Para Hydrogen Generator

BPHG 90

User Manual

Version 001

Innovation with Integrity

NMR

This manual was written by

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P/N: Z33081

DWG: Z4D12013

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Contents

1 About 7
1.1 This Manual 7
1.2 Policy Statement 7
1.3 Symbols and Conventions 7
2 Introduction 9
2.1 Main Components and Part Location 9
2.2 Principle of Operation 10
2.3 Installation and Initial Commissioning 10
2.4 Limitation of Liability 10
2.5 Copyright 11
2.6 Warranty Terms 11
2.7 Customer Service 11
2.8 EC Declaration of Conformity 12
3 Safety 17
3.1 General 17
3.2 Intended Use 17
3.3 Owner's Responsibility 17
3.4 Personnel Requirements 19
3.4.1 Qualifications 19
3.4.2 Unauthorized Persons 19
3.4.3 Instruction 20
3.5 Personal Protective Equipment 20
3.6 Location of the Safety Label 20
3.7 Basic Dangers 20
3.7.1 General Workplace Dangers 22
3.7.2 Dangers from Electric Power 23
3.7.3 Mechanical Dangers 24
3.7.4 Dangers from Magnetic Fields 24
3.8 Signage 25
3.9 Spare Parts 25
4 Technical Data 27
4.1 General Information 27
4.2 Connection Values 27
4.3 Operating Conditions 28
4.4 Rating Plate 28
4.5 Scope of Supply 29
4.5.1 The Optional Hydrogen Gas Supply Panel 29
4.5.2 The Optional PH2 Storage Kit 30
5 Transport, Packaging and Storage 31
5.1 Packaging 31
5.2 Symbols on the Packaging 31
5.3 Inspection at Delivery 33
5.4 Storage 33
6 Installation 35
6.1 Pre-installation Guidelines 35

Contents

6.1.1 Location of the BPHG 90 36
6.1.2 Hydrogen Gas Supply 36
6.2 Installation Procedure 36
6.2.1 Hydrogen Supply Installation 37
6.2.2 Exhaust Line Connection 38
6.2.3 Ethernet Connection 39
6.2.4 USB Connection 39
6.2.5 Mains Power Connection 39
7 Operation 41
7.1 Getting Started 42
7.1.1 System Check Window 42
7.1.2 Stand-by Mode 43
7.2 System Settings 45
7.2.1 Adjustable Parameters 46
7.2.2 Operating Information 46
7.3 Purge Function 48
7.4 Start Function 48
7.4.1 Cool-down 49
7.5 Parahydrogen Delivery Modes 49
7.5.1 Continuous Flow Mode 50
7.5.2 Batch Delivery Mode 51
7.6 Generator Shut-down 53
7.6.1 Warm Up 53
8 Troubleshooting 55
9 Maintenance 57
9.1 Periodic Maintenance 57
9.1.1 Hydrogen Gas Sensor 57
9.1.2 Vacuum Pump 57
9.1.3 Coolant Circuit 57
9.1.3.1 Coolant Replacement 57
9.1.3.2 Fans and Radiator 58
9.1.3.3 Coolant Pump 58
9.1.4 Gas Purifier Cartridge Replacement 58
9.1.5 Cryocooler 58
9.2 User Maintenance 58
9.2.1 Cleaning Instructions 58
9.2.2 Cryocooler Coolant Circuit 58
9.2.2.1 How to Drain the Cooling System 59
9.2.2.2 How to Fill the Cooling Circuit 60
9.2.3 Fuses Replacement 60
9.2.4 Replace Power Line Fuses 61
9.2.5 Replace Cryocooler Controller Fuse 61
9.2.6 Replace Cryostat Vacuum Gauge Fuse 62
9.3 Spare Parts 62
10 Dismantling and Disposal 63
10.1 Dismantling 63
10.2 Disposal 63

Contents

11 Contact 71
List of Figures 73
List of Tables 75
Index 77

Contents

1 About

1.1 This Manual

This manual is planned for use by the service engineer in the installation of the device or accessory, and for the user in performing existing experiments and for the setup of new experiments.

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

Warning 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.

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

2 Introduction

The **BPHG 90** Parahydrogen Generator is an instrument designed to enrich the content of parahydrogen (p-H2) in normal hydrogen gas. It is intended to be used for the generation of parahydrogen in a laboratory environment. An example of laboratory use of parahydrogen is for the production of hyperpolarized substrates for NMR spectroscopy or pre-clinical MRI.

2.1 Main Components and Part Location

The BPHG 90 consists of the following components, contained in a single metal cabinet:

- Solenoid valves making up the gas distribution circuit.
- A conversion chamber attached to a cryocooler cold head inside a vacuum enclosure.
- A vacuum pump.
- A closed loop water cooling circuit for the cryocooler.
- The control electronics.
- The instrument is connected to the mains power supply and a high purity hydrogen gas supply.

Figure 2.1: BPHG 90 Front View

Figure 2.2: BPHG 90 Rear View

2.2 Principle of Operation

Ortho- and parahydrogen are two spin-isomers of hydrogen which occur naturally in the ratio 3:1 at room temperature. Parahydrogen has a lower ground state energy than orthohydrogen and therefore the parahydrogen content increases at lower temperatures. The quantum-mechanical transition probability from ortho- to parahydrogen is very low unless the gas is brought into contact with a catalyst. In the BPHG 90, hydrogen gas at room temperature is cooled in the cryocooler to below 40K and fed through a conversion chamber with a catalyst, resulting in a parahydrogen content close to 90%.

2.3 Installation and Initial Commissioning

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 Biospin is not covered by your warranty.

2.4 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.

2.5 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.

2.6 Warranty Terms

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

2.7 Customer Service

Our customer service division is available to provide technical information. See the chapter Contact [▶71] for contact information.

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.

2.8 EC Declaration of Conformity

Certificate of Compliance

Certificate: NA201210159 **Date Issued:** March 16, 2012

Project: 163301-9.1

Issued to: Bruker BioSpin
34 rue de l'Industrie BP 10002
67166 Wissembourg Cedex
France

The products listed below are eligible to bear the CCL Mark

Issued by: Robert Keller, Senior Engineer/Safety Supervisor

Authorized by: Thomas Jackson, General Manager

PRODUCTS

MEASUREMENT, CONTROL, OR LABORATORY EQUIPMENT – Certified to US Standards

Product: Para Hydrogen Generator

Trademark: BRUKER

Model: PHG – Para Hydrogen Generator

Ratings: 0.5kVA, 100-240VAC, 50/60Hz, Class I

APPLICABLE REQUIREMENTS

UL Std. No. 61010-1 2nd Edition - Safety Requirements for Electrical Equipment for Measurement, Control, and Laboratory Use – Part 1: General Requirements

Nemko-CCL, Inc. 1940 West Alexander Street Salt Lake City, Utah 84119-2039 Tel (801) 972-6146 Fax (801) 972-8432

Supplement to Certificate of Compliance

Certificate: NA201210159 **Project:** 163301-9.1

The products listed, including the latest revision described below, are eligible to be marked in accordance with the referenced Certificate.

Product Certification History

Project	Date	Description
163301-9.1	March 16, 2012	**Original Certification: Model**: PHG – Para Hydrogen Generator **Ratings**: 0.5kVA, 100-240VAC, 50/60Hz, Class I

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Akkreditiert durch - *Accredited by*: DGA, Frankfurt/Main
DAR-Registriernummer: DGA-PL-203/05-01

Nemko EMV Testzentrum

PRÜFBERICHT - *TEST REPORT*

Elektromagnetische Verträglichkeit (EMV) - *Electromagnetic Compatibility (EMC)*

ANTRAGSTELLER - *APPLICANT*

Firma - *Company:*	**Bruker Biospin SA**
Anschrift - *Address:*	**34 rue de l'industrie** **F - 67166 Wissembourg**
Anwesende - *Witness(es):*	-

PRÜFLING (EUT) - *EQUIPMENT UNDER TEST*

Gerätebez. - *Equipment:*	**Para Hydrogen Generator (PHG)**
Modell/Typ - *Model/Type:*	**BPHG 90 - W118752**
Fertigungs Nr. - *Serial No.:*	**# 014**

PRÜFUNG - *TEST*

Anlieferung *Arrival of EUT:*	**06.12.2011**
Meßtermin(e) *Date of measurement:*	**07. + 08.12.2011**
Prüfungsgrundlage *Standards:*	***Störaussendung** - Emission:* **EN 61326-1:2006** **Klasse A** - *class A* **EN 61000-3-2:2006** **EN 61000-3-3:2008** ***Störfestigkeit** - Immunity:* **EN 61326-1:2006** **industrielle Umgebung**-*industrial locations*
Ergebnisse - *Results:*	**Anforderungen erfüllt** - *PASSED* **Details siehe Zusammenfassung** - *Details see test result summary*
Bemerkungen - *Remarks:*	**Erweiterung zu Prüfbericht FS-1012-163301 vom 04.04.2011.** ***Amendment to test report FS-1012-163301 dated 2011-04-04.***
Durchführung - *Performed by:*	**Dipl.-Ing. Th. W. Stein**

