BTpH

- Bruker Combined Titration pH Unit

User Manual

Version 002

Innovation with Integrity

NMR

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1 About

1.1 This Manual

This manual enables safe and efficient handling of the device.

This manual is an integral part of the device, and must be kept in close proximity to the device where it is permanently accessible to personnel. In addition, instructions concerning labor protection laws, operator regulations tools and supplies must be available and adhered to.

Before starting any work, personnel must read the manual thoroughly and understood its contents. Compliance with all specified safety and operating instructions, as well as local accident prevention regulations, are vital to ensure safe operation.

The figures shown in this manual are designed to be general and informative and may not represent the specific Bruker model, component or software/firmware version you are working with. Options and accessories may or may not be illustrated in each figure.

1.2 Policy Statement

It is the policy of Bruker to improve products as new techniques and components become available. Bruker reserves the right to change specifications at any time.

Every effort has been made to avoid errors in text and figure presentation in this publication. In order to produce useful and appropriate documentation, we welcome your comments on this publication. Support engineers are advised to regularly check with Bruker for updated information.

Bruker is committed to providing customers with inventive, high quality products and services that are environmentally sound.

1.3 Symbols and Conventions

Safety instructions in this manual are marked with symbols. The safety instructions are introduced using indicative words which express the extent of the hazard.

In order to avoid accidents, personal injury or damage to property, always observe safety instructions and proceed with care.

DANGER

DANGER indicates a hazardous situation which, if not avoided, will result in death or serious injury.

This is the consequence of not following the warning.

▶ This is the safety condition.

1. This is the safety instruction.

⚠ WARNING

WARNING indicates a hazardous situation, which, if not avoided, could result in death or serious injury.

This is the consequence of not following the warning.

▶ This is the safety condition.

1. This is the safety instruction.

⚠ CAUTION

CAUTION indicates a hazardous situation, which, if not avoided, may result in minor or moderate injury.

This is the consequence of not following the warning.

▶ This is the safety condition.

1. This is the safety instruction.

NOTICE

NOTICE indicates a property damage message.

This is the consequence of not following the notice.

▶ This is a safety condition.

1. This is a safety instruction.

SAFETY INSTRUCTIONS

SAFETY INSTRUCTIONS are used for control flow and shutdowns in the event of an error or emergency.

This is the consequence of not following the safety instructions.

▶ This is a safety condition.

1. This is a safety instruction.

This symbol highlights useful tips and recommendations as well as information designed to ensure efficient and smooth operation.

2 Introduction

The Bruker Titration and pH (*BTpH*) unit is designed to allow the operator to easily and precisely set the *pH* of a sample. The system is intended primarily for small sample volumes, usually in the 1 ml range.

2.1 Concept

The unit is designed to automatically titrate a buffer, acid, or base to samples in order to set their pH to a programmed value.

The unit is controlled using a touch panel controller, which interfaces to the main unit via a *USB* cable and a *DC* power cable. The +24V DC required by the touch panel is supplied from the Titration unit.

Refer to *Unit Description [30]* for a complete description.

Figure 2.1: The Titration pH Unit

2.2 Intended Use

In *NMR* fingerprinting with wine, the NMR peak spreads can be drastically reduced when the samples are all first set to have as near as possible the same pH. This system was developed to allow a large number of samples to be easily and quickly set to a reference pH prior to running the NMR measurements.

The system is primarily intended for use with water based solutions. For use with other solution types the user must first ensure that the liquid is safe for use with an *ISFET* type pH sensor and that the sensor can operate correctly in said solution. This is the responsibility of the user.

The system can be used for any generic titration pH operation for small sample volumes and small containers. The user must decide regarding the suitability of the system.

It' s not recommended to use a base and/or acid solution with a concentration not higher than 2 molar. Higher concentrated reagents will lead to a quicker wear-out of wetted parts.

It is also critical, for the liquids used, that the containers and fill volumes are such that the unit is capable of mixing the sample properly.

For example:

- If the container is too small the liquid may foam and overflow.
- If the container is too large then the mixing may be incomplete.
- If the sample contains alcohol or other volatile solvents, mixing and long open standing periods may cause these components to evaporate.

2.3 Limitation of Liability

All specifications and instructions in this manual have been compiled taking account of applicable standards and regulations, the current state of technology and the experience and insights we have gained over the years.

The manufacturer accepts no liability for damage due to:

- Failure to observe this manual.
- Improper use.
- Deployment of untrained personnel.
- Unauthorized modifications.
- Technical modifications.
- Use of unauthorized spare parts.

The actual scope of supply may differ from the explanations and depictions in this manual in the case of special designs, take-up of additional ordering options, or as a result of the latest technical modifications.

The undertakings agreed in the supply contract, as well as the manufacturer's Terms and Conditions and Terms of Delivery, and the legal regulations applicable at the time of the conclusion of the contract shall apply.

2.4 Copyright

This manual is protected by copyright and intended solely for internal use.

This manual must not be made available to third parties, duplicated in any manner or form – whether in whole or in part – and the content must not be used and/or communicated, except for internal purposes, without the written consent of the manufacturer.

Violation of the copyright will result in legal action for damages. We reserve the right to assert further claims.

2.5 Warranty Terms

The warranty terms are included in the manufacturer's Terms and Conditions.

2.6 Customer Service

Our customer service division is available to provide technical information. See for contact details. In addition, our employees are always interested in acquiring new information and experience gained from practical application; such information and experience may help improve our products.

2.7 EC Declaration of Conformity

EC-DECLARATION OF CONFORMITY

Bruker BioSpin GmbH

BRUKER

The undersigned, representing the following manufacturer

Manufacturer: **Bruker BioSpin GmbH**
Address: **Silberstreifen 4, 76287 Rheinstetten, Germany**

herewith declares that the product **BTpH H116356**

CE

is in conformity with the provisions of the following EC directives. (including all applicable amendments)

Reference no.	Title
2004/108/EC	Directive 2004/108/EC of the European Parliament and of the Council of 15 December 2004 on the approximation of the laws of the Member States relating to electromagnetic compatibility and repealing Directive 89/336/EEC, former 89/336/EWG
2006/95/EC	Directive 2006/95/EC of the European Parliament and of the Council of 12 December 2006 on the harmonisation of the laws of Member States relating to electrical equipment designed for use within certain voltage limits(Low Voltage Directive), former 73/23/EWG

This declaration is in conformity with the following standard(s) or other normative document(s)
Harmonized standards:

Standard	Title
EN 61010-1:2011	Safety requirements for electrical equipment for measurement, control and laboratory use - Part 1: General requirements (IEC 61010-1:2001)
EN 61326-1:2006	Electrical equipment for measurement, control and laboratory use - EMC requirements - Part 1: General requirements (IEC 61326-1:2005);
EN 61000-3-2:2010	Electromagnetic compatibility (EMC) - Part 3-2: Limits - Limits for harmonic current emissions (equipment input current = 16 A per phase) (IEC 61000-3-2:2005);
EN 61000-3-3:2009	Electromagnetic compatibility (EMC) - Part 3-3: Limits - Limitation of voltage changes, voltage fluctuations and flicker in public low-voltage supply systems, for equipment with rated current =16 A per phase and not subject to conditional connection (IEC 61000-3-3:2008);

Rheinstetten, Germany
(Place)

24.5.13
(Date)

D. MAIER, TECHNICAL DIRECTOR
(Name and function of the signatory empowered to bind the manufacturer or his authorized representative)

(Signature)

2.8 Konformitätserklärung

EG-Konformitätserklärung

Bruker BioSpin GmbH

Der Unterzeichner, der den nachstehenden Hersteller vertritt

Hersteller: **Bruker BioSpin GmbH**
Anschrift: **Silberstreifen 4, 76287 Rheinstetten, Germany**

erklärt hiermit, dass das Produkt **BTpH H116356**

in Übereinstimmung mit den Bestimmungen der nachstehenden EG-Richtlinien (einschließlich aller zutreffenden Änderungen) ist.

Referenz No..	Title
2004/108/EG	Richtlinie 2004/108/EG des Europäischen Parlaments und des Rates vom 15. Dezember 2004 zur Angleichung der Rechtsvorschriften der Mitgliedstaaten über die elektromagnetische Verträglichkeit und zur Aufhebung der Richtlinie 89/336/EWG (früher 89/336/EWG)
2006/95/EG	Richtlinie 2006/95/EG des Europäischen Parlaments und des Rates vom 12. Dezember 2006 zur Angleichung der Rechtsvorschriften der Mitgliedstaaten betreffend elektrische Betriebsmittel zur Verwendung innerhalb bestimmter Spannungsgrenzen (früher 73/23/EWG)

Standard	Folgende harmonisierte Normen wurden angewandt:
EN 61010-1:2011	Sicherheitsbestimmungen für elektrische Mess-, Steuer-, Regel- und Laborgeräte - Teil 1: Allgemeine Anforderungen (IEC 61010-1:2001); Deutsche Fassung EN 61010-1:2001
EN 61326-1:2006	Elektrische Mess-, Steuer-, Regel- und Laborgeräte - EMV-Anforderungen - Teil 1: Allgemeine Anforderungen (IEC 61326-1:2005); Deutsche Fassung EN 61326-1:2006
EN 61000-3-2:2010	Elektromagnetische Verträglichkeit (EMV) - Teil 3-2: Grenzwerte - Grenzwerte für Oberschwingungsströme (Geräte-Eingangsstrom = 16 A je Leiter) (IEC 61000-3-2:2005 + A1:2008 + A2:2009); Deutsche Fassung EN 61000-3-2:2006 + A1:2009 + A2:2009
EN 61000-3-3:2009	Elektromagnetische Verträglichkeit (EMV) - Teil 3-3: Grenzwerte - Begrenzung von Spannungsänderungen, Spannungsschwankungen und Flicker in öffentlichen Niederspannungs-Versorgungsnetzen für Geräte mit einem Bemessungsstrom =16 A je Leiter, die keiner Sonderanschlussbedingung unterliegen (IEC 61000-3-3:2008); Deutsche Fassung EN 61000-3-3:2008

Rheinstetten, Germany
(Place)

29.5.13
(Date)

D. MAIER, TECHNISCHER DIREKTOR
(Name und Funktion des vom Hersteller oder von seinem Bevollmächtigten zur Unterschrift berechtigten Person)

(Unterschrift)

Introduction

3 Safety

This section provides an overview of all the main safety aspects involved in ensuring optimal personnel protection and safe and smooth operation. Non-compliance with the action guidelines and safety instructions contained in this manual may result in serious hazards.

3.1 Intended Use

The device has been designed and constructed solely for the intended use described here in this manual. The BTpH must only be used for measuring a sample‘s pH and for titrating a sample to a target pH. Intended use also includes compliance with all specifications within this manual. Any use which exceeds or differs from the intended use shall be considered improper use. No claims of any kind for damage will be entertained if such claims result from improper use.

3.2 Owner‘s Responsibility

Owner

The term 'owner' refers to the person who himself operates the device for trade or commercial purposes, or who surrenders the device to a third party for use/application, and who bears the legal product liability for protecting the user, the personnel or third parties during the operation.

Owner‘s Obligations

The device is used in the industrial sector, universities and research laboratories. The owner of the device must therefore comply with statutory occupational safety requirements.

In addition to the safety instructions in this manual, the safety, accident prevention and environmental protection regulations governing the operating area of the device must be observed.

In this regard, the following requirements should be particularly observed:

- The owner must obtain information about the applicable occupational safety regulations, and - in the context of a risk assessment - must determine any additional dangers resulting from the specific working conditions at the usage location of the device. The owner must then implement this information in a set of operating instructions governing operation of the device.
- During the complete operating time of the device, the owner must assess whether the operating instructions issued comply with the current status of regulations, and must update the operating instructions if necessary.
- The owner must clearly lay down and specify responsibilities with respect to installation, operation, troubleshooting, maintenance and cleaning.
- The owner must ensure that all personnel dealing with the device have read and understood this manual. In addition, the owner must provide personnel with training and hazards information at regular intervals.
- The owner must provide the personnel with the necessary protective equipment.
- The owner must warrant that the BTpH is operated by trained and authorised personnel as well as all other work, such as transportation, mounting, start-up, the installation, maintenance, cleaning, service, repair and shutdown, that is carried out on the device.

- All personnel who work with, or in the close proximity of the BTpH device, need to be informed of all safety issues and emergency procedures as outlined in this user manual.
- The owner must document the information about all safety issues and emergency procedures in a laboratory SOP (Standard Operating Procedure). Routine briefings and briefings for new personnel must take place.
- The owner must ensure that new personnel are supervised by experienced personnel. It is highly recommended to implement a company training program for new personnel on all aspects of product safety and operation.
- The owner must ensure that personnel are regularly informed of the potential hazards within the laboratory. This is all personnel that work in the area, but in particular laboratory personnel and external personnel such as cleaning and service personnel.
- The owner is responsible for taking measures to avoid inherent risks in the handling of dangerous substances, preventing industrial disease, and providing medical first aid in emergencies.
- The owner is responsible for providing facilities according to the local regulations for the prevention of industrial accidents and generally accepted safety regulations according to the rules of occupational medicine.
- All substances needed for operating and cleaning the device samples, solvents, cleaning agents, etc. have to be handled with care and disposed of appropriately. All hints and warnings on storage containers must be read and adhered to.
- The owner must ensure that the work area is sufficiently illuminated to avoid reading errors and faulty operation.
- If the unit is connected to a nitrogen source the owner must ensure that the laboratory is equipped with a low oxygen warning device.

Furthermore, the owner is responsible for ensuring that the device is always in a technically faultless condition. Therefore, the following applies:

- The owner must ensure that the maintenance intervals described in this manual are observed.
- The owner must ensure that all safety devices are regularly checked to ensure full functionality and completeness.

3.3 Personnel Requirements

3.3.1 Qualifications

Only trained Bruker personnel are allowed to mount, retrofit, repair, adjust and dismantle the unit!

This manual specifies the personnel qualifications required for the different areas of work, listed below:

Laboratory Personnel

Laboratory personnel are health care professionals, technicians, and assistants staffing a research or health care facility where specimens are grown, tested, or evaluated and the results of such measures are recorded. Laboratory personnel are able to carry out assigned work and to recognize and prevent possible dangers self-reliant due to their professional training, knowledge and experience as well as profound knowledge of

applicable regulations. The workforce must only consist of persons who can be expected to carry out their work reliably. Persons with impaired reactions due to, for example, the consumption of drugs, alcohol, or medication are prohibited from carrying out work on the device. When selecting personnel, the age-related and occupation related regulations governing the usage location must be observed.

3.3.2 Unauthorized Persons

⚠ WARNING

Risk to life for unauthorized personnel due to hazards in the danger and working zone!

Unauthorized personnel who do not meet the requirements described in this manual will not be familiar with the dangers in the working zone. Therefore, unauthorized persons face the risk of serious injury or death.

1. Unauthorized persons must be kept away from the danger and working zone.
2. If in doubt, address the persons in question and ask them to leave the danger and working zone.
3. Cease work while unauthorized persons are in the danger and working zone.

3.3.3 Instruction

Personnel must receive regular instruction from the owner. The instruction must be documented to facilitate improved verification.

Date	Name	Type of Instruction	Instruction Provided By	Signature

3.4 Personal Protective Equipment

Personal protective equipment is used to protect the personnel from dangers which could affect their safety or health while working.

Personnel must wear personal protective equipment while carrying out the different operations at and with the device.

This equipment will be defined by the head of the laboratory. Always comply with the instructions governing personal protective equipment posted in the work area.

3.5 Basic Dangers

The following section specifies residual risks which may result from using the device and have been established by means of a risk assessment.

In order to minimize health hazards and avoid dangerous situations, follow the safety instructions specified here as well as in the following chapters of this manual.

Handling of the Flasks

⚠ CAUTION

Spillage Risk When Refilling/Replacing Liquid Storage Flasks

The storage flasks contain aggressive liquids (acid/base).

▶ These flasks should be refilled/replaced as described in this manual.

1. Always wear appropriate eye protection, gloves and protective clothing.
2. Ensure that suitable safety procedures for the handling, disposal etc of these materials are in place in your laboratory environment.

Refer to the section *Filling the Storage Flasks [▶ 33]* regarding the correct procedure.

The Step Motor Syringe Actuators

⚠ CAUTION

Accident hazard from movement of mechanical parts!

The fingers or hand may be pinched due to movement of mechanical parts.

1. Power off the unit before removing or replacing a syringe.
2. Do NOT put a finger into the vertical slots in which the syringe drive moves.

Use of Highly Concentrated Reagents

⚠ CAUTION

Reduction of product life through use of highly concentrated reagents

The use of reagents with a concentration higher than 2 molar may shorten the product life of wetted parts on the BTpH unit, especially with long term usage.

1. Use reagents with a concentration not higher than 2 molar.

3.5.1 General Workplace Hazards

Dirt and Scattered Objects

⚠ CAUTION

Danger of Injury from tripping over dirt and scattered objects.

Dirt and scattered objects may cause people to slip or trip. A fall may result in injuries.

1. Always keep the work area clean.
2. Wear non-slip shoes.
3. Remove objects which are no longer required from the work area and particularly from the floor.
4. Indicate unavoidable hazards using marking tape.

Electrical Current

⚠ WARNING

Danger of injury from electrical shock!

A life threatening shock may result when the housing is open during operation.

1. Only qualified personnel should open the housing.
2. Disconnect the device from the electrical power supply before opening the device. Use a voltmeter to verify that the device is not under power!
3. Be sure that the power supply cannot be reconnected without notice.

Biological and Chemical Hazards

⚠ WARNING

Biological, chemical hazard!

Infection, contamination, or other health endangerment as a result of contact with biological or chemical substances, e.g. from broken samples.

1. Clean the device before maintenance work and/or returning to Bruker for repair.
2. Prepare a list of materials in which the device came into contact with or measured.
3. A signed confirmation of correctly carrying out cleaning/disinfection is required from the customer. Without this confirmation the parts delivered for repair will be rejected and returned to the customer.

Electrostatic Discharge

Electrostatic discharge from friction may occur, resulting in an electric spark and loud bang. Use ESD flooring and wear ESD shoes.

Electronic discharge can seriously damage you pH probe. To minimize this risk use ESD flooring and wear ESD shoes.

Unit Overheating

NOTICE

Material damage hazard from unit overheating.

Material damage may occur when the unit overheats as a result of a blocked air inlet and/or outlet.

1. The unit must be mounted on a level and even surface.
2. At the rear of the unit there must be at least a 10 cm deep area clear of all obstructions.

Access to Mains Supply

SAFETY INSTRUCTIONS

Difficulties in fully disconnecting unit from mains supply

The unit is only completely separated from the mains power when the mains connector plug is removed.

1. The mains connector must be easily accessible at all times.

4 Technical Data

4.1 General Information

Main Unit - Bruker Part Number H116356

Data	Value	Unit
Weight (flasks empty)	11.6	kg
Width	31	cm
Depth (excluding strain relief)	24	cm
Height	21	cm

Table 4.1: Technical Data: Main Unit General

Touch Display Control Unit - Bruker Part Number H122316

Data	Value	Unit
Weight	0.8	kg
Width	20	cm
Depth	14	cm
Height	10	cm

Table 4.2: Technical Data: Touch Display General

4.2 Connection Data

Electrical (Main Unit)

Data	Value	Unit
Mains supply voltage	110-230	VAC
Apparent power consumption, maximum	100	VA
Circuit Protection (110 - 230V AC)	2 x 2.0 Slow Blow	A
Frequency	50/60	Hz
DC out voltage	24+0.5	V
DC out current	500	mA
DC out protection (multi fuse - solid state fuse)	500	mA

Table 4.3: Connection Data Main Unit

Electrical (Touch Panel)

Data	Value	Unit
DC in voltage	12 - 30	VDC
DC in current (maximum at 24 V)	500	mA
Apparent power consumption, maximum	12	W

Table 4.4: Connection Data Touch Display

pH Sensor

The pH Sensor comes complete in a case together with reference pH samples and a brush for cleaning the electrode tip.

Figure 4.1: The pH Sensor Kit

1.	pH Electrode #89008	5.	pH 7.00
2.	Brush #1807260	6.	*KCL* Special #H139244
3.	pH 2.00	7.	30 ml *PE* Bottles
4.	pH 4.00		

Technical Data

Parameter	Condition	Value Min.	Value Max.	Unit
Design				
Measuring Range	Between 0 °C And 40 °C	0	14	pH
Operating Temperature		0	40	°C
Storage Temperature (complete device)	During storage: Maintain temperature as stable as possible	10	40	°C
Storage Relative Humidity (complete device)	During storage: Maintain *RH* as stable as possible	30	80	%
Shipping Conditions	*ESD* proof bag, box or container	10	40	°C
Storage Conditions[1]	Wet	10	40	°C
Media Compatibility	Exposure to compounds normally used. For titration measurements	To be determined by customer (by means of validation)		
Weight	Including cable and connector, excluding external electronics	135	145	G
Diameter (barrel)		7.9	8.1	mm
Diameter (tip)		4.7	4.9	mm
Length (cable)		1200	1300	mm
Connector	Mini-din 7p.	n/a	n/a	
IP Class	Splash proof		IP 65	
Performance				
Parameter	Condition	Value Min.	Value Max.	Unit
pH Sensitivity[2]	0 =< pH =<14	52	52	Mv/pH
pH Offset	pH 7, 25 °C	1100	2400	Mv
Long Term Drift (0-24 Hours)[3]		0	0.15	pH
pH Accuracy[4]	25°C...35 °C	-0.02	0.02	pH
pH Calibration	Tree point calibration both, at factory and point of use data stored in probe eeprom	97	110	Slope %
Temperature Accuracy	25°C...35°C	-0.5	+0.5	°C
Temperature Calibration	Data stored in probe eeprom	25	35	°C

Parameter	Condition	Value Min.	Value Max.	Unit
[1] Probe must be stored wet. Short term storage (< 2 days) preferably in 3M KCl or pH7 in sample chamber. Long term storage (>2 days) with wetted protection cap. [2] The drift performance, probe accuracy and response time is dependent on correct handling of the probe. This involves among other things cleaning, storage and soaking. Typical drift over 24 hours is 0.06 pH. [3] Exposing the ISFET chip to UV-light will change the offset. Depending on the duration and strength this can cause the offset to go out of specifications. [4] Avoid direct sunlight or strong UV light shining directly on the ISFET during measurement or calibration.				

Table 4.3: pH Sensor Spcifications

4.3 Operating Conditions

Normal environmental conditions (CAN/CSA 61010-1-12; IEC 61010-1: 2010; ANSI/UL 61010-1)

- Indoor use only.
- Maximum operation altitude: 2000 m.
- Working temperature 5 °C to 40 °C.
- Maximum relative humidity 80% for temperatures up to 31 or decreasing linearly to 50% relative humidity at 40 °C.
- MAINS supply voltage fluctuations up to ±10% of the nominal voltage.
- TRANSIENT OVERVOLTAGES up to the levels of OVERVOLTAGE CATEGORY II.
- Pollution degree 2.
- Protection class IP20.

Environment (Main Unit, Touch Display and pH Sensor)

Data	Value	Unit
Storage temperature range	5 - 50	°C
Operating temperature range	5 - 35	°C
Relative humidity up to 31C, maximum	< 80	%
Decreasing linear till relative humidity < 50% at 40 °C, maximum	< 50	%

Table 4.6: Operating Environment

The pH measurements are temperature compensated. The best repeatability, however, is obtained when there is little temperature change during the course of a series of measurements/titrations.

4.4 Rating Plates

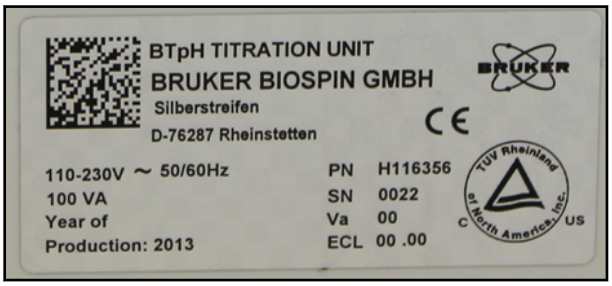


Figure 4.2: BTpH Titration Unit Rating Plate

Figure 4.3: BTpH Touch Panel Rating Plate

The rating plate for the main unit is located at the power input and the one for the touch display on the base of the touch display controller. They contain the following information:

- Manufacturer
- Type
- Voltage
- Frequency
- Apparent power consumption, maximum
- Year of Production
- PN: Part Number
- SN: Serial Number
- Va: Variant
- ECL: Engineering Change Level

4.5 Fluids Used

There are five storage flasks (2 x 250 ml and 3 x 100 ml) kept in the leakage bath on the top of the unit.

Demineralized water (100 ml flask):	Demineralized* water is stored here. This is used to wash out all the fluid paths.
Waste (250 ml flask):	Waste fluids pumped through the unit during the initialization and washing operations are stored here. This flask must be emptied in a regular basis and always whenever it is more than ca. 70% full. No measurement of the contents is performed by the unit. It is the responsibility of the user to monitor the level of its contents and empty it as and when required.
Buffer (250 ml flask):	The buffer to be used is usually determined by the desired target pH and the type of samples used. The user defines the buffer type. Normally a small amount of buffer solution (e.g. in wine analysis, it‘s 10% of the sample volume) is added to the sample at the start of the titration operation to adjust the pH of sample towards the target pH.
Acid (100 ml flask):	The acid type and strength is determined by the user (e.g. 1N *HCl*). Small volumes are added to the sample to move the sample‘s pH down toward the target pH.
Base (100 ml flask):	The base type and strength is determined by the user (e.g. 1N *NaOH*). Small volumes are titrated to the sample to move a sample‘s pH up toward the target pH.
* It is advisable to add a small amount of bactericide to the water to prevent fouling in the flask and tube paths. For instance 0.01% Sodium Azide (*NaN3*).	

Table 4.1: Fluids Used

See *Filling the Storage Flasks [▶ 33]*.

4.6 Materials Used in the Construction

All ‘wetted‘ materials (those materials which are normally in contact with any of the liquids used) are inert with respect to the normal liquids used during a titration operation.

Flasks:	PE
Tubing:	Teflon (*FEP*/*PEEK*)
Fitting Seals:	Teflon
Valves:	*Kel-F*, Teflon
Syringes:	Glass, Teflon, PEEK

The remaining materials may have occasional contact with the fluids due to leakage or spillage:

Main outer housing, drip trays for storage flasks and under the valves at the front of the unit:	Polestyrol
Front panel:	Polyester on an aluminium plate.
Fitting bodies:	Polycarbonate + stainless steel.

Ensure that any fluids you wish to use with the unit are, at least, safe to use with the wetted materials. In extreme cases (very common with the type of liquids used here) there may be minor damage to the non-wetted components. It is the users responsibility to ensure the fluids used are safe.

The inner housing is made from stainless steel. The unit is designed so that liquid from minor leakages or spillages is directed, either into one of the drip trays or, at the very least, away from the unit in order to minimize the likelihood of any liquid entering the inner housing.

The tubing and fittings are rated to > 20 bar. The valve and syringes are rated to approximately 5 bar. In the event of a blockage at the output, whereas the pressure in the syringe exceeds 5 bar, the selection valves will leak out the leakage port at the left of the valve. A tube is connected to this port to provide drainage to the drip dray.

4.7 Spare Parts

Article	Bruker PN
BTpH Unit Complete, including:	AH0173
BTpH Unit & BioShake 3000 BBIO	H116356
Touch Panel	H122316
Probe Adapter	H138865
pH Probe Stand	H123161
1.8 ml Cryo Vials (Pack of 100)	85372
pH Probe Complete	H126781
Consumables:	
pH Probe Complete	H126781
1.8 ml Cryo Vials (Pack of 100)	85372
3M KCl Wine Special 250 ml	H139244
Wine Reference Solution pH 3.1 100 ml	H140841
Wine Buffer Solution 100 ml	H140842
Combi-Pack Wine Ref. & Buffer Solution 100 ml each	H143305
Spare Parts & Accessories:	
BTpH Unit Shipping Box	1809153
BioShake 3000 BBIO including power supply	H139144
Syringe 100 µl ext. Tip	88810
Syringe 500 µl ext. Tip	88809
6 way Valve	88808
Bottle Capillary Set	1804523
PE Bottle 100 ml	89072
PE Bottle 250 ml	89073

Table 4.8: Spare Parts

5 Design and Function

5.1 Overview

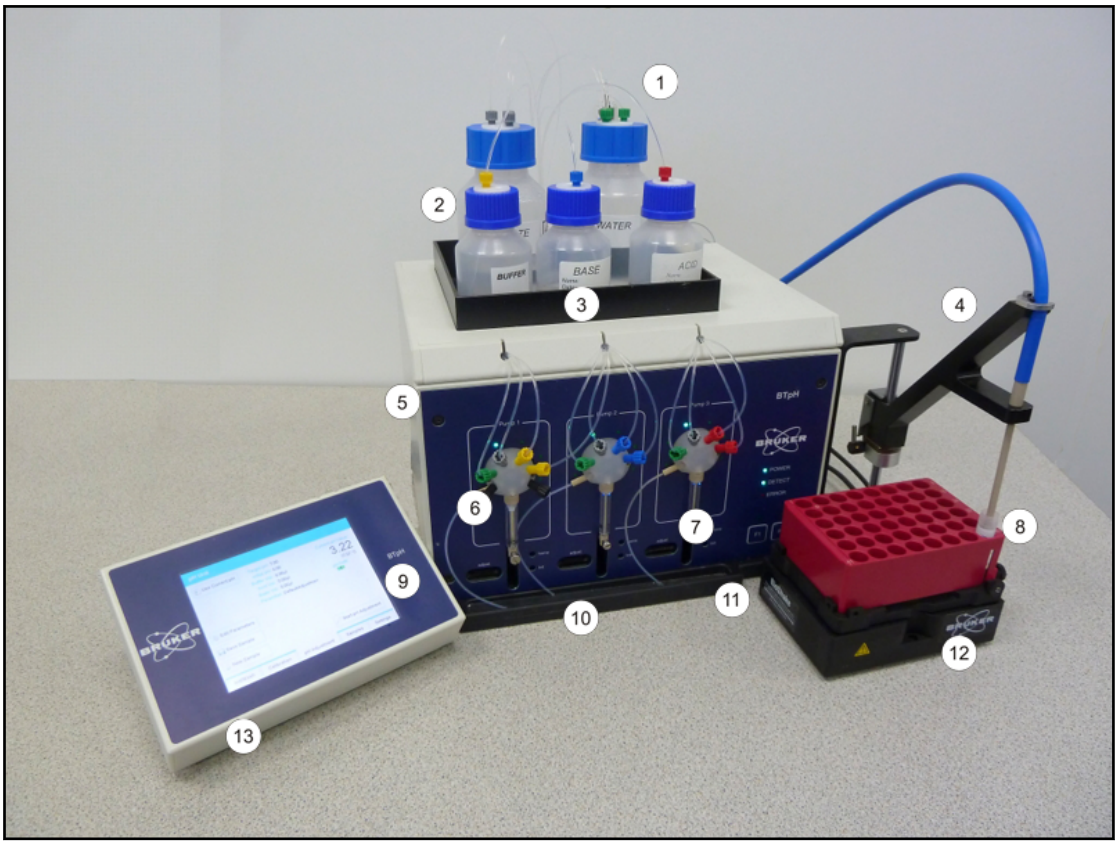


Figure 5.1: BTpH Unit Overview

1.	Teflon FEP Capillary Tubing	8.	pH Sensor
2.	Storage Flasks	9.	Touch Display
3.	Storage Flask Drip Tray	10.	Drip Tray
4.	pH Sensor Stand	11.	1/4“-28 Female Port for Overflow to Waste Tubing
5.	BTpH Titration pH Unit	12.	BioShake 3000 BBIO
6.	Selection Valve	13.	BTpH Touch Panel Controller
7.	Syringe		

5.2 Unit Description

The BTpH (Bruker Titration pH Unit) allows a user to adjust the pH of a sample to a predefined value. It has been specially designed to work with sample tubes or well plates with an internal diameter as small as 8 mm and sample volumes of < 1ml. The practical lower limit of approx. 400 µL is determined by the actual internal diameter of the sample tube. In order for the sensor to measure and adjust the pH, the ISFET sensor and reference must also both be in the liquid, and the base of the sensor should be just clear (approx. 1 mm) of the bottom of the sample tube.

The unit measures the pH, adds a (user) programmed volume of buffer, then adds base or acid until the (user) programmed target pH is reached.

A precision ISFET pH probe has been developed which has capillary tubes for the buffer, base, acid and mixing gas (normally *N2*) feeding through the probe to its tip.

While the adjustment is taking place, N2 is bubbled with a (user) programmed pressure out through the center capillary tube to continuously mix the sample. The titration is performed by 3 step motor driven syringe pumps, each with its own selection valve all controlled by the touch panel controller.

