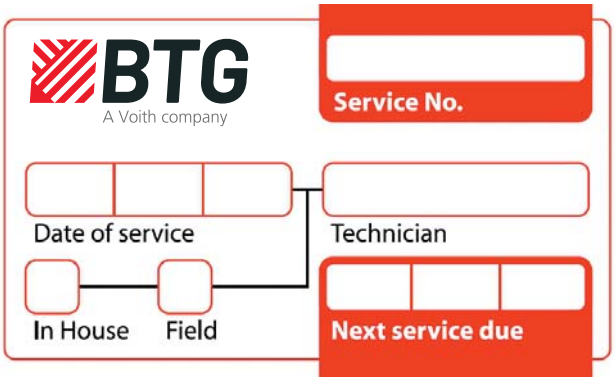


Fig. 2-7 Service sticker

2.4 Technical data

Dimensions	- Mütek™ PCD-06:	224 mm x 233mm x 335 mm (w x d x h)
Weight	- Mütek™ PCD-06 Light:	5.1 kg
	- Mütek™ PCD-06 Standard:	6.0 kg
	- Mütek™ PCD-06 Premium:	6.4 kg
Power supply	- External power supply Input:	100-240 V AC; 2.92 A; 50-60 Hz
Service conditions	- Ambient temperature:	+15 °C up to +40 °C
	- Storage/ transport temperature:	+5 °C up to +40 °C
Measured variables	- Streaming potential [mV] - pH	
Results	- Anionic/ cationic demand [ml] - Charge quantity [µeq/l] - pH - Isoelectric point (IEP)	
Sample volume	- Measuring cell: 10-50 ml	
Outputs	- Streaming potential (+/- 2500 mV) - Titrant consumption on the display of Mütek™ PCD-06 - pH on the display of Mütek™ PCD-06 with pH measurement option - USB interfaces of Mütek™ PCD-06 to store result, and parameter to an USB-memory	
Reproducibility	- Relative standard deviation SD(x): (results obtained by titration of anionic titrant against cationic tritrant) - ≤ 1%	
Detection limit	Up to 1 ppm depending on sample specification	
Standards	- Mütek™ PCD-06	EN 61326, EN 61010
Safety	Protection class III	

2.5 CE Declaration

When using the units in combinations other than those tested for, BTG can not guarantee CE directive conformity.

The units in combination with customer-installed external devices may conform with EMC and safety requirements when properly installed and CE-marked equipment is used.

The system operator is responsible for CE directive conformity. Conformity must be verified by inspection.

EU Declaration of Conformity (DoC)

CE Mark

PRODUCT: PARTICLE CHARGE DETECTOR

TYPE: PCD-06

BTG INSTRUMENTS AB
P.O. Box 602
661 29 Säffle
Sweden

This declaration of conformity is issued under the sole responsibility of the manufacturer.

We declare that the above Total Consistency Transmitter conforms to:

2014/30/EU	Electromagnetic Compatibility Directive, EMC
2014/35/EU	Low Voltage Directive, LVD
2011/65/EU+2015/863/EU	RoHS

The following harmonized standards have been practiced:

EN 61010-1:2010	Safety requirements for electrical equipment for measurement, control, and laboratory use. General requirements
EN 61326-1:2013	Electrical equipment for measurement, control and laboratory use - EMC requirements Part 1: General requirements

Authorized Signature:..... Date: 2021-06-28

Name: Björn Fahlin Position: Director of Operations

2.6 Scope of delivery

2.6.1 Mütek™ PCD-06

Standard delivery

Qty	Item
1	PCD-06
1	Measuring cell
1	Displacement piston
1	External power supply
1	Power cable
1	Piston brush
1	Cationic titrant bottle, 0.001N, 250 ml
1	Anionic titrant bottle, 0.001N, 250 ml
1	Beaker with screen

Optional

Qty	Item
1	Precision pipette, 2-10 ml
1	Plastic transportation box
1	pH set - pH electrode - buffer solution 4.01 - buffer solution 7.00 - buffer solution 9.21

III. System layout and functions

The Mütek™ PCD-06 Particle Charge Detector is available as Light, Standard, or Premium.

3.1 Description of PCD-06

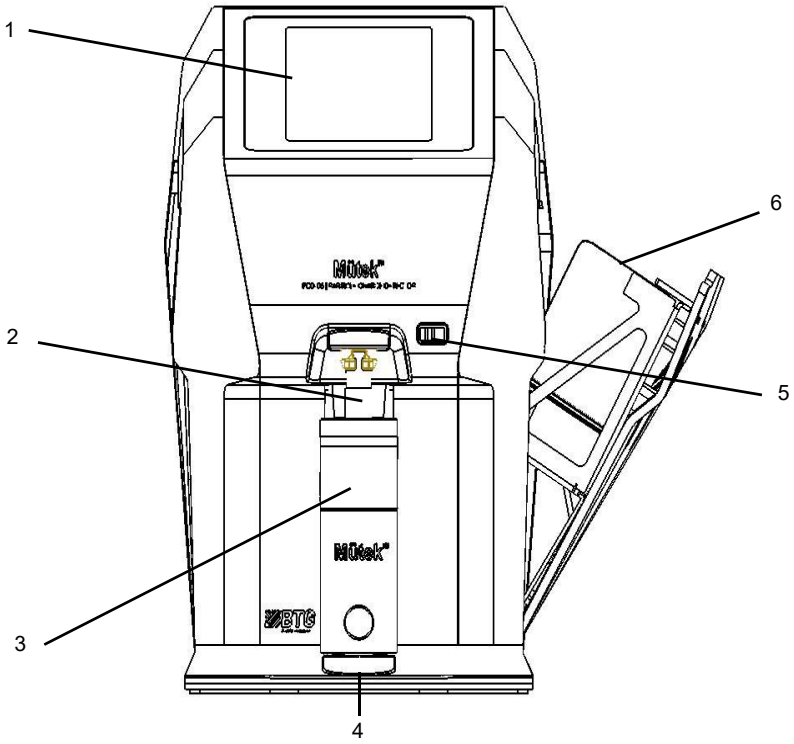


Fig. 3-1 Front view PCD-06

Designations

1 Display
2 Displacement piston
3 Measuring cell
4 Guide for measuring cell
5 USB interface for USB-Stick
6 Anionic (-) and cationic (+) titrant bottle

PCD-06 Light has no pump installed
PCD-06 Standard has one pump installed
PCD-06 Premium has two pump installed

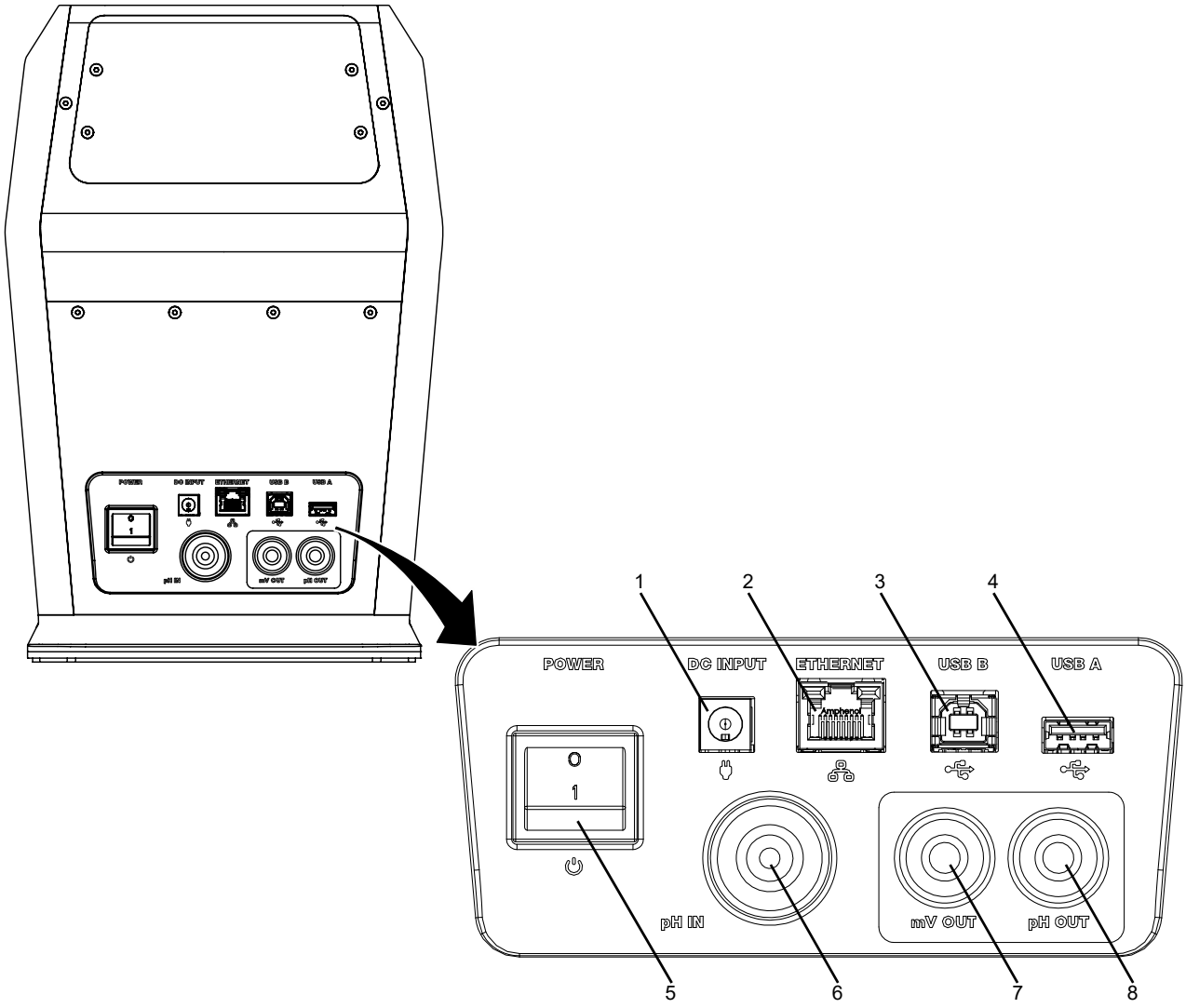


Fig. 3-2 Rear panel PCD-06

Designations

1 DC Input
2 Ethernet
3 USB B interface
4 USB A interface
5 Power ON/ OFF switch
6 pH Input
7 mV Output
8 pH Output

3.1.1 Measuring cell

Standard delivery of the Mütek™ PCD-06 includes a precision measuring cell and a displacement piston. Fitted in the measuring cell are two platinum electrodes for identifying the streaming potential.