PRÜFBERICHT - *TEST REPORT*

Identifikationsnummer *Identification No.:*	**FS-1012-163301-001**
Datum des Prüfberichts *Date of Report:*	**08.12.2011**
bearbeitet von - *Provided by:*	**Dipl.-Ing. Th. W. Stein** **Prüfer** - *Person responsible* — **Unterschrift** - *Signature*
überprüft von - *Approved by:*	**Dipl.-Ing. P. Lukas** **Prüfer** - *Person responsible* — **Unterschrift** - *Signature*

QMV-5.10-2 d-e / Rev 5.9

Nemko

Nemko Canada Inc

CERTIFICATE OF COMPLIANCE

Certificate Number	:	NA201210159 Issue : 1
Order Number	:	163301
Certificate Issued to	:	Bruker BioSpin 34 rue de l'Industrie BP 10002 67166 Wissembourg Cedex France
Product Name	:	Model: PHG – Para Hydrogen Generator Rated: 0.5kVA, 100-240VAC, 50/60Hz, Class I
Equipment Description	:	Para Hydrogen Generator
Applicable Standards	:	CAN/CSA-C22.2 No. 61010-1-04 Second Edition Safety Requirements for Electrical Equipment for Measurement, Control, and Laboratory Use-Part 1: General Requirements

The products listed above have been certified as being compliant with all applicable requirements of the specifications listed and are eligible to bear the following certification mark:

c Nemko

This certificate is issued on condition that the holder complies and will continue to comply with the requirements of the above mentioned specifications and pursuant to the terms and conditions specified in the Certification Agreement.

Date of Issue: **20 March 2012**

Authorized by: **For Nemko Canada Inc**

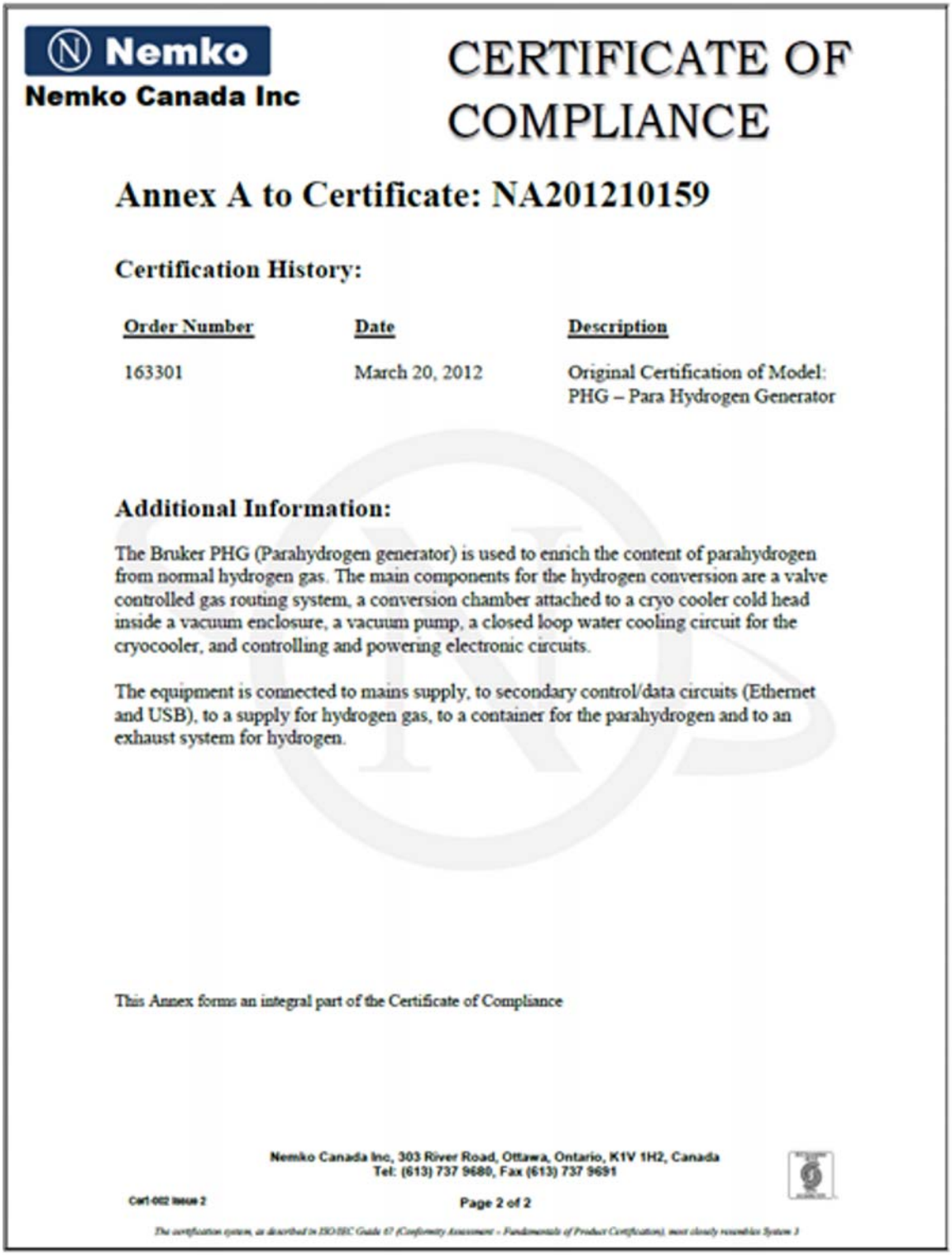
S.C. Beck, Director of Certification

Nemko Canada Inc, 303 River Road, Ottawa, Ontario, K1V 1H2, Canada
Tel: (613) 737 9680, Fax (613) 737 9691

Cert-002 Issue 2

Page 1 of 2

The certification system, as described in ISO/IEC Guide 67 (Conformity Assessment – Fundamentals of Product Certification), most closely resembles System 3



Nemko

Nemko Canada Inc

CERTIFICATE OF COMPLIANCE

Annex A to Certificate: NA201210159

Certification History:

Order Number	Date	Description
163301	March 20, 2012	Original Certification of Model: PHG – Para Hydrogen Generator

Additional Information:

The Bruker PHG (Parahydrogen generator) is used to enrich the content of parahydrogen from normal hydrogen gas. The main components for the hydrogen conversion are a valve controlled gas routing system, a conversion chamber attached to a cryo cooler cold head inside a vacuum enclosure, a vacuum pump, a closed loop water cooling circuit for the cryocooler, and controlling and powering electronic circuits.

The equipment is connected to mains supply, to secondary control/data circuits (Ethernet and USB), to a supply for hydrogen gas, to a container for the parahydrogen and to an exhaust system for hydrogen.

This Annex forms an integral part of the Certificate of Compliance

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Cert-002 Issue 2

Page 2 of 2

The certification system, as described in ISO/IEC Guide 67 (Conformity Assessment - Fundamentals of Product Certification), most closely resembles System 1

3 Safety

This section provides an overview of all the main safety aspects involved in ensuring optimal personnel protection, as well as safe and smooth operation.

Non-compliance with the action guidelines and safety instructions contained in this manual may result in serious hazards.

3.1 General

Before opening the device, be aware of the high 230/115V voltages. Even if these voltages are protected by security features to avoid any physical contact, it is still possible that the voltage sources can be unintentionally touched with a tool, object, etc.

Therefore, always verify if you really need the power supply to be switched on during your work. Otherwise turn the device off and disconnect the power cable from the wall socket to the device. Safeguard that no one is able to re-power the system without your approval.

3.2 Intended Use

The device has been designed and constructed solely for the intended use described here.

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.

3.3 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 device 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 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, gases, 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.
- The owner must ensure that the laboratory is equipped with an oxygen warning device, in case the device is operated with nitrogen.

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.

3.4 Personnel Requirements

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

3.4.1 Qualifications

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.

3.4.2 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.

3.4.3 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

3.5 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.

3.6 Location of the Safety Label

The laboratory supervisor is responsible for ensuring that all the warning labels are maintained in their proper place any time that the device is used.

3.7 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.

DANGER

Danger of injury from flammable gas

Hydrogen is an extremely flammable gas that burns with an invisible flame. It can form explosive mixtures with air. The Lower Explosion Limit / Lower Flammability Limit (LEL/LFL) is 4%.

1. The device should be installed in a sufficiently spacious, well ventilated room equipped with a permanent H2 alarm gas sensor.
2. The user is referred to MSDS safety data sheets for hydrogen gas for further information.