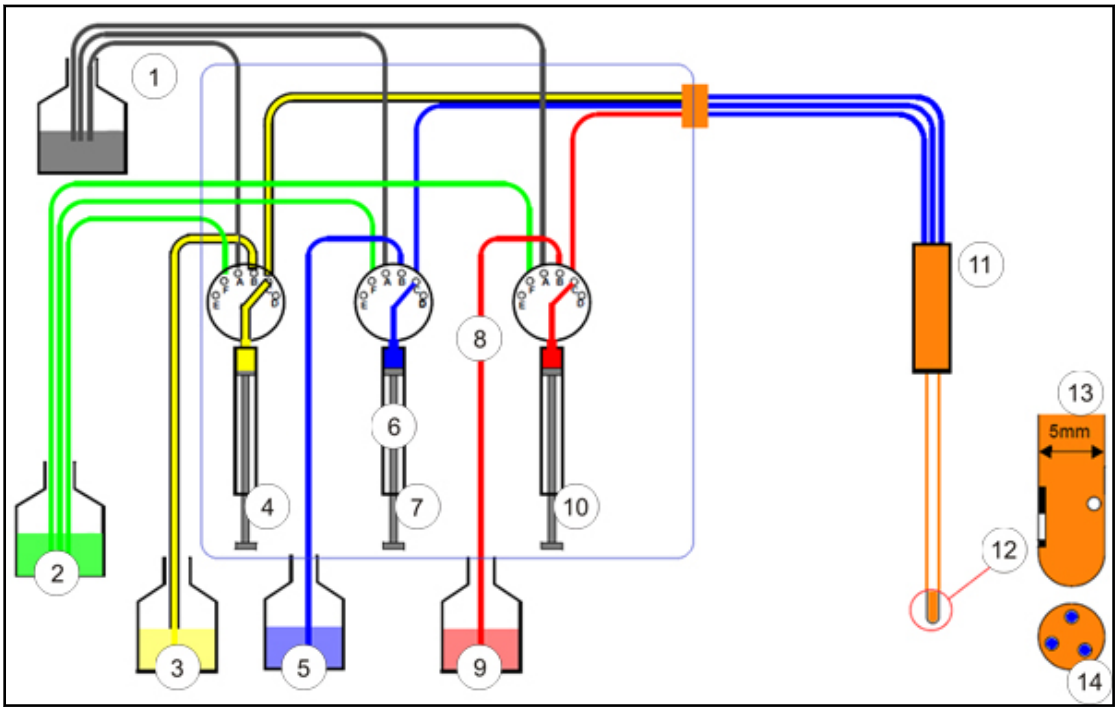


Figure 5.2: Titration Unit Block Diagram

1.	Waste	8.	Selection Valves
2.	H20	9.	Acid (e.g. HCl)
3.	Buffer	10.	100 µL Syringe
4.	500 µL Syringe	11.	pH Probe
5.	Base (e.g. NaOH)	12.	pH Probe Tip
6.	Syringe Pumps with 6-way Valve: Kel-F/Teflon	13.	pH Probe Tip with ISFET and Reference diaphragm
7.	100 µL Syringe	14.	Capillary Outlets

For proper mixing of the sample the BioShake 3000 *BBIO* is used. This shaker is integrated into the software and allows reproducable mixing during titration.

Figure 5.3: BioShake 3000 BBIO

See also

Spare Parts [▶ 28]

5.3 Connecting the Unit

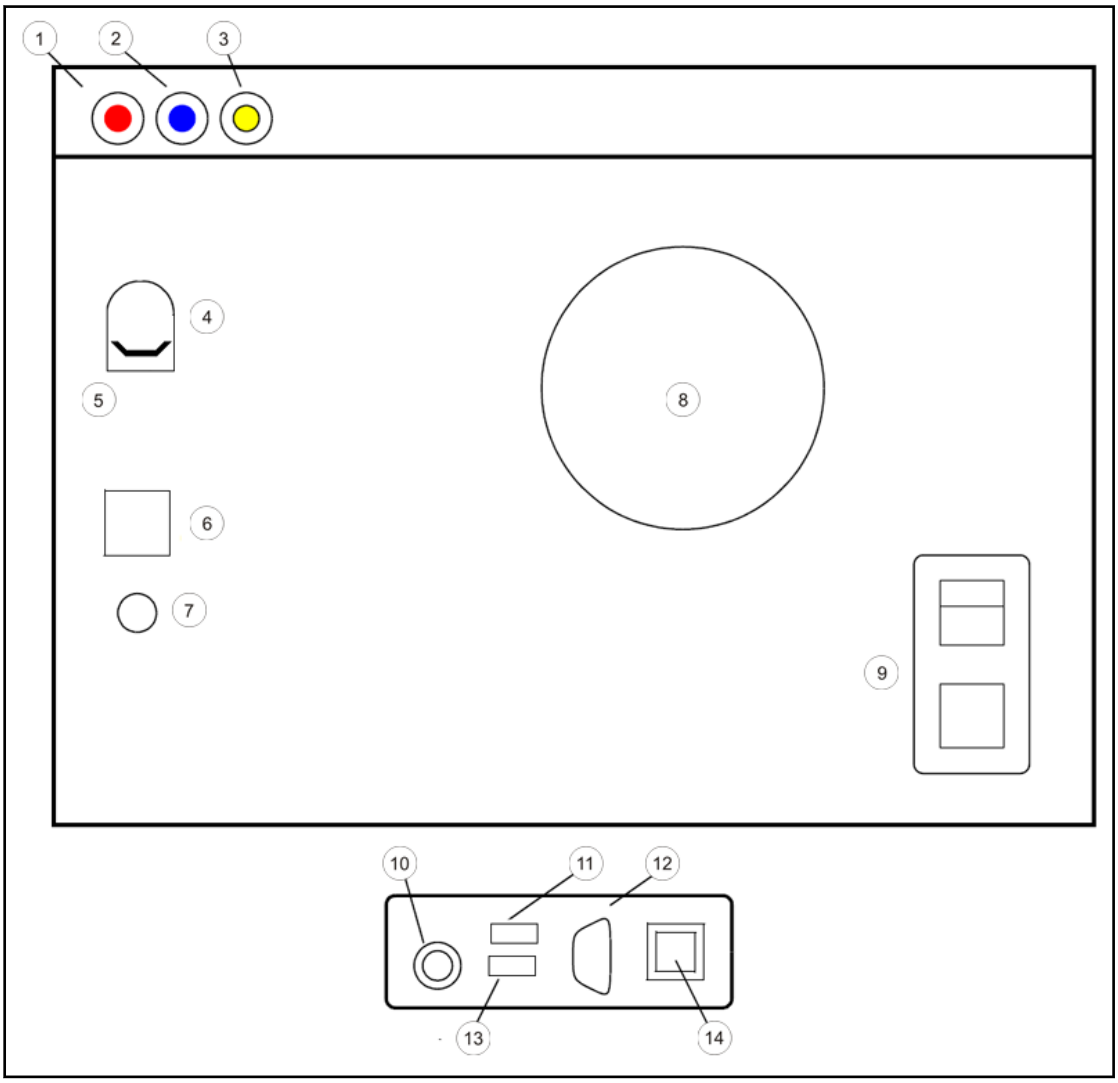


Figure 5.4: Main Unit Rear Connections

1.	Acid Outlet to pH Probe (red connector)	8.	Fan Outlet
2.	Base Outlet to pH Probe (blue connector)	9.	Mains Input with ON/OFF Switch
3.	Buffer Outlet to pH Probe (yellow connector)	10.	AC-In Connected to Main Unit 24V DC Out
4.	pH Probe Connection	11.	USB Connected to Barcode Reader
5.	Support Bracket with Ground Connection	12.	*RS232*
6.	USB to Controller	13.	USB Connected to Main Unit
7.	24V DC-Out to Controller	14.	Ethernet

1. Ensure that the mains switch is in the ‘Off‘ position (Press in the ‘0‘ side of the switch).

2. Using the cables provided, connect the Touch Panel Control to the main Unit (USB and DC Power).
3. Secure the pH Probe in the stand provided and connect its electrical and tubing connectors. The tubing connectors are color coded. Secure the support bracket to the pH Probe cable.
4. Plug the mains cable into the unit then into the mains.
5. The tubing connections from the storage flasks to the selection valves and from the selection valves to the outlet connections will have been done correctly in house. If for any reason you have disconnected and fittings these must be reconnected as shown in *Unit Description [30] [29]*.
6. You can now fill the storage flasks with your fluids of choice. See *[29]Filling the Storage Flasks [33]*

5.4 Filling the Storage Flasks

⚠ CAUTION

Spillage Risk When Refilling/Replacing Liquid Storage Flasks

The storage flasks contain aggressive liquids (acid/base).

▶ These flasks should be refilled/replaced as described in this manual.

1. Always wear appropriate eye protection, gloves and protective clothing.
2. Ensure that suitable safety procedures for the handling, disposal etc of these materials are in place in your laboratory environment.

It' s not recommended to use a base and/or acid solution with a concentration not higher than 2 molar. Higher concentrated reagents will lead to a quicker wear-out of wetted parts.

1. Ensure you are wearing appropriate safety clothing (protective gloves, glasses etc.).
2. Unscrew the cap with fittings from the flask which you wish to refill (or empty in the case of the waste flask) taking care not to knock the flask over.
3. Replace this cap with a standard cap (supplied with the unit).
4. Carefully carry the flask to the safe area or lab where you can refill it with the fluid of your choice or simply replace it with an identical flask already filled with the correct fluid. Or empty the waste flask into the container in your lab designed and reserved for such waste.
5. Replace the flask in the drip tray.
6. Carefully remove the cap and replace it with the cap and fittings.
7. Repeat for all flasks needing filled.

Note: To stop fouling in the water flask and the fluid paths you should add a bactericide to the demineralized water. (For example 0.01% Sodium Azide is effective and will not be seen in an NMR spectrum). As the bactericide can be highly toxic this should only be prepared by suitably trained personnel. If you do not wish to use a bactericide then the water should be replaced and all paths thoroughly washed at least every two days.

5.4.1 The pH Sensor Stand

The sensor stand can be mounted on the right or left hand side of the unit to accommodate right or left handed operators.

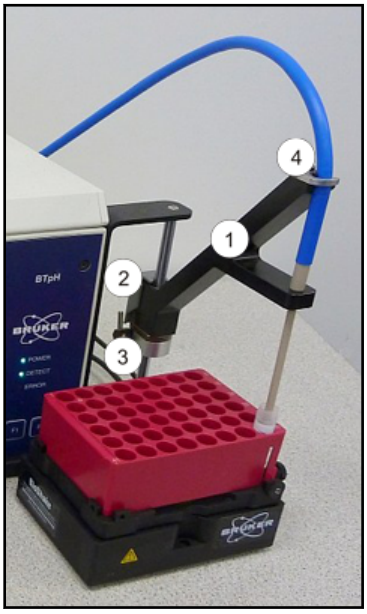


Figure 5.5: The pH Sensor Stand

1.	Sensor Holder	3.	Bottom Clamp
2.	Sensor Clamp	4.	Sensor Cable Clip

The sensor clamp can be released by gripping the sensor holder opposite the clamp and squeezing the clamp lever. Now the sensor holder is free to be moved up or down on the guide rod. If you release the sensor clamp the sensor holder will stay in that position.

The bottom clamp can be released and secured with the fixing screw at the right of the clamp. The clamp should be positioned such that with the sensor holder set completely down into the clamp the tip of the pH sensor is at exactly the right position and depth in the sample tube.

If you always place your sample tubes at the same position this provides a simple method of ensuring that the pH sensor tip always sits at the same position in each sample tube.

5.4.2 Starting the Unit

The Touch Pad Controller, the Main Unit and the pH Probe must first be connected as described ([29]Connecting the Unit [32]) and the flasks filled (Filling the Storage Flasks [33] [29]).

Power on the main unit (press the 'I' on the mains switch). This powers up both the main unit and the touch pad controller.

On the Main Unit:

The power LED lights and the pumps can be heard to make a slight noise as the valves are moved to their start position (syringe input connected to port A - 3rd from left).

The unit runs an auto-detect of the pumps. During this time the blue syringe LEDs move up in a chain sequence (all lit, then bottom LED goes out, etc) and the green valve LEDs do the same in a clockwise move. This is repeated three times. Once completed the syringe LED's stop with only the top LED being lit and the valve LED's stop with the LED adjacent to the selected port (here port A) being lit.

At the same time the unit checks if it is connected over USB. The detect LED lights on a valid USB detect.

The unit checks if a pH sensor is connected. If so, the assembly and calibration information are read from an EEPROM in the pH sensor tip.

The unit then checks if the shaker is powered up and connected correctly.

On the Touch Display:

The startup display is shown. The unit model number and the correct main unit firmware level are displayed. If the main unit does not have the correct firmware level this is then downloaded automatically from the touch pad controller.

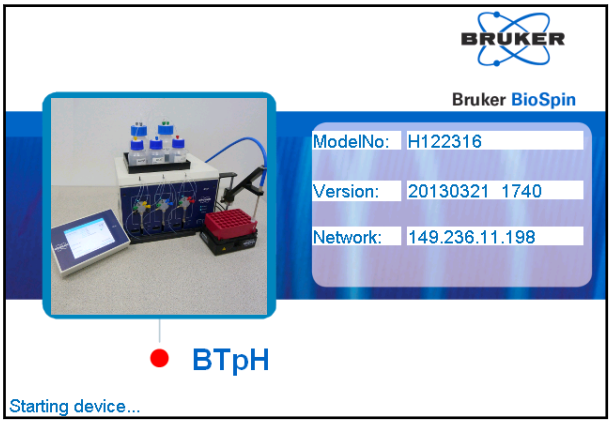


Figure 5.6: The Startup Display

A network address is only shown if the unit is connected to a network and is allocated an address (DHCP) - only needed at present when downloading the sample data.

The touch controller now performs the basic hardware initialization of the unit:

On each pump the valves are set to the initialise position (here port A) and the syringe is set to find its 'home' position then on to position '0'.

The home position is detected by a sensor in the pump and corresponds to a position a few mm. before the upper end stop. The upper end stop is that position where the syringe piston is completely pushed in.

The initialise position is saved in the pump memory and corresponds to a position just down from the physical endstop.

Note: The start position for each pump MUST be reset whenever a syringe or valve is replaced. See *Resetting the Pump Home Position [107]* for help.

The touch display software finishes the internal software initialization and displays the main screen. The unit is now ready for use: See *Operating The Unit [41]*.

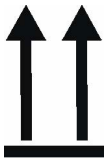
6 Transport, Packaging and Storage

The BTpH unit is delivered with all major components already assembled except for auxiliary parts such as the probe stand, touch panel and the pH probe. Keep the original container and packing assembly, at least as long the warranty is valid, in case the BTpH unit has to be returned to the factory.

Installation, initial commissioning, retrofitting, repairs, adjustments or dismantling of the device must only be carried out by Bruker Service or personnel authorized by Bruker. Damage due to servicing that is not authorized by Bruker is not covered by your warranty.

6.1 Symbols on the Packaging

The following symbols are affixed to the packaging material. Always observe the symbols during transport and handling.

Top		The arrow tips on the sign mark the top of the package. They must always point upwards; otherwise the content may be damaged.
Fragile		Marks packages with fragile or sensitive contents. Handle the package with care; do not allow the package to fall and do not allow it to be impacted.
Protect Against Moisture		Protect packages against moisture and keep dry.
Attach Here		Lifting gear (lifting chain, lifting strap) must only be attached to points bearing this symbol.
Center of Gravity		Marks the center of gravity of packages. Note the location of the center of gravity when lifting and transporting.
Weight, Attached Load		Indicates the weight of packages. Handle the marked package in accordance with its weight.

Permitted Stacking Load		Indicates packages which are partially stackable. Do not exceed the maximum load-bearing capacity specified on the symbol in order to avoid damaging or destroying the content.
Do not Damage Air-tight Packaging		The packaging is air-tight. Damage to the barrier layer may render the contents unusable. Do not pierce. Do not use sharp objects to open.
Component Sensitive to Electrostatic Charge		The packaging contains components which are sensitive to an electrostatic charge. Only allow packaging to be opened by trained personnel. Establish potential equalisation before opening.
Protect from Heat		Protect packages against heat and direct sunlight.
Protect from Heat and Radioactive Sources		Protect packages against heat, direct sunlight and radioactive sources.

Table 6.1: Symbols on the Packaging

6.2 Inspection at Delivery

Upon receipt, immediately inspect the delivery for completeness and transport damage.

Proceed as follows in the event of externally apparent transport damage:

- Do not accept the delivery, or only accept it subject to reservation.
- Note the extent of the damage on the transport documentation or the shipper's delivery note.
- Initiate complaint procedures.

Note: Issue a complaint in respect to each defect immediately following detection. Damage compensation claims can only be asserted within the applicable complaint deadlines.

6.3 Packaging

About Packaging

The individual packages are packaged in accordance with anticipated transport conditions. Only environmentally friendly materials have been used in the packaging.

The packaging is intended to protect the individual components from transport damage, corrosion and other damage prior to assembly. Therefore do not destroy the packaging and only remove it shortly before assembly.

Handling Packaging Materials

Keep the original container and packing assembly, at least as long the warranty is valid, in case the BTpH unit has to be returned to the factory. When the packaging material is no longer needed dispose of in accordance with the relevant applicable legal requirements and local regulations.

6.4 Storage

Storage of the Packages

Store the packages under the following conditions:

- Do not store outdoors.
- Store in dry and dust-free conditions.
- Do not expose to aggressive media.
- Protect against direct sunlight.
- Avoid mechanical shocks.
- Storage temperature: 15 to 35 °C.
- Relative humidity: max. 60%.
- If stored for longer than 3 months, regularly check the general condition of all parts and the packaging. If necessary, top-up or replace preservatives.

Note:

Under certain circumstances, storage instructions may be affixed to packages which expand the requirements specified here. Comply with these accordingly.

Transport, Packaging and Storage

7 Operating The Unit

7.1 Powering Up the Unit (for the first time)

When the unit is used for the first time this means that the contents of all the storage flasks have been replaced (filled for the first time - refer to the **Refill** section in *Initialize the Unit [42]*).

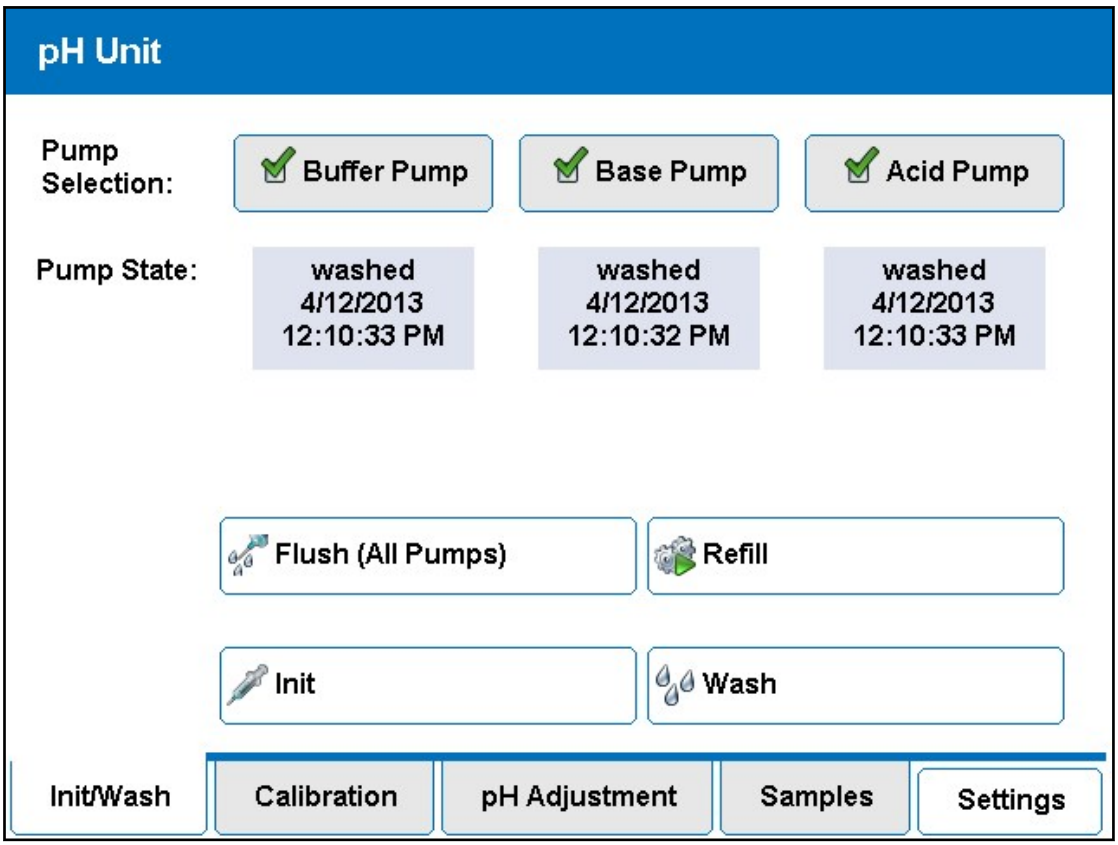


Figure 7.1: Init/Wash Page

With the unit powered up (see *The pH Sensor Stand [34]*) and once the start up checks have been completed, the following screen appears.

On power up the unit always starts with the Init/Wash page as the unit has to be initialized and the probe calibrated before it is used.

The text under each of the pump identifiers (Buffer, Base and Acid Pump) shows the present pump initialization states. After a power up or reset this is ‘unknown’.

7.1.1 Initialize the Unit

There are 3 distinct types of initialization.

Init

This is used when you first use the unit after it has been out of use for a while (e.g. switched off at the end of the day) In this case the storage flasks were already filled with the correct liquids and the last time it was in use these liquids were used. You would normally select all 3 pumps as shown and run 1 Init cycle.

Once selecting any of these options you will be asked to ensure that the pH Probe is located above a waste container. Do this and press OK to confirm.

The **purpose** of the 'init' operation is to:

- Fill the flow paths from the storage flasks to the syringes with the buffer, base or acid from the storage flasks.
- Wash out the syringes with the buffer, base or acid.
- Remove any gas bubbles from the syringes.
- Fill the flow paths from the syringes to the pH Probe tip with the buffer, base or acid.

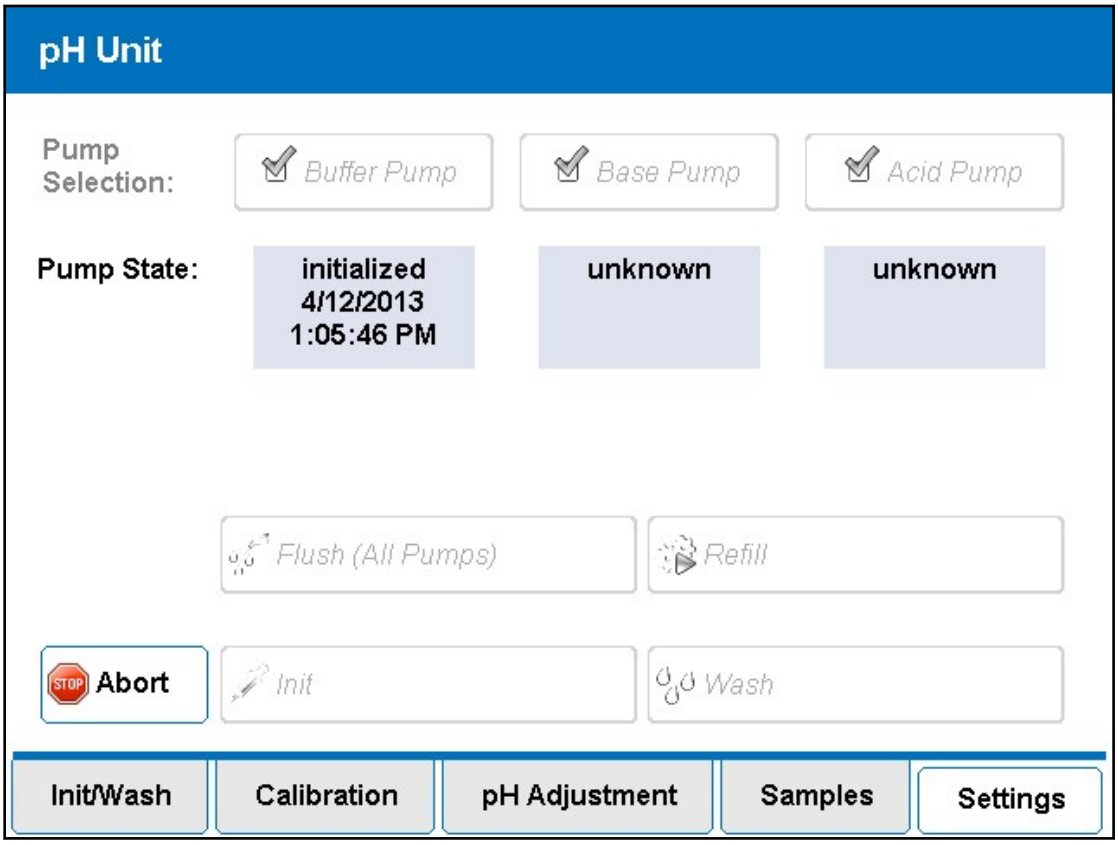


Figure 7.2: Init/Wash Abort

If the unit has not been used for an extended period, or you can clearly see gas bubbles in one or more of the syringes, you can repeat the Init action. For example, the first time you use the unit. If you have refilled one of the storage flasks (buffer, base or acid) you should also run **Refill** on the associated pump.

During this and the wash and refill operations you can abort at any time.

Simply press the **Abort** button and the operation stops soon afterwards.

Wash

This MUST be used when you have finished using the unit for the day and it is effectively to be set in a storage mode.

The purpose of the ‘Wash‘ operation is to:

- Fill the flow paths from the storage flasks to the syringes with (distilled) water.
- Wash out the syringes with the (distilled) water.
- Fill the flow paths from the syringes to the pH Probe tip with (distilled) water.

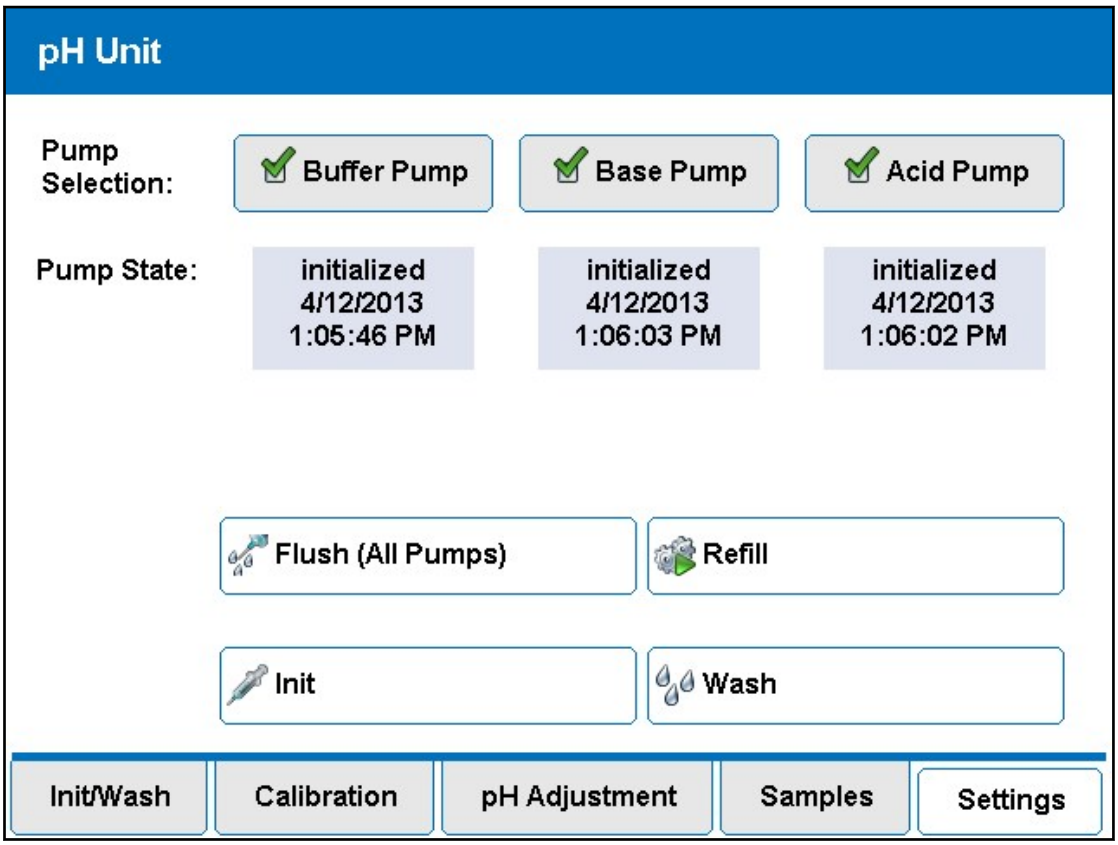


Figure 7.3: Init/Wash Completed

NOTICE

Material damage due to agressive reagents or clogged capillaries.

Constant contact with aggressive reagents may result in clogged capillaries or degradation of sensor sensitivity.

1. Always execute a wash cycle after finishing a titration series.

Refill

This MUST be used when you replace one or more of the buffer, base or acid flask contents with a **different** buffer, base or acid. This need only be done for the associated pump.

The purpose of the ‘Refill‘ operation is to:

- Wash the old liquid from all the fluid paths associated with the new liquid.
- Fill the same paths with the new liquid.

This is mostly a wash followed by an Init operation. If you have simply renewed a buffer, base, acid flask with the same contents then an **Init** operation is sufficient.

Once the Init, Wash or Refill operation has been done the Pump State for each of the pumps is indicated as shown above.

Flush Function

Flush is an action cleaning the capillaries in the probe tip and preventing a carryover in pumping a small quantity of the currently connected liquids.

To adjust the amount of flush liquid see *Extras [98]* - Flush Settings.

7.2 Calibrating the pH Probe

The pH probes are factory calibrated, however, in order to maintain its high precision each probe should be frequently recalibrated, at least once a day. When a pH Probe is successfully calibrated the calibration information together with the time and date of the calibration are saved to the probe‘s EEPROM.

If you attempt to perform a pH titration adjustment and the probe‘s calibration is overdue a warning message appears. You can choose to accept (OK) or ignore the warning.

If you press ignore, you can continue with your adjustment, but the warning will appear again later. A 15 minute time-out till the next warning, is started.

If you press OK then you must calibrate the pH probe before continuing - otherwise the warning will immediately re-appear.

Note: If the pH probe has not been used for a long period of time (e.g. a few days), it should be ‘conditioned‘ before use. See *Conditioning the pH Probe [103]*.

7.3 Calibration Operation

On the main page press the ‘Calibration‘ Tab to select the calibration page.

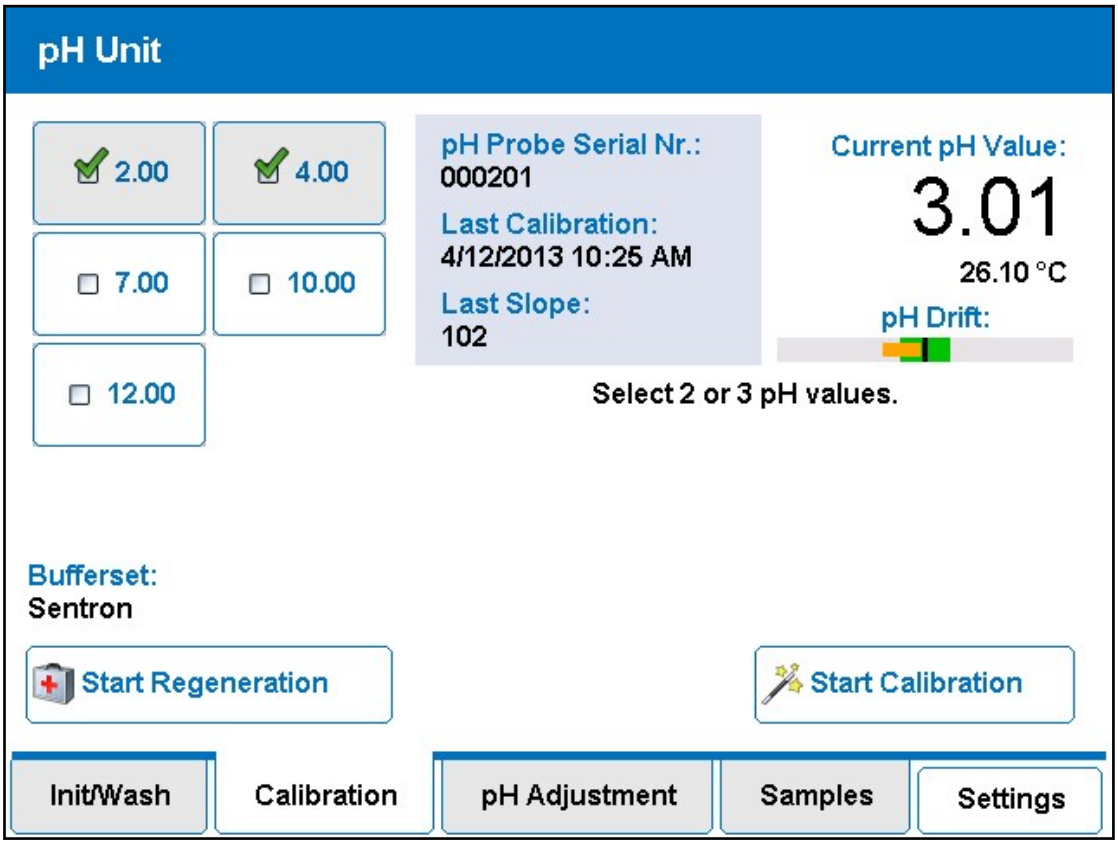


Figure 7.4: Calibration

Select 2 or 3 pH values to calibrate to. If most of your intended titration adjustments are to fall in the same range then 2 calibration values should be sufficient. One of the values should lie close to your desired pH adjustment range in order to give the most accurate results.

Here pH 4.00 was selected followed by pH 2.00. The calibration will be carried out in the same order in which the calibration values were selected. If you wish to change this order, deselect all pH values and re-enter them in your desired order.

NOTE: The calibration list displayed here is for the ‘Sentron‘ buffer set. Also other calibration standards, like *NIST*, *JIST*, or *DIN* can be selected. These can be changed in the ‘Settings‘ pages. See the section *Calibration Settings [▶ 95]* for a full explanation.

Press the ‘Start Calibration‘ tab to start the calibration.

You are now requested to wash the probe prior to the calibration. Simply move the probe above a suitable waste container and squirt the tip with distilled water. Wipe this off with a lint free, soft tissue and repeat.

Now you can move the probe into the pH calibration standard solution as requested.