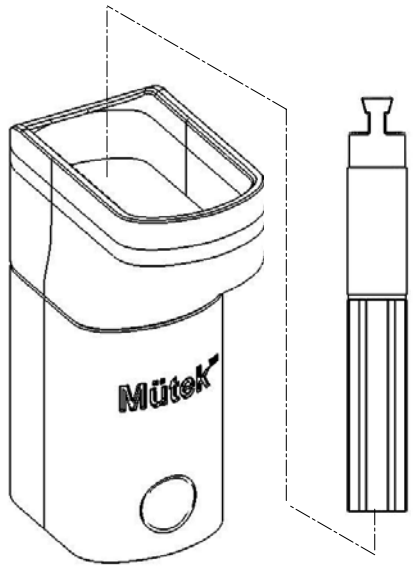


Fig. 3-3 Measuring cell and displacement piston

3.1.2 pH set (optional)

The Mütek™ PCD-06 Smart identifies not only the charge level but also the pH of a sample.
In combination with a Travel Titrator module or an external Titrator, the instrument can determine the isoelectric point of a sample via the pH electrode.

Install the pH electrode in the measuring cell on the wall opposite the titrant tubes, that is at a maximum distance from the tube tips.

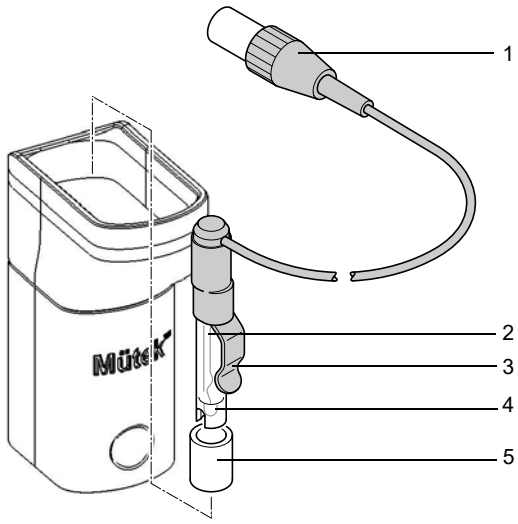


Fig. 3-4 Measuring cell and pH electrode

Designations

1 Electrode cable with A2 connector
2 Plexiglass shaft
3 Clip
4 Ceramic diaphragm
5 Electrode cap

The pH electrode must immerse in the sample solution. Therefore it is necessary to use an increased sample volume of 30 ml for isoelectric point measurements. Use more sample material and do not dilute the sample with water!

When just one pH value has to be measured (no pH curve or IEP measurement) the pH of the 10 ml sample can be measured outside before filling into the measuring cell.

IV. Installation

This chapter covers the installation of the Mütek™ PCD-06 Particle Charge Detector and the necessary preparations. In the course of installation, all required settings have to be made.

4.1 Safety rules

Please adhere to the applicable safety rules during preparatory work and when commissioning the system.

4.2 Personnel requirements

The Mütek™ PCD-06 may only be installed by qualified and skilled personnel.

4.3 Preparations onsite

Prior to installation of the Mütek™ PCD-06, all preparations mentioned in chapter 4.4 "Installation environment", page 23 must be made and the ambient conditions met.

Allow the Mütek™ PCD-06 to adapt to ambient temperature before commissioning.

4.4 Installation environment

4.4.1 Installation site

⚠ Hint

To warrant safe and reliable operation of the Mütek™ PCD-06, check on the following conditions before commissioning the instrument:

- ❑ Ambient temperature: +15°C up to +40°C
- ❑ Place the device on a stable and flat surface
- ❑ The room where the Mütek™ PCD-06 is set up must be clean and dry
- ❑ Do not operate the device while it is exposed to direct sunlight
- ❑ Never use the instrument when it is or was directly exposed to water
- ❑ Never use the device in explosion prone rooms
- ❑ Never use the instrument if it is or was exposed to aggressive chemical substances
- ❑ If you notice the presence of condensed water either during transport or after storage, allow approximately two hours before turning on the instrument

4.4.2 External power supply

⚠ Hint

Before starting the instrument, check if the local mains voltage is identical to that indicated on the type plate.

The Mütek™ PCD-06 is equipped with an external power pack which connects to all power voltages within the range indicated on it.

- ❑ Input voltage: 100 - 240 V AC
- ❑ Frequency: 50 - 60 Hz
- ❑ In the room where the device is set up, electrical installations must conform to current regulations
- ❑ Be sure to connect the power supply to an approved earthed socket
- ❑ Do not undo any earthing contacts
- ❑ The guarantee by BTG Instruments AB expires if the device is used with an inappropriate voltage or if it is not connected to the external power supply that comes with the instrument

4.4.3 Sample specification

General

Basically, nearly every aqueous sample - including original concentrations - can be measured. The measurability of a sample may be verified by the Mütek™ PCD-06 Particle Charge Detector. To this end, fill a sample into the measuring cell, install the cell together with the displacement piston in the Mütek™ PCD-06 and turn on the motor. Measurement may be started as soon as the motor runs smoothly and silently and the signal keeps comparatively stable (at approximately ±50 mV).

The limit values indicated in this chapter are not absolute values, but are merely indicative of sample characteristics that might lead to measuring problems.

If there are doubts about whether or not a substance can be measured, conduct a test measurement and, if necessary, condition the sample accordingly.

Solvents

Charge measurement is possible after functional groups are dissociated. Since this dissociation can only take place in aqueous solutions, the substance to be tested must be available in this form or be dispersed in water.

If required, the substance to be tested may be dissolved in lower alcohols (methanol, ethanol, isopro-panol) before it is diluted with de-ionized water for subsequent titration.

The proportion of organic solvents depends on the sample and should be determined in pretests. To begin with, a blank reading should be obtained for the solvent concerned. For this purpose, fill 10 ml of the solvent (de-ionized water or a water/ alcohol mixture) into the measuring cell and titrate up to the point of zero charge.

Even if the cell is filled with de-ionized water only, the streaming potential will not be at 0 mV as might be expected, but will show a comparatively high anionic signal. This signal drops to zero as soon as a minimum amount of a cationic titrant is added.
If de-ionized water is titrated, approximately 0.02 ml cationic titrant is normally required.

The blank value thus obtained is to be deducted from the results of all further titrations.

Particle sizes

Solid particle sizes in a sample should not exceed 500 µm. The minimum detectable limit value is 1 nm.

Viscosity

Suitable for measurement are all samples with a viscosity below 6000 mPa*s. Viscosities exceeding this value impede the oscillating movement of the displacement piston in the measuring cell. This restriction is still aggravated by the flocculation effect typically occurring toward the end of titration.

Accordingly, samples with a viscosity exceeding 6000 mPa*s should conveniently be diluted with de-ionized water.

If highly viscous samples are to be titrated, mixing the titrant with the sample takes longer than with low-viscosity samples.

Molecular weight

The minimum molecular weight of polyelectrolytes to be tested should be at approximately 300 g/mol, since with smaller molecules, their adsorption on the cell wall by van-der-Waal forces is no longer ensured. Ions present in a sample due to dissolved inorganic salts are below the detection limit.

At the upper end of the scale, there is no limiting molecular weight of colloidally dissolved substances.

Conductivity

The conductivity of a sample decisively impacts the streaming potential: the higher the conductivity, the lower the electrical resistance in the sample will be and the more the streaming potential will decrease without, however, influencing the titration results.

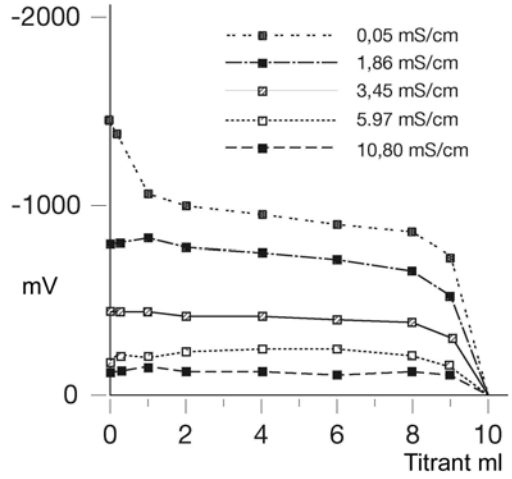


Fig. 4-1 Conductivity vs. streaming potential

In Fig. 4-1, various different NaCl proportions were added to anionic titrant 0.001 N and the samples were titrated with cationic titrant 0.001 N.

Hint

If conductivities are excessively high (20 mS/cm - 50 mS/cm), the streaming potential may fluctuate between anionic and cationic so than an endpoint detection and thus titration is no longer possible. In these cases, reduce the conductivity by diluting the sample with de-ionized water.

Information

Samples that do not meet the above specifications may be prepared as outlined in chapter 5.4 "Sample preparation", page 31.

4.5 Solutions

4.5.1 Titrants

Titrants must always carry the opposite charge of the sample.

The sample concentration (dilution) should optimally be selected to keep the titrant consumption between 0.2 and 10 ml.

Polyelectrolytes

BTG Instruments AB recommends the use of BTGs anionic and cationic titrant as standard reagents, since their charge densities are practically pH-independent.

Anionic and cationic titrant are available in 250 ml or 1000 ml volume for ready to use. As well it can be ordered as concentrate to be prepared on site.

Hint

MSDS for concentrates are available on: www.btg.com under MyBTG.

4.5.2 Acids/bases

Use diluted acids as titrants for anionic samples, and diluted bases for cationic samples.
Hydrochloric acid, sulphuric acid and sodium hydroxide solution have proven useful in this context and are normally applied in concentrations of 0.1 - 0.01 mol/l.

4.5.3 Cleaning solution

⚠ Warning

Acids and bases must only be handled by qualiefied personal.
Acids and bases must be reomoved from the unit before shipment.

⚠ Warning

Acetone and the cleaning solution are highly inflammable.

⚠ Warning

Please follow the safety instructions in chapter 1.5 "General safety directions", page 9.

The cleaning solution for manual cleaning of the measuring cell of the Mütek™ PCD-06 is available from BTG Instruments AB or can be produced from 50 parts sodium bromide (NaBr) and 50 parts acetone plus 125 parts water.

- Prepare the above proportions.
- Fill water into a suitable vessel.
- Add sodium bromide and dissolve.

⚠ Hint

Take care that sodium bromide dissolves completely.

- Add acetone and mix gently.

ⓘ Information

Selection of an optimum detergent depends on the sample concerned.

4.6 Start-up procedure

The PCD-06 is available in three variants:

- PCD-06 Light with no titration pump installed
- PCD-06 Standard with one titration pump installed
- PCD-06 Premium with two titration pump installed

4.6.1 PCD-06 Standard and Premium

- Connect Power supply to PCD-06.

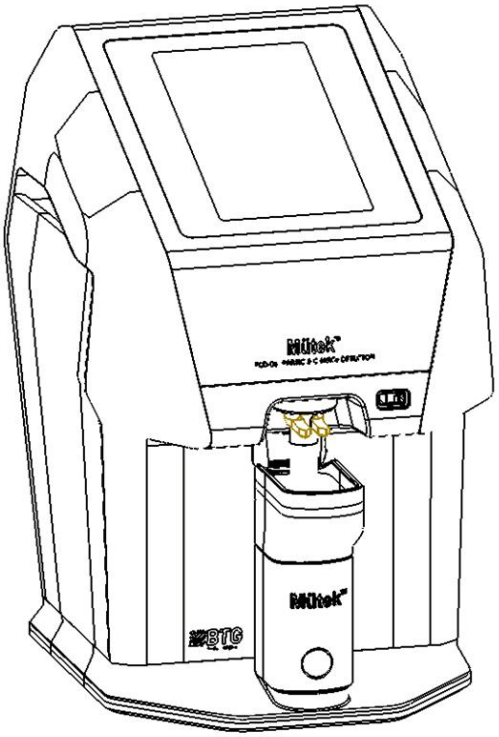


Fig. 4-2 PCD-06

4.6.2 Connect PCD-06 Light

The Mütek™ PCD-06 can be operated with an external automatic titrator.