DANGER

Danger of Suffocation from Hydrogen Gas

If excessive amounts of H2 gas is released in an enclosed space, hydrogen will decrease the amount of available oxygen and may cause suffocation.

1. The device should be installed in a sufficiently spacious, well ventilated room equipped with a permanent H2 alarm gas sensor.
2. The user is referred to MSDS safety data sheets for hydrogen gas for further information.

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 to life from strong magnetic fields!

Strong magnetic fields may cause serious injuries or death and significant damage to property.

1. Persons fitted with heart pacemakers must be kept away from the device. The functionality of the heart pacemaker could be compromised.
2. Persons with metal implants must be kept away from the device. Implants may heat up or be subject to magnetic attraction.
3. Ferromagnetic materials and electromagnets must be kept away from the magnetic source. Such materials could be subject to magnetic attraction and may fly around the room, injuring or killing people. Minimum distance 3 meters.
4. Remove magnetic items (jewelry, watches, pens etc.) before carrying out maintenance work.
5. Keep electronic equipment away from the magnetic source. Such equipment could be damaged.
6. Keep storage media, credit cards etc. away from the magnetic source. Data could be erased.

Note: The device does not produce a high magnetic field.

3.7.1 General Workplace Dangers

⚠ WARNING

Danger to life from nonfunctional safety devices!

If safety devices are not functioning or are disabled, there is a danger of serious injury or death.

1. Check that all safety devices are fully functional and correctly installed before starting work.
2. Never disable or bypass safety devices.
3. Ensure that all safety devices are always accessible.

⚠ CAUTION

Danger of injury from tripping over dirt and scattered objects!

Dirt and scattered objects may cause people to slip or trip, resulting in personal injuries.

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

NOTICE

Material damage due to a software error!

Samples or the device may be damaged due to a software error causing malfunction of the control system. Users may also be shocked by abrupt malfunction or unexpected system start.

1. Dummy samples must be used during installation and service.
2. Personnel should be alerted to unexpected malfunctions.

NOTICE

Material damage hazard due to impacting the magnet!

Impacting the magnet may result in a quench.

1. Mount the device carefully on the magnet.
2. Avoid banging the magnet during installation and operation, e.g. when replacing the sample carousel.

NOTICE

Material damage due to the use of genuine samples during installation and maintenance!

Using genuine samples during installation and maintenance may result in material damage.

1. Use only dummy samples during installation and maintenance.

3.7.2 Dangers from Electric Power

DANGER

Danger to life from stored charges!

Electric charges may be stored in electrical components even after the system has been switched off and disconnected from the power supply. Contact with these components may result in serious or fatal injury.

1. Before working on the specified components, ensure that they have been completely disconnected from the power supply.
2. Allow 10 minutes to elapse in order to ensure that the internal capacitors have been fully discharged.

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 to life from residual electrostatic potentials!

Friction between material being conveyed may result in significant development of electrostatic potential. Therefore, contact with parts immediately following the conveying operation may be life-threatening.

1. Potential equalisation must be ensured before making contact with parts, unless such equalisation is provided by the customer.

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

WARNING

Danger to life from contact voltage!

Absent or faulty protective earth conductor may result in contact voltage. This may pose a risk of injury or death.

1. Before the initial commissioning of the device, connect the main power supply to the socket and verify the complete functionality of the protective earth conductor.

3.7.3 Mechanical Dangers

⚠ CAUTION

Accident hazard from movement of mechanical parts!

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

1. Shut off the device before accessing.

⚠ CAUTION

Accident and material damage hazard from falling objects!

Equipment may fall down during assembly, retrofitting, or dismantling. This may result in personal injury or equipment damage.

1. If necessary, assemble/disassemble the device in multiple parts.
2. Use a platform with railings instead of a ladder to reach the assembly area.
3. Avoid working over the head. When this can not be avoided, wear a protective hard hat.
4. Follow the mounting instructions in the installation manual.

3.7.4 Dangers from Magnetic Fields

⚠ WARNING

Danger to life from strong magnetic fields!

Strong magnetic fields may cause serious injuries or death and significant damage to property.

1. Persons fitted with heart pacemakers must be kept away from the device. The functionality of the heart pacemaker could be compromised.
2. Persons with metal implants must be kept away from the device. Implants may heat up or be subject to magnetic attraction.
3. Ferromagnetic materials and electromagnets must be kept away from the magnetic source. Such materials could be subject to magnetic attraction and may fly around the room, injuring or killing people. Minimum distance 3 meters.
4. Remove magnetic items (jewelry, watches, pens etc.) before carrying out maintenance work.
5. Keep electronic equipment away from the magnetic source. Such equipment could be damaged.
6. Keep storage media, credit cards etc. away from the magnetic source. Data could be erased.

The magnetic field of the device does not cause any personal injuries or property damage. For further information see the manual of the magnet used.

3.8 Signage

The following symbols and information signs can be found in the work area. They refer to their immediate surroundings.

The identification and placement of warning labels are included in the manual. The laboratory supervisor is responsible for ensuring that all the warning labels are maintained in their proper place any time that the device is used.

Electrical Voltage

Only qualified electricians are permitted to work in a work room marked by this sign. Unauthorized persons must not enter the workplaces thus marked and must not open the marked cabinet.

Danger Spot

Warning indicating a danger spot in work rooms.

The warning label may be ordered using Bruker Part Number 67470.

3.9 Spare Parts

Loss of Guarantee

The use of non-approved spare parts will invalidate the manufacturer‘s guarantee.

Purchase spare parts from authorised dealers or directly from the manufacturer. See Contact for manufacturer's address.

4 Technical Data

4.1 General Information

Data	Value	Unit
Weight	approx. 52	kg
Length	66.5	cm
Width	46.5	cm
Height	68.5	cm

Table 4.1: Technical Data: General Information

4.2 Connection Values

Electrical

Data	Value	Unit
Voltage	100-240	V AC
Apparent power consumption, maximum	500	VA
Circuit protection	4 A T/250V	A
Frequency	50/60	Hz

Table 4.2: Electrical Connection Values

Inlet Gas

Data	Value	Unit
Operating pressure	6 bar min, 10 bar max.	bar
Inlet gas	H_2 gas high purity >= 99.999	%.
Inlet gas fittings	G1/8	“

Table 4.3: Inlet Gas Values

Outlet Gas

Data	Value	Unit
p-H_2 outlet flow rate	<= 0.2 (STP)	l/min
Gas outlet	Parahydrogen fraction >= 90	%.
Outlet gas fittings	G1/8	“

Table 4.4: Outlet Gas Values

4.3 Operating Conditions

Environment

Data	Value	Unit
Temperature range	15-32	°C
Relative humidity	10-90% non condensing	%
Operating altitude	<= 2000	m

Table 4.5: Operating Environment

Cooling Down Process

Data	Value	Unit
Conversion temperature	36 to 40	K
Cool-down time	<1 from room temperature	h

Table 4.6: Cooling Down

Warm Up Process

Data	Value	Unit
Target temperature	300	K
Warm up time	< 20	min

Table 4.7: Warm Up

4.4 Rating Plate

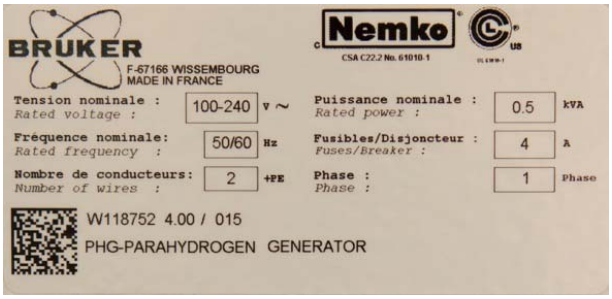

Figure 4.1: Rating Plate Example

The rating plate is located at the power input and includes the following information:

Example

- Manufacturer: Bruker Biospin France
- Rated Voltage: 200-240
- Frequency 50/60 Hz
- Wires: 2
- Rated power: 500 VA

- Fuses: 4A
- Phase: 1
- W118752 / 04 / 015
- Part Number: W118752
- Engineering Change Level (ECL): 04
- Serial Number: 015

4.5 Scope of Supply

The parahydrogen generator **BPHG 90** (P/N W123776**)** is delivered with the following accessories set:

P/N	Description	Quantity
3000	Power supply cable 3 m 3 x 1.0 mm² (for Europe)	1
2257	Fuse mini 5 x 20 mm 4.0AT	2
1803847	Stainless steel gasket	2
W131932	Nipple with hose nozzle with tubing D = 8 mm Black L = 1 m	1
1806787	IM personal H_2 gas detector with power supply.	1
W127310	CD Manual H_2 Gas Detector - English	1
W131933	Tubing l = 10 m with pump silencer	1
1803111	Brass “SWAGELOK” tube fitting 1/8 inch	2
1803121	Stainless steel tubing 1/8, L = 6 m	1
1807578	Stainless steel hose dn6 f1/8bsp-f1/8bsp L = 4 m	1
1806418	Coupling G1/8 - G1/8 stainless steel conical	1
39930	PTFE tape 12 mm	1
W127477	Label "DANGER HYDROGEN"	2
1807106	Wrench 7/16”	1
1808576	Wrench 1/2 x 9/16”	1
1904	Straight male pipe coupling 8 mm - 1/8” cylindrical	2
W131498	BPHG Technical Manual Z33015	1
1808869	Fuse mini 5 x 20 mm 10.0 AT	2

4.5.1 The Optional Hydrogen Gas Supply Panel

The optional H_2 supply panel contains:

- a shut-off valve,
- a pressure regulator,
- a pressure gauge, and
- a flow limiter.