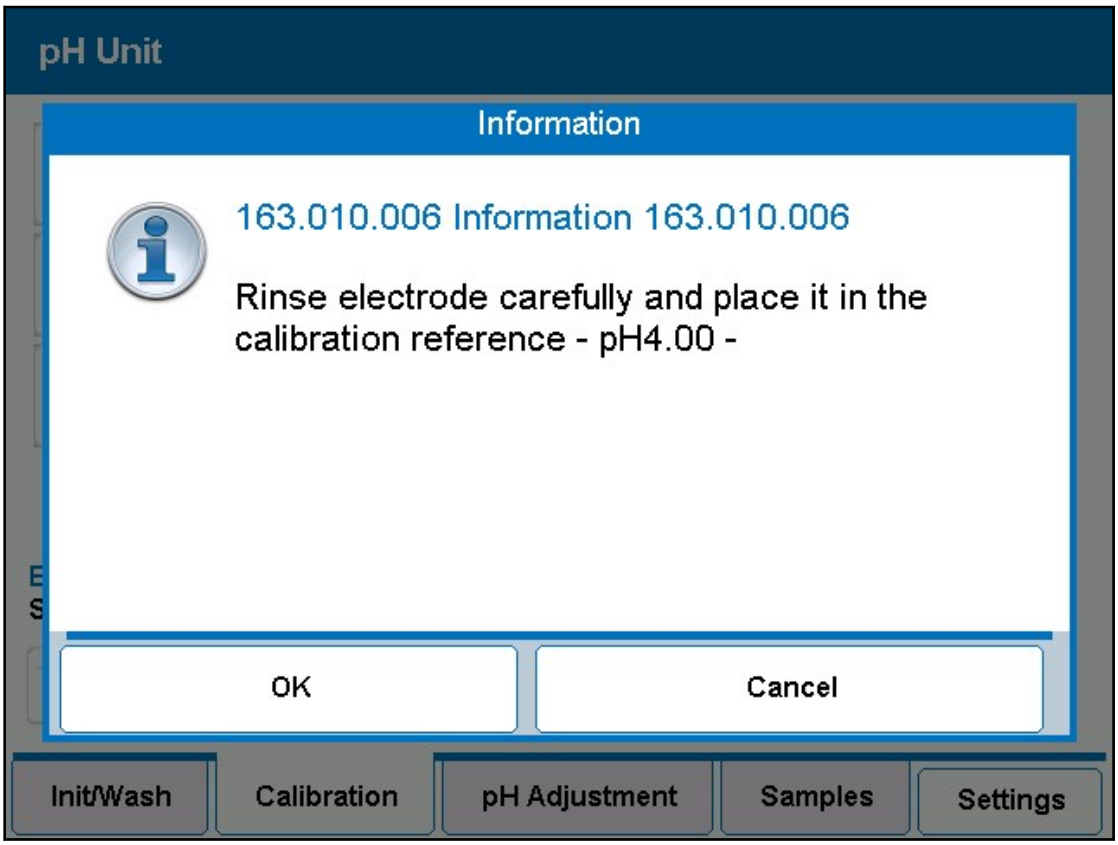


Figure 7.5: Rinse Information Window pH 4.00

Press ‘OK‘ to start the first part of the calibration procedure.

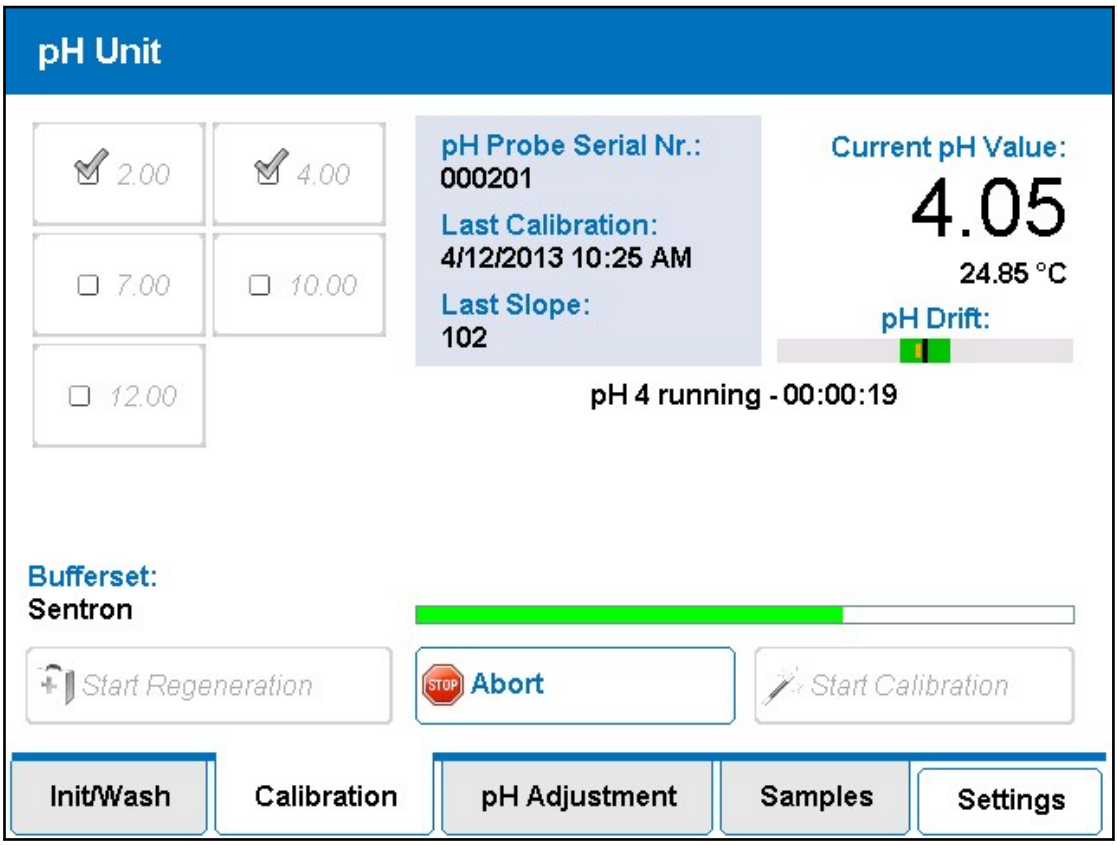


Figure 7.6: Calibration to pH 4.00 Running

The present measured pH value (using the values from the last calibration) is displayed as well as a 'pH Drift' bar. This shows the small background changes in pH (down to < 0.001). A green area in the middle indicates an acceptably small value for this jitter. The narrow orange bar shows the trend. The pH reading is taken to be stable once the drift is very small. To the left is drift down to the right is drift up.

Note: Jitter/Noise is treated differently from true drift. With drift the polarity is mostly +ve or mostly -ve indicative of a real drift in one direction. With Jitter/Noise the polarity jumps frequently between +ve and -ve.

You can abort the calibration at any time by pressing the **Abort Calibration** button. In the status window the run time of this phase of the calibration routine is also displayed.

The longer bar above the tabs is a calibration progress indicator. This will slowly increase (the green bar increases in size from left to right) as the calibration operation continues. At first, if the pH value is still drifting, the bar may grow larger and then jump back down again. Once the reading is very stable the bar moves fairly swiftly to the right.

Once completed the pH display will show the correct pH (same as reference) and a popup window again requests you to rinse the probe and place it in the next reference solution.

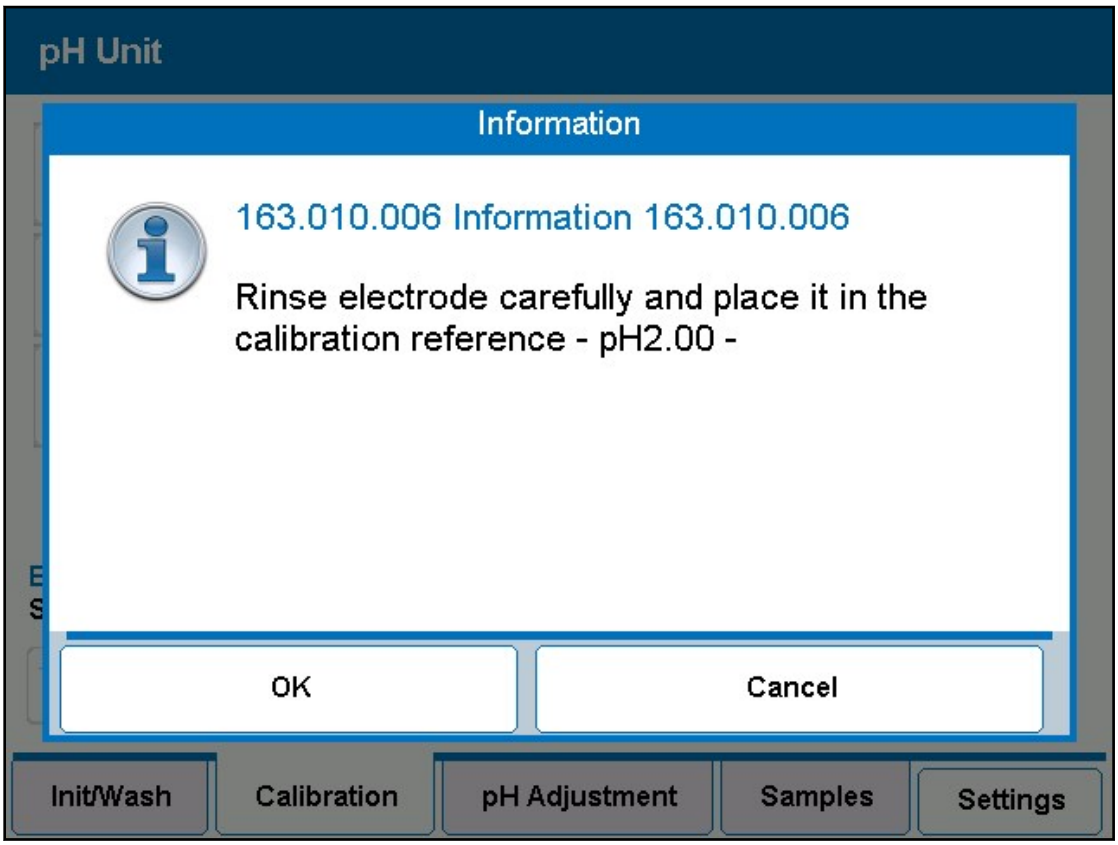


Figure 7.7: Rinse Information Window pH 2.00

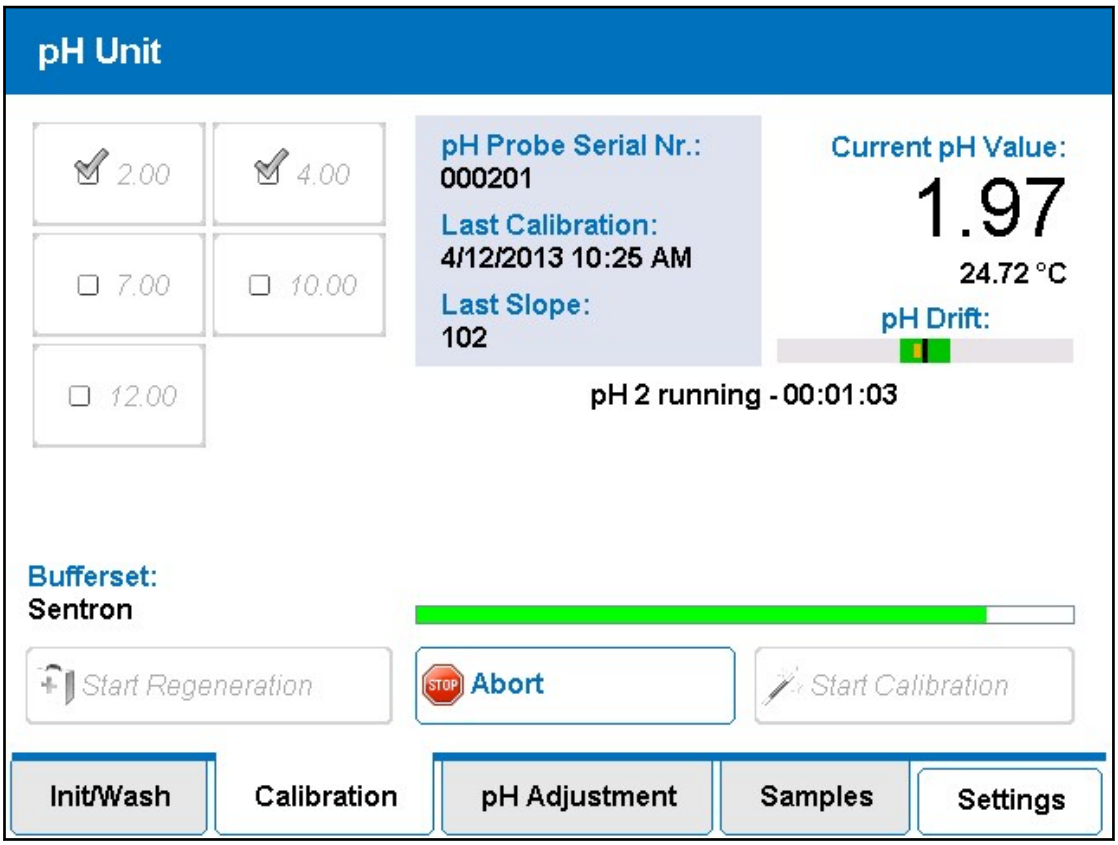


Figure 7.8: Calibration to pH 2.00 Running

Remove the probe from the present reference solution. Replace the cap on the reference solution and place it to one side. Thoroughly rinse and dry the probe tip as before and place it in the next reference solution as requested.

Press ‘OK‘ to start this next phase of the operation.

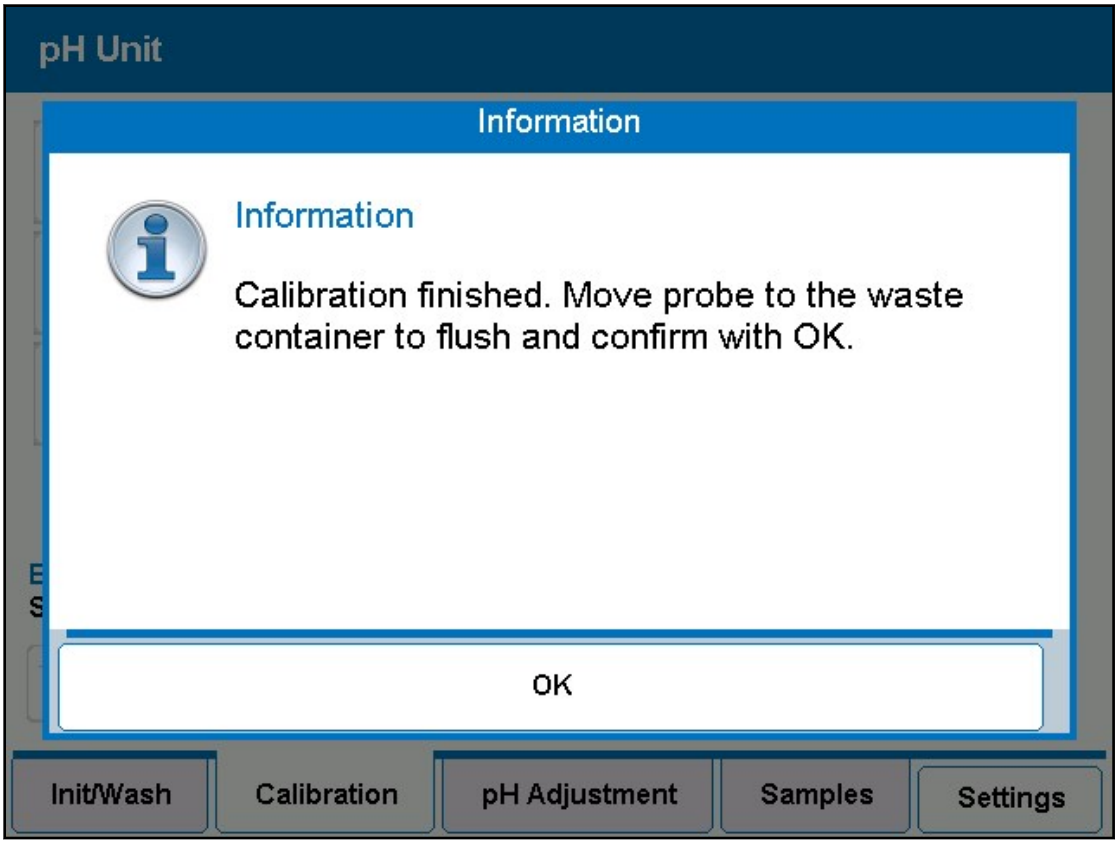


Figure 7.9: Calibration Finished Message

This runs exactly the same as for the first phase of the calibration procedure.

Flush Function

Flush is an action cleaning the capillaries in the probe tip and preventing a carryover in pumping a small quantity of the currently connected liquids.

To adjust the amount of flush liquid see *Extras [▶ 98]*.

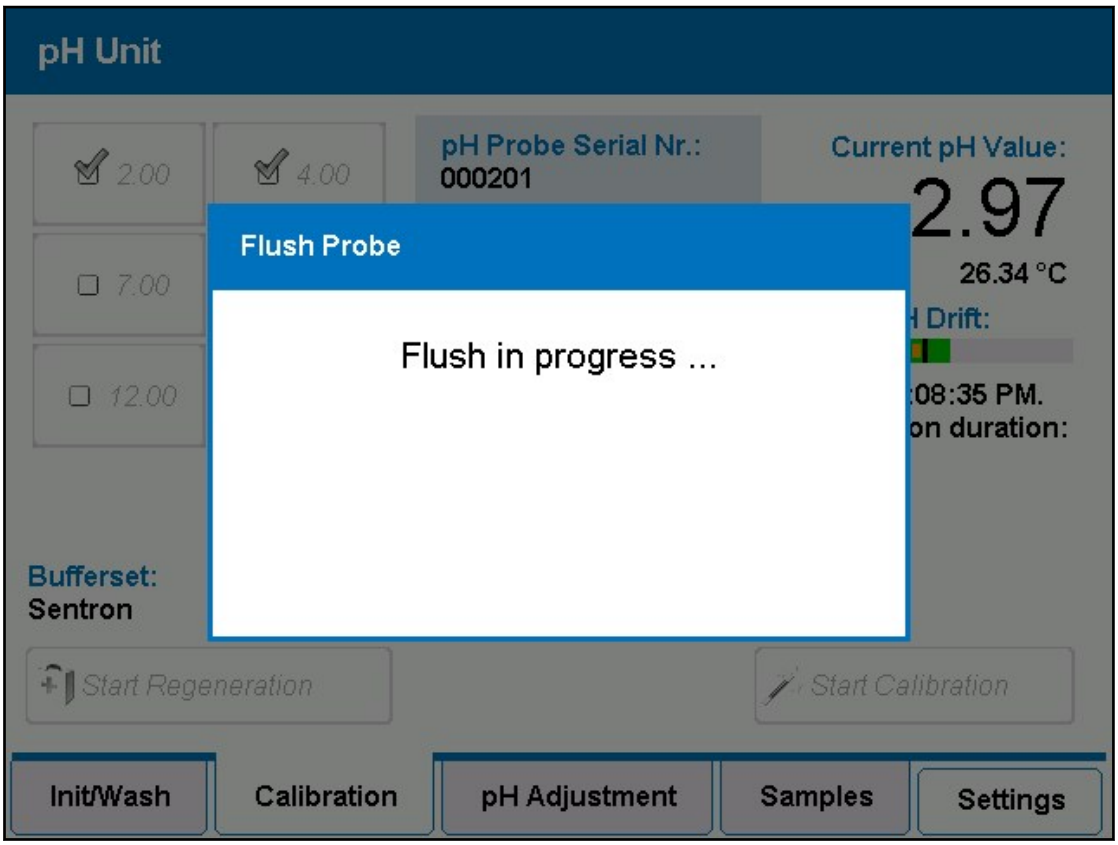


Figure 7.10: Flush in Progress Message

Once it is finished you may have a 3rd calibration phase or, if you selected only 2 calibration references, you will now be finished. This is indicated by a ‘Calibration finished at‘ message on the display.

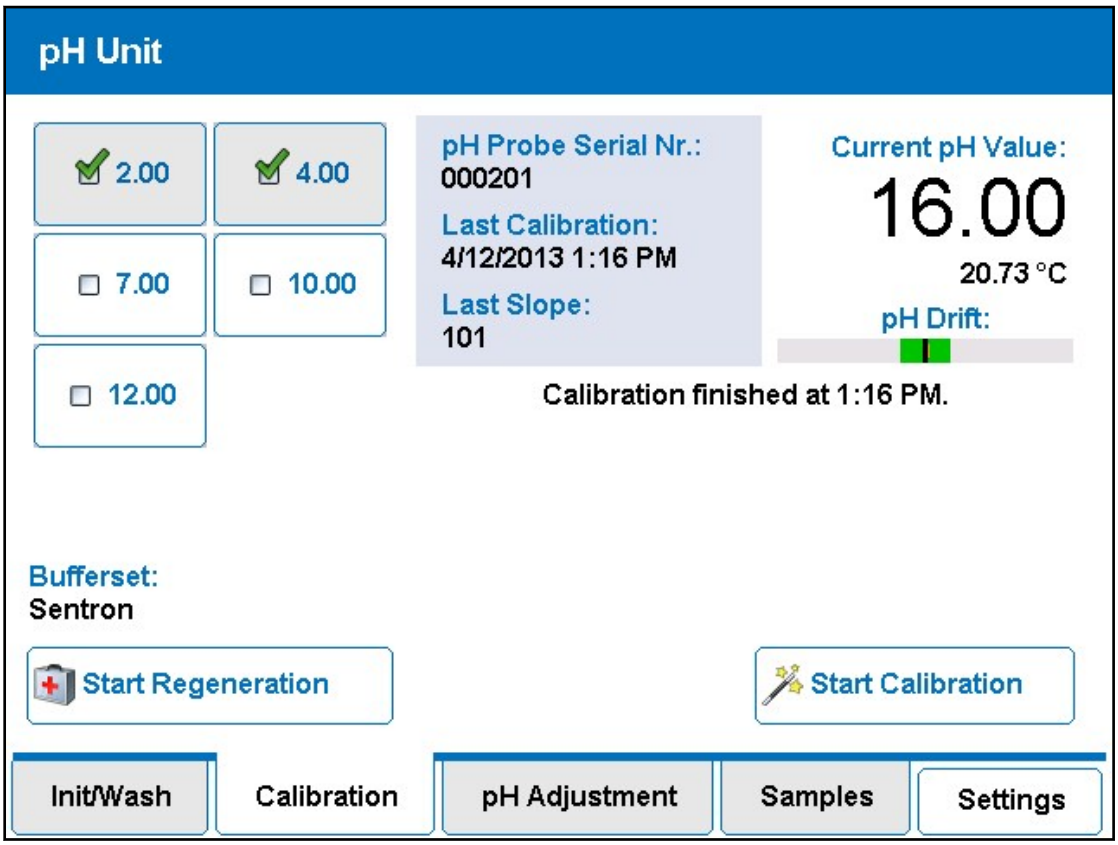


Figure 7.11: Calibration Completed

At this stage, if the calibration has been finished successfully, the calibration data and the date and time of calibration are saved to the pH Probe.

The new 'Last calibrated on value' as well as the new slope value are displayed on the 'Status' block.

The slope value should in the range 93 -108. Basically, the nearer to 100 the better the probe is working.

Out with this range and you may have to replace the probe or at the very least, thoroughly clean its ISFET sensor and the reference diaphragm. See *pH Sensor [▶ 102]*. Once again remove the probe from the reference solution and rinse/dry it thoroughly. You can now start adjusting the pH of your samples.

You should, in any case, regularly regenerate the probe especially if you notice that the slope is far away from 100: Press 'Start Regeneration' and follow the instructions.

See also

- Settings [▶ 84]
- Adjustment Settings [▶ 89]

7.3.1 Regeneration

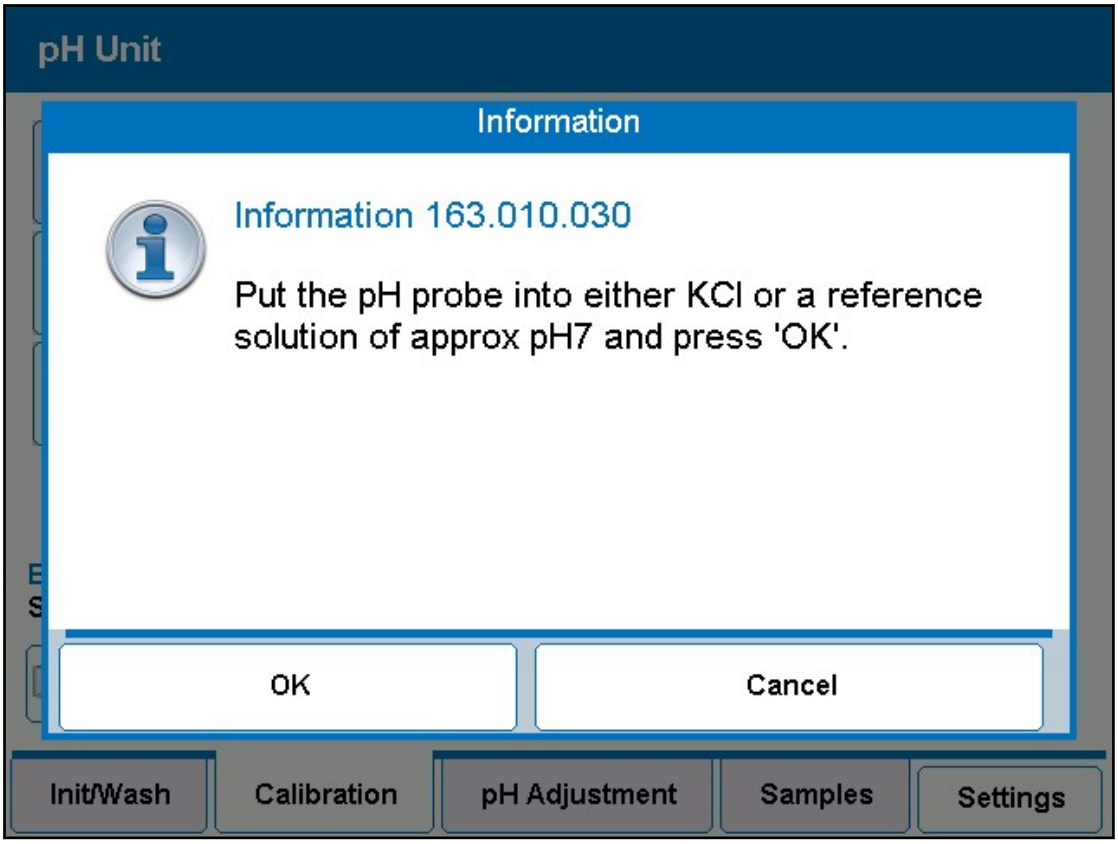


Figure 7.12: Calibration Regeneration Start

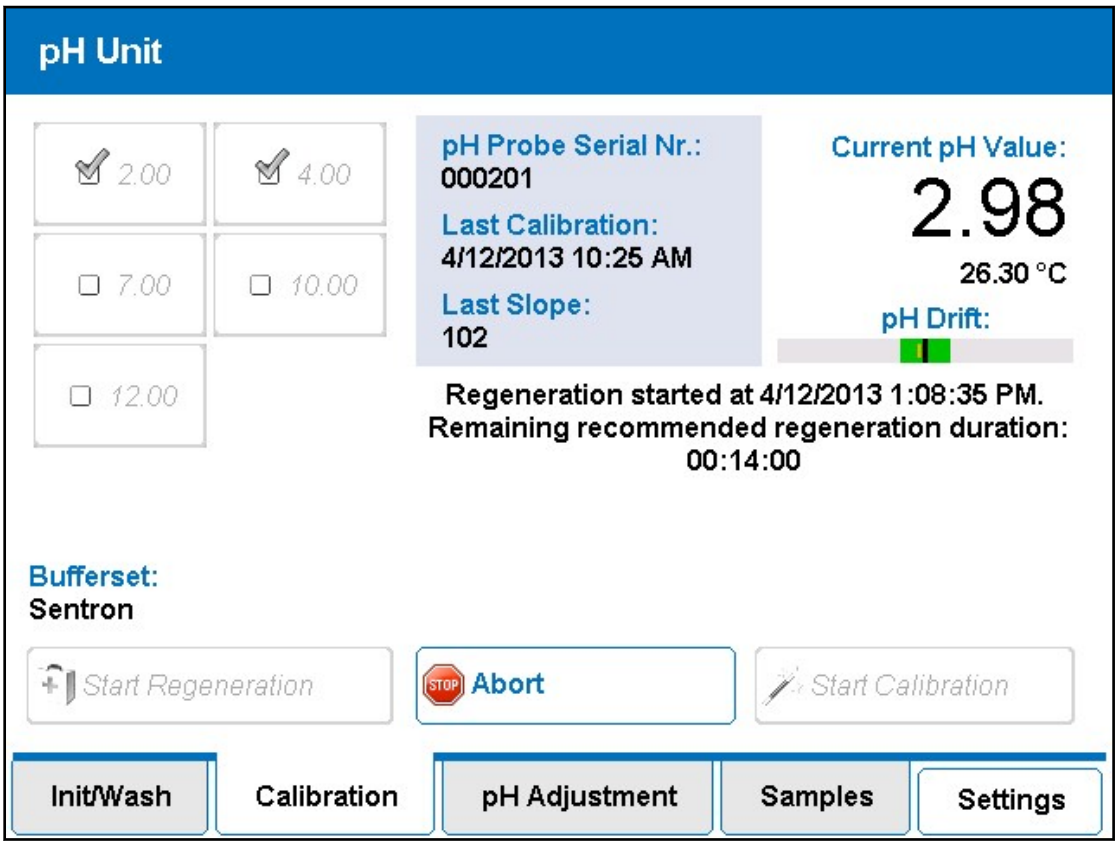


Figure 7.13: Calibration Regeneration Running

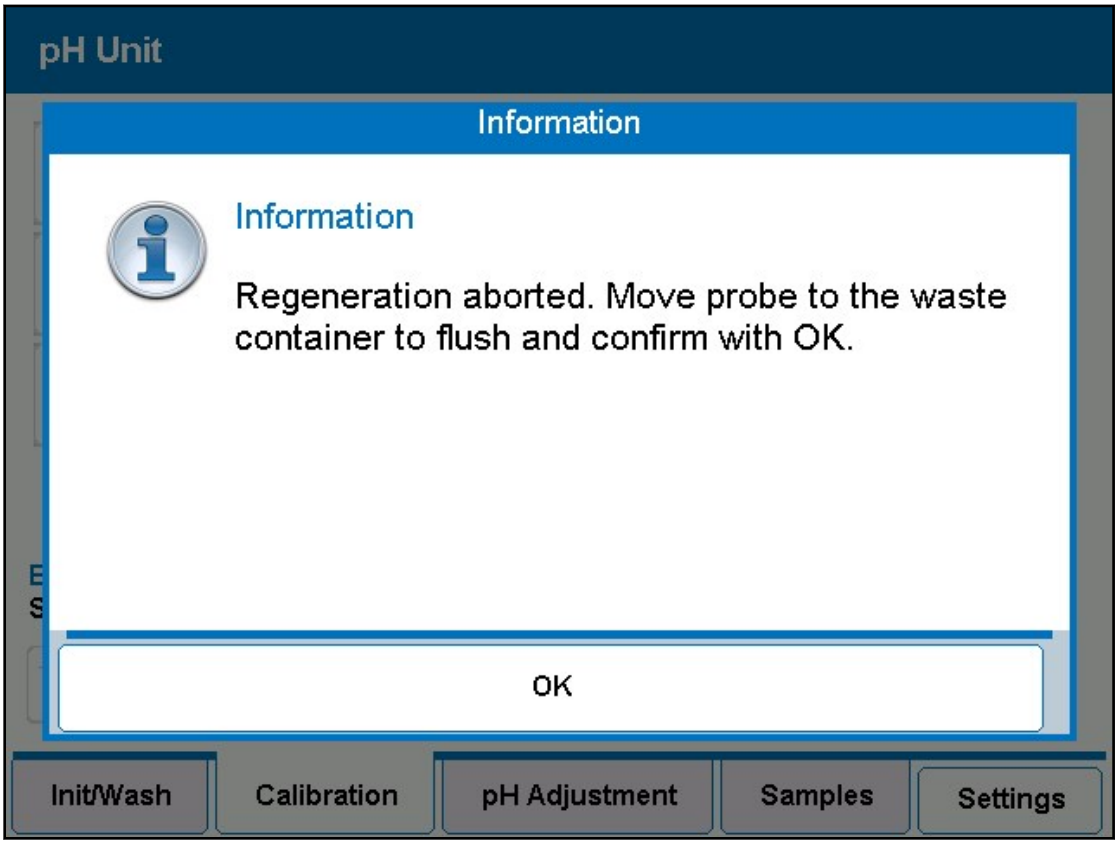


Figure 7.14: Calibration Regeneration Aborted

7.4 pH Adjustment of a Sample

Press the, ‘pH Adjustment’ tab. The display switches to pH adjustment mode.

Press ‘New Sample’.

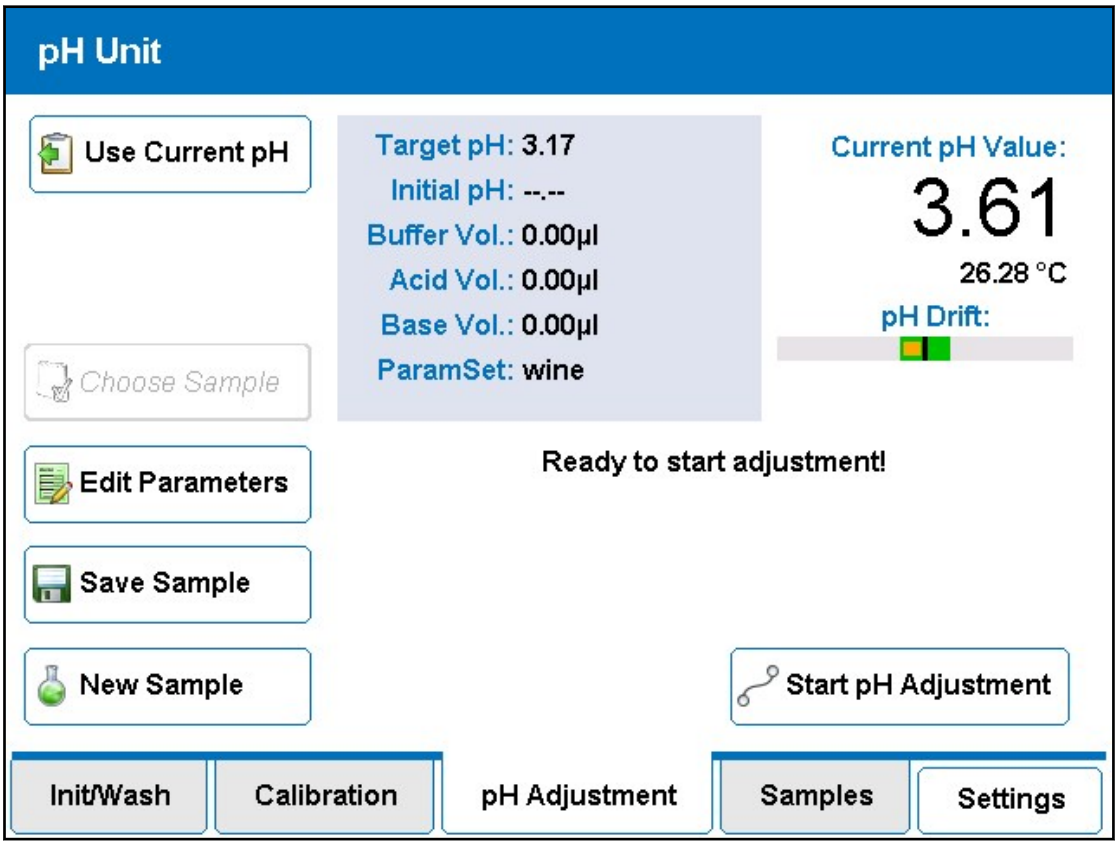


Figure 7.15: pH Adjustment Mode Window

You must now choose the Parameter Set applicable to this sample.