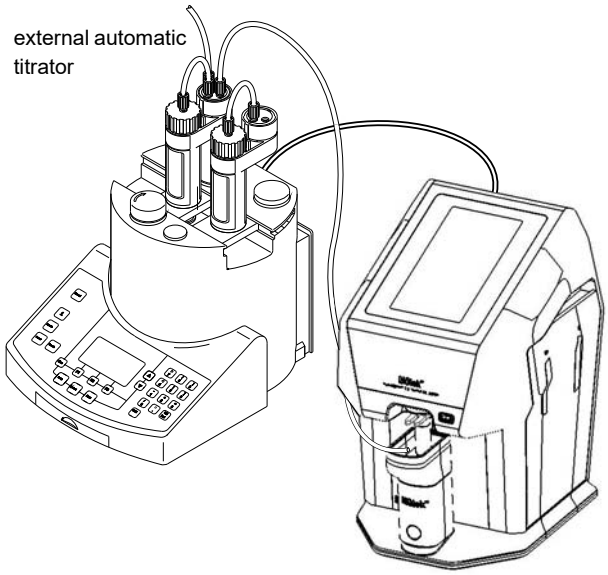


Fig. 4-3 PCD-06 combined with an external titrator

4.6.3 Replace a Titrant

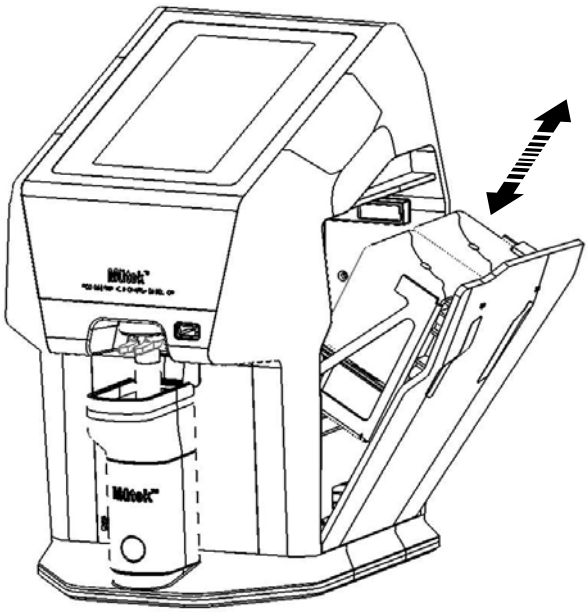


Fig. 4-4 Replace a titrant

1. Open the side door.
2. Turn the bottle movement protection to outside.
3. Screw the bottle lid from the old bottle to the new one.
4. Push the new bottle into the bottle holder.
5. Turn back the bottle movement protection.
6. Rinse the system.

Hint

There will stay some liquid in the bottle holder this can be easily removed with an tissue or just rinse the system with no bottle inserted.
Use PPE to remove the remaining titrant in case Acid or base was used.

4.6.4 Rinse the System

Hint

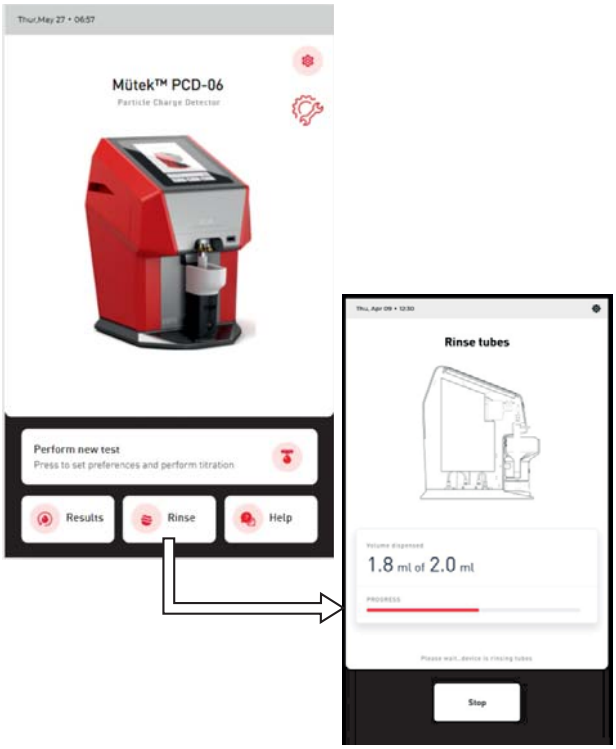
Rinse the dosing tubes in order to assure that they are bubble-free, before starting the measurement.

Procedure

1 Turn on the Mütek™ PCD-06.
2 Insert the measuring cell.
3 Press the rinse button.
4 The system will be rinsed with 2 ml.

Information

The rinsing process can be interrupted at any time by pressing the stop button.



V. Measuring

Information

The Mütek™ PCD-06 measures the streaming potential of an aqueous sample. To detect its charge level a titration is performed until reaching a streaming potential of 0 mV. Titration may be performed manually or better with help of an automatic titrator.

For manual operation of the Mütek™ PCD-06 Particle Charge Detector, use a measuring pipette to transfer titrant drop by drop into the measuring cell (see "Fig. 5-4", page 34). The endpoint, that is the point of zero charge, is reached when the streaming potential is at 0 mV.

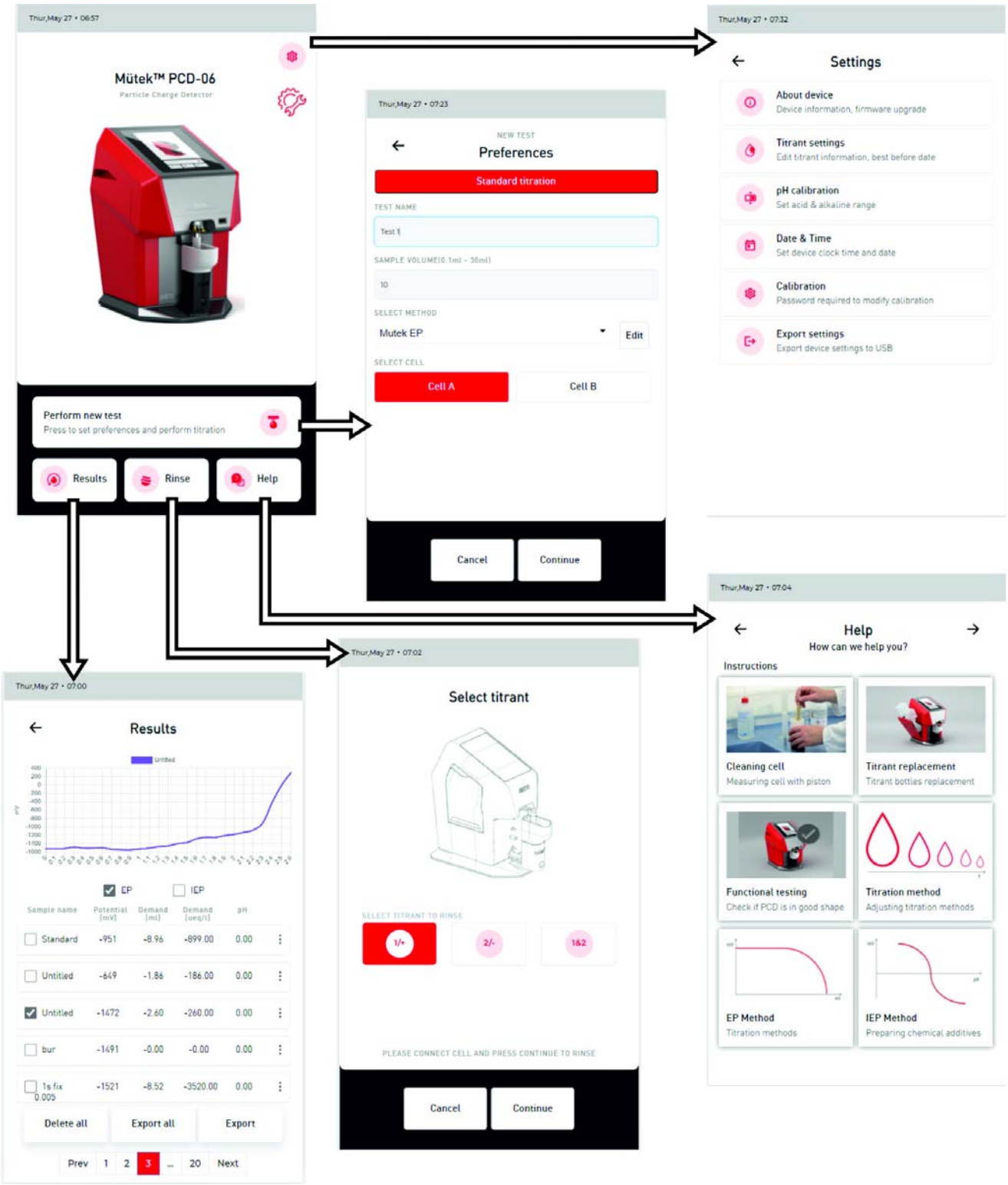
5.1 Safety rules

Be sure to follow the chapter I. "Basic safety instructions", page 7 before commissioning the Mütek™ PCD-06.

5.2 Personnel requirements

Only qualified and skilled personnel are allowed to operate the Mütek™ PCD-06.

5.3 Overview Main Screens



5.4 Sample preparation

For stock systems including both colloidally dissolved material and solid particles, different ways of sample preparation will give different data on the charge conditions prevailing in a sample (see 4.4.3 "Sample specification", page 24).
Generally speaking, results are only directly com-parable if the samples are prepared in an identical manner. Test results obtained for samples of the same kind but which were treated by different methods may yield additional information about, for example the charge conditions of solids.

In each case, quantitative sampling must be ensured to obtain reproducible values.

5.4.1 Filtration

To separate larger solid particles from a sample, a whiteband filter is normally used. However, this may falsify results because polyelectrolytes tend to be adsorbed at the filter paper. We therefore strongly advise against using paper filters.

The use of ceramic filters by comparison is normally problem-free.

5.4.2 Centrifugation

The centrifugation period must be identical for all samples.

5.4.3 Screening

In the majority of cases, it will be sufficient to separate long fibres and larger particles by means of a nylon screen.

Quick and reproducible sampling is achieved by immersing a Mütek™ filtration beaker with screen in the sample and removing the supernatant liquid with a measuring pipette.