This optional panel can be used to supply the BPHG 90 with hydrogen gas.

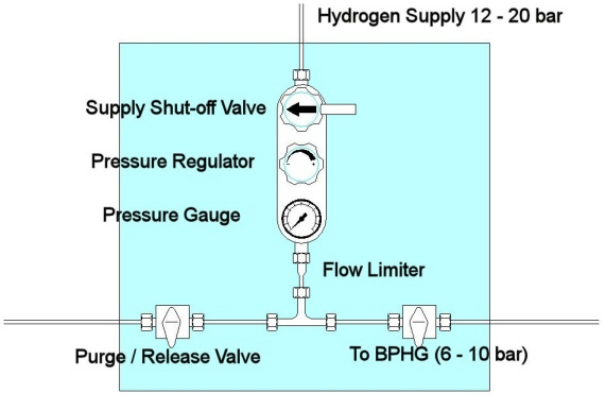

Figure 4.2: H_2 Gas Supply Panel P/N W126956

4.5.2 The Optional PH2 Storage Kit

The optional storage kit (0.8 liter thick wall aluminium bottle) is available containing a manual valve, pressure gauge, 2 meter stainless steel hose and fittings.

The container is intended to store gas PH2, temporarily, before being used in another place distant from the generator.

Figure 4.3: 2 p-H_2 Storage Kit P/N W126957

5 Transport, Packaging and Storage

5.1 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

Dispose of packaging material in accordance with the relevant applicable legal requirements and local regulations.

5.2 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 5.1: Symbols on the Packaging

5.3 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.

5.4 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: Maximum 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

6 Installation

Installation, initial commissioning, retrofitting, repairs, adjustments or dismantling of the device must only be carried out by employees of the manufacturer or persons authorized by the manufacturer.

6.1 Pre-installation Guidelines

Note: For the installation of the BPHG 90 it is necessary to select an appropriate location and make the required provisions for the safe operation of the instrument.

⚠ WARNING

Particular attention is required for the safety aspects of working with hydrogen gas.

Before installation, check to insure the following minimum conditions are provided:

1. The room where the BPGH 90 is to be installed must be equipped with a permanent hydrogen detector to monitor the air and give a visual and acoustic alarm if hydrogen gas concentration is detected is higher than a safe limit.
2. The permanent H_2 sensor should be subject to regular maintenance to ensure its safe operation.
3. The room must be well ventilated and the air should renewed by appropriate means.
4. A provision must be in place to allow the safe removal of waste hydrogen gas from the room, e.g. through a fume cupboard. The BPHG 90 is delivered with 10 m flexible exhaust tubing for this purpose.
5. The room must be equipped with a hydrogen gas supply (99.999%; 6 to 10 bar) to which the generator will be connected. Provisions have to be made with regards to the supply of hydrogen gas to ensure that no failure can cause a sudden release of H_2 in quantities such that the LOWER EXPLOSION LIMIT (LEL) is exceeded. The LEL for Hydrogen is 4% by volume. Large capacity hydrogen containers should be stored outside the room.
6. Open flames or other ignition sources in the room are prohibited and a corresponding warning sign should be displayed.
7. Warning signs must be visible near the position of the BPHG 90 and on the main entrance door. At the entrance to the room an appropriate flammable gas warning sign (H_2 flammable gas DANGER sign, of which two are delivered with the generator) must be placed and the room must be identified as a non-smoking area.
8. The BPHG 90 must be connected to the mains 100-230 VAC 50/60 Hz, 500 Watt.

6.1.1 Location of the BPHG 90

The BPHG 90 must be installed indoors and in a location where there is no danger that it can be exposed to water or rain. It can be installed close to a wall or possibly below a desk (a 20 cm clearance must be observed to the rear and above, for the proper cooling of the generator). The power and Ethernet connections and the supply of H_2 gas are made on the rear of the instrument, whereas the pH_2 outlet is located on the front.

If the gas supply line is attached to the wall, the rear side of the generator must be accessible to permit the easy attachment of the gas capillary tube supply line with a flat wrench.

The front panel must be easily accessible and visible for the operation of the generator.

The generator must be installed on a flat surface. Once installed the front wheels must be blocked and the instrument must not be moved afterwards without disconnecting and reconnecting the gas supply line.

6.1.2 Hydrogen Gas Supply

The most critical provision for the BPHG 90 is the supply of pure hydrogen and prior to the installation of the instrument; provisions for the safe delivery of hydrogen gas have to be made.

Different sources of hydrogen gas can be used to supply the generator:

1. An independent electrolytic hydrogen gas generator producing very pure hydrogen, 99.999% at sufficient pressure (P > 6 bar) and flow (≥ 0.4 NL/min).
2. A pressurised cylinder (200 bar) located in a remote room or outside the building. The cylinder with a regulator supplies a metal distribution line made of stainless steel or copper tubing. The room where the BPHG 90 is installed is equipped with an H_2 gas outlet, a manual stop valve, a pressure regulator, a pressure gauge, a purge valve, and a flow limiter. The optional hydrogen supply panel contains all necessary provisions (see section The Optional Hydrogen Gas Supply Panel [▶29])
3. A pressurised cylinder (200 bar max.) with a suitable pressure regulator. The cylinder must be handled and secured according the prevalent safety regulations and connected to the BPHG 90 in the same way as a remote cylinder (see point 2 above).

The connection between the hydrogen supply and the BPHG 90 is made by means of a flexible stainless steel tube (supplied with instrument).

The BPHG hydrogen inlet pressure must be between 6 and 10 bar.

6.2 Installation Procedure

The BPHG 90 will normally be installed and tested by a trained Bruker service engineer. Whenever the instrument needs to be moved to another location, care should be taken to follow the correct installation procedure.

6.2.1 Hydrogen Supply Installation

An 1/8” outside diameter stainless steel capillary tube with two “Swagelok” type 1/8” fittings and flat seals is delivered with the generator. An existing H_2 gas source may be used, such as:

- a supply panel,
- a H_2 gas cylinder, or,
- a hydrogen gas generator able to deliver 500 ml/min gas at 10 bar.

Step1: Connect the capillary SS tube to the gas source

1. Determine the length of the tube needed to reach the source from the generator and add 1m more to form a free loop that can be left behind the generator. The loop will make it possible to move the generator slightly without having to remove the capillary tube. Cut the tube with a saw and deburr the tube ends.
2. Connect the capillary tube to the H_2 gas source with a Swagelok fitting.
3. Attach the tube to the wall.

Step2: Connect the capillary tube to the generator

Figure 6.1: H_2 Gas Inlet Port

1. Wrap 1-2 turns of PTFE tape around the fitting thread and screw the Swagelok fitting onto the H_2 inlet on back panel.
2. Open the H_2 gas valve and the pressure regulator to purge the capillary line for a few seconds.
3. Close the H_2 gas valve.
4. Insert the capillary tube in the fitting and tighten by hand. With a wrench tighten a further **1¼ turn** to make a leak free connection.

Once the connection to the hydrogen supply has been made, the generator should only be moved over very short distances to avoid compromising the gas connections.

6.2.2 Exhaust Line Connection

In normal operating mode, small amounts of hydrogen can be released through the vacuum pump or through the safety valves and will be routed to the hydrogen vent outlet at the rear side of the instrument.

Figure 6.2: H2 Gas Vent Outlet

Ten meters of 8 mm flexible plastic tubing are delivered with the generator. The tubing must be connected to the hydrogen gas vent port on the rear side (labeled H_2 vent) to release the hydrogen safely outside the room, e.g. by leading it into a fume cupboard or outdoors.

Once all services have been connected, move the generator to its final position and lock the two front wheels.

⚠ WARNING

Equipment displacement can damage the hydrogen gas supply line.

Once connected to the H2 gas supply line, the generator should only be moved over very short distances to avoid compromising the gas connections.

⚠ CAUTION

Risk of appliance overheating and malfunction

A clearance of 10 cm behind the rear panel and above and to both sides of the BPHG90 are required for the correct cooling of the generator.

⚠ CAUTION

Risk of hydrogen gas leak (risk of explosion/risk of fire)

Tighten gas connections fittings and check all with soapy water or a special H2 gas leak detector.