A parameter set consists of a set of variables which are used specifically for the pH adjustment. The variables can be ‘fine-tuned’ to allow particular sample types (for example, white wine or apple juice) to be adjusted as quickly and as accurately as possible.

See *Adjustment Settings [89]* for a full explanation.

If you are doing a series of similar adjustments you would simply use the same parameter set each time.

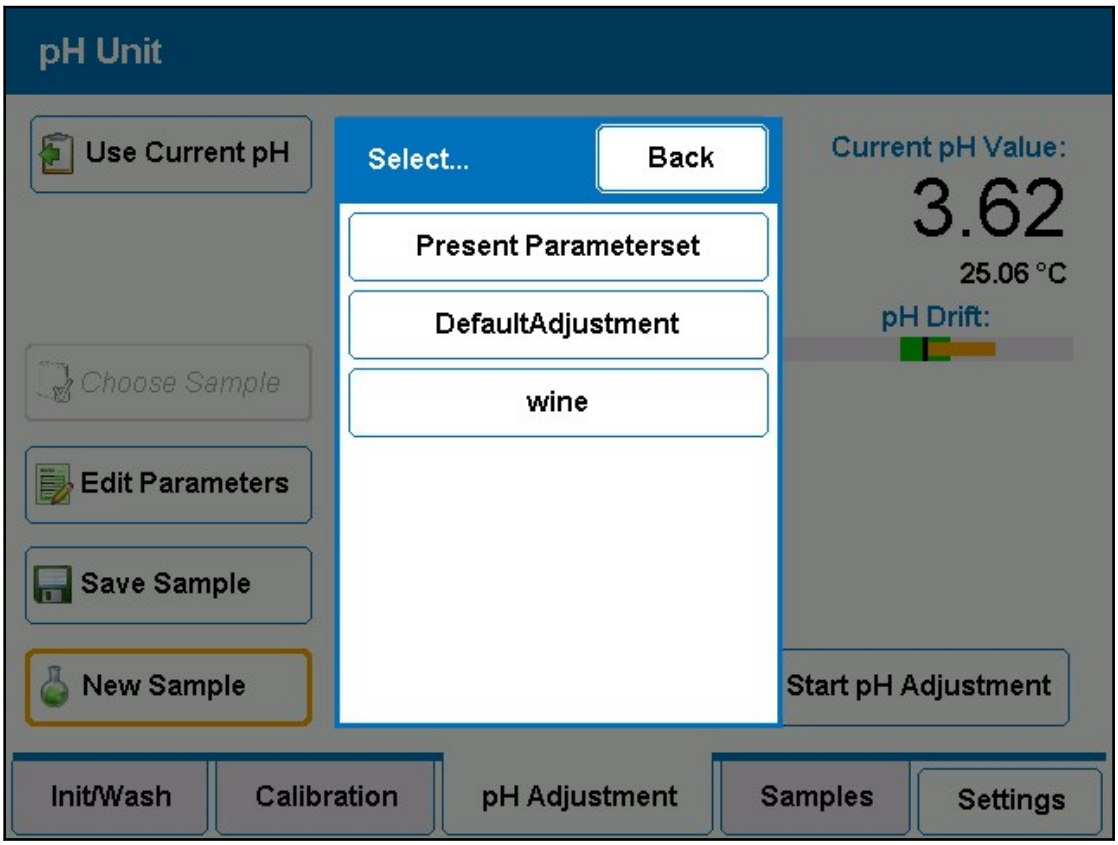


Figure 7.16: New Sample: Choose Parameter Set

So mostly you would simply press ‘Present Parameter Set‘. The unit clears any data collected during the previous sample adjustment and is now ready to adjust this sample. However, if you do wish to change the parameter set, press one of the sample set buttons to select that sample set. This clears the present sample data and returns to the Adjustment window.

Pressing ‘Back‘ returns to the adjustment window without changing anything.

You may, however, want to adjust a single parameter for this next sample (or samples). For instance, to set a different target pH or a different volume of buffer to be added. In this case, in the adjustment mode window, press the **Edit Parameters** button to call up a mask displaying those parameters selected at present.

Changing single parameters at this point won't overwrite the original 'Parameter Set' but is only temporarily used. The Parameter Set name will get an "*" so it's always clear that it's not the original "Parameter Set".

See *Adjustment Settings [▶ 89]* for a full explanation of each of the parameters. If you are unsure of the meaning of a parameter then do not change it!

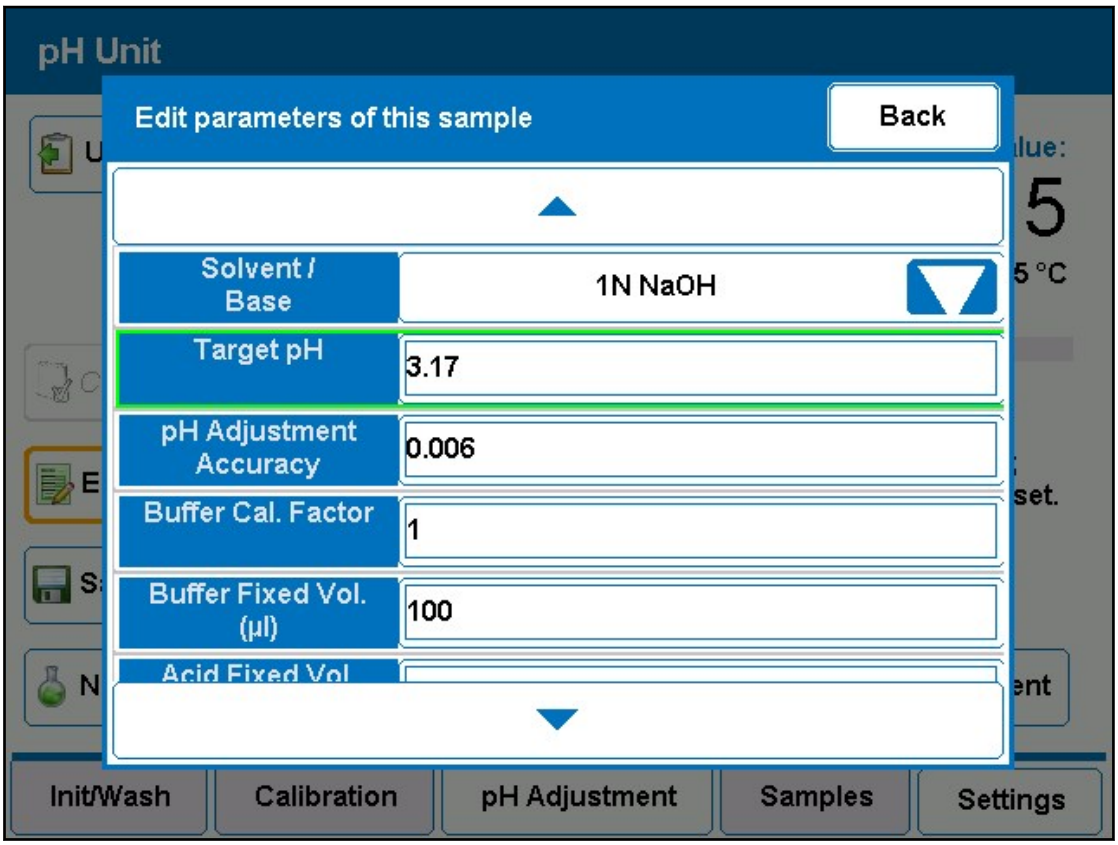


Figure 7.17: New Sample: Edit Parameters Window

Use the blue up and down arrows to move up and down through the edit options. Press the option value you wish to change. For example, to change the target pH, press on the present target pH, and a numeric (alphanumeric for some other options) keypad is displayed.

Enter the new target pH value of pH. Press ‘Enter’ to use this new value or ‘Back’ to leave the value unchanged.

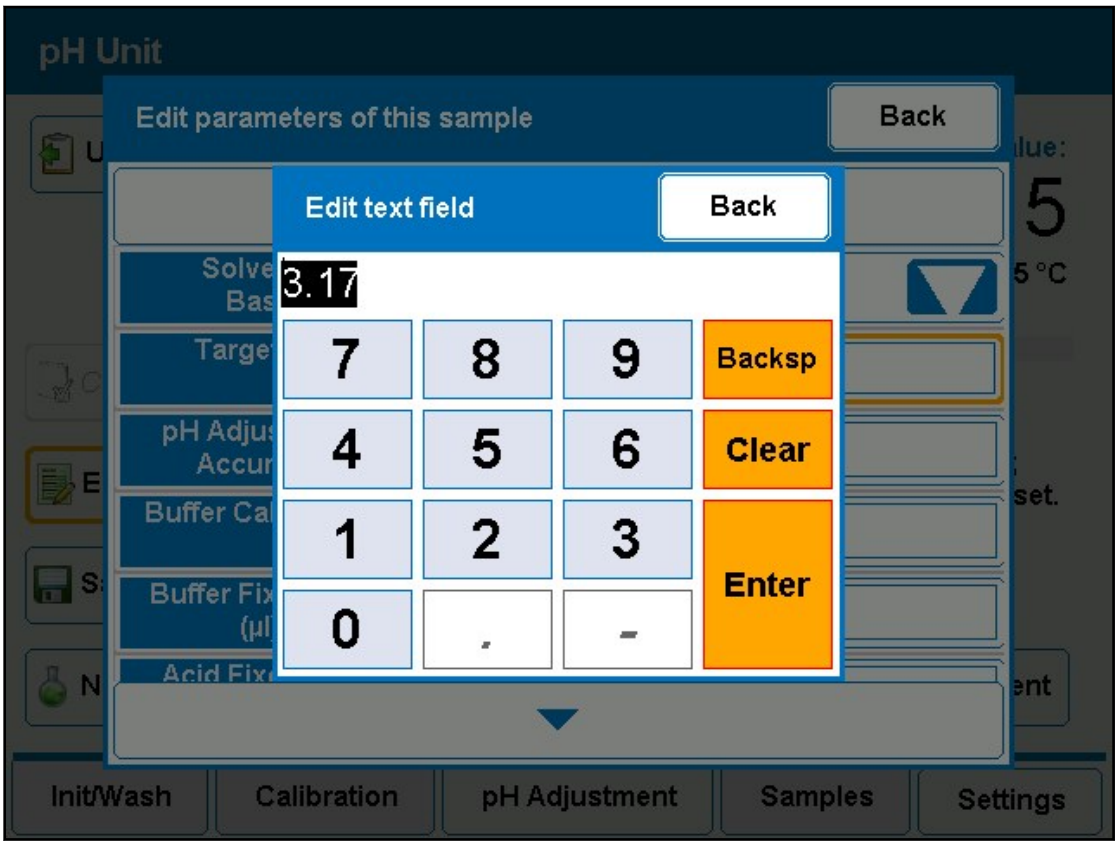


Figure 7.18: New Target pH

This new value now appears In the Edit Parameters window.

Press the **Back** button to implement this modification and to return to the Adjustment mode window.

These changes are shown in the status block:

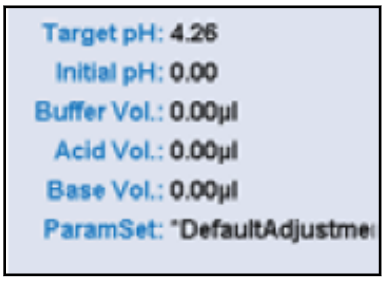


Figure 7.19: Adjustment Mode: Status Block

The target pH changes to the new value, and the data relating to the previous sample has been cleared (the initial pH appears once the adjustment is started). As the selected parameter set has been modified this is indicated by the '*' prefix to the parameter set.

A final option is if you wish to set your new sample to have the same pH as some reference you have prepared yourself. Simply wash and dry the probe and place it in your reference. Press the **Use Current pH** button. The unit waits till the reading is stable and sets this value to be the target pH.

Remove the pH probe from your reference, clean it, dry it and place it in your sample to be adjusted. In the 'Adjustment Mode Window' press **Start Adjustment**.

7.4.1 Adjustment Started: Warnings and Errors

If you have neglected to calibrate the pH Sensor before starting the pH measurements then you will get the following warning:

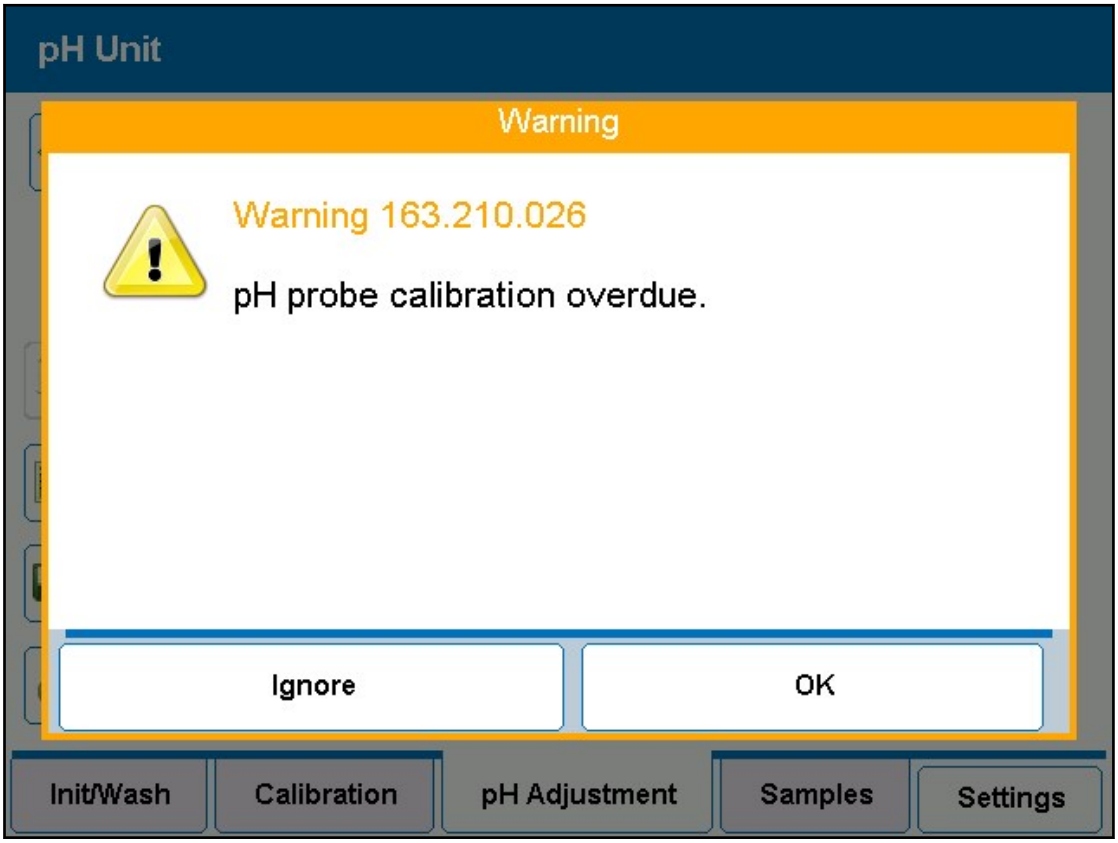


Figure 7.20: Calibration Warning

You should normally press ‘OK‘ and then calibrate the pH Sensor as defined in *Calibration Operation [▶ 45]*. However, if you are satisfied that a calibration is not needed at this stage (e.g. you calibrated the sensor earlier in the same day and are now wishing to do the last few of a large number of pH adjustments) simply press ‘Ignore’ to continue the adjustment. This resets the next warning time out to 15 minutes.

If you try to start a further adjustment after this period, the warning will be repeated. This continues until the calibration is carried out.

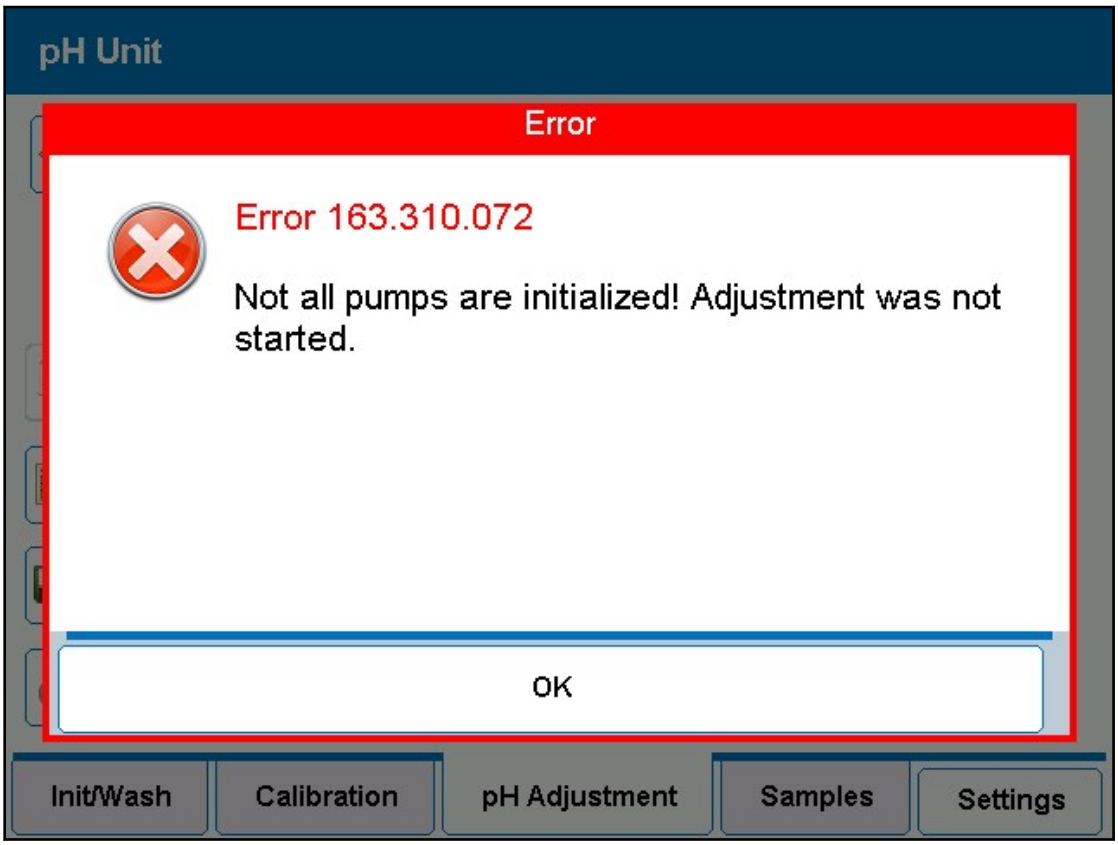


Figure 7.21: Not Initialized Error

If the pumps have not been initialized the error message to the left appears.

It is not possible to adjust the pH of a sample without first initializing the pumps.

You must press 'OK' and initialise the pumps before proceeding. See *Initialize the Unit [▶ 42]*.

Other similar warnings may also occur, usually because you have defined a parameter to be used during the adjustment and the system cannot use the value so defined: For example:

- Buffer, base or acid not the same as entered in the solvent setup.

Adjust the parameter accordingly and try again.

7.4.2 Use Current pH (target pH)

The "Use Current pH" is used to set the target pH in the pH reference solution. In doing so, you are compensating pH offsets and pH drift of the system. The result of the titration will be more correct and reproducible.

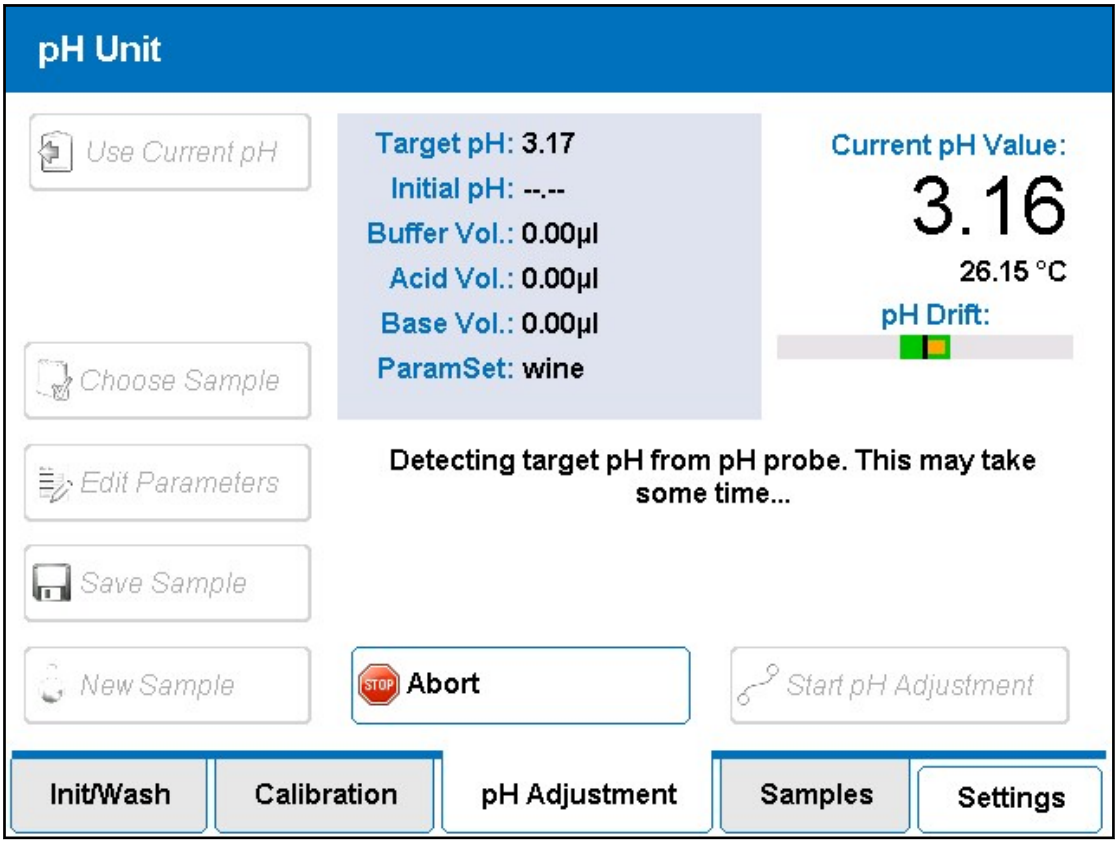


Figure 7.22: Adjustment Target pH Running

Insert the pH probe in your pH reference solution and press **Use Current pH** button. The shaker will start with the frequency set for the parameter set chosen.

After determination of the pH the value of the target pH will be changed to the currently measured pH value and used for the next titration.

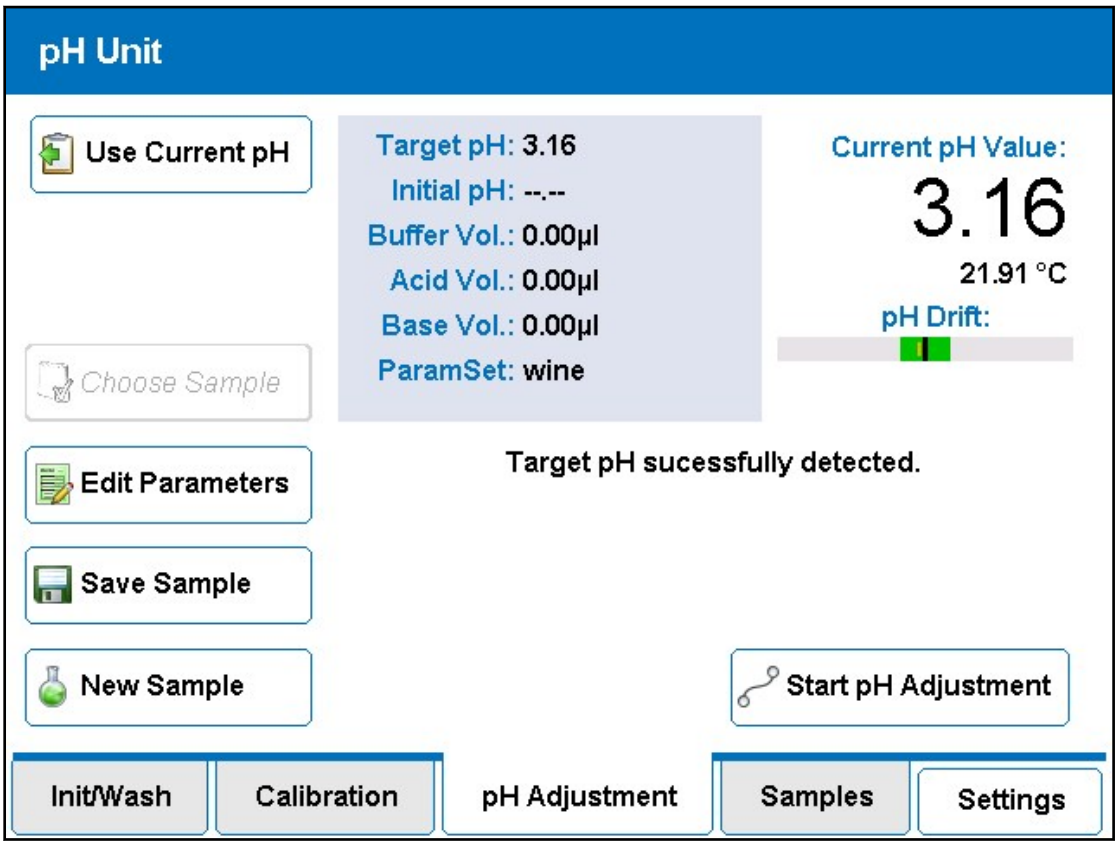


Figure 7.23: Adjustment Target pH Finished

7.4.3 Adjustment Running

After checking that all conditions required to run an adjustment have been met, the system starts the adjustment operation. The shaker starts and is set to the programmed value and the sample pH is read until it is stable. This value is then displayed as the Initial pH value in the status block.

During the adjustment the info text shows the status of the adjustment operation.

The values in the status block change as buffer, acid and/or base are added to the sample.

Adjustment Process (as displayed in the info text):

- Read initial pH value.

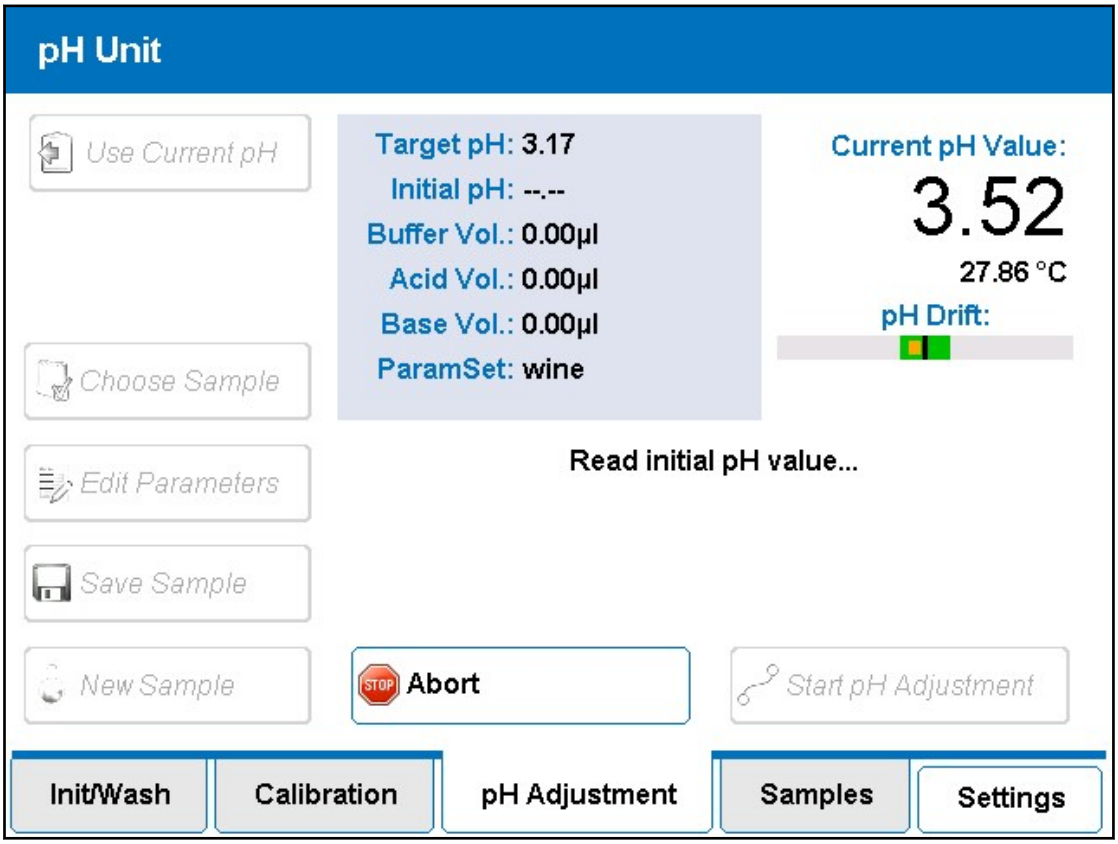


Figure 7.24: Adjustment Process: Read Initial pH Value

- Adding Buffer.

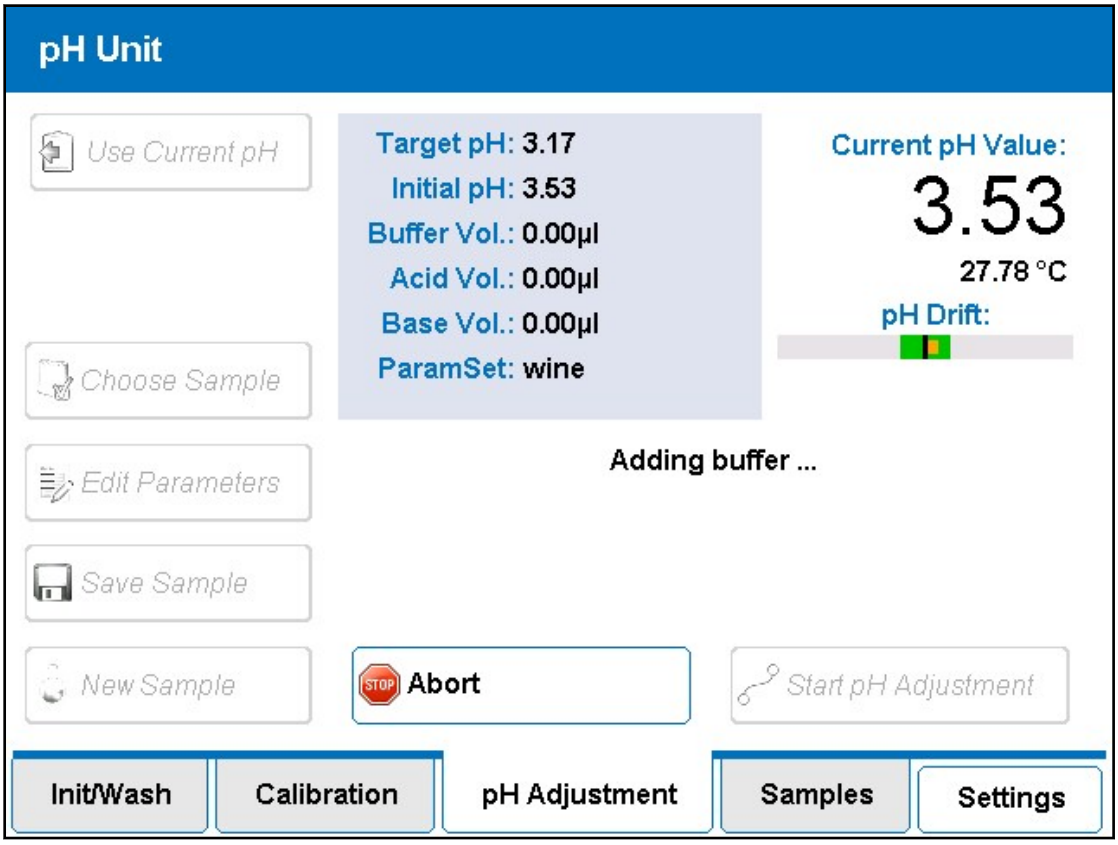


Figure 7.25: Adjustment Process: Adding Buffer

- Buffer added, waiting for stable pH.
- Adding Base/Acid (‘Base/Acid Name‘, step max. xxµL).