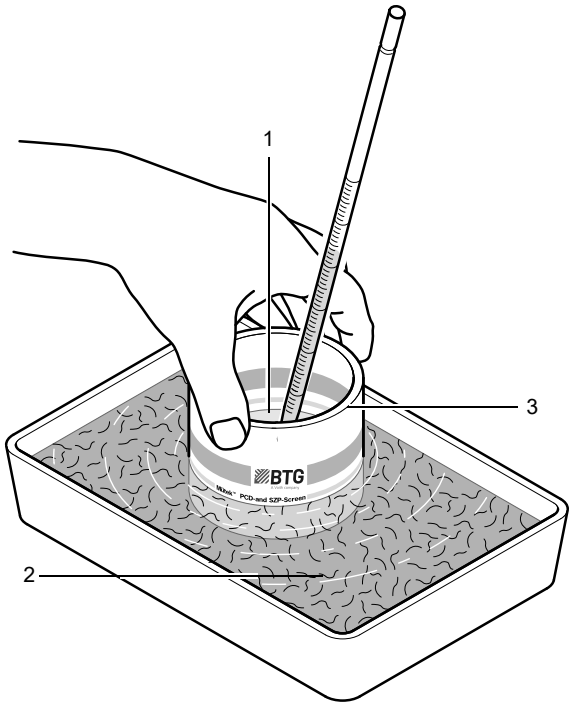


Fig. 5-1 Screening of samples

Designations

1 Rising measurable sample
2 Original sample, for example thick stock, or coated broke
3 Filtration beaker with 300 µm screen

5.4.4 Back titration

This method of sample preparation suggests itself in the following cases:

- Due to their structure, the samples cannot be transferred to the cell directly which would enable quantitative determination of the charge level of larger particles
- The reaction of the substance to be tested with the titrant is too slow for direct titration

5.4.4.1 Example for back titration

Sample

The total charge of an anionic solid sample is to be determined. In view of the high solids content or excessive particle sizes, direct measurement in the measuring cell is not feasible. Instead, back titration is recommended.

Procedure

Take 20 g of the anionic sample to be measured and fill up to 100 g with cationic titrant 0.001 N. Stir this mixture for approximately 2 hours with a magnetic stirrer. During this period, the cationic cationic titrant will neutralize all anionic charges of the sample. Since there is an excess amount of cationic titrant, a residual cationic charge will remain in the sample.

After the reaction period, remove the solids from the mixture by screening. Pipette 10 ml of the solution into the measuring cell and titrate up to the point of zero charge with anionic titrant 0.001 N.

Result V1: Consumed anionic titrant 4.83 ml

In addition, titrate 10 ml of fresh cationic titrant solution with anionic titrant up to the point of zero charge.

Result V2: Consumed anionic titrant 10.68 ml

Calculation of charge density q:

$$q = \frac{(V2 - V1) * c}{m}$$

V2 - V1: The difference between the charges of the fresh cationic titrant and the reacted cationic titrant indicates the charge amount neutralized in the sample.

c: Concentration of the titrant, here 0.001 eq/l

m: 2 g of the original sample were used for titration. (20 g were filled up to 100 g of which 10 g were used for titration.)

$$q = \frac{(0.01068 - 0.00483)\ [l] * 0.001\ [eq/l]}{2\ [g]}$$

$$q = 2.925 * 10^{-6}\ [eq/g]$$

Result: The charge of the sample under test is 2.925 µeq/g.

5.5 Fill sample into the measuring cell

Preparation

Information

Since both electrodes inside the measuring cell must be covered with liquid, the sample volume should be at least 10 ml. To precisely dose the sample volume for charge calculation, weigh in the sample or use a measuring pipette.

Information

Good reproducibility of results is only achieved if the sample is appropriately homogenized for quantitative sampling.

- Insert the displacement piston in the measuring cell.
- Fill a sample into the measuring cell.
- Move the measuring cell along the guide up to the stop.
- The piston will automatically be lifted and locked.
  - Assure that the marking facing the front

Fig. 5-2 Measuring cell and displacement piston

- If applicable, install pH electrode in the measuring cell (see Fig. 5-4 "Insert pH electrode", page 34).

Hint

The pH electrode must immerse in the sample solution. Therefore it is necessary to use an increased sample volume of 30 ml for isoelectric point measurements. Use more sample material and do not dilute the sample with water!
When just one pH value has to be measured (no pH curve or IEP measurement) the pH of the 10 ml sample can be measured outside before filling into the measuring cell.

5.6 Measuring with the Mütek™ PCD-06 Light

Information

The display is equipped with a screen saver which automatically appears after 20 min. To deactivate the screen saver just touch the screen.
The time when the screensaver should start can be adjusted under "Calibration".

Information

The pH screen shown on the following screens is only available to users who have ordered the option "pH Set" with the instrument.

5.6.1 Manual Titration

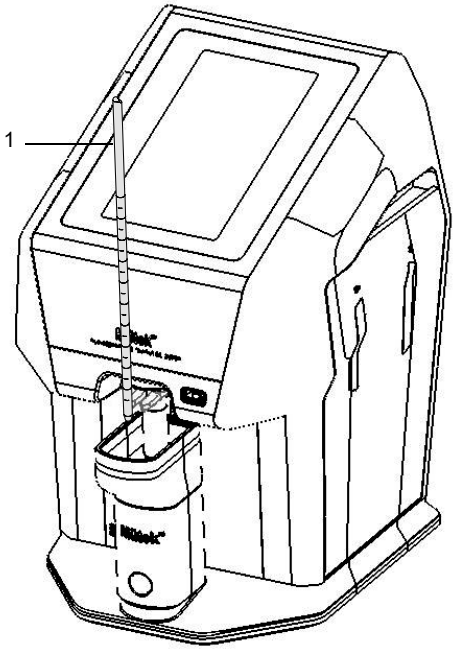


Fig. 5-3 Manual operation

Designation

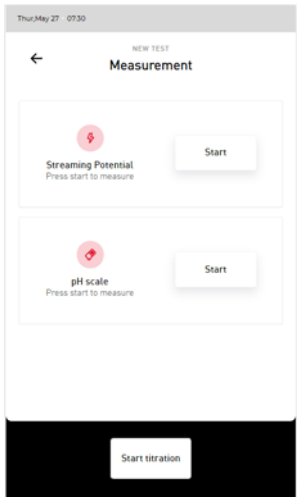
1 Measuring pipette

If you conduct an acid/base titration with the Mütek™ PCD-06, the pH display indicates the isoelectric point (pH value at 0 mV streaming potential).
Manual titration with a measuring pipette should solely be carried out for pretests.

5.6.1.1 Start measurement

- Assemble the Mütek™ PCD-06 Light according to chapter 4.6 "Start-up procedure", page 27.
- Switch on the PCD-06 Light
- Choose New test
- The Preferences screen appears
- Select Cell A or Cell B and press Continue

The measure screen will appear:



- Fill in measuring sample and install the measuring cell according to chapter 5.5 "Fill sample into the measuring cell", page 33.
- With pH option, insert the pH electrode into the measuring cell and press the pH "START" button (see Fig. 5-4) before starting the potential measurement.

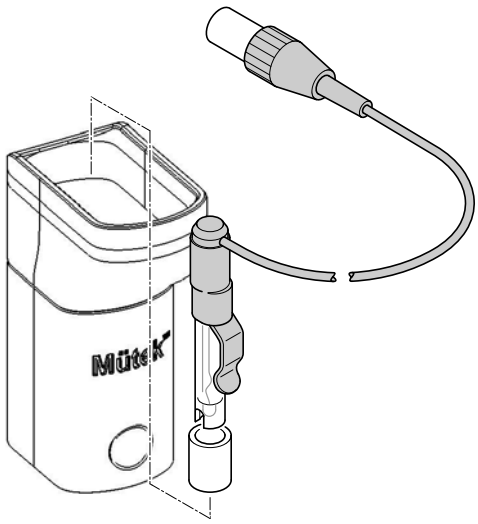


Fig. 5-4 Insert pH electrode

- To start the potential measurement in mV, press the button "START".
  - This will cause the displacement piston to oscillate and the streaming potential to be displayed in mV
- Wait for approximately 30 seconds until the signal has stabilized.
  - For later data storage note down the streaming potential start value mV (here: -935 mV)

5.6.1.2 Start titration

- Use a pipette to dose oppositely charged titrant until point of zero charge 0 mV is reached (see 5.6.1 "Manual Titration", page 34).
- Note down dosage amount ml and pH value (option) at point of zero charge as your measuring result.

Information

Since the displacement piston continues its oscillating movement, the streaming potential and pH signal (optional) may change. This is a normal effect without any influence on measurement results.

- Press "Off" (to the left of the "Potential" field) to stop the displacement piston from oscillating.

5.6.2 Automatic titration with external titrator

Optionally the Mütek™ PCD-06 can be combined with an automatic titrator.

Information

Streaming potential and pH signal (optional) will be send to the titrator.

Information

For more detailed information about the titration setting parameters and data storage, please refer to the manual of your external titration system.

5.6.2.1 Start measurement

- Start the potential measurement according to (see 5.6.1.1 "Start measurement", page 34).

5.6.2.2 Start titration

- Select oppositely charged titrant on your titrator
  - sample charge: anionic = - mV signal, select cationic titrant
  - sample charge: cationic = + mV signal, select anionic titrant
- Select endpoint titration 0 mV on your external titration system.
- Insert the dosing tube into the measuring cell.
- Press START on your external titrator.
  - The titrator stops automatically at the endpoint 0 mV streaming potential
- Note down the indicated ml result shown on your external titrator system.
- Based on consumption, the charge level can be quantitatively calculated especially in the case of a polyelectrolyte titration. See chapter 2.1.2 "Calculation of charges", page 13.

Information

Since the displacement piston continues its oscillating movement, the streaming potential and pH signal (optional) may change. This is a normal effect without any influence on measurement results.

- Press "Off" ("Potential" field) to stop the displacement piston from oscillating.
- Press "Off" ("pH" field) to stop the pH measurement (optional).

5.7 Measuring with the Mütek™ PCD-06 Standard or Premium.

Information

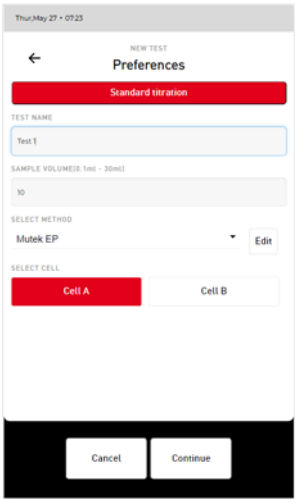
The display is equipped with a screen saver which automatically appears after 20 min. To deactivate the screen saver just touch the screen.

Information

The pH display shown on the following screens is only available to users who have ordered the option "pH Set" with the instrument.

5.7.1 Start Measurement

Press "Perform new test" in the landing page to come into the "Preferences" screen.