6.2.3 Ethernet Connection

The BPHG 90 can optionally be connected via an Ethernet link to an external computer. Connect the Ethernet cable on the rear panel (labeled ETH) to a free Ethernet port on the external computer, using a standard RJ45 Ethernet cable.

6.2.4 USB Connection

The generator has an USB port on rear panel for updating the firmware. This port is reserved for use by trained service personnel only.

6.2.5 Mains Power Connection

The BPHG 90 is connected to the mains using a 3-wire grounded power cord rated for 600 W. Plug the power cord into the rear power connector and connect the other end to the wall outlet. The BPHG 90 can now be switched on. The rear power switch on the socket must be in **ON** position.

The line socket contains two **T 4A /250V** fuses in a small drawer. If the fuses need to be replaced, always use the same type and rating.

The red power switch on the front is used to switch the instrument **ON** and **OFF**. A red light in the front power switch indicates that line voltage is present.

7 Operation

The BPHG 90 has been designed to operate in a largely automatic manner. The user has access to different modes of operation via a touch panel at the front of the instrument. The touch panel presents the user with information on operational parameters via a number of screens. A typical screen layout will present a title bar at the top of the screen, possibly some operational parameters in the main field and some buttons for control over the operation (see the next figure). Most operating screens feature a **Settings** button at the lower right hand corner, giving access to instrument set-up parameters (see the section System Settings [▶45].

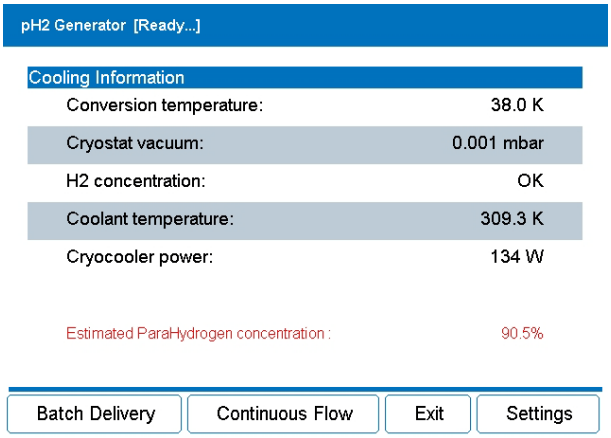

Figure 7.1: Touch Screen for Instrument Operation

The main operating parameter of the **BPHG 90** is the conversion temperature at which ortho hydrogen is converted to parahydrogen. This temperature determines the yield of parahydrogen (see the next figure).

The instrument is designed to operate between 36 and 40 K, producing H_2 gas with more than 85% parahydrogen. The conversion temperature is a user adjustable parameter (see the section Adjustable Parameters [▶46]).

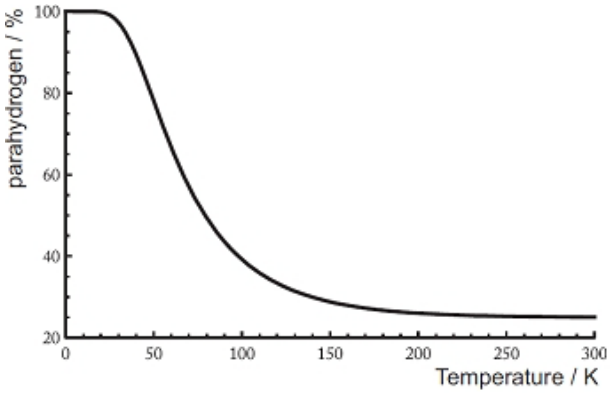

Figure 7.2: Parahydrogen Fraction Function of Conversion Temperature

7.1 Getting Started

1. Open the H_2 gas supply valve and ensure that the hydrogen inlet pressure is in the correct range: 6 to 10 bar.
2. Switch on the generator using the mains power switch on the front panel (see the next figure).

Next to the power on switch on the front panel of the BPHG 90 are two LEDs that provide basic status information.

The green **Ready** LED indicates normal operation of the BPHG 90. During the cooling down of the conversion chamber, it blinks, and when the pre-set conversion temperature is reached, it will stay permanently ON.

The red **Error** LED indicates that an error condition is present (e.g. a low coolant level, low input pressure, etc.).

Figure 7.3: Power Switch and Panel LED's

7.1.1 System Check Window

When the BPHG 90 is switched on, the instrument briefly shows a welcome screen and then performs a pre-operation **System Check** of the main components. At the end of the system check, the system displays the **System Check** window. If any of the tests does not pass, the failed test result is highlighted in RED(see the figure below) and operation of the instrument is not possible until the user has taken remedial action.

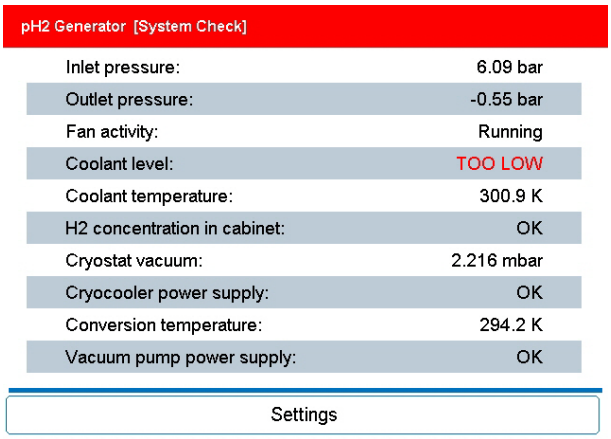

Figure 7.4: System Check with Low Coolant Level Warning

A special case is the test for the hydrogen sensor that measures the H_2 concentration in air inside the instrument. This sensor needs about a minute to warm-up and this is indicated by a message in ORANGE(see the figure below).

When all tests pass, the **System Check** window disappears after a few seconds and the system enters **Standby** mode (see section Stand-by Mode [▶43]).

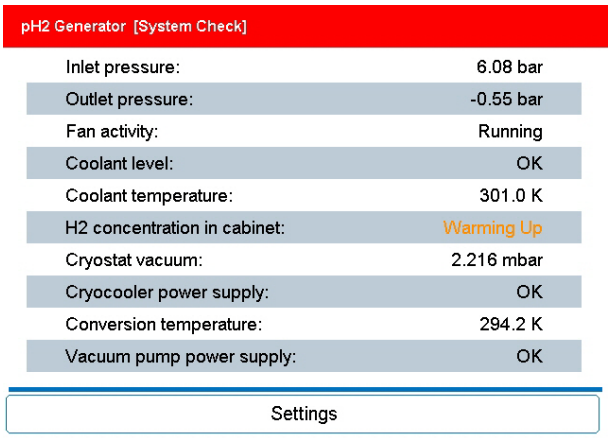

Figure 7.5: Hydrogen Gas Sensor Warming Up

7.1.2 Stand-by Mode

Once the instrument has successfully passed the initial automatic system check, it goes into **STANDBY** mode. First, a confirmation screen is displayed (see the next figure), asking the user to verify that the exhaust hydrogen gas tube is safely routed outside the room or to a fume cupboard.

The BPHG 90 is delivered with 10 meter of flexible exhaust plastic tubing, which is connected to the rear of the instrument and routed, for example to a fume cupboard or outside the room (see section Exhaust Line Connection [▶38]).

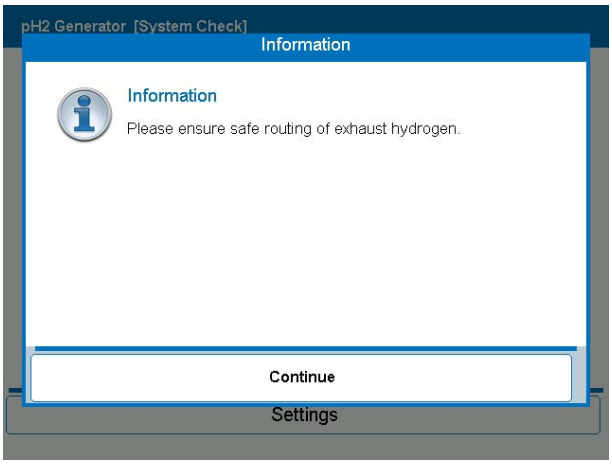

Figure 7.6: Exhaust Gas Warning Message

⚠ WARNING

Hydrogen is a very flammable gas.

The exhaust hydrogen gas must be safely routed outside the room using the tubing supplied.

Once the safety check message has been confirmed, the system switches to the **Standby** screen in which the user can select between the three main operation modes of the instrument (see the next figure): **Start**, **Purge**, and **Warm Up**.

In addition, **Settings** gives the user access to various operational information and set-up parameters.