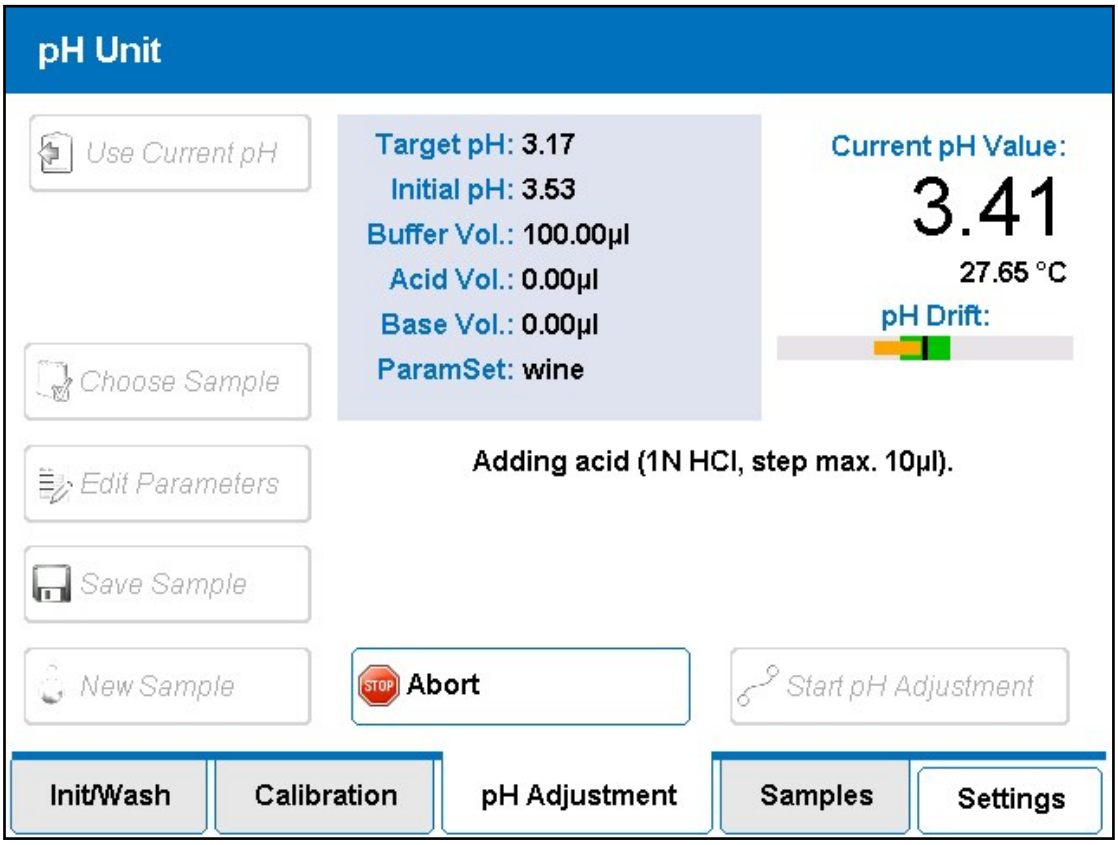


Figure 7.26: Adjustment Process: Adding Base/Acid

- Acid/Base added, waiting for stable pH.

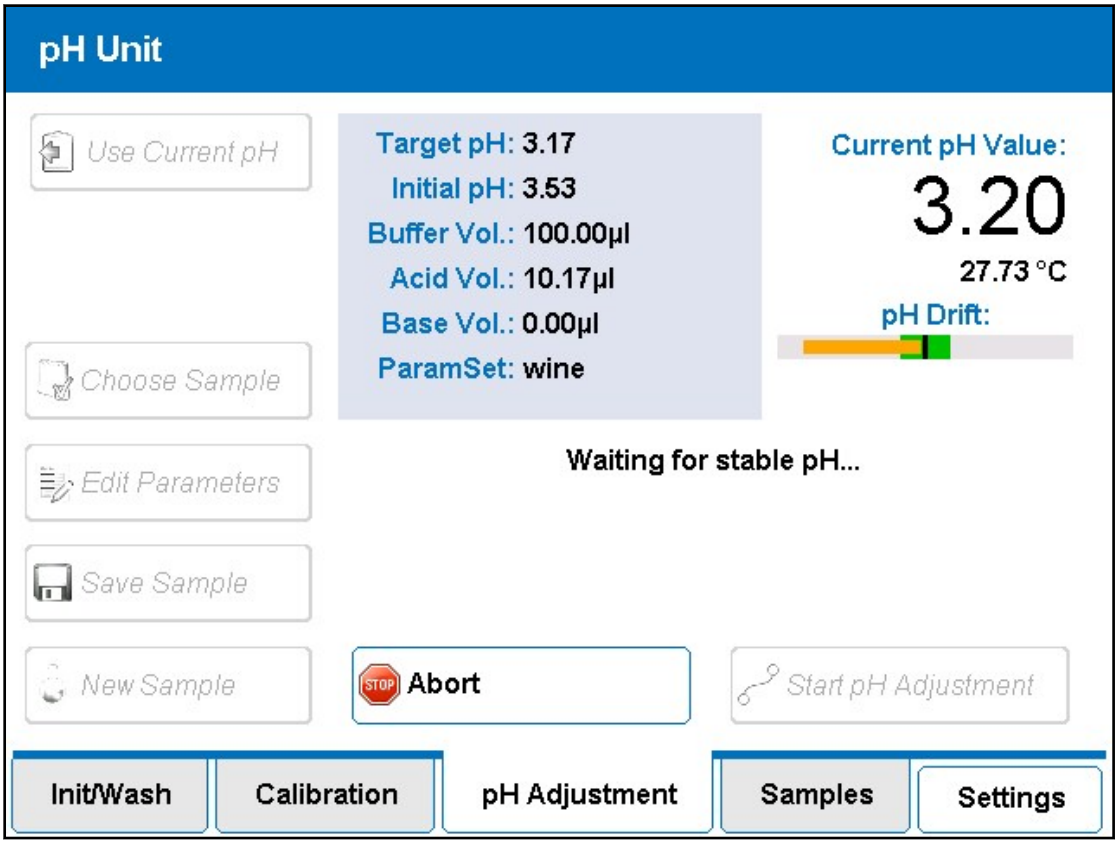


Figure 7.27: Adjustment Process: Acid/Base Added, Waiting for Stable pH

There are more messages than this. The meanings are self explanatory. While the base/ acid is being (slowly) added, the system monitors the pH. When either the step max value of xxµL has been added or the pH is within a particular regulation distance from the target (determined by the regulation factor parameter) the system stops and waits until the pH value stabilizes. This continues until the pH read is very close to the target pH. If the pH read is within the programmable fine range distance (usually 0.05), the system switches its control mode:

- The pH is within the fine adjustment range. Adding 0.5 µl base (Base Name).

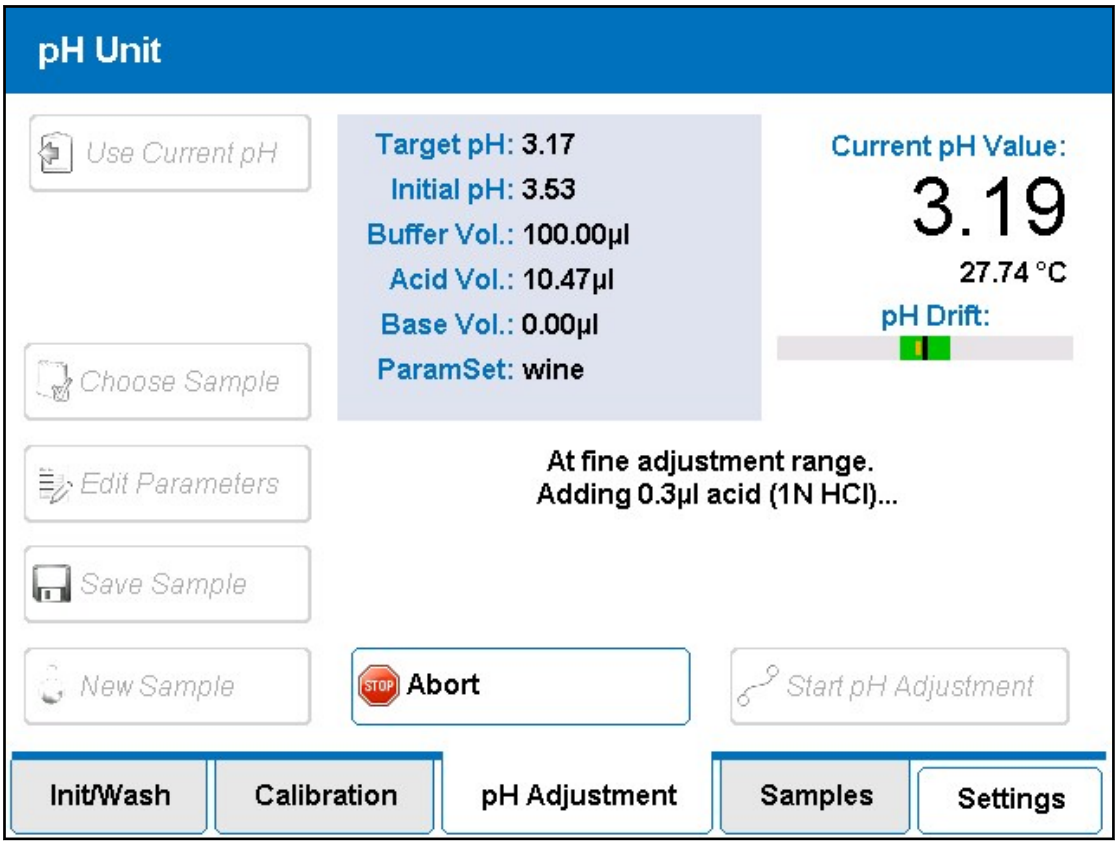


Figure 7.28: Adjustment Process: Adding 0.5 µL Base

This continues until the target pH (+ the tolerance defined by the Adjustment Accuracy parameter):

- The target pH range has been reached, waiting till the pH reading is stable.

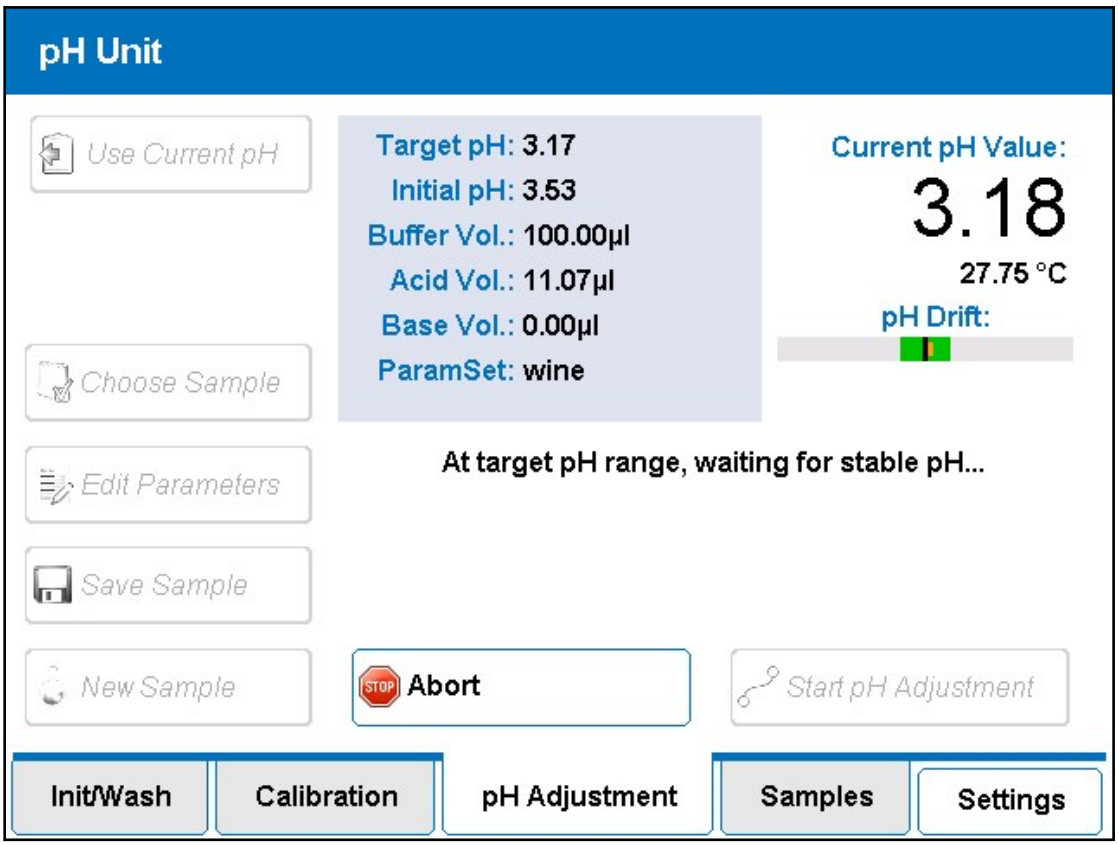


Figure 7.29: Adjustment Process: Target pH Range Reached, Waiting Til pH Reading Stable

- The pH adjustment finished in *hh.mm.ss*.

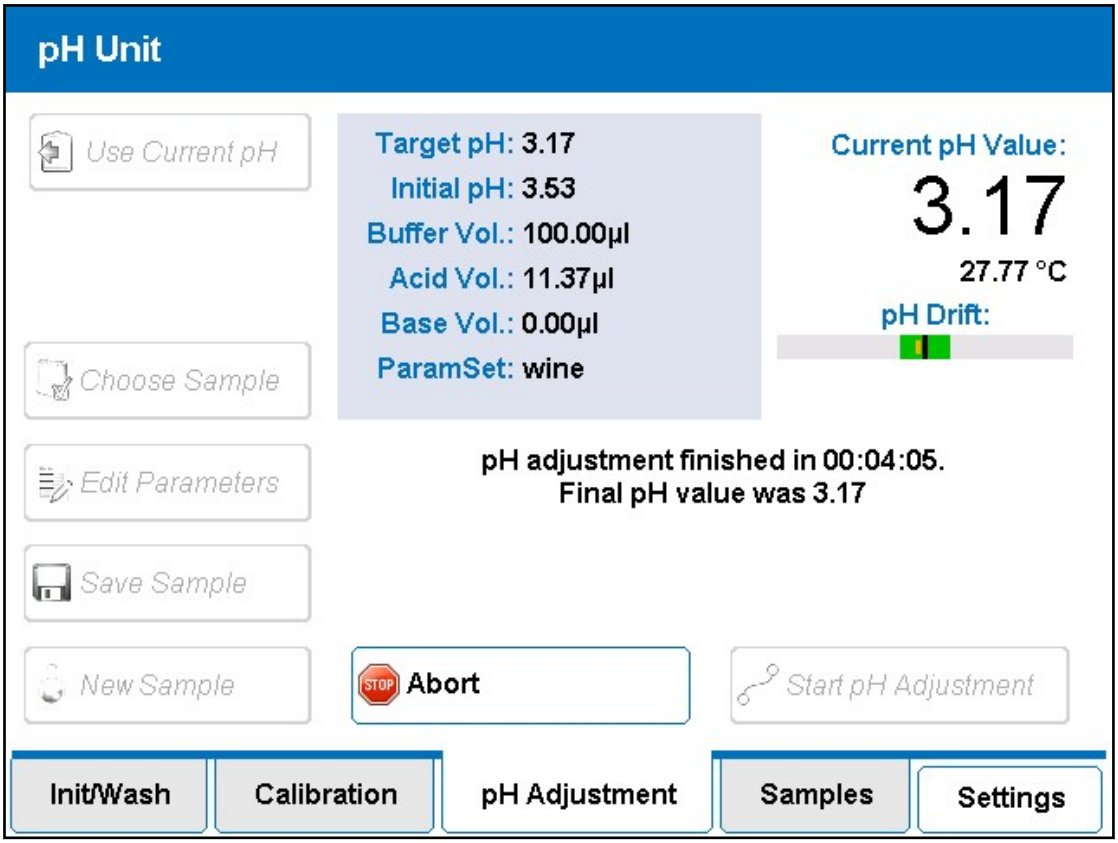


Figure 7.30: Adjustment Process: pH Adjustment Finished

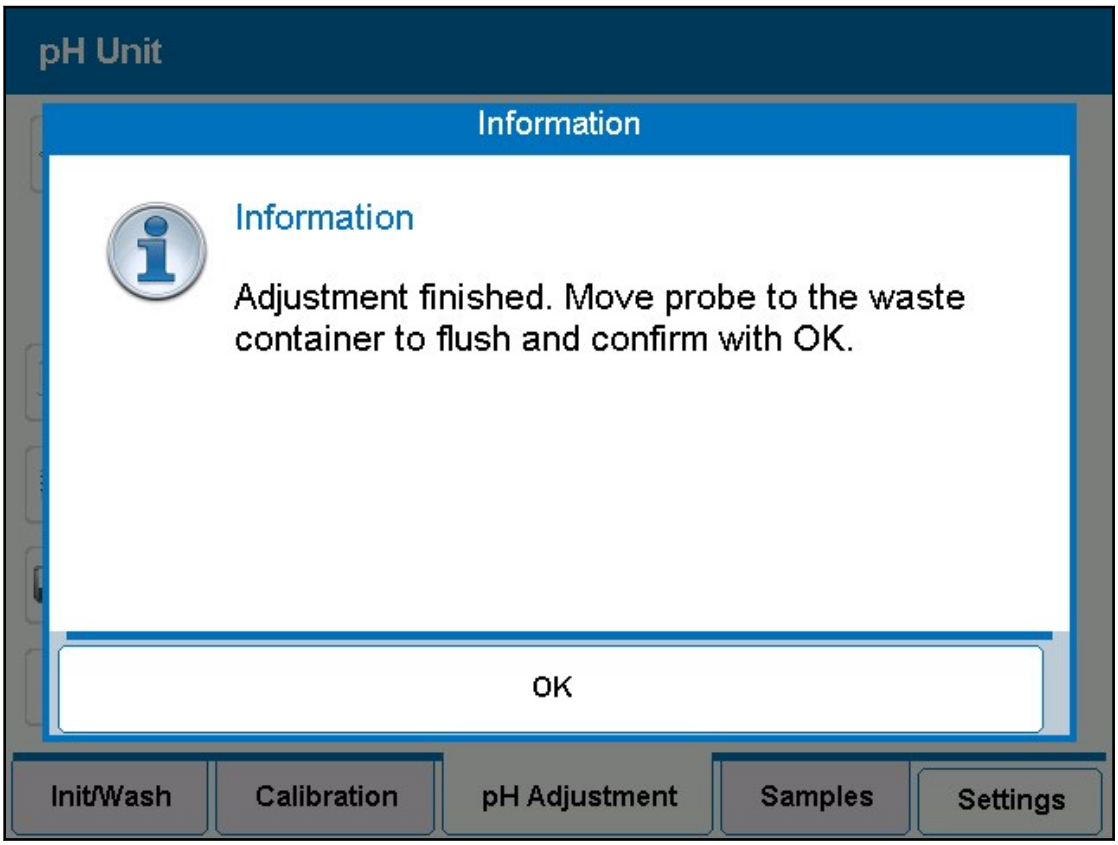


Figure 7.31: Adjustment Finished Information Window

When finished the time taken (hh.mm.ss) to carry out the adjustment is shown. Depending on how well the adjustment parameters have been set this should take approximately 2 - 5 minutes. See *Adjustment Settings [▶ 89]* for more detailed information.

At this stage you can save the adjustment information by pressing the **Save Sample** button.

See also

- Operating The Unit [▶ 41]
- Settings [▶ 84]
- Extras [▶ 98]

7.5 Sample Data

7.5.1 Save Sample

Press the **Save Sample** button.

Enter a name for this sample (here ‘my sample’), and press the **Enter** button to finish.

Figure 7.32: Save Sample: Enter Name

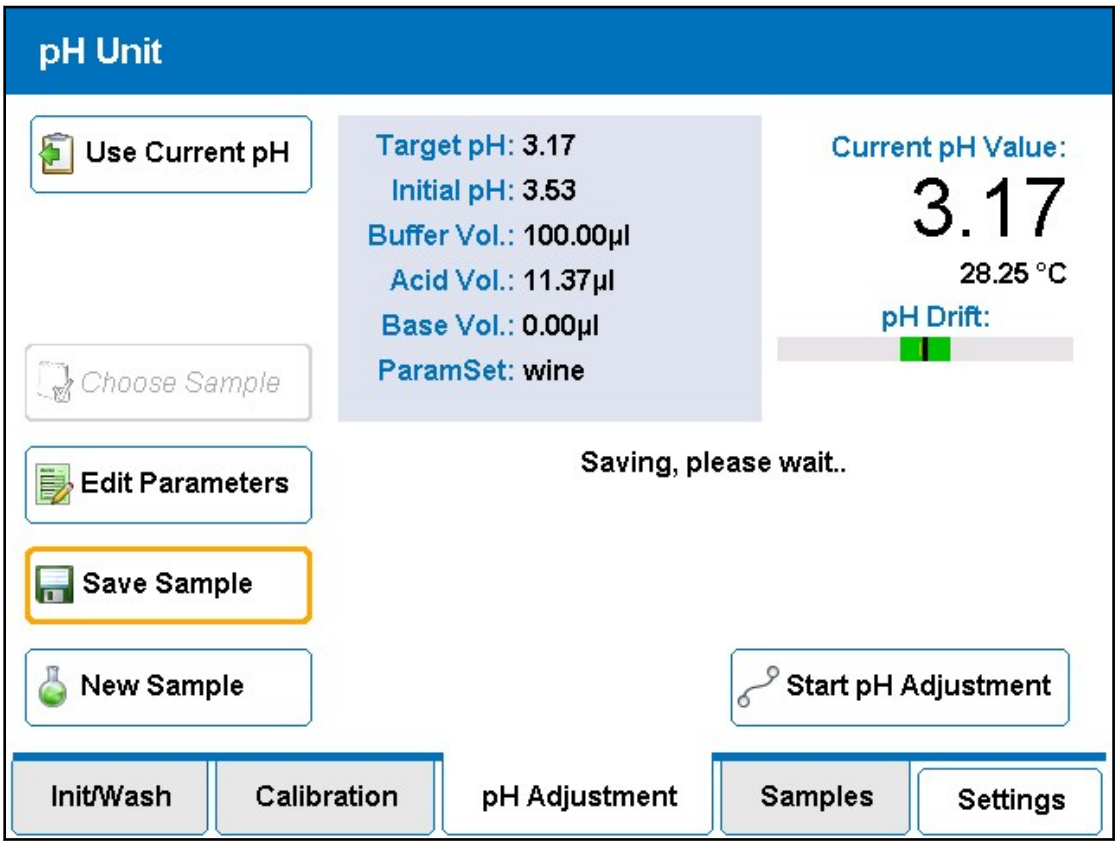


Figure 7.33: Save Sample Button

The adjustment details are now saved to system flash memory and are accessible via the **Samples** button or can be downloaded via the ethernet connection. See *Download Sample List [▶ 75]*.

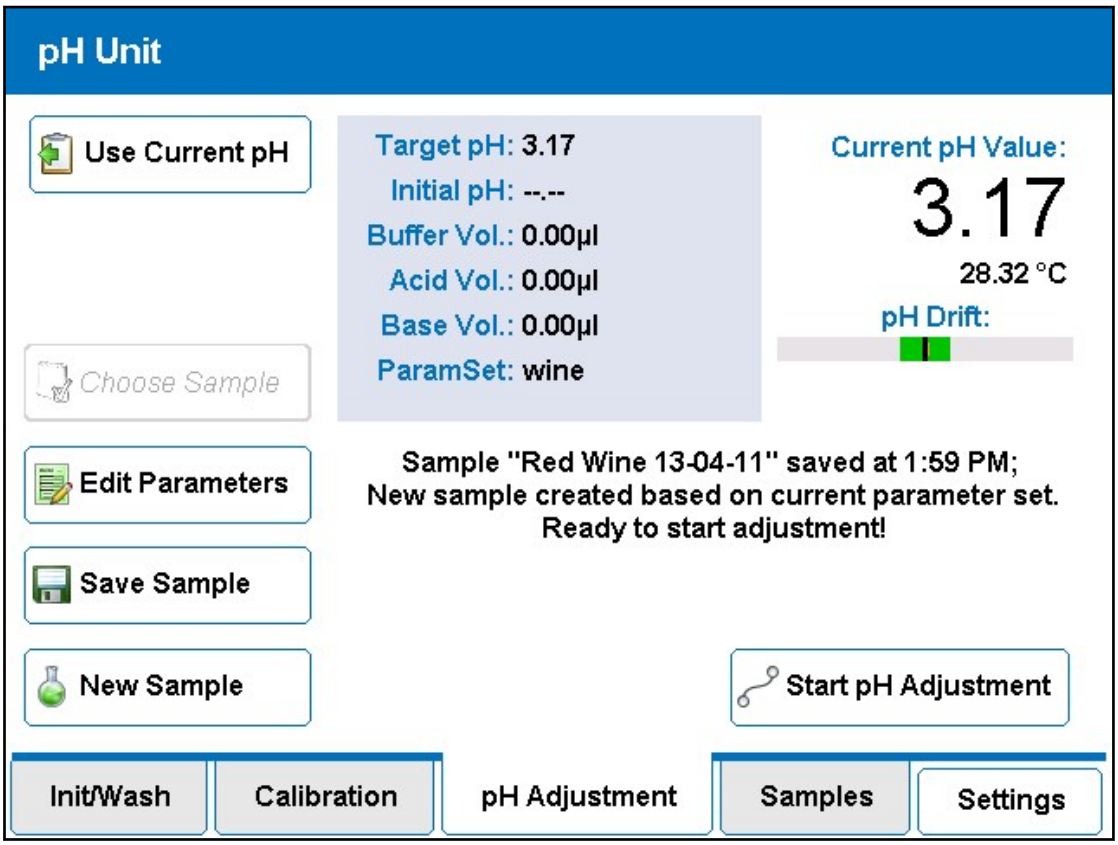


Figure 7.34: Save Sample Finished

The sample list is not lost when the unit is powered down.

7.5.2 Display Sample Data

Press the **Samples** button to display a list of samples saved.

Use the up and down arrows to scroll through the list of samples. Press the name of the sample for which you wish to display the information.

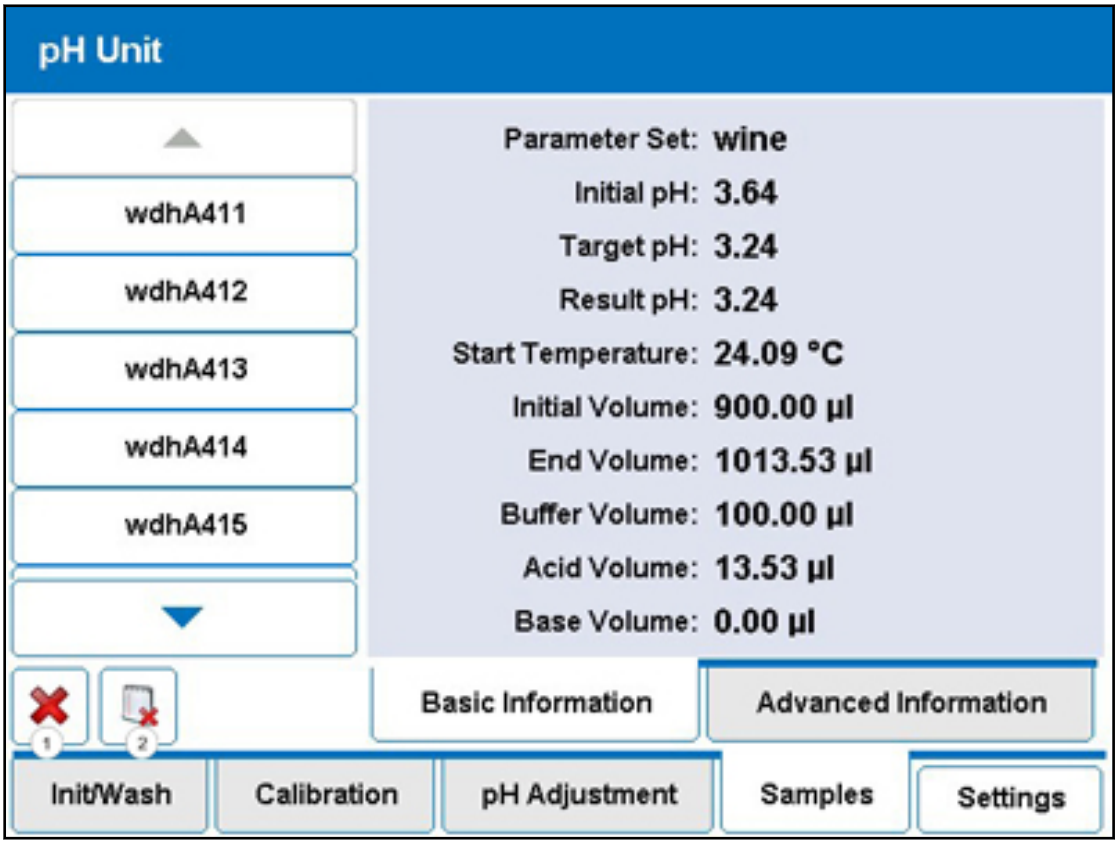


Figure 7.35: Sample Data: Basic Information

1.	Delete Selected Sample Button	2.	All Samples Button

The information is split into 2 sections accessible via the ‘Basic Information’ and ‘Advanced Information’ tabs.

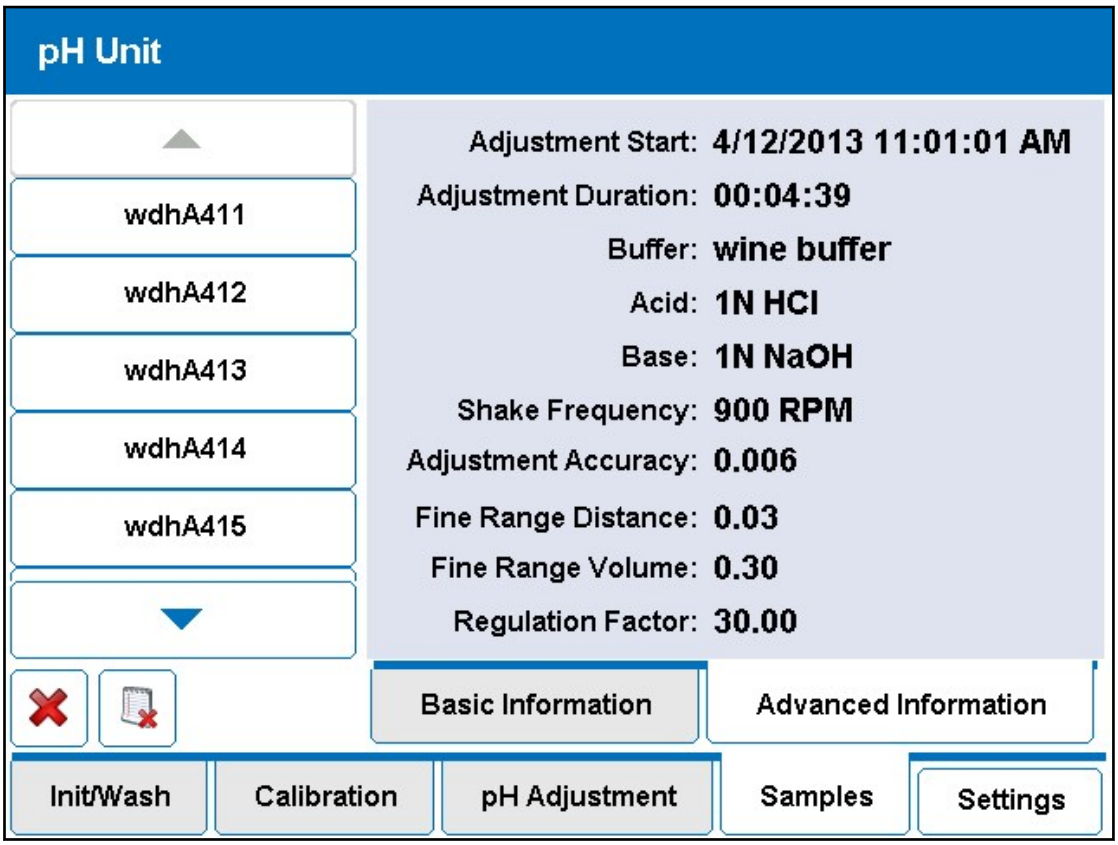


Figure 7.36: Sample Data: Advanced Information

The buttons at the bottom left allow you to delete the selected sample or all samples in the list. You are asked to confirm this or cancel.

Note: More than 35 system and application parameters are saved, but not all parameters are displayed.

7.5.3 Download Sample List

In order to do this the touch display controller must be connected to a network or computer on which a DHCP program is running (This is a program which allocates IP addresses to network connected systems). If you cannot connect the unit to your work's network you can simply install a suitable (there are many *freeware* versions available) DHCP server program on your PC or laptop.

The touch display software only requests an *IP address* if it identifies an ethernet connection during its initialization phase (i.e. while powering up). Connect the touch display controller and a suitable PC via an ethernet cable then reset the touch display (remove/ replace its DC power cable).

If you have a DHCP server running on the device connected over this ethernet cable the allocated IP address is displayed on the initialization screen. Occasionally the DHCP server is too slow to allocate the address and the 'no network' message is displayed instead of the IP address. However, once the unit has started you can check the IP address via the touch panel - Settings - General - About Device. Also your DHCP server may well have a 'Log' window you can check. The allocated IP address will also be displayed here (see *General Settings [85]*).

1. Start your browser of choice and enter this address in the *URL* field.
2. Click on ‘Download‘ then on the text ‘Download samples list as a *CSV*‘.