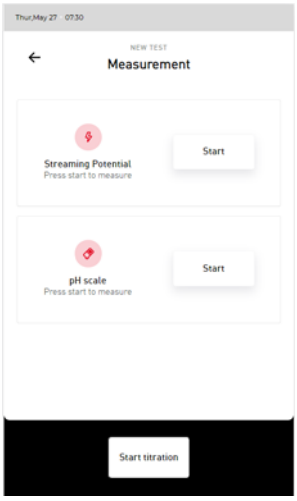


Set all required measurement preferences like:

- Sample name
- Sample volume
- Titration method
- Cell A or B

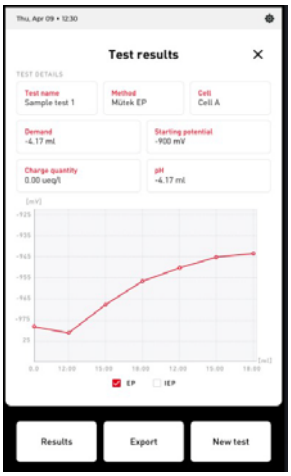
Press "Continue" to come into the "Measurement" screen.

Press "Start" in Streaming Potential Field.



- This will cause the displacement piston to oscillate and the streaming potential to be displayed in mV.

- Wait for approximately 30 seconds until the signal is stabilized.
  - The alternative is to set a time when the titration will start.
- Fill the sample with the adjusted volume into the measuring cell.
- Press "Start titration".



- A titration curve will be shown in real time until the measurement reach the end point criteria.
- In the Test result screen you will see all relevant measurement settings as well as the titration result in demand and charge quantity.
- All test result including measurement parameters will be stored automatically.
  - To make a new measurement press "New Test" to come back to "Preferences" screen.
  - To Export the measurement to an USB stick press "Export".
  - To view historical measurements press "Results".

Information

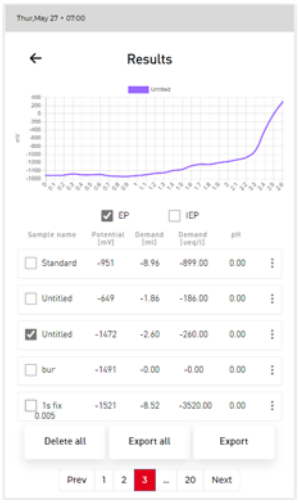
Since the displacement piston continues its oscillating movement, the streaming potential and pH signal may change. This is a normal effect without any influence on measurement result.

Information

When reaching the end point criteria the result will be stored automatically. The final demand will calculate back to 0 mV.

5.7.2 Result handling Mütek™ PCD-06

All measurement results will be stored in the internal memory. During measurement the diagram screen will show the measurement progress:



For parameter transfer to an USB stick refer to chapter 5.7.3.1 "Transfer Parameter to a USB stick", page 38.

5.7.3 Result transfer to a USB stick

For result transfer to a USB stick, plug in a USB stick in the front USB slot of the instrument:

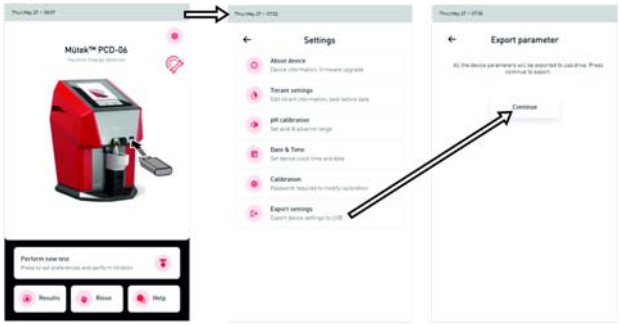
Open the menu “Results” from landing page.

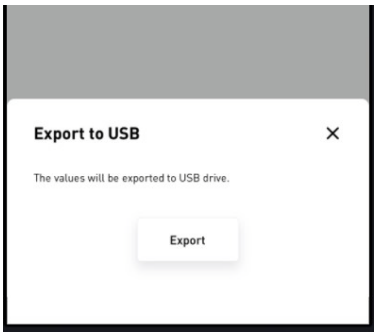
Press either “Export all” to transfer all measurement or “Export” to transfer only the marked measurements.

5.7.3.1 Transfer Parameter to a USB stick

For Parameter transfer to a USB stick, plug in a USB stick in the front USB slot of the instrument.

- Open the menu by pressing the setting symbol at the top of landing page.
- Press "Export Settings".



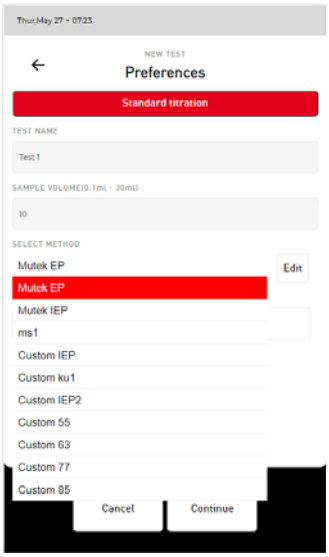


- All results and parameters will be transferred to the plugged in USB stick as a .csv-file.

5.8 Methods

Adjusting titration methods

The PCD-06 features two factory set, non-editable methods:
one for endpoint titrations (EP) and one for iso-electric point titrations (IEP).
To open and edit a method, open the drop down menu, select any method and press "Edit".



5.8.1 Setting of standard methods

Method 900:

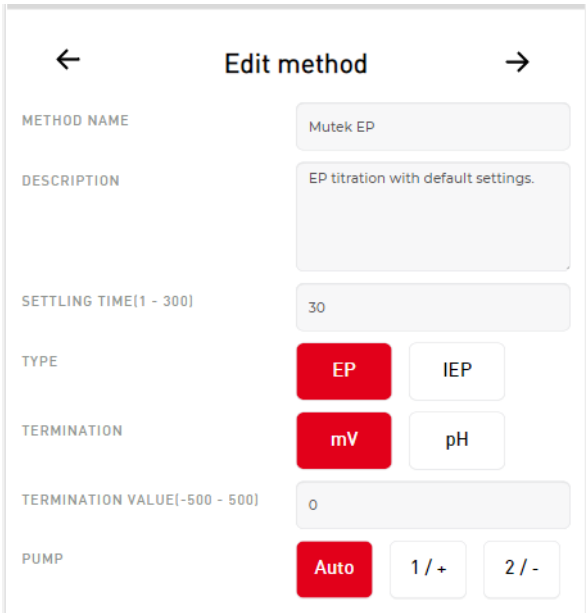
Termination	mV	**EP**	0 mV
Pump	Automatic		
Type	EP		
Meas. Mode	fixed	**Increment**	3 s
Addition	Dynamic	**dE (set)**	8 mV
		dV (min)	0,01 ml
		dV (max)	0,1 ml

Method 901:

Termination	pH	**EP**	7,00 pH
Pump	Automatic		
Type	IEP		
Meas. Mode	Adaptive	**dE**	1,0 mV
		dt	1 s
		t (min)	3 s
		t (max)	15 s
Addition	Dynamic	**dE (set)**	8 mV
		dV (min)	0,005 ml
		dV (max)	0,05 ml

Editable method parameters are as follows:

5.8.2 General Method Settings



Method name

Name or rename a method here by pressing the text field.

Short description

Here you can describe the method in a few words. Your colleagues will honor this!

Settling time

A time to stabilize the mV signal at the beginning of a test. After starting the measurement, the unit will count down the settling time and then automatically start the titration.

⚠ Hint

Use this function when the sample needs a certain time to stabilize - and to make sure, that this time is always the same in every test. No matter who is testing.

Type

EP stands for endpoint titration. Results are titrant demand [ml] and charge density [µeq/l]. Default diagram type is mV/ml.

IEP stands for iso-electric point titration. Result is the IEP [pH] and the default diagram type is mV/pH.

Termination

Select if the titration endpoint should be at a mV or a pH value.

Termination value

Adjust the value (mV or pH) you want the titration to end.

⚠ Hint

The typical termination value of an EP titration is 0 mV. However, there is no sharp cut at 0 mV, instead the signal will cross the zero line and show the opposite sign. There is some over-titration taking place, but no worries - the result will be calculated back to 0 mV.

⚠ Hint

The point of neutral charge is also the point of highest instability of the sample. Flocks and coagulates form and might break again under the strokes of the PCD piston. This instability can cause the mV signal to return, even if it has already crossed the 0mV mark. In such a case you should consider setting a termination value, that is beyond the point of zero charge (for example +50 mV for a negatively charged sample). This makes sure, that all sample charge is neutralized by the titration.

Pump

Select how titrant pumps are to be used:

Auto: the PCD will check the sign of the mV signal and will automatically use the pump with the opposite sign.

1/+ or 2/-: independent from the mV signal, only the selected pump will be used for titration.

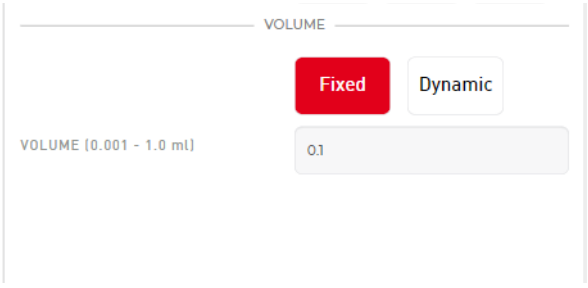
⚠ Hint

Apply this function in case you use two cationic titrants, for example one higher concentrated in position 1/+ and one lower concentrated in position 2/-.

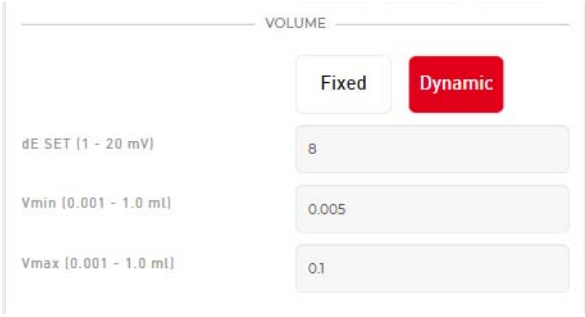
5.8.3 Volume

Here you can program, how much titrant is to be injected per dosing step.

Fixed:
The pump will deliver the same amount of titrant in every dosing step.



Dynamic:
Selecting this mode, the dosage will be set under dynamic control. Parameters for controlling the dosage are ΔE, Vmin and Vmax. ΔE is the mV signal change between two dosage steps, and Vmin Vmax specify, how the titrant pump should respond to this signal change.



Example: ΔE be set to 8 mV. If the signal changes more than 8 mV, the pump will deliver less titrant with a minimum of Vmin. If the signal change is smaller than 8 mV, the pump will deliver more titrant with a maximum of Vmax.

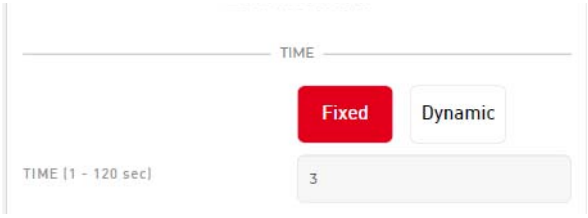
⚠ Hint

If you want titrations to be more precise, go for lower values in ΔE, Vmin and Vmax. And vice versa, if not accuracy but speed is the primary goal: set ΔE, Vmin and Vmax to higher values.