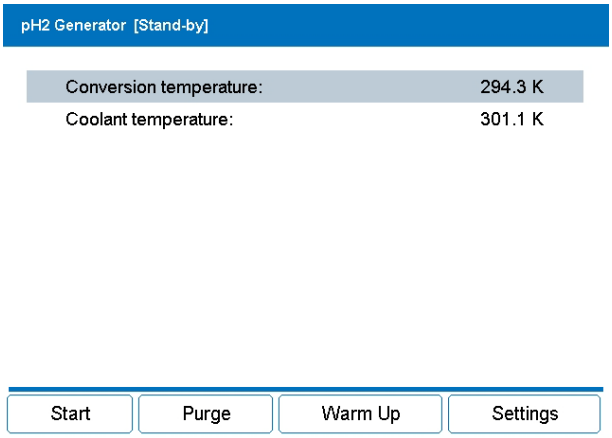

Figure 7.7: Stand-by Screen with Warm-up Conversion Chamber

The main operation of the BPHG 90 can be (re-)started by clicking the **Start** button. This action will start the cryocooler and cool down of the conversion chamber (see section Start Function [▶48]).

The **Purge** function is used to clear the working volume and all internal tubing of the instrument from air or other contaminants (see section Purge Function [▶48]). It is recommended to execute a purge procedure before starting the cooling of the conversion chamber. Once the conversion chamber is cold (i.e. below 280 K), a purge cannot be performed and the corresponding button is greyed-out.

Warm Up executes a fast, forced warm-up of the conversion chamber with a dedicated heater (see section Generator Shut-down [▶53]). It is recommended that a warm-up be performed before the system is powered down.

The BPHG 90 monitors the operating times of maintenance critical components and checks them against their preset service intervals. If any of the service intervals are exceeded while the BPHG 90 is in operation, then a **Maintenance Warning** is issued (see section Operating Information [▶46]). It is still possible to operate the system after a non-critical maintenance warning has been issued. After a critical maintenance error, the instrument shuts down.

7.2 System Settings

The **BPHG 90** operations are controlled by a number of parameters which are factory preset. These parameters are accessible via the **Settings** function (see the next figure) which can be called up by tapping the **Settings** button at the bottom right hand corner of most operating screens. The main Settings access window has two tabs:

- **User** settings.
- **Service** settings.

The **Service (BRUKER)** is intended only for trained service personnel and is password protected.

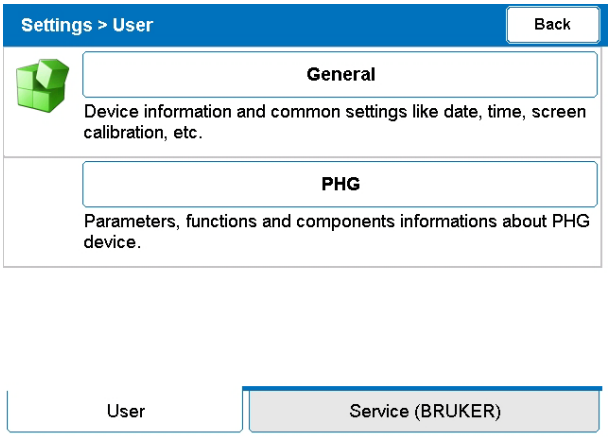

Figure 7.8: System Settings and User Settings Interface

The user has access to two settings screens: **General** or **PHG**.

The **General** button offers general system set-up functions for e.g. touch screen calibration and system date and time settings.

The **PHG** button presents the user accessible operating parameters. This screen has two tabs: **Settings** and **Operating Information**.

7.2.1 Adjustable Parameters

In the **PHG > Settings** window, the user has access to the following operational parameters:

- Conversion temperature set-point (36...40K), default 36 K.
- Batch delivery target pressure (0...6 bar), default 3 bar.
- Filling container delivery timeout (0... 60 min), default 60 min.

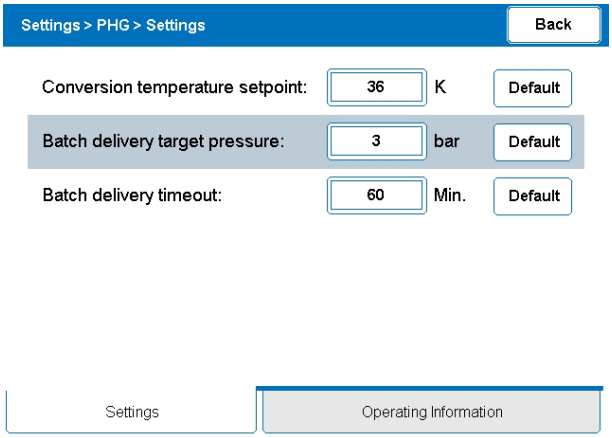

Figure 7.9: User Settings Menu with Adjustable Parameters

In order to change a parameter value, tap the parameter field. This will bring up a numerical keypad to enter the new value. The user accessible parameters have a limited value range and entering values outside this range will cause the nearest valid value to be loaded. The parameter values are stored in a non-volatile memory and user defined values will survive a power-down of the instrument. Factory preset values can be restored by tapping the **Default** button next to the value field.

7.2.2 Operating Information

The BPHG 90 has built-in timers for monitoring the operation of maintenance critical components. The tab **Operating Information** brings up an information window with the values of the operating times of these components and of the main operational parameters.

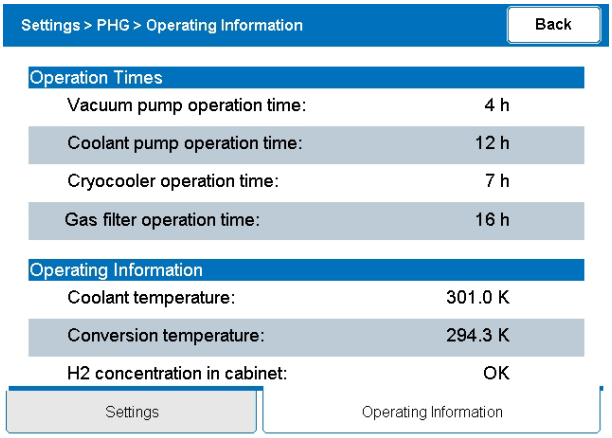

Figure 7.10: Operation Information Screen

At start-up, when the operating time of any of the maintenance critical components is within 90% of the preset service interval, then a maintenance warning is given, but the system can continue to operate. In this case, the operating information screen will display a warning triangle next to the component indicating it is near its service interval maintenance. Warnings are repeated at regular intervals, prompting the user to take remedial action before a critical maintenance error occurs.

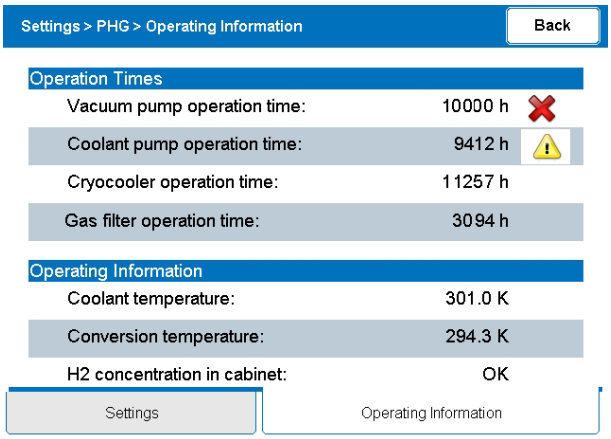

Figure 7.11: Example of Non-critical Warnings

At start-up or during operation, when any of the operating times have become equal to 100% of the preset service interval, a critical maintenance warning is issued (see the next figure) and the system shuts down. The device can not be operated until the user has taken remedial action. The source of the critical maintenance error is indicated in the operating information screen by means of a red cross.

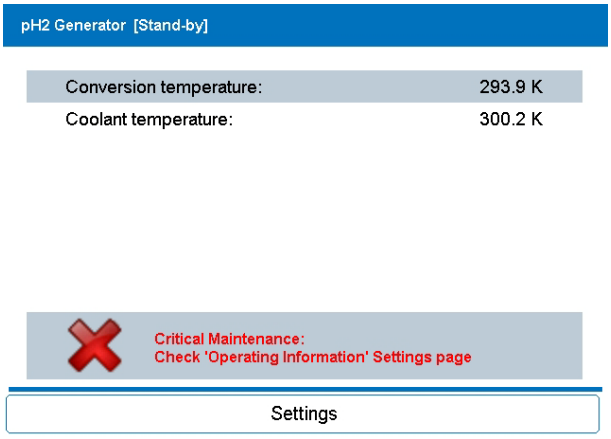

Figure 7.12: Critical Maintenance Warning

7.3 Purge Function

Before the BPHG 90 can be cooled down for the first time, the instrument must ensure that no gases other than H_2 will be present in any internal tubing and in the conversion chamber. This is to avoid a blockage caused by the freezing of other gases such as CO_2, N_2, etc. For this purpose the working volume is first evacuated and then filled with H_2 gas. The process is executed twice and takes a few minutes. The user can select this purge procedure by tapping the **Purge** button on the **Standby** screen (see figure Stand-by Screen with Warm-up Conversion Chamber [▶44]).