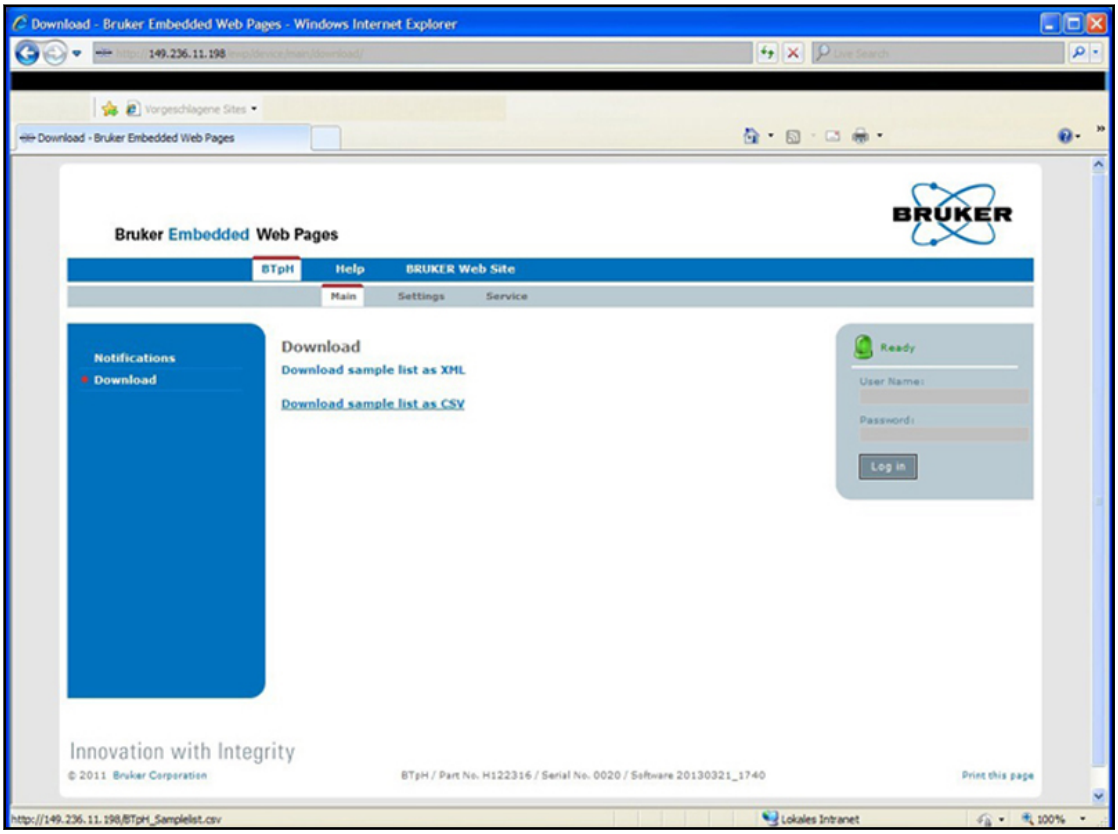


Figure 7.37: Web Page at Base IP Address

3. Save the file. Do not open the file yet as Excel will almost certainly not format the columns exactly as you wish.

See also

Opening a file using the template [▶ 77]

7.5.4 Importing Data into Excel

The precise menu text and format are dependant on your Excel version and language. This is only an approximate guide. This only has to be done once. After which you can save the file as a ‘template’ to be used for future files.

1. Start Excel and open a new file.
2. Click on **Data** - **Get External Data** - **Import File**.
   - ➡ This opens up the select file window.
3. Navigate through this till you find and select the file you wish to open.
   - ➡ The Text Conversion Assistant opens.
4. Click on **Separated** to select the type of import, then **Continue**.
5. Select the **Tabstop** as the separator then continue.
6. Click on **More** then set the decimal and thousandth separator to be ‘.’ and ‘, ‘ and enable negative numbers, then click **OK** to return.

➨ You will see a short display of the file contents to be imported.

7. You can set the format for each column. Set this to ‘Standard’ for all the columns you do wish to import and to ‘do not import’ for the others.
8. Click on **Finished** then select the data to be imported as a new file.
9. If you have inadvertently imported too many columns simply delete these you do not want. If you have forgotten to import some columns it is probably easiest to start anew.
10. Select the columns with numbers (e.g. Initial pH, Result pH), right click and select **Format Cells**. Select **Numbers** and the number of decimal places you wish to see (normally 2 places for pH and 1 for volumes). Then click on **OK**.

    ➨ Your file is now formatted as you wanted.
11. Save the file by selecting **File** - **Save As**, then select the standard data type (*xls*, *xml*), and a file name.

7.5.5 Generating an Excel Template

Save the file you have generated above as a ‘Template’.

Click on **File** - **Save As** then select the template data type *.xlt

Enter a suitable name (e.g. BTpH_Template) then save the file. You will be asked if you want the data to be deleted and re-opened with this template - click **OK**.

7.5.6 Opening a file using the template

In Excel open the template file and click on **Activate Automatic Update**. Now you can select your new *.csv file to be opened using this template. The File opens formatted exactly as in your template.

You can now save the file as an *.xls or *.xml type.

7.5.7 Interface with Optional SampleTrack and Barcode Reader

The BTpH also provides interface with optional SampleTrack and a barcode reader.

Figure 7.38: The Optional Sample Barcode Reader

When the handheld barcode reader is connected to the second USB port of the touch panel, and the unit is networked with a SampleTrack PC, the display on the pH Adjustment page shows an additional button named **Choose Sample**:

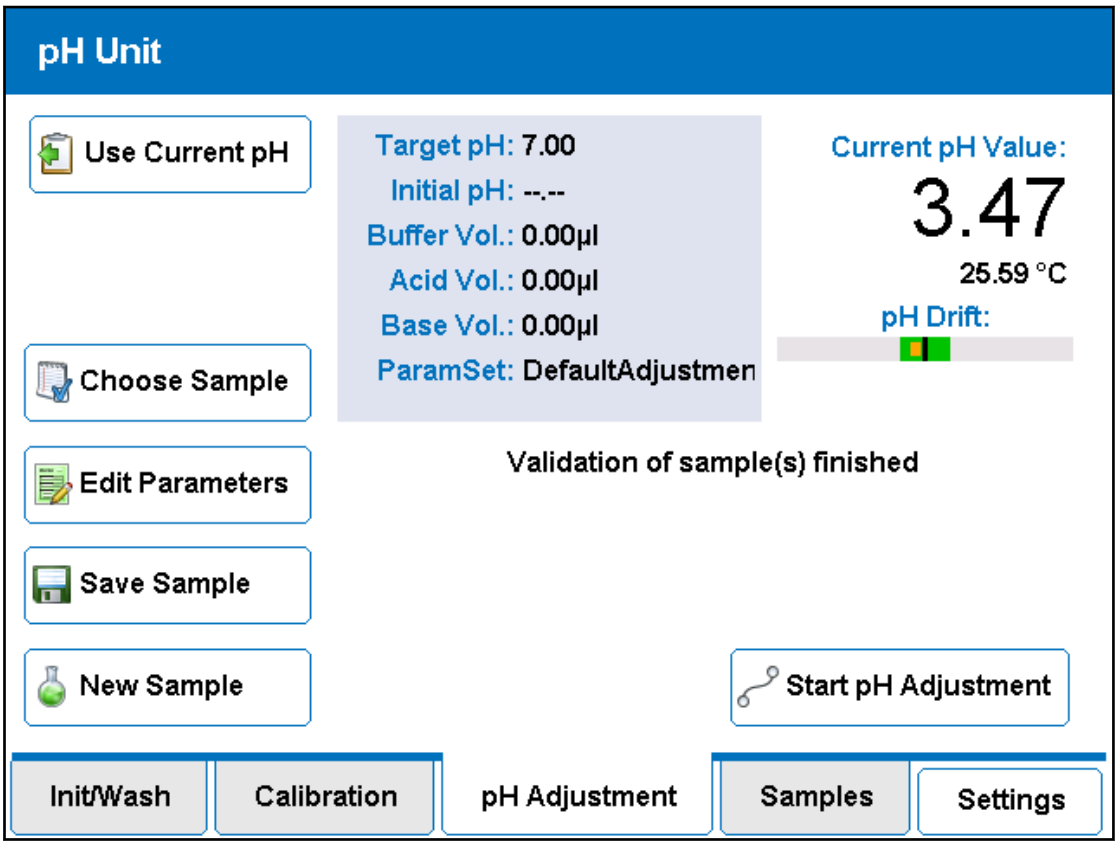


Figure 7.39: pH Adjustment: Choose Sample Button

When a sample list is set up in SampleTrack and verified, the Sample ID's will be transferred automatically to the BTpH unit.

Note: The BTpH unit must be initialized and the pH probe calibrated, otherwise SampleTrack will receive an error and can't verify the run(s).

When you press **Choose Sample** the list that is sent from SampleTrack will be displayed. Now you can choose a sample either by pressing the corresponding sample ID, or using the barcode reader (the safer way). As this list can be rather long you have the chance to filter your sample(s) of choice by using the **Enter Filter Value….** button.

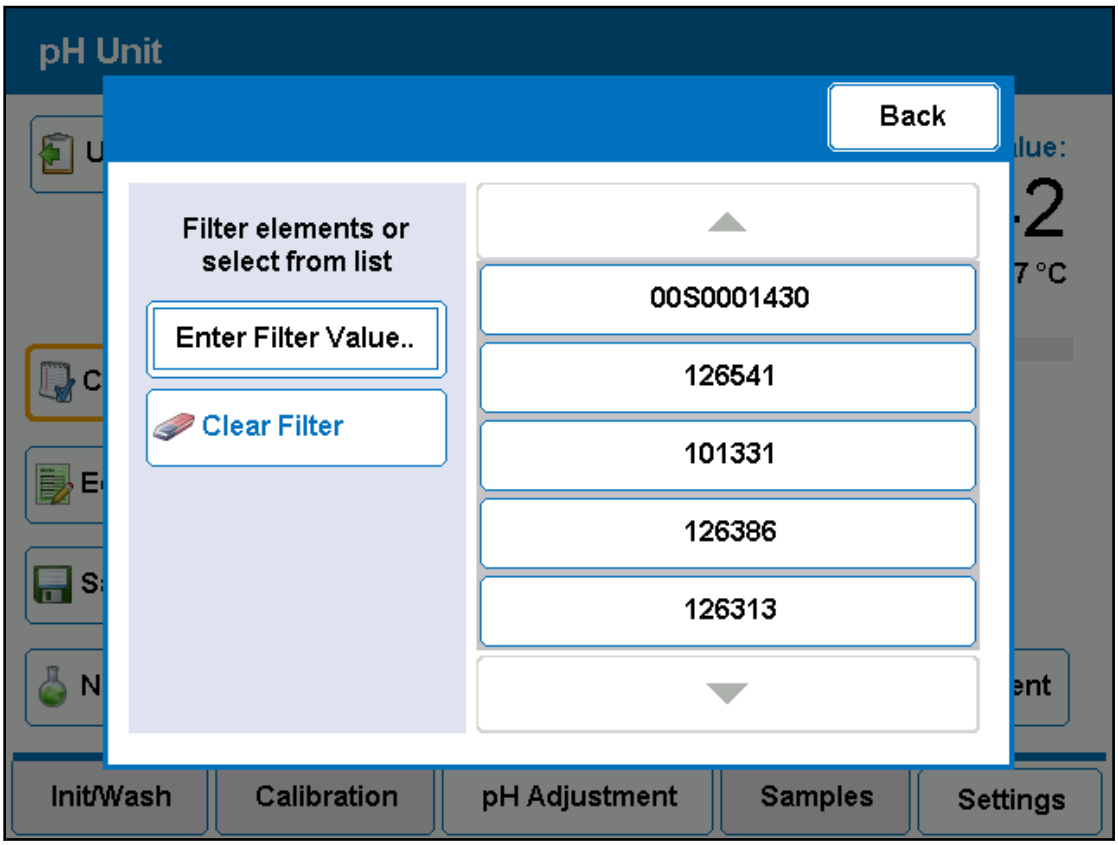


Figure 7.40: pH Adjustment: Choose Sample Window

When you press the **Enter Filter Value** button, a specific value (e.g. '13') can be entered, whereas only samples including the numbers *13* will be displayed:

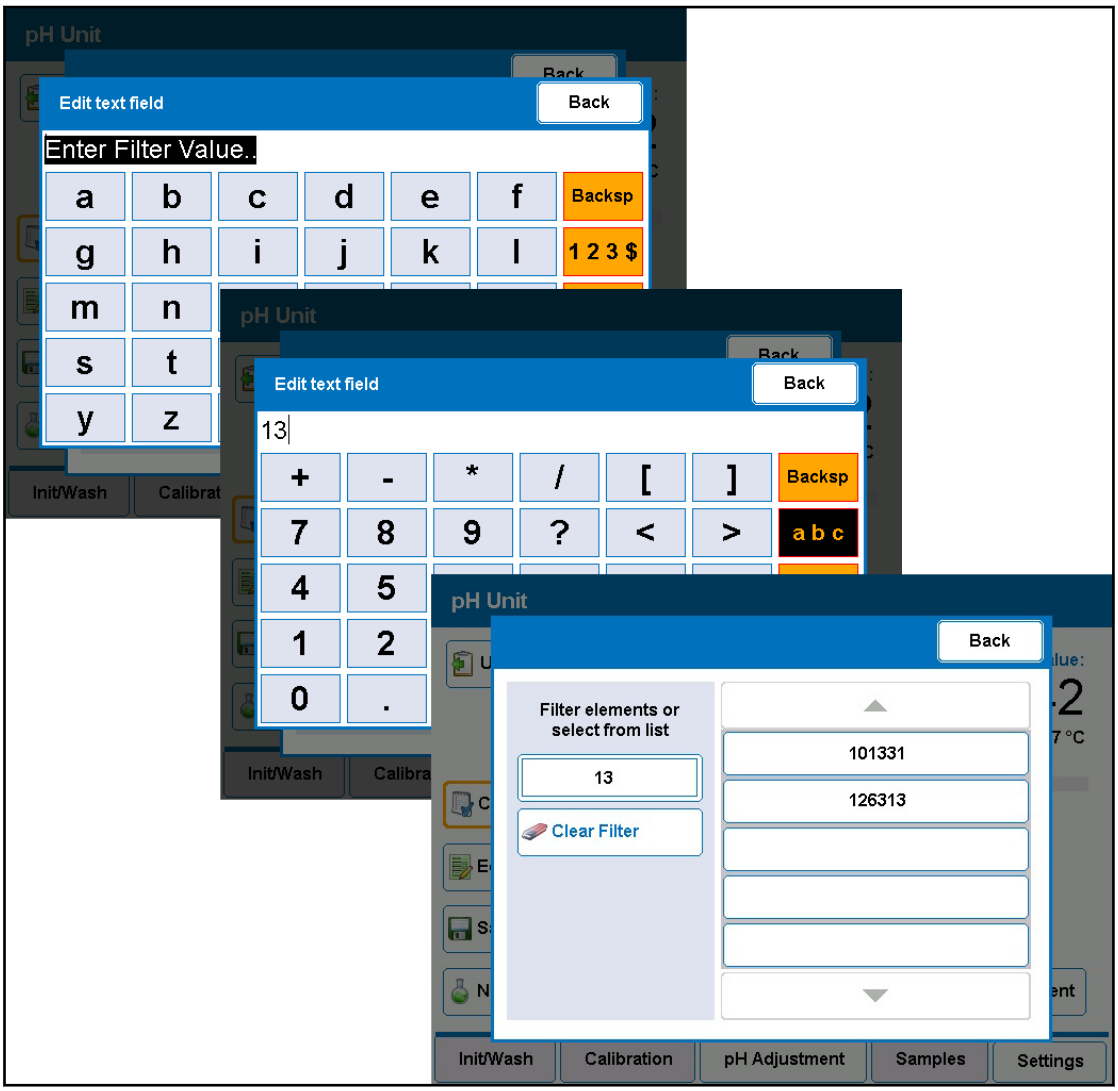


Figure 7.41: pH Adjustment: Entering Filter Value

After the filter is selected the system will scan for your sample and the corresponding parameter set will be loaded.

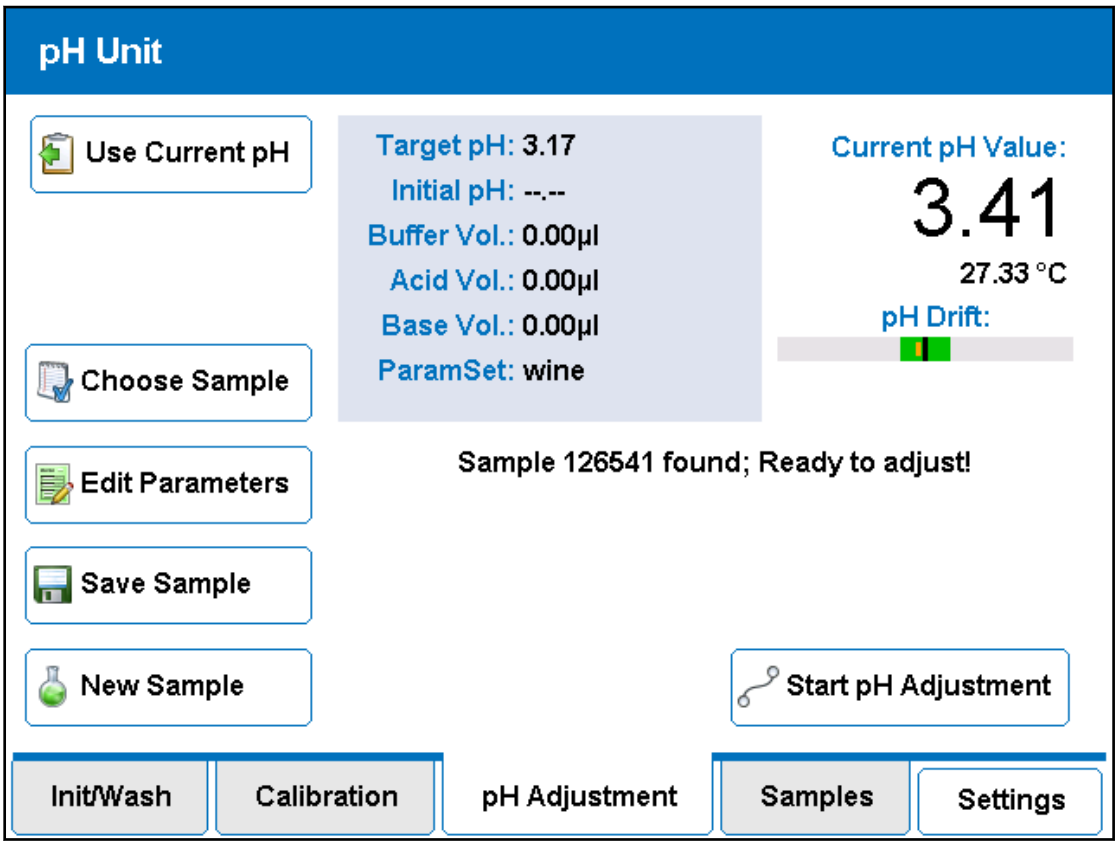


Figure 7.42: pH Adjustment: Scanning for a Sample

The unit is then ready to start the titration.

After titration is finished, all parameter used and created during the endpoint titration will be send automatically to SampleTrack. No manually stored sample will be overwritten.

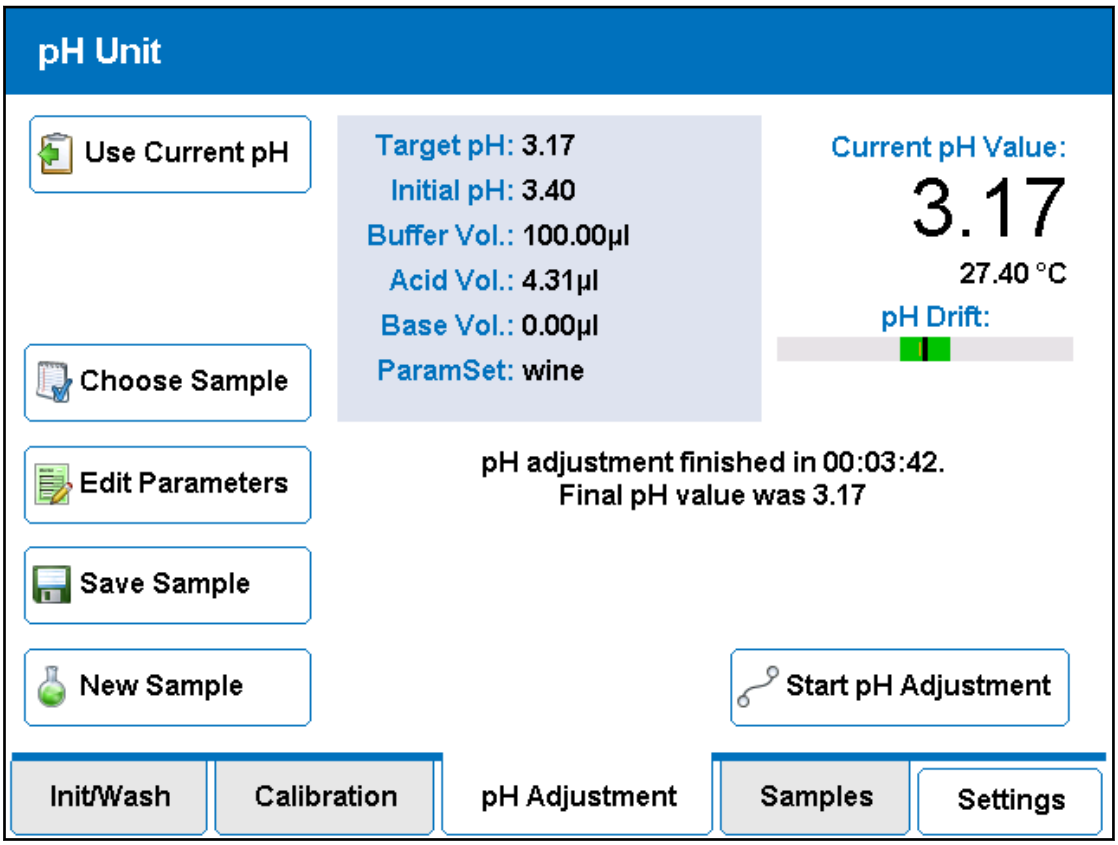


Figure 7.43: pH Adjustment: Adjustment Finished

Using a reference sample, e.g. each 5th or 10th sample, you can load the correct parameter set manually and choose the 'Present Parameter Set' in SampleTrack.

7.6 Settings

Press the settings tab to enter the settings pages. The unit may take a few seconds to complete the switch.

From there you can select either **General Settings** to change the clock time, calibrate the touch pad etc. (self explanatory), or **BTpH Settings** to change parameters sets and liquid control.

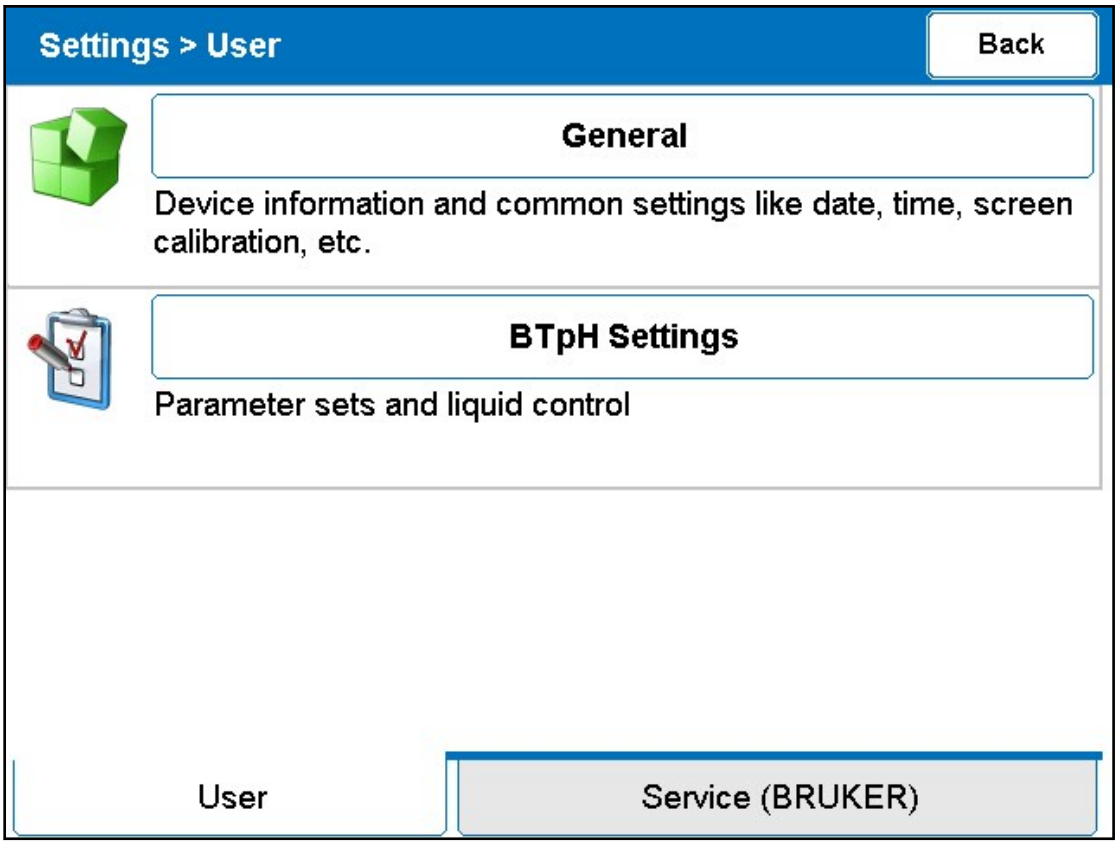


Figure 7.44: Settings: User Window

7.6.1 General Settings

Touch Screen

You can adjust the touch panel to your personnel finger print. Press the **Start calibration** field and follow the instructions.

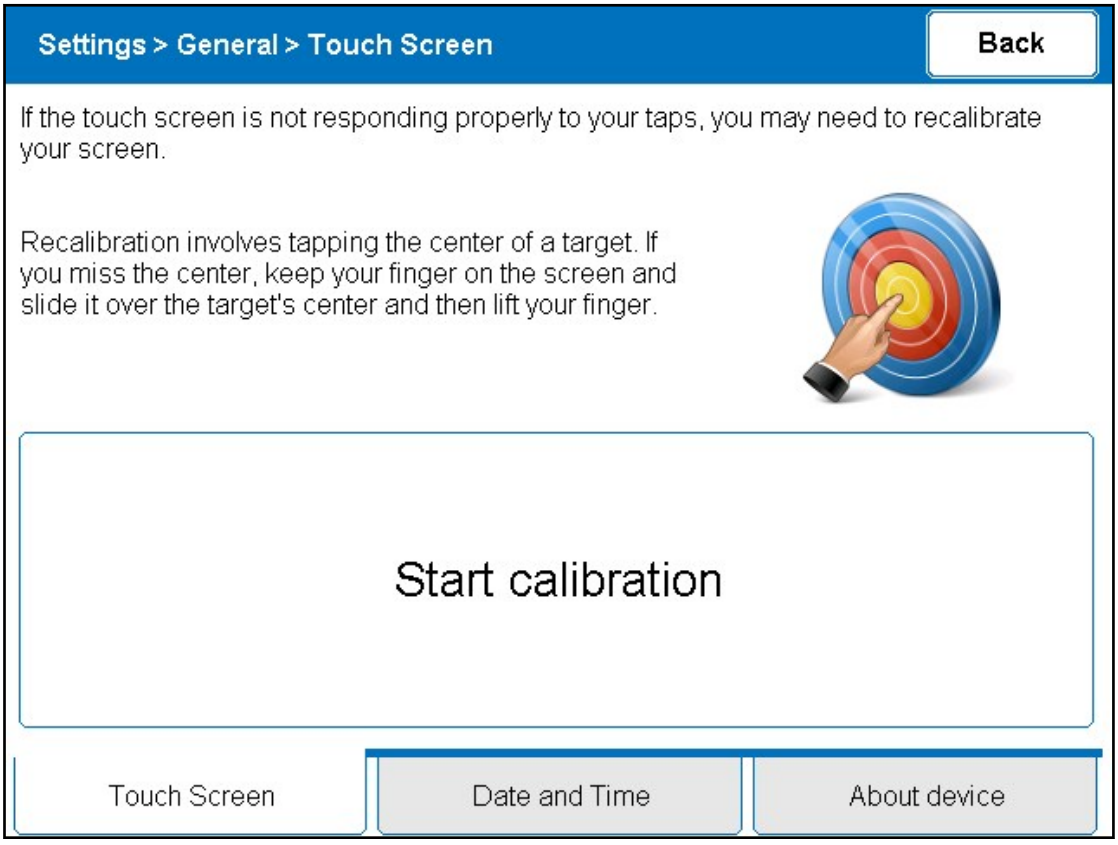


Figure 7.45: Settings: Calibration Touch Screen

Date and Time

To ensure correct documentation, the correct date and time should be set in the Date and Time tab:

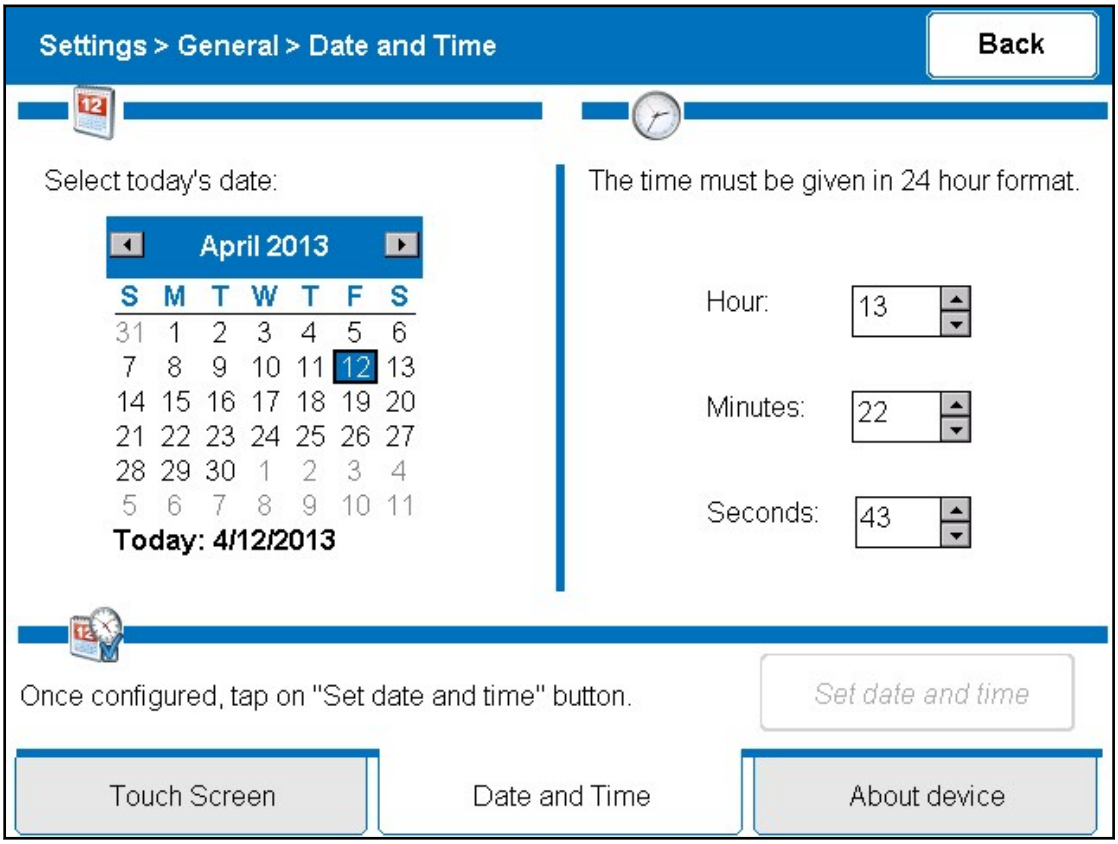


Figure 7.46: Settings: Date and Time

About Device

The About Device tab shows information concerning the device and the network IP address used for data transfer to a connected PC.

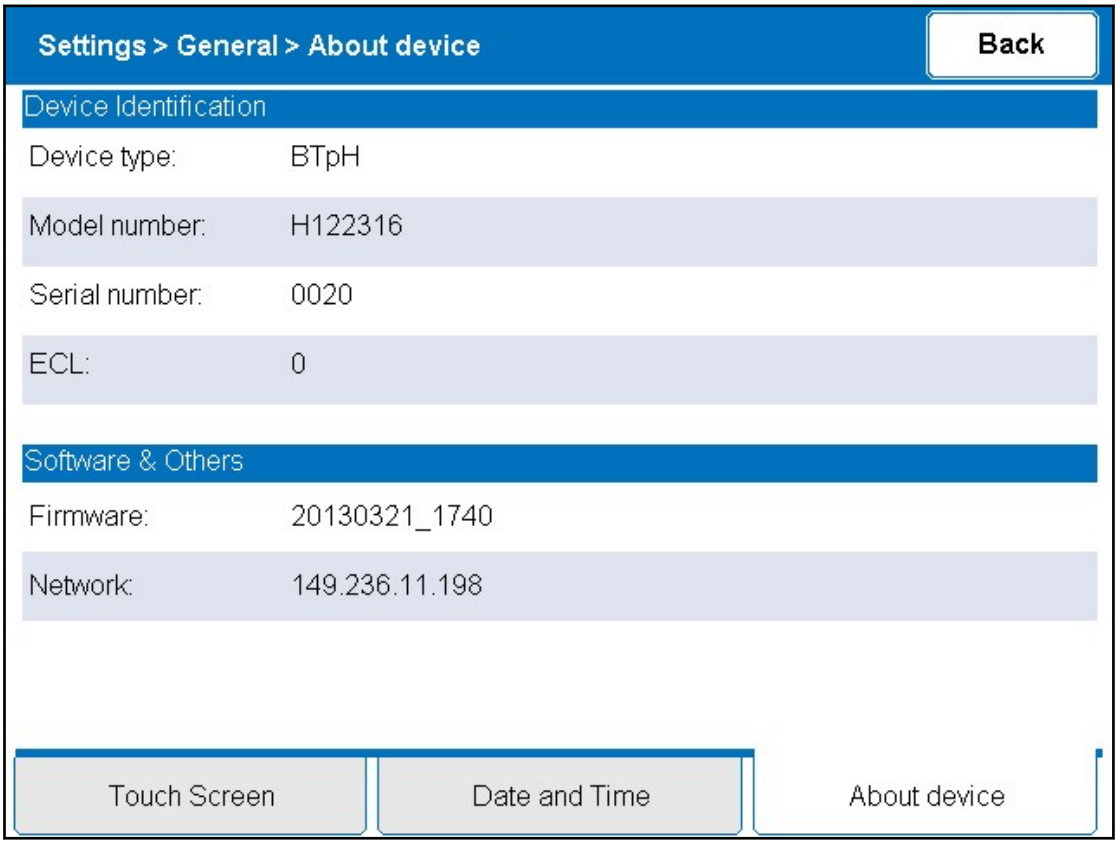


Figure 7.47: Settings: About Device

Service (BRUKER)

The Service area is only for Bruker Service purposes and therefore protected with a password.

Figure 7.48: Settings: Service Area

7.6.2 BTpH Settings

7.6.2.1 Adjustment Settings

The adjustment settings allow you to define the set of values used by the unit while adjusting a sample.