5.8.4 Time

Here you can program the time interval between two titrant dosing steps.

Fixed:
A dosage will take place every time as programmed here.

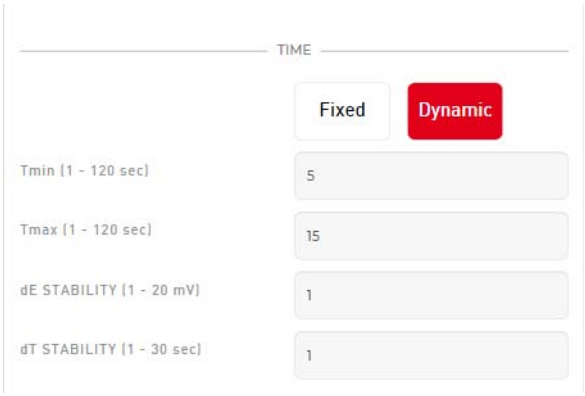


Hint

You want to make sure, that the mV signal has stabilized after a titrant dosage. If you notice that the mV signal is still busy stabilizing while the next titrant injection already takes place, select a higher value here.

Dynamic:
Selecting this mode, the time interval between two dosing steps will be set under dynamic control. Parameters for controlling the time interval are ΔE (Stability), Δt (Stability), tmin and tmax. The approach is to achieve a certain signal stability - defined via ΔE (Stability) and Δt (Stability) - until the next dosage can take place. The minimum and maximum waiting times for this stability are tmin and tmax.

The increment is within the range between minimum (dVmin) and maximum(dVmax) increment.



Example:
ΔE (Stability) is 1 mV, Δt (Stability) is 1 s, tmin is 5 s and tmax is 15 s. Means that the PCD continuously checks for the defined stability criterion: the signal must not swing more than 1 mV within 1 s.
And it waits at least 3 s, but not longer than 15 s for this stability criterion to be achieved.

Hint

Dynamic time control is the preferred mode for IEP titrations. Acid or base additions can make the mV signal swing strongly. A further acid or base injection should only take place when the signal swinging is over.

Start dynamic control at:

You can adjust, at which mV value all dynamics programmed under "Volume" and "Time" should start.

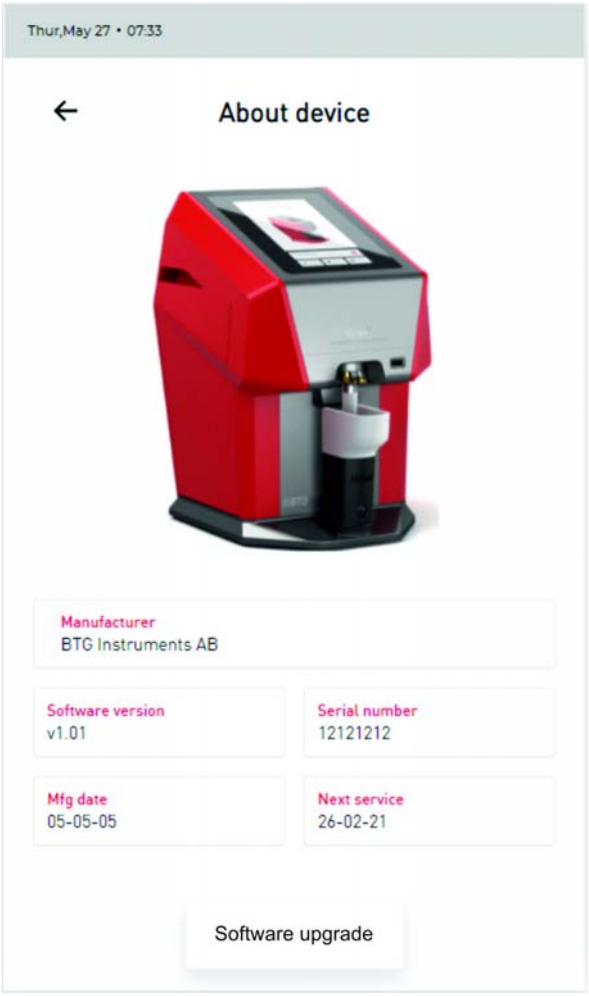
Hint

Titrations oftentimes have a distinct constant part and become variable when approaching the point of neutral charge. You might want to bridge the constant part fast and the dynamics to kick in only when the signal becomes variable. So, observe the titration curve and read out the mV value where the slope starts to change. Set this value as starting point for the dynamic control.

If no dynamics (DYN) are selected under "Volume" or "Time", this feature is hidden.

5.9 Settings

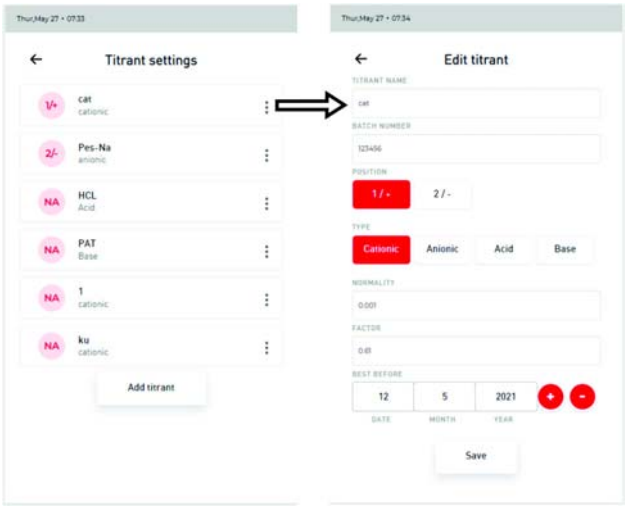
5.9.1 About device



Manufacturer	BTG Instruments AB
Software version	Shows the software version of the base unit
Serial number	Serial number of the PCD-06
Manufacturing date	Shows the date when the PCD-06 was produced
Next Service	Shows the next recommended service date
Service Settings	By entering a password 4374 further calibration can be done, this should be done only from advanced users, see chapter 5.9.4, page 45
Software upgrade	A upgrade can be done when a USB stick with Software was inserted at the device

5.9.2 Titrant Settings

Select the titrant for those settings to be changed, press on the three dots to open the submenu.



The following parameters can be adjusted:

Titrant name	Opens a keyboard to enter any name
Batch number	Opens a keyboard to enter a batch number
Position	Enter where the titrant is mounted at the device
Type	Select the titrant type
Normality / Molarity	For cationic or anioic titrant enter the normality For Acid or base enter molarity
Factor	Correction factor for result calculation
Best before	Enter the date of opening the titrant bottle plus 6 month
Delete	Delete this titrant
Save	Save the titrant settings

5.9.3 pH Calibration (optional)

Information

If the Mütek™ PCD-06 is connected to an external titrator system, the pH electrode has to be simultaneously calibrated on the titrator and Mütek™ PCD-06.

For calibration, use two commercial buffer solutions, one of them with pH 7.0 and the other with pH 4.01 or pH 9.21.

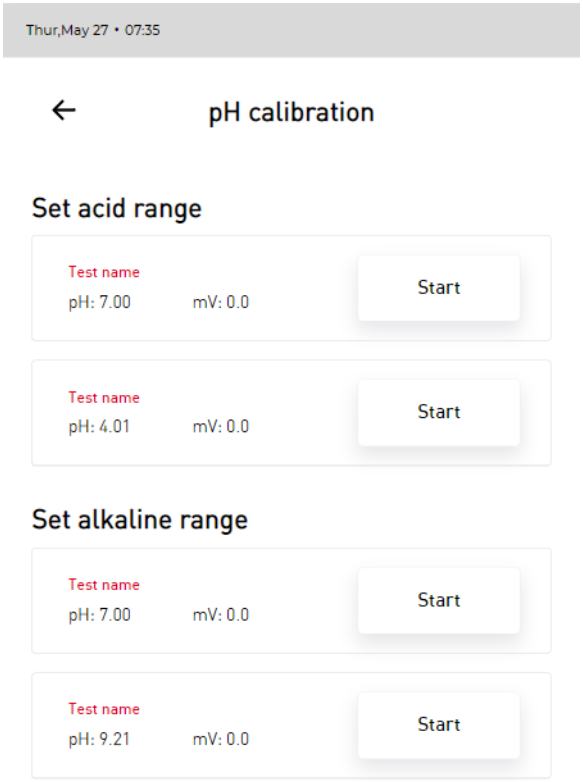
Information

The buffers used for calibration should correspond to the pH range of the samples.

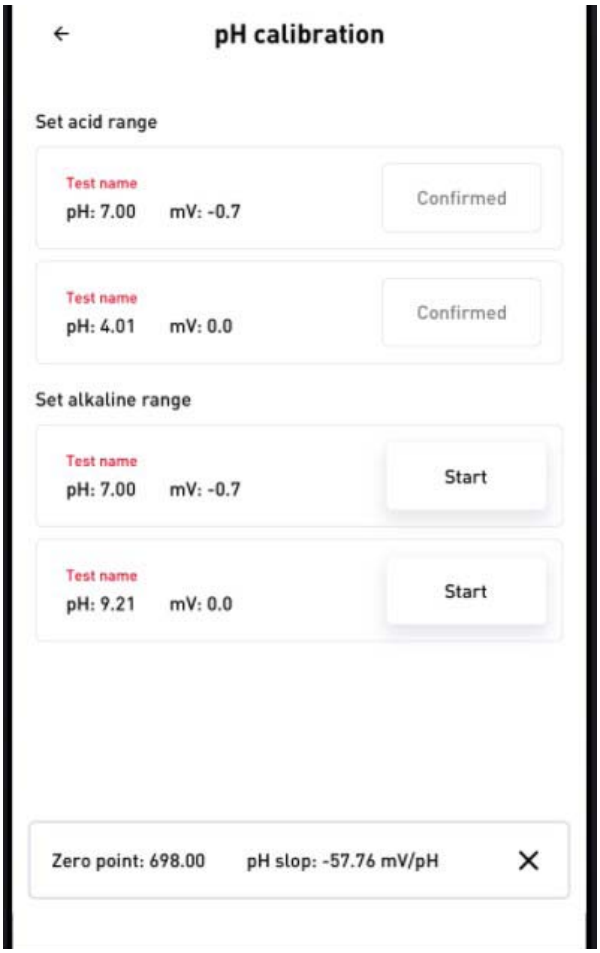
Procedure

- Under the tab "Settings", select "pH calibration".