During the process, the instrument reports progress through the **Purge in progress** screens, indicating the pass number in the title bar. The purge process can be aborted by tapping the **Abort Purge** button (see the figure *System check with low coolant level warning* in the section System Check Window [▶42]).

Whenever a maintenance operation is performed (e.g. a change of hydrogen supply bottle, purifier cartridge replacement) and there is the possibility that foreign gasses have entered the generator, then a purge operation must be carried out.

A purge operation cannot be carried out when the system is cold. In this case the **Purge** button in the **Standby** screen is greyed-out and the system needs to be warmed up first (see section Generator Shut-down [▶53]).

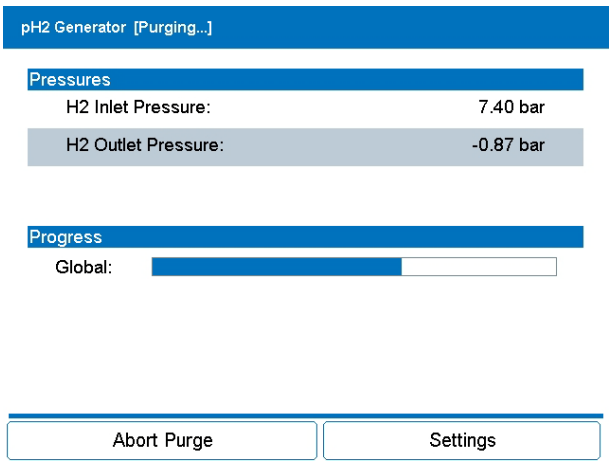

Figure 7.13: Purge in Progress Screen

7.4 Start Function

Once the working volume of the BPHG 90 has been purged, the **Standby** screen (see the figure *Stand-by Screen with Warm-up Conversion Chamber* in the section Stand-by Mode [▶43]) reappears and the instrument is ready to be cooled down. The cool down procedure is initiated by tapping the **Start** button.

The conversion chamber in the BPHG 90 is contained within the vacuum space of the cryostat, to provide an effective thermal isolation from the environment. It is essential for the efficient low temperature operation of the instrument that the residual pressure in the vacuum chamber is below a pre-set limit (5 mbar) before the conversion chamber is cooled down. The system starts the cool-down procedure by testing and, if necessary, improving the vacuum. Pre-pumping of the cryostat vacuum, by the internal diaphragm pump, will take a few minutes.

7.4.1 Cool-down

When the cryostat vacuum is good (below 5 mbar), the cryocooler is automatically started.

When the cryocooler is functioning, it emits an audible noise, this noise is normal. The sound level of the BPHG generator is below 70 dB at 1 m.

While the BPHG 90 is cooling down, relevant parameters are displayed on the cool-down screen.

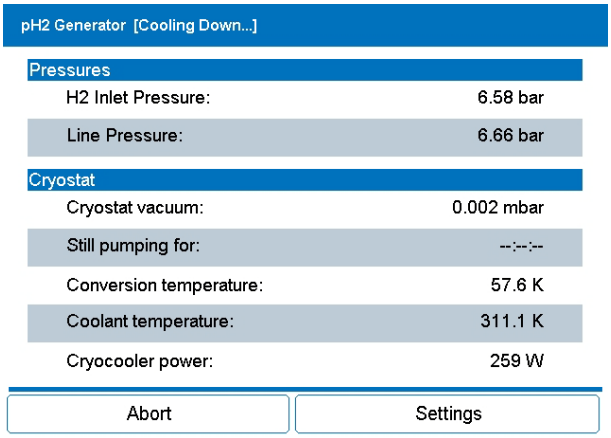

Figure 7.14: Cool Down Screen

The cool-down process can be interrupted using the **Abort** button. When this button is pressed, the user is asked to confirm his action before the cool-down is aborted. The system then returns to the **Standby** screen.

Once the system has reached the operating temperature, the **Ready** screen appears with the main operating buttons **Batch Delivery** and **Continuous Flow** enabled. The **BPHG 90** is then ready to deliver parahydrogen. The time required to cool the system down from room temperature is typically less than one hour.

7.5 Parahydrogen Delivery Modes

Once the conversion chamber has reached the desired conversion temperature, the BPHG 90 is ready to deliver parahydrogen in two different modes:

1. **Batch Delivery**
2. **Continuous Flow**

The **Batch Delivery** mode serves to store the produced parahydrogen gas in a container or a bottle for subsequent use at another location.

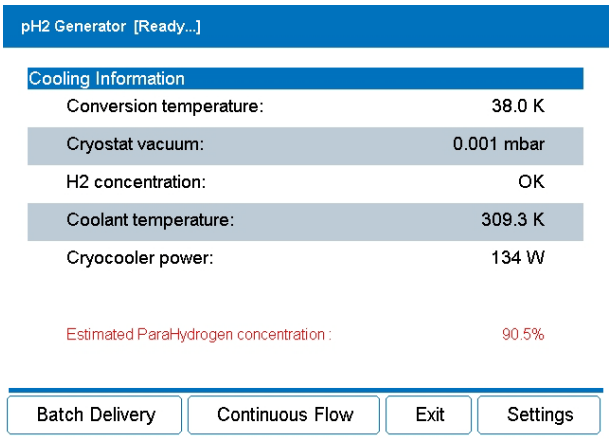

Figure 7.15: Ready Screen with Access to Main Operating Functions

In **Continuous Flow** mode, the instrument will continuously deliver parahydrogen to the outlet. It is therefore essential that the outlet is connected to a consumption device, such as the Bruker parahydrogen polarizer, and that any waste gas from this device is routed safely out of the laboratory, e.g. through a fume cupboard.

WARNING

The BPHG90 has NO means of verifying that the delivered gas is handled in a safe manner.

It is the responsibility of the user to ensure that the delivered gas is handled safely.

7.5.1 Continuous Flow Mode

In continuous flow mode, the instrument is capable of maintaining a flow of 0.2 L/min (STP) of Para hydrogen with an inlet pressure of 10 bar. The output pressure will depend on the back pressure of the device to which the BPHG 90 is connected. During delivery the **Continuous Flow** screen (see figure Continuous flow progress screen [▸50]) shows operational parameters and a running chronometer (labelled **Delivery time**).

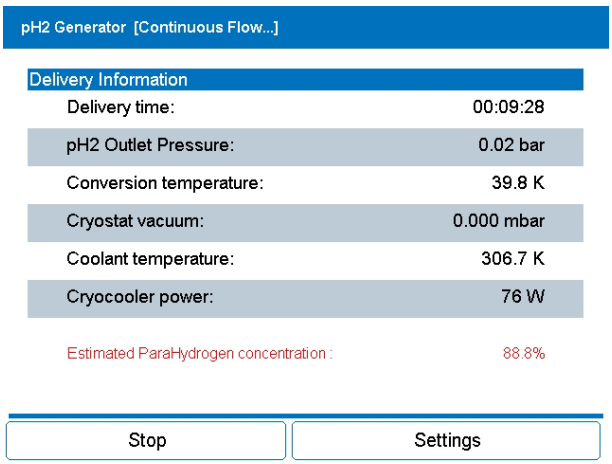

Figure 7.16: Continuous Flow Progress Screen

The user can stop parahydrogen gas delivery by pressing the **Stop** button where upon the system returns to the **Ready** screen.

Based on the conversion temperature, the estimated pH_2 yield is indicated as a percentage.

7.5.2 Batch Delivery Mode

In batch delivery mode, the BPHG 90 assumes that an **EMPTY** container is connected to the pH_2 gas outlet on the front panel of the instrument. Ensure that the container used is parahydrogen compatible, i.e. does not contain materials that can quench the hydrogen polarization (e.g. paramagnetic materials). Aluminium bottles are recommended (a suitable aluminium bottle is an optional accessory for the BPHG 90, see section Scope of Supply [▶29]). When the user presses the button Batch Delivery, the BPHG 90 displays a dialogue screen (see figure *Container Connection Dialogue Window*) asking for confirmation that an empty container has been connected to the p-H_2 outlet of the front panel.

The instrument then proceeds with evacuating the internal delivery lines and checking the internal pressure of the attached container. If the pressure in the container is too high, a warning window is displayed (see figure Container High Pressure Warning Screen [▶51] and the batch delivery cannot proceed. The user must then disconnect the container and reconnect an empty one.

Once the BPHG 90 has detected a low enough pressure in the container, the container is completely evacuated using the vacuum pump of the BPHG and the filling process with parahydrogen begins.