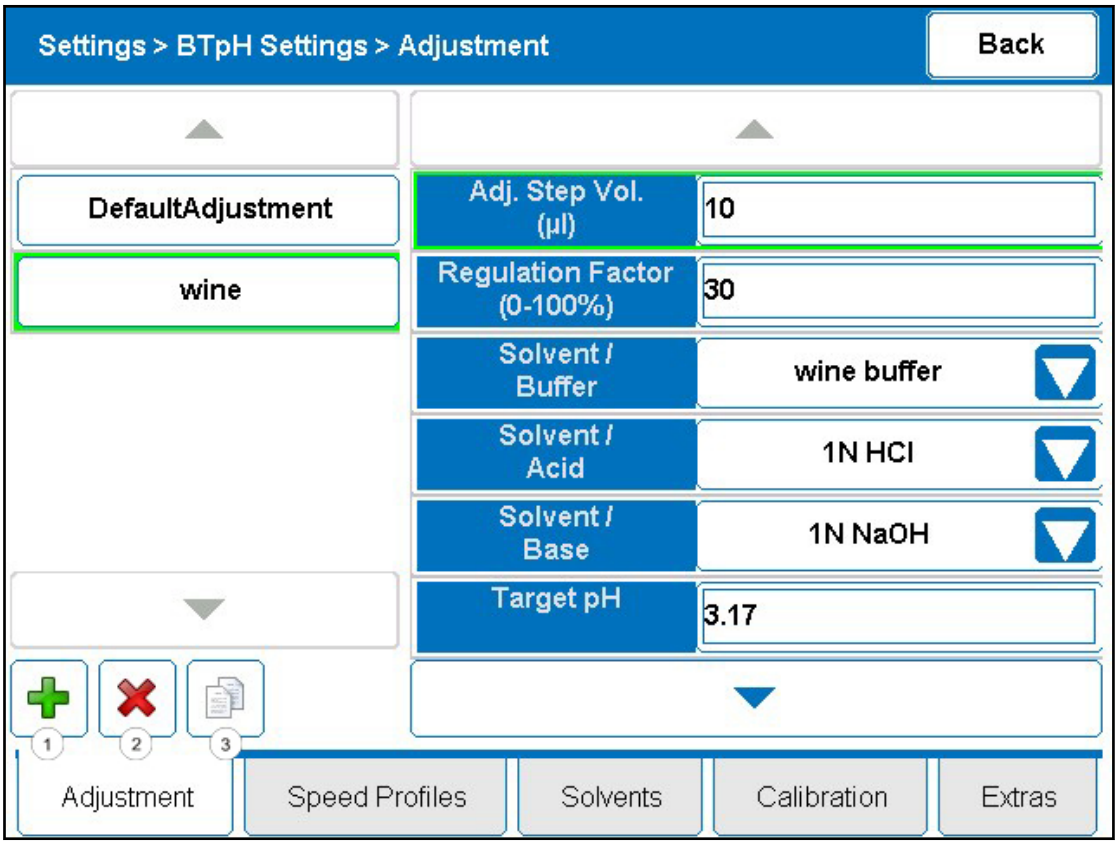


Figure 7.49: Adjustment Settings

1.	Add Button	3.	Clone Button (used to copy and save as a new file)
2.	Delete Button		

You would normally only change these or add a new set when you are preparing a set of samples all of a similar type.

The ‘Default Adjustment’ set is a suitable start for most titrations.

To generate a new set, select a set whose values are broadly similar to what you want (for example, often you want only a new target pH value).

Press the **Clone** button and save this set under a new name: here ‘my samples’.

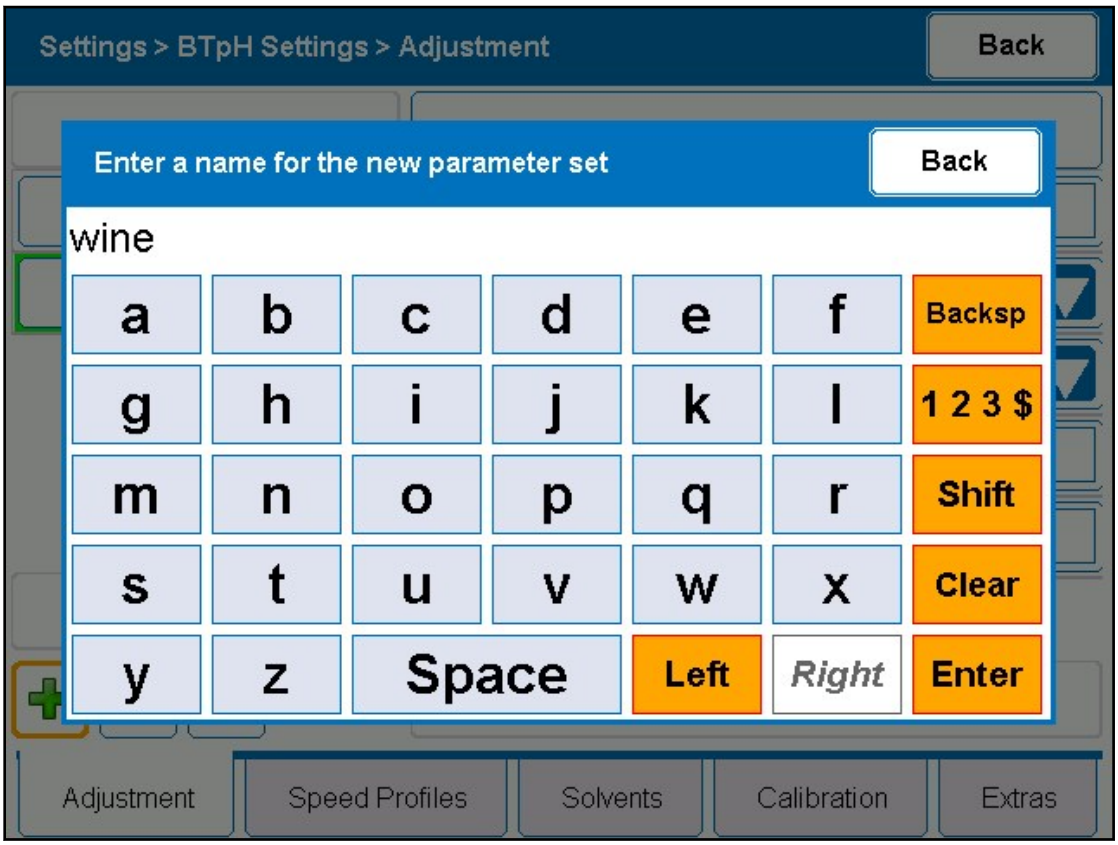


Figure 7.50: New Adjustment Profile

Now you can edit the individual settings for this adjustment set.

Mostly the default values do not need to be changed. Only do so if you fully understand their meaning and how they influence the pH titration operation.

Use the blue up and down arrows to move up and down through the edit options. Press the option value you wish to change. For example, to change the target pH, press on the present target pH (here 3.17) and a numeric (alphanumeric for some other options) keypad is displayed. Type in the new value and press 'Enter' to save it. Press 'Back' if you do not wish to change the value.

Option	Description
Adj. Step Vol.:	This is the maximum volume (in µL) delivered at each step phase while the unit is adding acid or base to the sample. While dosing, if the pH reaches one of the other 'limits', the dose step is halted. In any case, after each dose step, the unit waits a slightly longer time for the pH reading to become stable. Default Value: 10 µL.
Regulation Factor:	This directly influences the regulation operation. A lower value may be faster but is more likely to overshoot. Default Value: 40.
Solvent Buffer:	Allows you to select from the list of buffers you defined in *Define Buffer, Acid and Base [▶ 93]*. Be aware that the buffer you select here MUST also be the buffer that you have defined as in use at present, otherwise you will get an error when you attempt an adjustment.
Solvent Acid:	As for buffer.
Solvent Base:	As for acid and buffer.
Target pH:	Sets the nominal target pH. Default Value: 7 (pH).

Option	Description
pH Adjustment Accuracy:	During a pH adjustment, when the pH is within this set amount of the target pH then the target is deemed to have been reached. Default Value: +/- 0.02 (pH)
Initial Sample Vol. (µL):	Is the volume of sample you start with. It is noted in the sample data but has no influence on the regulation. Default Value: 900 µL
Buffer Cal Factor:	Allows an external Calibration of (only) the dispensed buffer volume. This should normally NOT be changed from the default value. Default Value: 1
Buffer Fixed Vol. (µL):	Defines the fixed volume of buffer which is added to the sample at the start of the adjustment. This is added before the pH regulation starts. Default Value: 100 µL
Acid Fixed Vol. (µL):	Defines the fixed volume of acid which is added to the sample at the start of the adjustment.This is added before the pH regulation starts. Default Value: 0 µL
Base Fixed Vol. (µL):	Defines the fixed volume of base which is added to the sample at the start of the adjustment. This is added before the pH regulation starts. Default Value: 0 µL
Shaker Frequency (RPM):	Defines the shaking frequency during the pH adjustment. Default Value 850 RPM.
pH Fine Range Distance:	During an adjustment operation, when the pH measured is within this amount of the target pH then the regulation switches over to a fine step mode. Default Value: 0.1 (pH)
Fine Range Vol. (µL):	In the fine step mode this volume is dispensed on each operation. Default Value: 0.5 µL
Titration Limit Vol. (µL):	To avoid overfilling the maximum volume which can be titrated to the sample is defined. Once this volume is reached the adjustment operation aborts.
Speed Profile Adjustment:	Defines the Speed profile to be used during the pH adjustment operation (mostly acid/base dispense) as defined in *Speed Profiles - Meaning [91]*.
Speed Profile Operation:	Defines the Speed profile to be used during the pH adjustment operation where no adjustment is being made (filling syringes, dispensing fixed volumes) as defined in *Speed Profiles - Meaning [91]*. The profile used can allow faster dispenses than in the Adj. Profile.
Flush Buffer Vol. (µL):	Defines the flush volume for the buffer capillary after pH adjustment. Default value 10 µL.
Flush Reagent Vol. (µL):	Defines the flush volume for the acid & base capillary after pH adjustment. Default value 5 µL.

Table 7.1: Adjustment Profile Options

7.6.2.2 Speed Profiles - Meaning

The ‘InitWash’ speed profile is always used during the initialise and wash operations. You can change the values here but you cannot allocate any other profile to the initialise and wash operations.

The ‘Adjustment’ profile is nominally used during the adjustment phase of a pH Titration adjustment operation.

The ‘Default’ speed profile is used for any other dispense and aspiration operations.

7.6.2.3 Speed Profiles - Modify

Push the ‘Speed Profiles’ Tab and the following window is displayed.

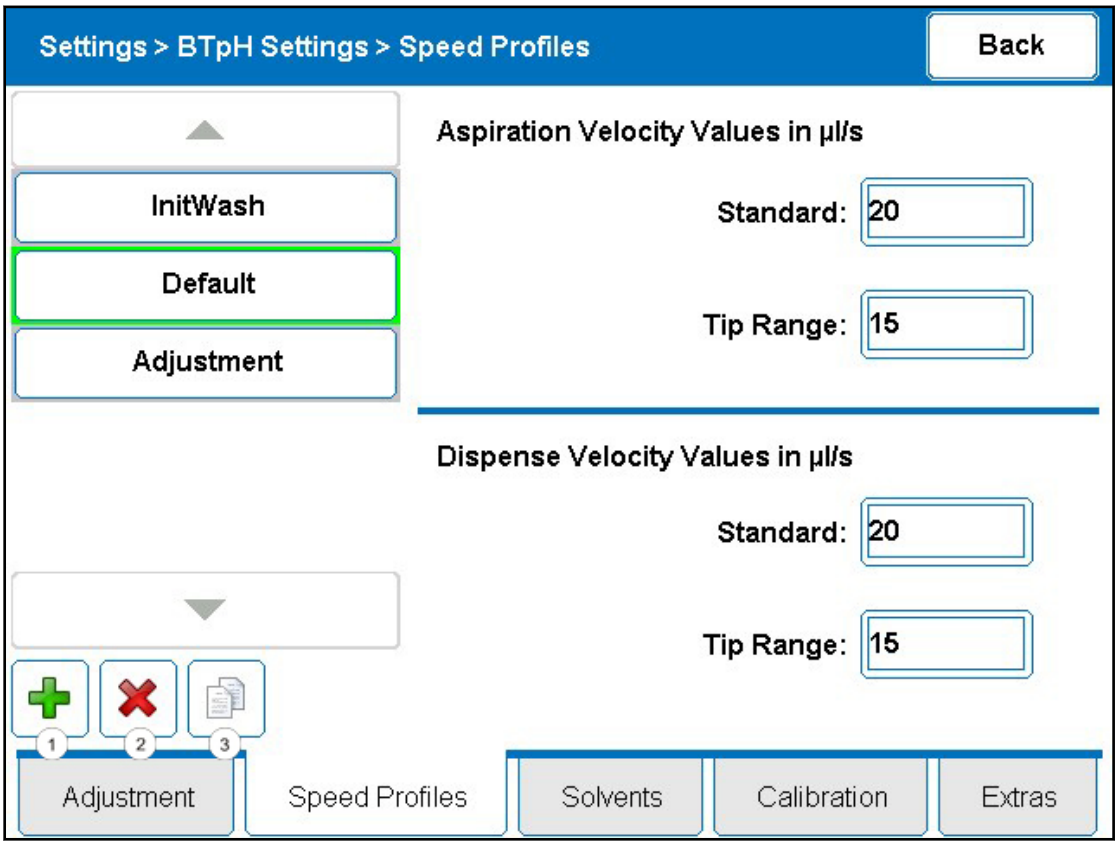


Figure 7.51: Speed Profiles

1.	Add Button	3.	Clone Button (used to copy and save as a new file)
2.	Delete Button		

Here you can set the pump speed profiles for different operations. Three standard profiles are available. You can either use these or add your own.

‘Standard’ and ‘Tip Range’ refer to the position of the syringe plunger. The syringes used are a special type whereby the tip of the plunger protrudes through the end of the syringe and into the body of the valve. This allows the syringe volume to be more completely cleared when dosing. ‘Tip Range’ refers to the upper part of the syringe movement where the tip is moving through the end of the syringe. As the flow is more restricted here the flow rate should be restricted. The values selected are the maximum aspiration (i.e. sucking in) and dispensing flow rate in µL per second.

Use the **Add** or **Clone** buttons to generate a new profile. Enter the new name in the popup keypad then select the profile and edit the values here.

NOTE: Don’t change these values unless you fully understand their meanings. The standard values defined here are suitable for most adjustments.

The speed values defined apply to all 3 pumps.

You cannot define speed profiles on a single pump basis.

7.6.2.4 Define Buffer, Acid and Base

In the Settings page press the '**Solvents**' Button. Here you define and select the name of the Buffer, Acid and Base you are using for titrations.

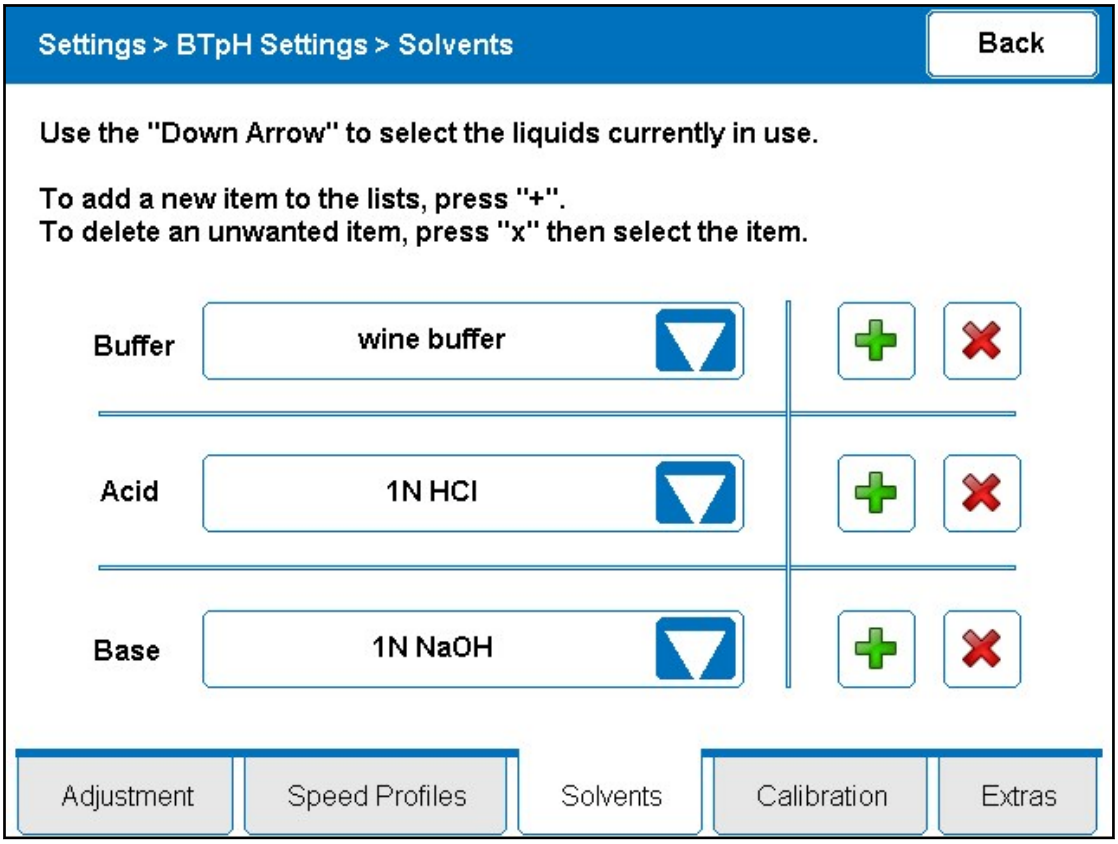


Figure 7.52: Setup: Liquids in Use

The Buffer, Acid and Base listed here are those defined to be in use by the system at present. Their main purpose is to appear in the sample list so that you know what buffer, acid and base were used for any particular pH titration.

Press the arrow to the right of 'Selected Buffer to obtain a list of available names. Press on the name you wish to use to select it. If the liquid you are actually using is not listed here you will have to define it yourself.

Press the green **+** button to the right of 'Buffer'.

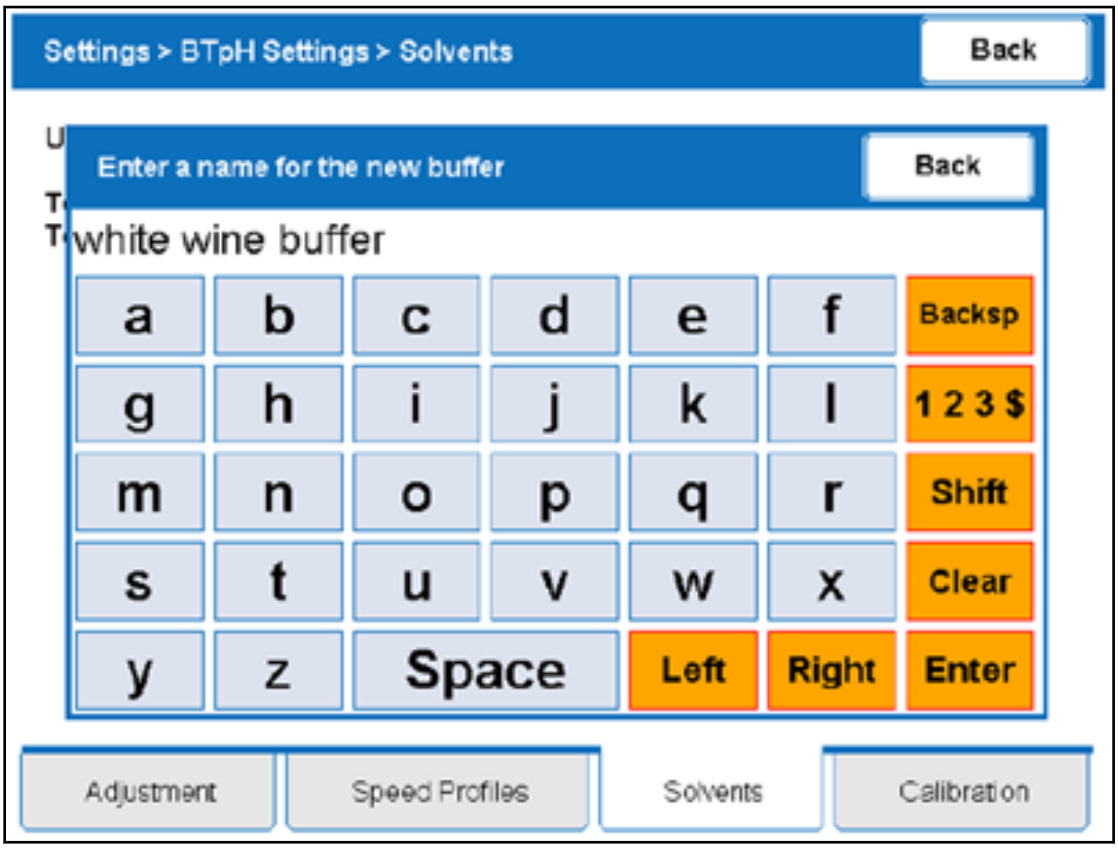


Figure 7.53: Define Name of Buffer in Use

In the keypad which pops up type in the name of your new buffer and press enter.

You can now select this buffer.

NOTE: This procedure tells the system that the operator has filled the 'Buffer' flask with a buffer called in this case 'white wine buffer'.

The names the operator gives here are simply arbitrary names defined to identify the buffer (acid, base) actually used for titrations.

In the adjustment settings, see *Define Buffer, Acid and Base [▶ 93]*, you must define the same buffer as here otherwise when you try to start an adjustment an error will be flagged that the buffer (acid, base) selected for the adjustment is not available.

Repeat this process for the acid and the base.

See also

Adjustment Settings [▶ 89]

7.6.2.5 Calibration Settings

This displays the calibration options (below).

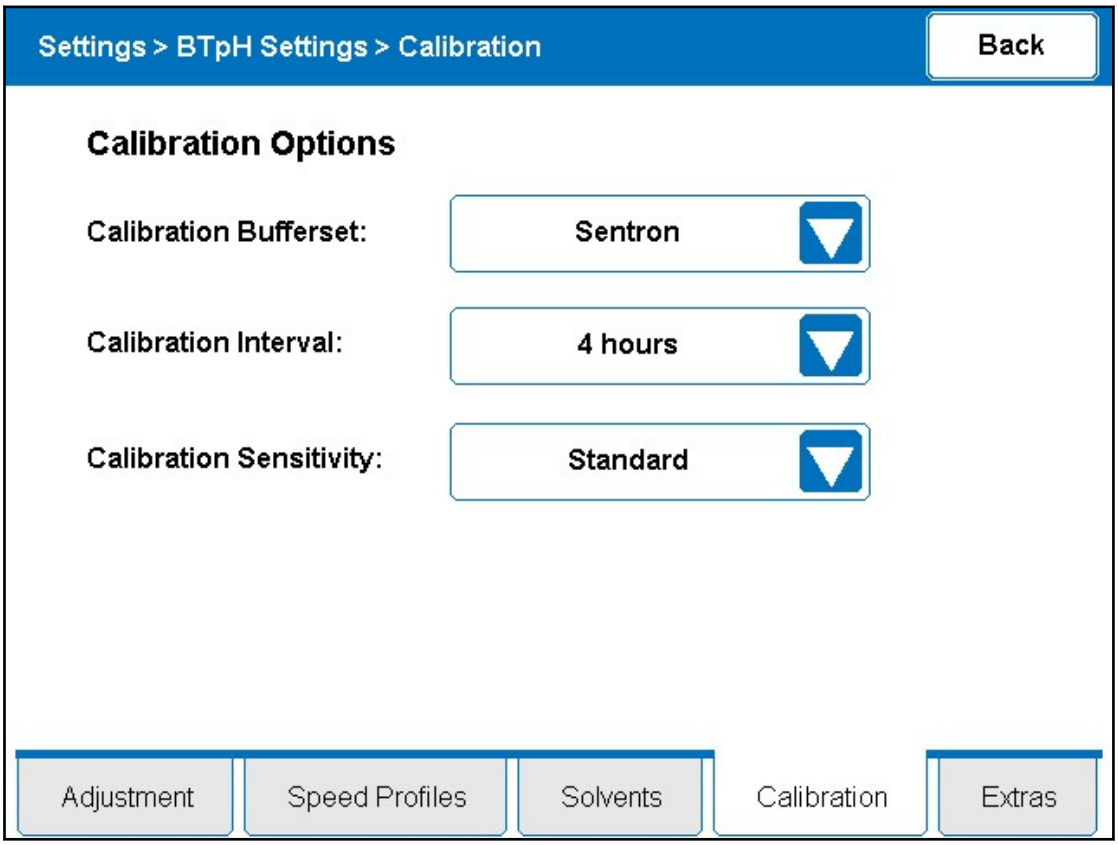


Figure 7.54: Calibration Settings

Press the selection arrow to the right of the selected buffer set. The popup list displays the standard calibration buffers supported by the unit. Select the calibration buffer set you wish to use. This determines which pH calibration values are listed in the calibration page.

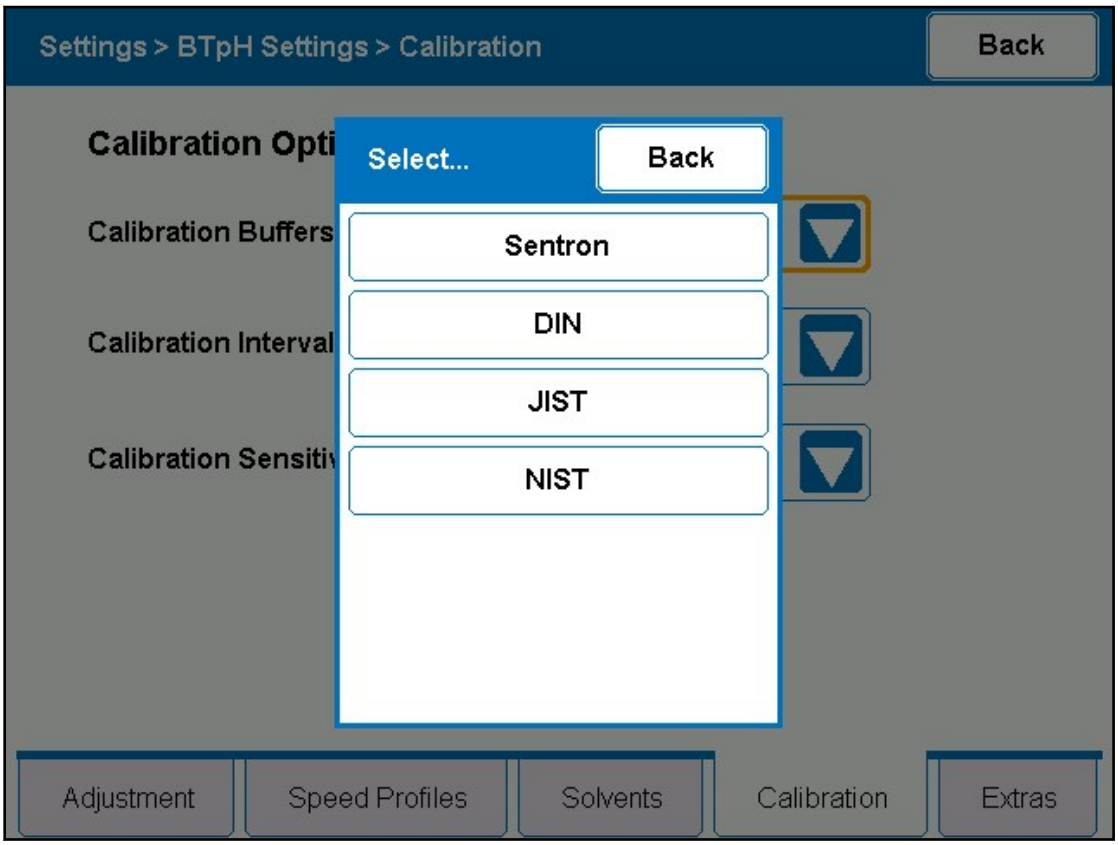


Figure 7.55: Calibration Settings: Select Calibration Buffer

The calibration interval defines how long after a calibration before the unit starts to display calibration reminder messages.

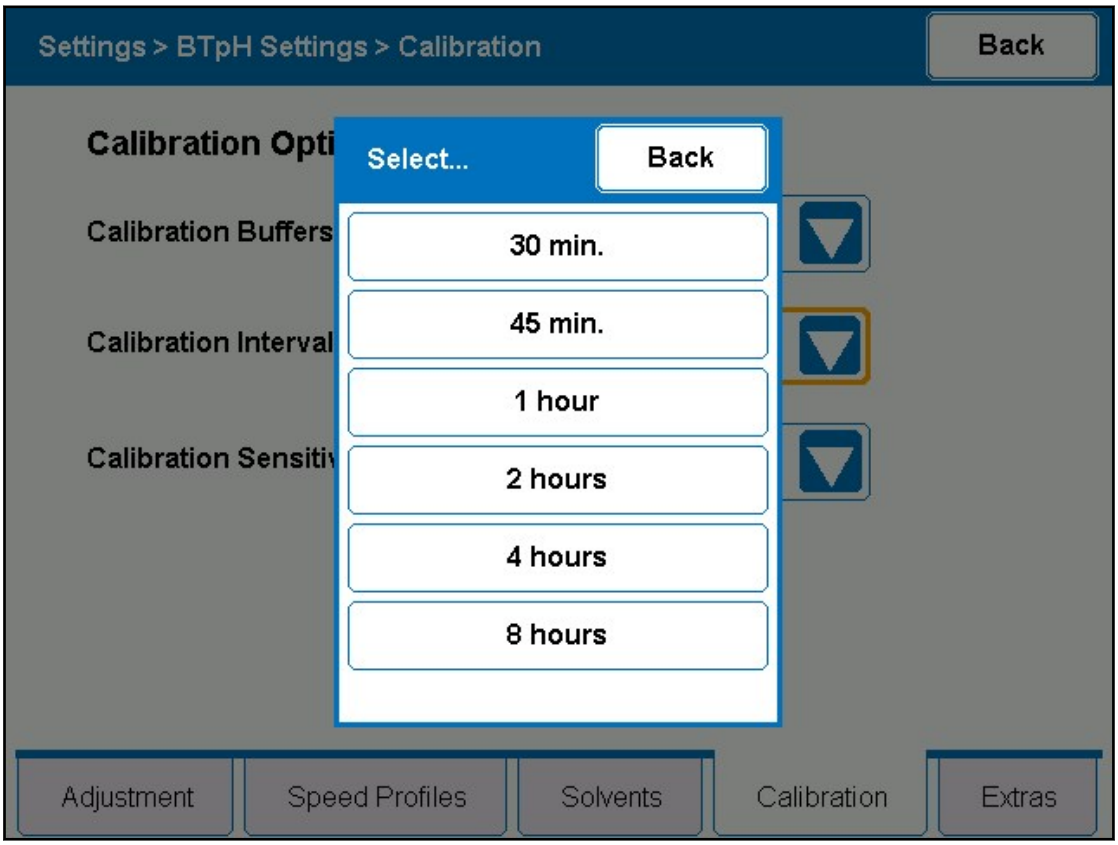


Figure 7.56: Calibration Settings: Select Calibration Interval

The calibration sensitivity can be set to ‘Fine’, ‘Standard’ or ‘Course’.

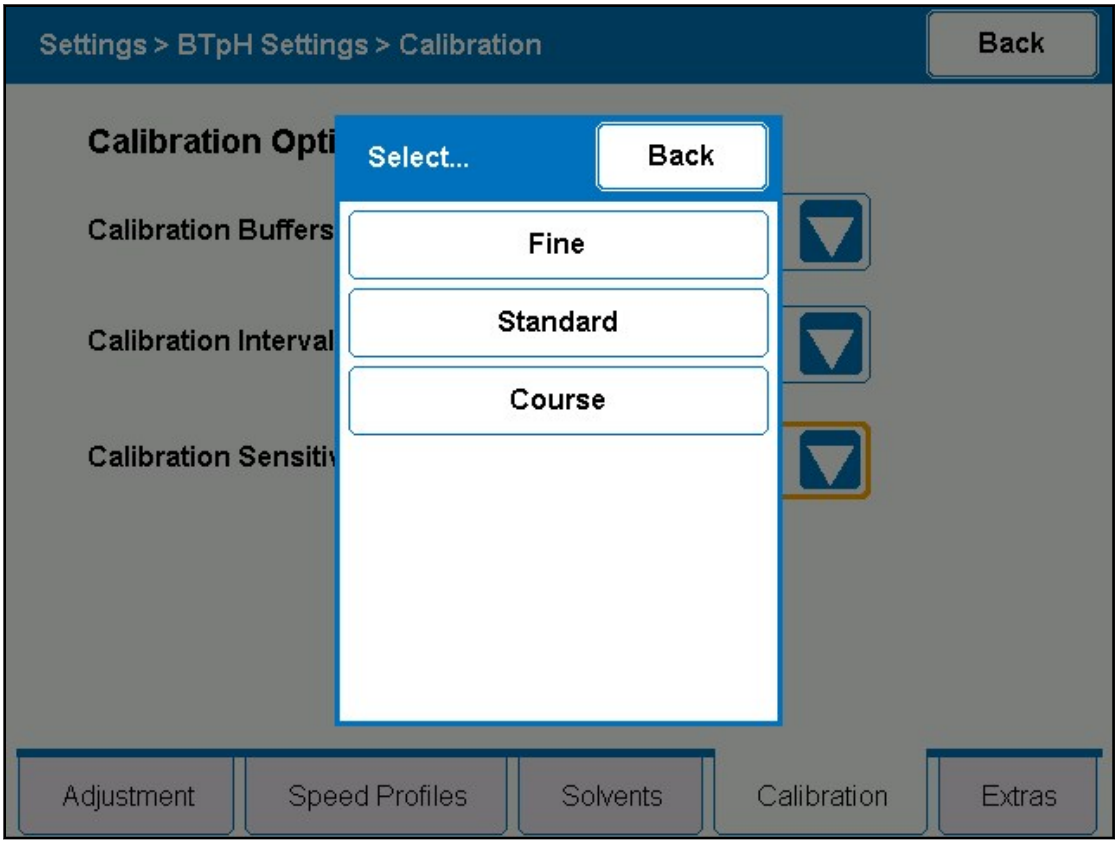


Figure 7.57: Calibration Settings: Select Calibration Sensitivity

This defines the acceptable level of drift and noise in the pH voltage signal. Some sensors and/or environments are inherently very noisy. If you are having difficulty in calibrating the sensor you can select ‘Course’ here. Conversely you may wish to do some especially precise measurements so, if conditions permit, you can select ‘Fine’ here. The default value ‘Standard’ is usually quite suitable.