Depending on the range of the second buffer solution you wish to use, touch the appropriate field (acid pH 4.01 or alkaline pH 9.21)



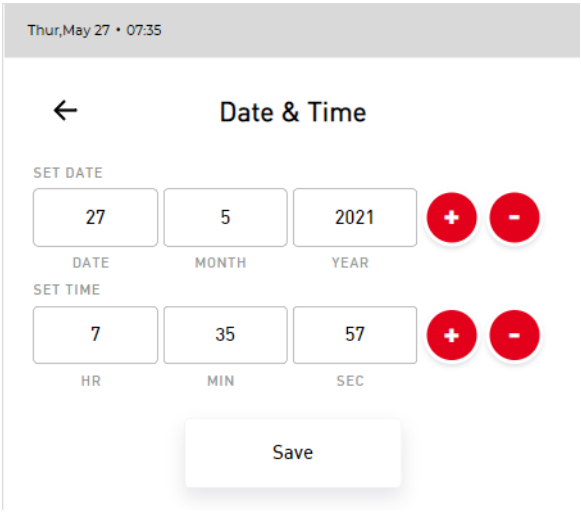
- This will open the following screen:



- Plug the pH electrode at the rear panel of the Mütek™ PCD-06.
- Remove the electrode cap and rinse the electrode with deionized water.
- Immerse the pH electrode in the pH 7.00 buffer solution and select "Confirmed".
- Wait until the displayed mV value has stabilized and select "Confirmed".
- Rinse the electrode with deionized water again.
- Then immerse the pH electrode in a buffer solution with pH 4.01 for acid range or pH 9.21 for alkaline range and select "Start".
- Wait until the displayed value has stabilized and select "Confirmed".

5.9.4 Date and Time

Adjust date and time in this menu:

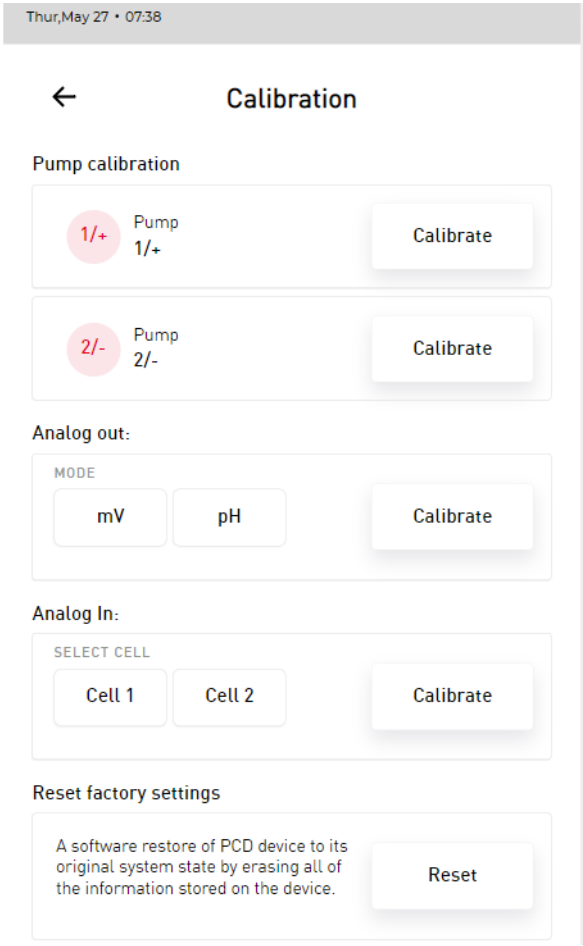


Date format:
Choose the required date format
Set date:
Select "Date", "Month" and, "Year", adjust with + or -
Set time:
Select "Hour", "Min", and "Sec", adjust with + or -

5.9.5 Calibration

Information

With the following settings the instruments calibration will be changed, this has a direct influence to the measurement results. This setting should be changed only from a skilled user.



Pump calibration:

1 Press the "Calibrate" button on the required pump
2 Start the dosing
3 Weigh the volume and enter it to the screen
4 Repeat it two times
5 The device will calculate the average and store the new value

Analog input potential:

1. Select either cell 1 or cell 2 depending which cell you want to calibrate
2. Clean measuring cell manually
3. Fill in 10 ml 0.001 N anionic titrant
4. Insert measuring cell and hook piston
5. Press "Calibrate" in the "Analog In potential" field
6. Wait until it becomes stable, then press "Confirmed"

Analog output:
In case the PCD-06 is connect to an external titrator the output signal can be adjusted to this titrator.

1. Display the potential value in the titrator (for that refer to the titrator manual).
2. Press the "Calibrate" button for Analog out in the PCD-06 screen
3. Select -1.6 V and start the calibration
4. At the titrator it should show now -1.6 V if not it can be adjusted with + and -
5. Same procedure should be done with 0 mV and +1.6 V

VI. Diagnostics and troubleshooting

6.1 Safety instruction

If errors occur that are not listed in the table below, disconnect the Mütek™ PCD-06 and tag it as "defective". Contact your local BTG division to ensure quick corrective action.

6.2 Personnel requirements

Troubleshooting may only be performed by qualified and skilled personnel.

6.3 Table of malfunction

6.3.1 Error messages on the PCD-06 Smart

Error message	Cause	Corrective action
Warning: No endpoint detected	- Maximum titrant volume of 20 ml reached - pH end point is reached before 0 mV is reached	• Verify correct position of anionic and cationic titrant.
		• Rinse titrant tubes thoroughly by filling them with titrant.
		• Increment too low adjust method
Slope out of range! The values are not saved	- pH electrode contaminated or defective	• Clean pH electrode, if necessary replace it.
Error: Transfer failed!	- USB stick not identified or writeable	• Unplug and replug the USB stick, retry transfer.
		• Control USB stick format.
		• Control USB stick regarding partitioning. - An USB stick with partitioning does not work

6.3.2 Basic faults

The following basic faults may be experienced with the Mütek™ PCD-06 also when used in combination with an external titrator:

Problem	Cause	Solution
Heavy fluctuations of streaming potential after sample is filled in	- Excessive conductivity of the sample	• Dilute sample.
	- Insufficient sample volume in measuring cell	• Weigh in once again or use pipette.
	- High solids in the sample	• Dilute the sample or centrifuge/ screen it to remove coarse particles.
Potential reverses after reaching the endpoint	- Very slow reaction between titrant and sample	• Titrate more slowly or perform back titration.
		• If necessary, check sample preparation for homogeneity.
At the start of titration, potential increases instead of decreasing	- Wrong titrant	• Select titrant with opposite charge to that of sample.
	- Adsorption equilibrium not attained	• Proceed slowly with titration.
	- Sample concentration too high or titrant concentration too low	• Dilute sample.
Potential remains essentially unchanged during titration	- Sample concentration very high	• Dilute sample or use a higher strength titrant.
	- Wet contacts on Mütek™ PCD-06	• Dry contact pins with a soft cloth.
	- Wet electrodes on the outside of the measuring cell	• Dry electrodes with a soft cloth.
	- Measuring cell contaminated	• Thoroughly clean measuring cell and displacement piston.
Sign of displayed potential not in conformity with sample specification	- Sample charge is pH dependent	• Check on pH dependence of sample.
	- Excessive viscosity	• Dilute sample.
	- Measuring cell contaminated	• Thoroughly clean measuring cell and displacement piston.

VII. Maintenance

7.1 Cleaning

7.1.1 Cleaning the instrument

Use a soft and lint-free cloth to wipe the outer surfaces of the instrument. Take care not to scratch the touch-panel. If contamination persists, moisten the cloth with soap water. A special cleaning procedure is required for the measuring cell.

7.1.2 Cleaning the measuring cell

Clean the measuring cell with water and cleaning solution only.

After each measuring cycle, go through the following cleaning routine:

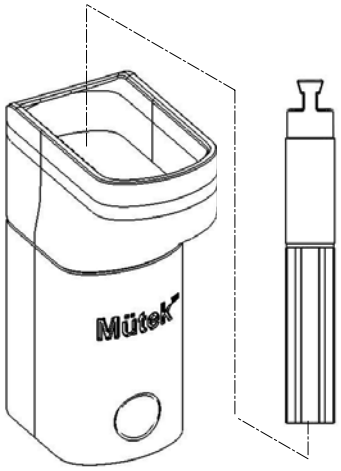


Fig. 7-1 Measuring cell and displacement piston

- Empty the measuring cell.
- Rinse it with water.
- Use a brush and cleaning solution (see chapter 4.5.3 "Cleaning solution", page 26) to clean measuring cell and displacement piston.
- Rinse both with de-ionized water.
- Empty the measuring cell.
- Use a clean paper cloth to dry the outside of the measuring cell - but NOT the inside of the cell or the displacement piston.

⚠ Hint

Not cleaning the measuring cell after each measurement will lead to measuring errors and prevent reproducibility of results.

To avoid deposits in the measuring cell and on the replacement piston, fill the freshly cleaned cell with water and leave overnight.

7.2 Maintenance

7.2.1 Personnel requirements

Maintenance shall exclusively be carried out by qualified and skilled personnel.

7.2.2 Functional testing

To avoid measuring errors caused by faulty operation of the instrument or contamination of the measuring cell, functional testing can be performed by different methods.

7.2.2.1 Basic test of PCD-06 without measuring cell

- Turn on power switch
- Switch on displacement piston drive
  - A streaming potential of 0 mV will be displayed

7.2.3 Standard titration

Standard titration with Mütek™PCD-06 Light

Fill exactly 10 ml of the anionic standard anionic titrant 0.001 N into the measuring cell and titrate with cationic titrant 0.001 N up to the point of zero charge.

The consumption of cationic titrant should be in the range of **9.75 - 10.25 ml.**

[i] Information

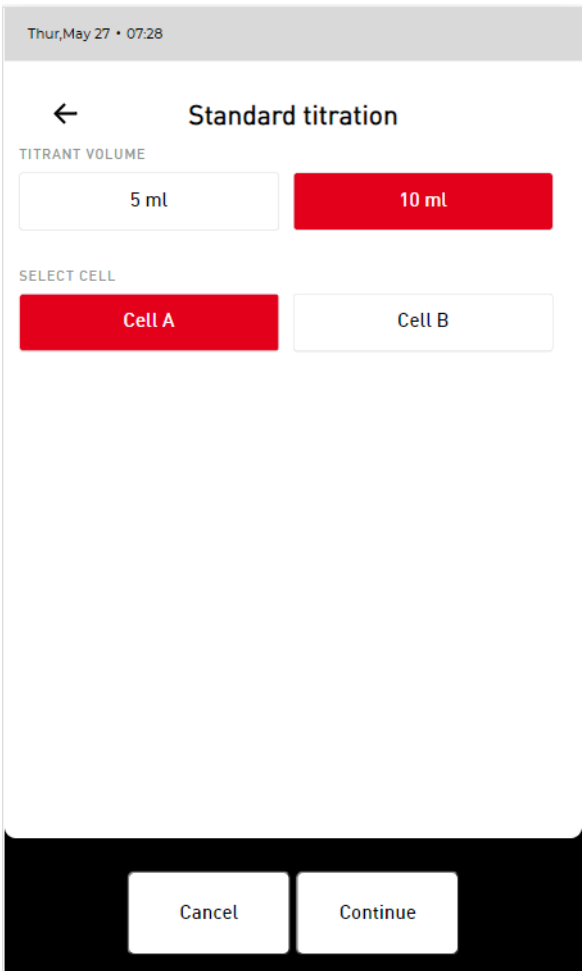
With the Mütek™PCD-06 a standard titration can be performed automatically, as described below.