7.6.2.6 Extras

Shaker Settings

Regenerating Shaking Frequency (RPM): Default value for regenerating frequency of BioShake 3000 BBIO.

Note: The mixing frequency for titration will be adjusted in the parameter set under ‘Settings – Adjustment’

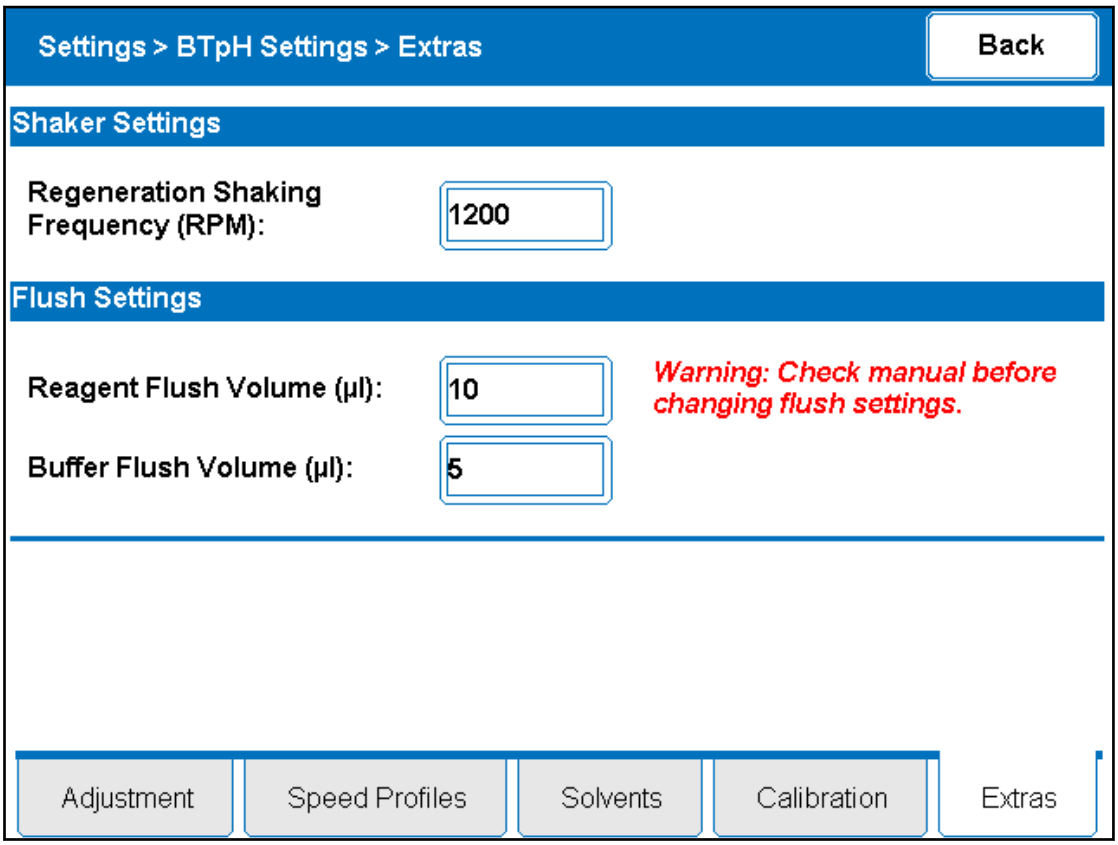


Figure 7.58: BTpH Settings Extras Window

Flush Settings

Reagent Flush Volume (µl): Used to set the default volume of the “Flush” for the base and acid capillaries to clean the tip capillaries between sample changes and after the calibration routine.

Buffer Flush Volume (µl): Used to set the default volume of the “Flush” for the buffer capillary to clean the tip capillaries between sample changes and after the calibration routine.

Note: Due to the strong salt gradient between the high concentrated buffer solution and the sample and calibration solution there might be an osmoses effect which leads to a liquid penetration into the capillaries. To avoid a carryover it’s recommended to flush the pH-probe tip capillaries always between sample changes.

8 General Maintenance

8.1 Cleaning the Main Unit and Touch Display

Disconnect the mains supply cable before cleaning the unit. Use a clean damp cloth to wipe of any residue from the outside of the unit. Wipe of excess moisture immediately with a dry cloth.

For non water-soluble marks, wipe using a cloth with a few drops of isopropyl alcohol.

Remove all cables to the touch display before cleaning. This can also be cleaned by wiping with a damp (not wet) cloth or with isopropyl alcohol.

Only reconnect the cables to the units once they have been wiped dry.

8.2 Replacing the Battery in the Touch Display

The touch display controller has a battery to back up the power to the real time clock while the display is not connected to an external power source. This should normally last many years, however, should the time function no longer operate then you will have to replace the battery (a Lithium 3V Button Cell type CR2032).

1. Remove all the cables to the unit.

⚠ WARNING

Electrical hazard from electrical shock.

A life threatening shock may result from contact with the high voltage to the touch panel backlight.

1. Always remove the cables to the touch panel before removing the rear panel.

2. Remove the 4 securing screws and carefully remove the base of the touch panel.

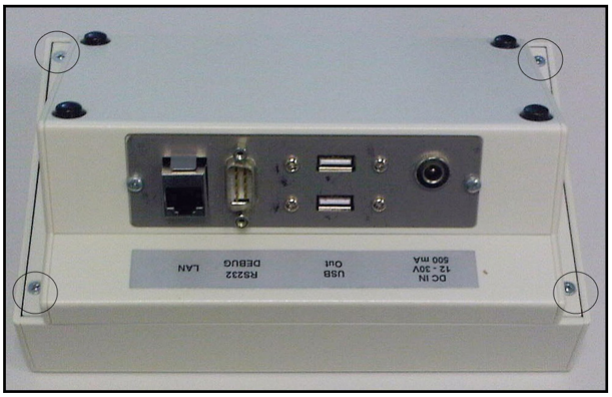


Figure 8.1: Touch Panel Base

➨ The cables are still attached.

3. Put the base panel to one side.
4. Remove the old battery using a flat screwdriver to lever it up and out (refer to the following figure).

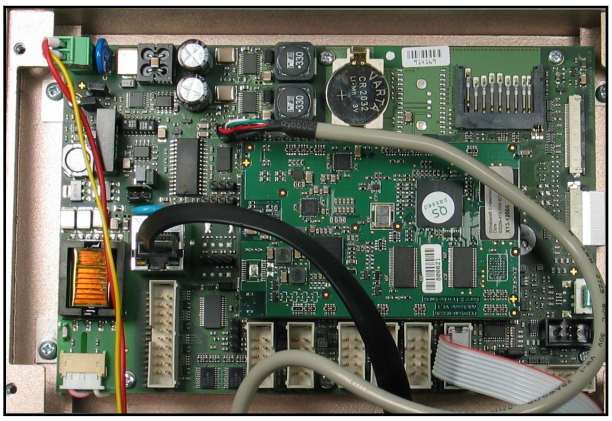


Figure 8.2: Location of the Battery on the Touch Display

5. Replace the battery ONLY with a battery of the same type.
6. Replace the base panel and the 4 securing screws.

The next time you power up the unit you will have to reset the time and date. See *General Settings [85]*for instructions on how to do this.

8.3 pH Sensor

The pH probe must be cleaned regularly to prevent the diaphragm of the reference from getting clogged and blocking the electrical contact to the internal electrode. No, or poor contact, causes the probe to work incorrectly.

Figure 8.3: The pH Probe Tip

The frequency with which you should clean the probe depends on how often you use the probe and the type of samples being measured.

If you use colored reference pH solutions the diaphragm (white spot in the figure above) can become slightly discolored. This has no appreciable influence on the function of the sensor. When the diaphragm is new and clean it should be white.

After each calibration the ‘slope’ of the sensor is displayed. Ideally this should be 100, but when the value is less than 93 or greater than 108 you should perform at least a regeneration. If this does not help, then you should clean the probe as described in the following chapter.

8.3.1 Conditioning the pH Probe

Conditioning the pH probe consists of placing the probe (tip) in either a neutral pH7 salt solution, or in a 3M KCl solution for about 15 minutes. This helps to restore the electrolyte conduction through the reference diaphragm.

After conditioning the probe, remove the probe from the solution and rinse/dry it thoroughly.

8.3.2 Cleaning pH Titration Probes

Clean the ISFET probe regularly to avoid malfunction. If the slope is < 93%, or if the drift has increased such that calibration of the probe takes a very long time in the standard calibration mode, an extensive cleaning is recommended.

Over periods of >3 days the probe has to be stored dry with wetted protection cap. However, its lifetime can be extended by regularly soaking it in a 3M KCl solution. If this is not readily available a pH7 solution can be used.

Soapy Water Method for Cleaning the Probe

1. Put the probe in hot (50 °C) soapy water for at least 5 minutes.
2. Try to keep the water temperature at 50 °C.
3. Remove the probe from the soap solution an place it immediately into a 3M KCl solution at 20 °C (room temperature) for about 30 minutes.
4. You can then clean the tip (the reference diaphragm and the ISFET sensor) with a soft WET brush (provided with the pH probe).

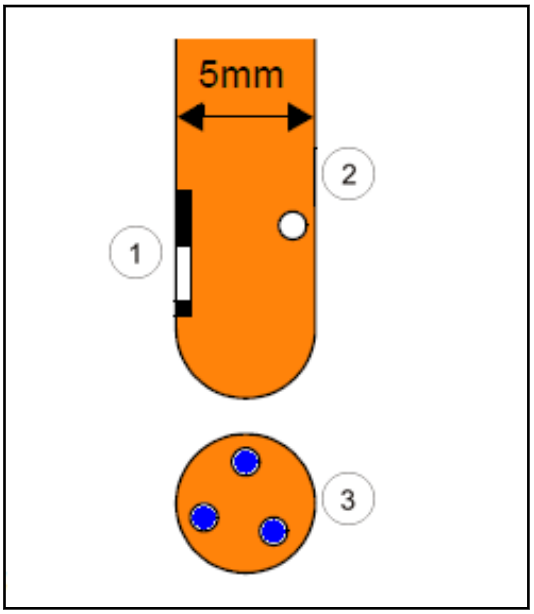


Figure 8.4: The ISFET Sensor

1.	ISFET Sensor	3.	Capillary Outlets
2.	ISFET Reference Diaphragm		

NOTE: Always soak the probe in the hot soapy water prior to cleaning it with a brush. Failing to do this may result in the surface of the ISFET or the epoxy being scratched due to hard crystal deposits left on the probe tip. Such damage can also cause incorrect readings.

8.3.3 Regenerating the pH Probe

This consists simply of placing the probe (tip) in a (warmed to 40 °C if possible) pH10 salt solution overnight followed by placing it in a (again warmed to 40 °C if possible) pH2 salt solution for several hours. This helps to promote the electrolyte conduction through the reference diaphragm.

NOTE: The pH sensor is a consumable and has a limited life. Under proper use and careful maintenance a pH probe will be good for at least for 4000 pH titration actions or respective 6 months of use. The life time will be reduced if crude samples or highly concentrated solutions are used.

8.4 Syringes

Two syringe sizes are used in the BTpH:

- 500 µL for the buffer solution (pump 1 - yellow).
- 100 µL for the base and acid solutions (pumps 2 and 3 - blue and red).

Figure 8.5: Syringes

The syringes are consumables and wear out with use. Replacements can be ordered from Bruker, see *Spare Parts [▶ 28]*.

8.4.1 Replacing a Syringe

Run a wash operation on the pump before replacing a syringe.

1. Power off the unit.
2. Turn the adjustment knob to move the syringe piston and mounting screw down about 10 - 15 mm from its top position.
3. Unscrew and remove the syringe mounting screw.
4. Slightly loosen (do not remove) the nylon lock screw.
5. Unscrew the syringe from the valve port.
6. Completely remove the syringe.

7. Carefully screw the new syringe into the valve port (it should be firmly finger tight - do not use tools!).
8. Lightly tighten the lock screw.
9. Move the piston end to the correct position and screw in the mounting screw.

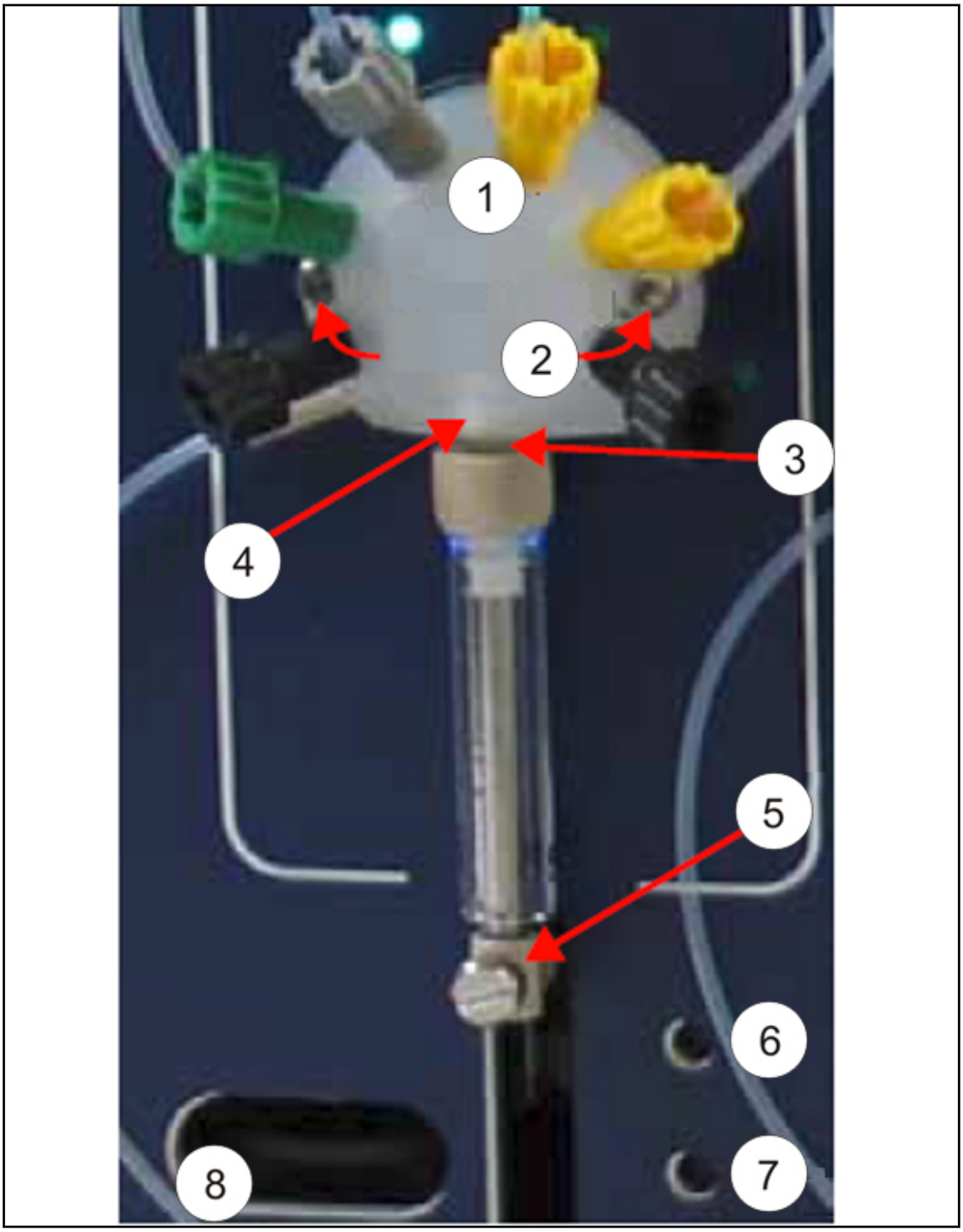


Figure 8.6: Syringe Replacement

1.	Valve	5.	Syringe Mounting Screw
2.	Mounting Screws	6.	Home Button
3.	Syringe Port in Valve	7.	Init Button

4.	Lock Screw	8.	Adjustment Knob

8.4.2 Resetting the Pump Home Position

After replacing a syringe the pump home position must be reset.

Note: These steps must be done whenever you change (or remove and reattach) a syringe. This should ONLY be done under these circumstances, it does not need to be done on a regular basis.

1. Remove the DC power cable to the touch controller (see *Connecting the Unit [▸ 32]*).
2. Power on the BTpH.
   - ➨ After a few seconds you can hear the valve at the top of each pump being initialized (valve set to waste position).
3. Use a pointed object (e.g. pencil, pen) to press the Init button beside the syringe that was replaced.
   - ➨ This causes the pump drive to move up or down until its internal initialize position is found (a light barrier near the top of the pump drive movement is detected).
4. Turn the adjustment knob slowly to the left to move the piston up until the mechanical end stop is reached. As you move the knob you will see that the syringe slowly moves upward. The resistance at the knob will noticeably increase as the end stop is reached.
5. Move the syringe a short distance back down from the end stop by turning the knob to the right.
6. Use a pointed object (e.g. pencil, pen) to press the Home button beside the syringe that was replaced.
   - ➨ This will cause the pump drive to move back down to the initialise position, then back up to the position you previously moved it to. This position is then saved internally as the ‘home’ position.
7. Replace the DC power cable.
   - ➨ The touch controller should now power up.
8. Once the touch controller has reached the startup page, select the Init/Wash function and start an Init operation for the pump where you have just changed the syringe. Alternatively if you are not intending to use the unit for a while, run a ‘wash’ operation (see *Initialize the Unit [▸ 42]*).
9. While the initialization or wash operation is running, examine the fluid flow into and out of the syringe.
   - ➨ By the time the init/wash is finished there should be no sign of any gas bubbles at the top of the syringe. If this is not the case, then ensure that the syringe is correctly and fully screwed into the valve port, then repeat the init or wash procedure.

8.4.3 Refilling the Flasks

Refer to *Filling the Storage Flasks [33]*

⚠ CAUTION

Spillage Risk When Refilling/Replacing Liquid Storage Flasks

The storage flasks contain aggressive liquids (acid/base).

▶ These flasks should be refilled/replaced as described in this manual.

1. Always wear appropriate eye protection, gloves and protective clothing.
2. Ensure that suitable safety procedures for the handling, disposal etc of these materials are in place in your laboratory environment.

8.5 Selection Valves

At the top of each pump is a 6 way selection valve. The valve serves to connect the syringe port with one of the 6 ports. The ports are labelled clockwise from left to right:

E F A B C D (see *Connecting the Unit [32]*)

At present only 4 ports are used, the ports E and D are blocked. The ports are connected as follows (clockwise left to right):

E	Not used (plug)	black fitting
F	From the water supply in the flask	green fitting
A	Out to the waste flask	gray fitting
B	From supply liquid in flask (buffer, base or acid)	yellow, blue or red fitting
C	Out to pH probe (buffer, base or acid)	yellow, blue or red fitting
E	Not used (plug)	black fitting

When the unit is turned on, the pump controller moves the valve to the initialize position ‘A’, so the syringe is connected to the waste output.

The valves are consumables and wear out with use, replacements can be ordered from Bruker. See *Spare Parts [28]*.

8.5.1 Replacing a Selection Valve

Run a wash operation on the pump before replacing the valve.

1. Turn off the unit.
2. Turn the unit on and after a few seconds, turn it back off again.
   ➨ This ensures that the valves are all set to their initialise position (Port A).
3. Remove the syringe from the valve you wish to replace (see *Replacing a Syringe [104]*).

4. If not already done, set the valve to its initialize position by rotating the connection axis on the rear of the valve with a pair of pliers so that the marking faces upwards as shown in figure below. This is the default factory position so normally this is already correct.

Figure 8.7: Valve Orientation

5. With the fittings to the old valve still in place, remove the valve mounting screws (use a 3/32“ Allen hex key to loosen 1 screw slightly, then the other before removing both screws completely).
6. Pull the old valve out of its mounting and set it slightly to one side.
   - ➨ Leave the fittings and tubing connected.
7. Mount the new valve in the vacant valve mounting, ensuring that the alignment pin fits correctly into the rear of the valve, and that the valve axis goes correctly into the drive axis at the pump.
   - ➨ The marking visible at the top of the valve drive axis should be visible when the valve is at its initialize position.
   - ➨ If this is not the case turn the valve drive axis using a screwdriver until the marking faces upwards. This is normally not required as power running the unit (above) causes the valves to be set to this position.
8. Screw in the mounting screws firmly but do not overtighten. The valve is made of plastic (Kel-F) and can be warped slightly if the screws are too tight.
9. Remove the fittings one at a time from the old valve and connect them immediately to the new valve. Very small amounts of liquid may drop out the end of the fittings.
10. Re-attach the syringe as described in *Replacing a Syringe [▷ 104]*.
11. Reset the home position as described in *Resetting the Pump Home Position [▷ 107]*.

8.6 Standard Operating Procedures for Wine Applications

8.6.1 pH Probe - Treatment in Wine Application

In wine-applications 3M KCl Special (BBIO # H140256) has to be used for storage and cleaning treatment of BTpH titration probes.

Prior to Starting a Titration Series:

Regeneration/Conditioning:

1. Place the probe for 15 minutes in a newly filled “3M KCl Special“ solution at a shake frequency of 1200 *RPM*.
2. Rinse the pH probe with demineralized H2O.
3. Place the probe for 15 minutes in a newly filled wine-reference solution at a shake frequency of 850 RPM.
4. Rinse the pH probe with demineralized H2O.
5. Initialize unit with BTpH “INIT” routine.

** For the case the probe was stored overnight in “3M KCl Special” this step will be obsolete.

Calibration:

Run a two point calibration at pH4.0 & pH2.0 without shaking.

Storage for breaks < 15 minutes during a titration series

Place the probe in a wine-reference solution at a shake frequency of 850 RPM.

Storage at breaks >15 minutes during a titration series

1. Place the probe for 15 minutes in a newly filled “3M KCl Special“ solution at a shake frequency of 1200 RPM.
2. Leave the probe in the “3M KCl Special “ solution.

Overnight storage:

1. Place the probe for 15 minutes in a newly filled “3M KCl Special“ solution at a shake frequency of 1200 RPM.
2. Store the probe overnight in a “3M KCl Special” solution, no shaking.

Weekend/holiday storage

1. Place the probe for 15 minutes in a newly filled “3M KCl Special“ solution at a shake frequency of 1200 RPM.
2. Rinse the pH probe with demineralized H2O.
3. Wash the pH probe using the BTpH “Wash” routine.
4. Fill the protection cap with a few droplets of demineralized H2O.
5. Place the wetted protection cap over the tip of the pH probe.

Cleaning pH probe (when the Slope < 93%)

1. Place the probe for 5 minutes in a 0,1% Pepsin/HCl solution at a shake frequency of 1200RPM.
2. Rinse the pH probe with demineralized H2O.
3. Place the probe for 5 minutes in a solution of warm (ca. 35°C) soapy water.
4. Rinse the pH probe with demineralized H2O.
5. Clean the chip at the tip of the pH probe with a soft brush.
6. Rinse the pH probe with demineralized H2O.
7. Place the probe in a “3M KCl Special“ solution, in a warm (ca. 35°C) water bath.
8. Let it cool down to ca. 25°C.
9. Wash the pH probe using the BTpH “Wash” routine.
10. Fill the protection cap with a few droplets of demineralized H2O.
11. Place the wetted protection cap over the tip of the pH probe.

9 Dismantling and Disposal

Following the end of its operational life, the device must be dismantled and either returned to Bruker, or disposed of in accordance with local environmental regulations.

9.1 Safety

⚠ WARNING

Danger of injury from electrical shock!

A life threatening shock may result when the housing is open during operation.

1. Only qualified personnel should open the housing.
2. Disconnect the device from the electrical power supply before opening the device. Use a voltmeter to verify that the device is not under power!
3. Be sure that the power supply cannot be reconnected without notice.

⚠ WARNING

Danger of injury due to improper dismantling!

Stored residual energy, angular components, points and edges on and in the device or on the tools needed can cause injuries.

1. Ensure sufficient space before starting work.
2. Handle exposed, sharp-edged components with care.
3. Dismantle the components properly.
4. Secure components so that they cannot fall down or topple over.
5. Consult the manufacturer if in doubt.

9.2 Dismantling

Before starting dismantling:

1. Shut down the device and secure to prevent restarting.
2. Physically disconnect the power supply from the device; discharge stored residual energy.
3. Remove consumables, auxiliary materials and other processing materials and dispose of in accordance with the environmental regulations.
4. Clean assemblies and parts properly and dismantle in compliance with applicable local occupational safety and environmental protection regulations.

9.3 Disposal

If returning the unit to Bruker follow the instructions found in *Returning the Unit to Bruker [114]*.

If no return or disposal agreement has been made, send the dismantled components for recycling.

- Scrap metals.
- Send plastic elements for recycling.
- Sort and dispose of other components in accordance with their material composition.

NOTICE

Danger to the environment from incorrect handling of pollutants!

Incorrect handling of pollutants, particularly incorrect waste disposal, may cause serious damage to the environment.

1. Always observe local environmental regulations regarding handling and disposal of pollutants.
2. Take the appropriate actions immediately if pollutants escape accidentally into the environment. If in doubt, inform the responsible municipal authorities about the damage and ask about the appropriate actions to be taken.

9.3.1 Returning the Unit to Bruker

If you do have to return the unit to Bruker for any reason ensure that:

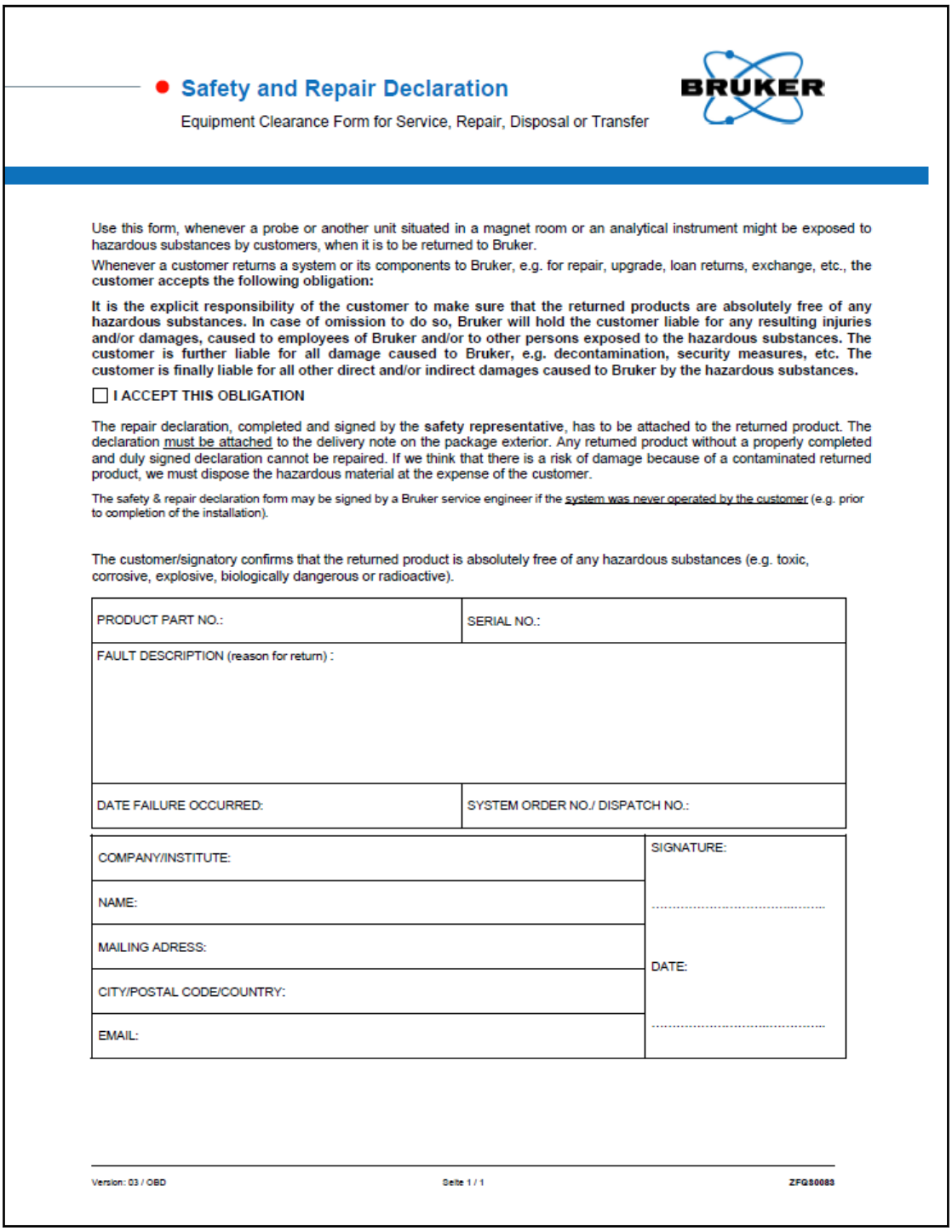
1. All fluid paths have been washed with distilled water.
2. All storage flasks are empty
3. The unit is well packed such that it is unlikely to be damaged in transport. The original packing is best suited for this.

NOTICE

Hazard from contamination.

Before returning a unit to Bruker the Customer is required to confirm in writing that the unit has been thoroughly cleaned and all sources of contamination removed: This is particularly important in respect to bio- and radioactive contaminants.

4. A copy of the following Safety and Repair Declaration must be filled out and returned with the unit. Please contact Bruker for information on obtaining this form:



Safety and Repair Declaration

Equipment Clearance Form for Service, Repair, Disposal or Transfer

BRUKER

Use this form, whenever a probe or another unit situated in a magnet room or an analytical instrument might be exposed to hazardous substances by customers, when it is to be returned to Bruker.

Whenever a customer returns a system or its components to Bruker, e.g. for repair, upgrade, loan returns, exchange, etc., **the customer accepts the following obligation:**

It is the explicit responsibility of the customer to make sure that the returned products are absolutely free of any hazardous substances. In case of omission to do so, Bruker will hold the customer liable for any resulting injuries and/or damages, caused to employees of Bruker and/or to other persons exposed to the hazardous substances. The customer is further liable for all damage caused to Bruker, e.g. decontamination, security measures, etc. The customer is finally liable for all other direct and/or indirect damages caused to Bruker by the hazardous substances.

☐ **I ACCEPT THIS OBLIGATION**

The repair declaration, completed and signed by the **safety representative**, has to be attached to the returned product. The declaration must be attached to the delivery note on the package exterior. Any returned product without a properly completed and duly signed declaration cannot be repaired. If we think that there is a risk of damage because of a contaminated returned product, we must dispose the hazardous material at the expense of the customer.

The safety & repair declaration form may be signed by a Bruker service engineer if the system was never operated by the customer (e.g. prior to completion of the installation).

The customer/signatory confirms that the returned product is absolutely free of any hazardous substances (e.g. toxic, corrosive, explosive, biologically dangerous or radioactive).

PRODUCT PART NO.:	SERIAL NO.:
FAULT DESCRIPTION (reason for return) :	
DATE FAILURE OCCURRED:	SYSTEM ORDER NO./ DISPATCH NO.:

COMPANY/INSTITUTE:	SIGNATURE:
NAME:	.....
MAILING ADRESS:	DATE:
CITY/POSTAL CODE/COUNTRY:	
EMAIL:	.....

Version: 03 / OBD Seite 1 / 1 ZFQ80083

Figure 9.1: Safety and Repair Declaration

9.3.2 Disposal of Packaging Material

Keep the original container and packing assembly, at least as long the warranty is valid, in case the BTpH unit has to be returned to the factory. When the packaging material is no longer needed dispose of in accordance with the relevant applicable legal requirements and local regulations.

Dismantling and Disposal

10 Contact

Manufacturer:

Bruker BioSpin NMR
Silberstreifen
D-76287 Rheinstetten
Germany
Phone: +49 721-5161-6155
http://www.bruker-biospin.com

NMR Hotlines

Contact our NMR service centers.

Bruker BioSpin NMR provide dedicated hotlines and service centers, so that our specialists can respond as quickly as possible to all your service requests, applications questions, software or technical needs.

Please select the NMR service center or hotline you wish to contact from our list available at:

http://www.bruker.com/service/information-communication/helpdesk.html

Contact

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Glossary

AC

Alternating Current

BBIO

Bruker BioSpin

BTpH

Bruker endpoint Titration and pH Unit

CSV

Comma Separated Value

DC

Direct Current

DHCP

Dynamic Host Configuration Protocol

DIN

"Deutsches Institut für Normung", which means "German Institute for Standardization".

EEPROM

Electrically Erasable Programmable Read-Only Memory

ESD

Electrostatic Discharge

FEP

Fluorinated Ethylene Propylene

freeware

Software that is available for use at no cost or for a nominal usually voluntary fee.

HCl

Hydrochloride

hh.mm.ss

Hours.Minutes.Seconds

IP address

Internet Protocol address

ISFET

Ion Selective Field Effective Transistor

JIST

Japanese Institute of Standards and Technology

KCL

Potassium Chloride

Kel-F

Poly-Chlor-Trifluor-Ethylen

LED

Light Emitting Diode

N2

Nitrogen Gas

NaN3

Sodium Azide

NaOH

Sodium Hydroxide

NIST

National Institute of Standards and Technology

NMR

Nuclear Magnetic Resonance

PE

Polyethylene

Glossary

PEEK

Poly-Ether-Ether-Keton

pH

The decimal logarithm of the reciprocal of the hydrogen ion activity, aH+, in a solution.

RH

Relative Humidity

RPM

Revolutions Per Minute

RS232

RS-232 is the traditional name for a series of standards for serial binary single-ended data and control signals connecting between DTE (data terminal equipment) and DCE (Data Communication Equipment).

URL

Uniform Resource Locator

USB

Universal Serial Bus

xls

Microsoft Excel Spreadsheet Format

xml

Extensible Markup Language

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