Standard Titration with Mütek ™ PCD-06 Premium

- Press "New test"
- Select "Standard titration"

[i] Information

If you select 5 ml anionic titrant, fill 5 ml de-ionized water into the measuring cell.

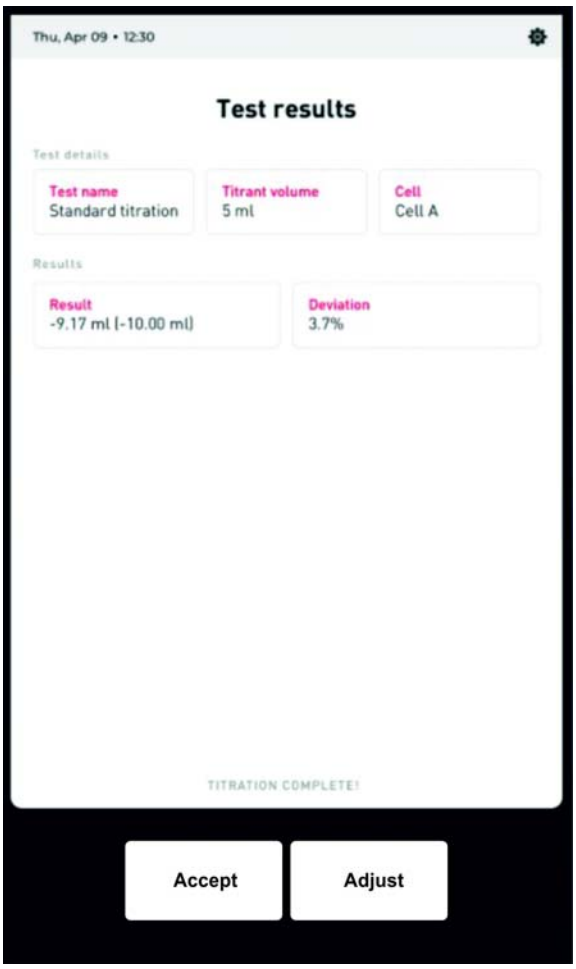


- Select 5 or 10 ml

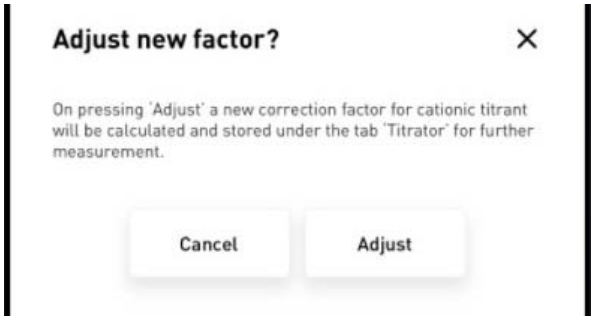
Information

If you select 5 ml fill 5 ml de-ionized water into the measuring cell.

- Press "Continue"
  - Anionic titrant will be dosed into the measuring cell
  - Titration will start automatically
  - Test result screen will be displayed



- Press "Accept" to return to "NEW TEST" without any changes
- Press "Adjust" to set a new factor for the cationic titrant.



Information

A standard titration verifies functionality of the Mütek™ PCD-06 and checks on the concentation of the used titrants.

7.2.4 Checking the streaming potential

If a freshly cleaned measuring cell is filled with 10 ml of anionic titrant 0.001 N repeatedly in succession, the streaming potential measured should be roughly identical. Depending on the instrument, the streaming potential is generally between -1200 mV to +1800 mV.

If the cell is used very frequently, this value will diminish over time due to wear. This does not affect titration results.

However, if the streaming potential suddenly drops by several 100 mV from one measurement to the next, the measuring cell is probably soiled. To avoid measuring errors, clean the cell thoroughly (see chapter 7.1.2 "Cleaning the measuring cell", page 49).

7.2.4.1 Electrostatic charging of the measuring cell

When the mV signal remains extremely stable during the titration process and you are in doubt if the Mütek™ PCD-06 receives a signal from the measuring cell, test the instrument functionality as follows:

Switch on the motor and rub your hand quickly up and down on the outside of the cell. This causes the signal to bounce. If no such reaction is observed although the motor is running, there is probably an electronics failure. In this case please contact the Service Department of BTG Instruments or your local agent of BTG Instruments .

7.2.5 Functional testing of the pH electrode

Before starting a test series, measure a buffer solution by means of the pH electrode which should respond within 10-30 seconds. If it does not, clean and re-calibrate the electrode. If the fault persists, replace the pH electrode.

The pH-electrode is a consumable goods.

Its life time is guaranteed approximately 6 months.

7.3 Proper handling of the pH electrode

The pH electrode must not fall dry. Therefore always put on the protection cap, when not using the electrode. Alternatively use a tiny beaker with 3M KCl solution for parking position inbetween measurements.

Always fill up the protection cap with fresh 3M KCl solution when you've finished operation. This will regenerate the electrode ever time after use and will prolong the life time of the electrode. The pH electrode will consum the KCl ions, therefore it is needed to always use fresh KCl solution.

Be carefull when laying the pH electrode on a table, in order not to break the diaphragm. It is recommend always to insert the pH electrode into a tiny beaker with 3M KCl solution for parking position inbetween measurements.

Always calibrate the pH electrode (see chapter 5.9.3 "pH Calibration (optional)", page 44) prior to your measurements (at beginning of every working day with the unit).

The buffer solutions supplied are meant as single use solutions. In order to assure reproducible results always use new/ stable buffer solutions.

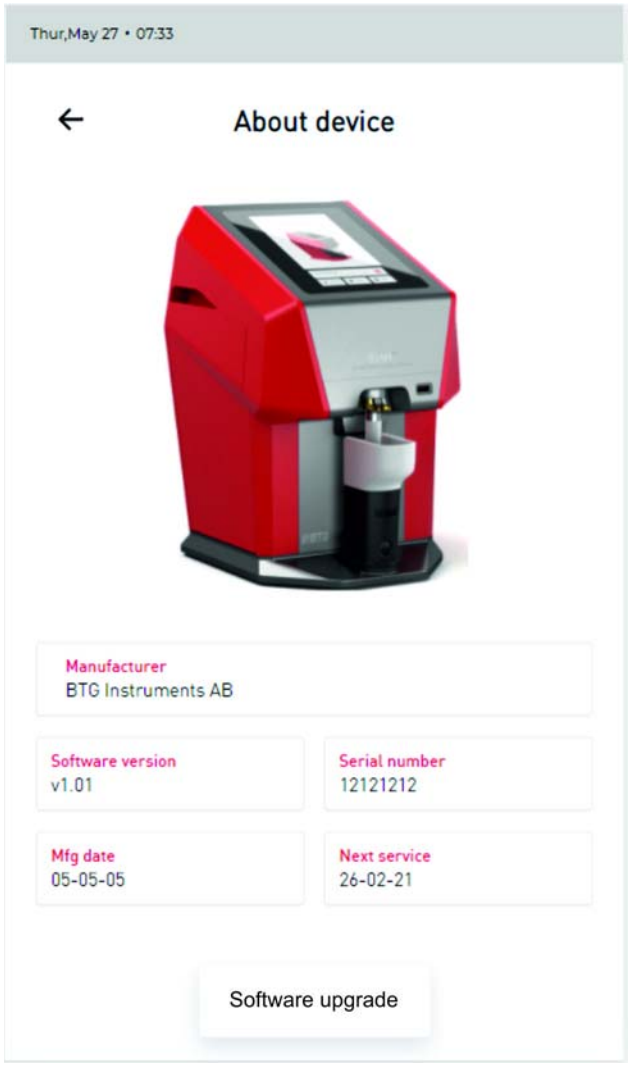
7.4 Yearly LAB Check™ inspection

To guarantee reliable measurement from the start, every Mütek™ laboratory instrument undergoes thorough testing before leaving the factory.

BTG's maintenance program LAB Check™ keeps your laboratory device in perfect working order year after year. Feel free to call in your local service team or send in your system for factory maintenance.

To keep track of the next inspection interval, the display on Mütek™ PCD-06 indicates a reminder message:

The next service date can bee seen in the "About device" screen.



VIII. Shutting down the system, storage and disposal

8.1 Safety instructions

Please adhere to the applicable safety rules when shutting down the system.

8.2 Personnel requirements

The system shall only be shut down by qualified and skilled personnel.

8.3 Shutting down the system

To place the Mütek™ PCD-06 out of operation, proceed as follows:

- Turn off the instrument.
- Disconnect the instrument from the mains.
- If applicable, disconnect the Mütek™ PCD-06 from the titrator.
- Clean the measuring cell (see chapter 7.1.2 "Cleaning the measuring cell", page 49).

If you use a pH electrode:

- Clean the pH electrode with a 3 M KCl solution.
- Prevent the diaphragm of the pH electrode from drying up. Fill the cap of the pH electrode with 3 M KCl solution and keep the electrode in this cap in times of disuse.

8.4 Storage

Store the Mütek™ PCD-06 in a suitably protected place. Do not keep outdoors.

8.5 Disposal

8.5.1 Official Regulations

Be sure to meet the current local regulations governing the disposal of the Mütek™ PCD-06.

IX. Parts list

9.1 Spare parts list

Item	Description	Part No.
1	Measuring cell for Mütek™ PCD-06	HA2004839
2	Displacement piston for Mütek™ PCD-06	PA2005535
3	External power supply	P00X50359
4	Piston brush for cleaning the measuring cells, d = 20 mm	P00X10158
5	Beaker with screen	P00X18977
6	Precision pipette 2-10 ml	P00X18772
7	Plastic tips for precision pipette 2-10 ml	P00X18773
8	Seal for precision pipette	P00X18776
9	pH electrode, plexiglass shaft, ceramic diaphragm, gel filling	H00X09115
10	pH electrode, glass shaft, glass section diaphragm, gel filling	H00X09133

9.2 Options

Item	Description	Part No.
1	Plastic transportation box PCD-06	PA2006019
2	pH set	H00X20130

9.3 Consumables

Item	Description	Remarks	Part No.
1	Cationic titrant bottle 0.001 N, 250 ml	Ready-to-use titrant for Mütek™ PCD-06	HB2000369
2	Cationic titrant bottle 0.001 N, 4x250 ml	Ready-to-use titrant for Mütek™ PCD-06	HB2000370
4	Cationic titrant 0.001 N, 1000 ml	Ready-to-use titrant for Mütek™ PCD-06	H00X10217
5	Anionic titrant bottle 0.001 N, 250 ml	Ready-to-use titrant for Mütek™ PCD-06	HB2000368
6	Anionic titrant 0.001 N, 1000 ml	Ready-to-use titrant for Mütek™ PCD-06	H00X10216
7	Buffer solution	pH 4.01	P00X17592
8	Buffer solution	pH 7.00	P00X17593
9	Buffer solution	pH 9.21	P00X17594
10	Cleaning solution for manual cleaning, 250 ml	Ready to use	H00X10091

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