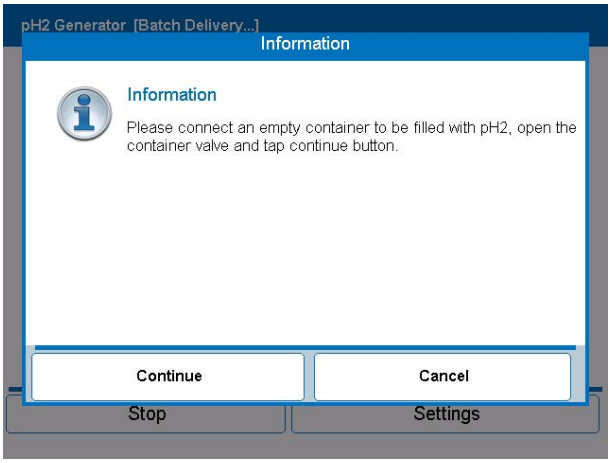

Figure 7.17: Container Connection Dialogue Window

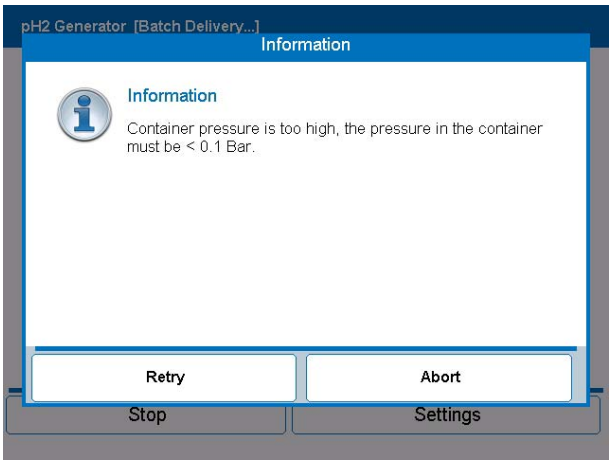

Figure 7.18: Container High Pressure Warning Screen

WARNING

The generator has no means of detecting the nature of the residual gas is in the container.

1. It is the responsibility of the user to ensure only to connect containers that have been used exclusively for hydrogen.
2. Connecting containers with residual gases other than hydrogen can lead to contamination and blockage of the BPHG 90.

The batch delivery continues until a pre-defined pressure has been reached or until aborted by the user. The delivery process also stops when the preset time-out has elapsed. Progress is indicated in the Batch Delivery screen (see the figure Batch delivery progress screen [▶51]).

The pressure and time-out parameters can be set by the user (see the section Adjustable Parameters [▶46]). During the procedure, the elapsed time and the pressure in the container are displayed on the **Batch Delivery** screen. After completion, the user is asked to confirm that the container has been removed (see the figure End of Batch Delivery Process [▶51]).

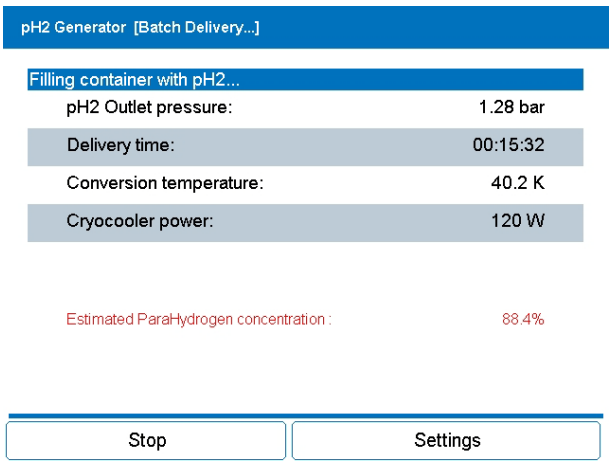

Figure 7.19: Batch Delivery Progress Screen

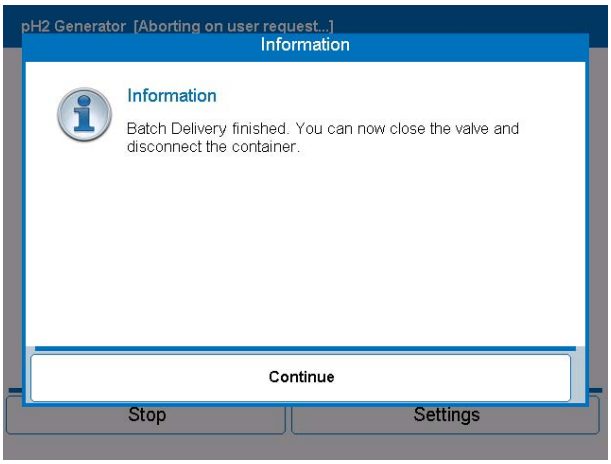

Figure 7.20: End of Batch Delivery Process

7.6 Generator Shut-down

The BPHG 90 can be left in **Ready** mode without detrimental effects to the instrument. If, however, it is envisaged that no parahydrogen is required for a prolonged period of time, it is recommended that the system be switched off. It is not recommended, however, that the BPHG 90 is switched off by means of the main power switch when the conversion chamber is cold. This could lead to pressure build up in the conversion chamber and (a small amount of) H_2 gas being released through the pressure safety valves in the instrument.

The preferred procedure for switching the system off is by first warming it up as described in the following section.

7.6.1 Warm Up

Upon terminating either of the two delivery modes, the BPHG 90 returns to the **Ready** screen. When the user presses the **Exit** button on this screen the system will return to **Standby** from which a forced warm-up can be initiated by tapping the **Warm Up** button.

The **Warm Up** procedure allows the user to warm the system up quickly to a preset temperature in order to clear a suspected conversion chamber blockage, or in preparation for a system shut-down.

A forced warm-up takes typically less than 20 minutes. When the **Warm-up** button is pressed, the system asks for a user confirmation (Warm up dialogue and information screen [▶53]) and then proceeds to heat the conversion chamber under continuous pumping on the working volume.

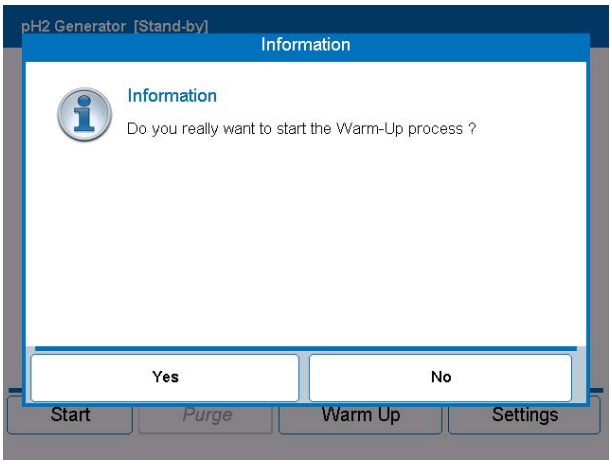

Figure 7.21: Warm-up Dialogue and Information Screen

The warm-up procedure stops when the preset warm-up temperature has been reached. Progress is indicated in the **Warming up** screen and the process can be aborted by tapping the **Abort** button (see Warm up progress screen with abort function [▶53]).

Once the warm-up preset temperature has been reached, the system returns to the **Standby** screen. Then the generator can be safely turned off.

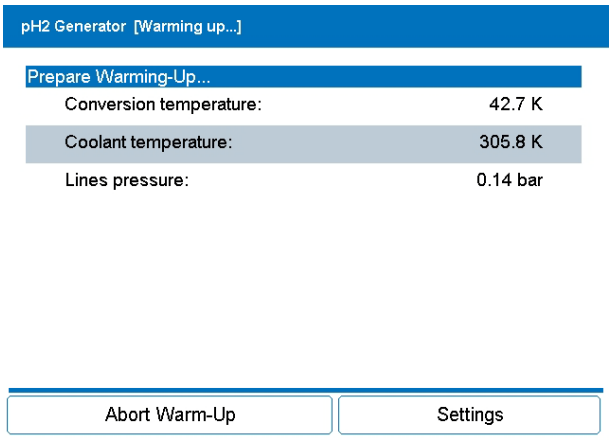

Figure 7.22: Warm-up Progress Screen with Abort Function

8 Troubleshooting

The following table list potential problems and indicates possible causes and remedies.

Problem	Cause	Remedy
High H_2 gas alarm.	Internal hydrogen gas leak.	Shut off the hydrogen supply and contact Bruker service.
	Hydrogen gas sensor failure.	Contact Bruker service.
	Control electronics failure.	Contact Bruker service.
Main display does not come on.	Rear power switch is not on.	Switch on the rear power switch (section Mains Power Connection [▶39]).
	Mains fuses are blown.	Replace the line fuses with the same fuse type (section Fuses Replacement [▶60]).
	Controller or display failure.	Contact Bruker service.
System check does not pass.	The hydrogen inlet pressure is out of range.	Adjust the inlet pressure.
	Low coolant level in reservoir.	Fill the coolant (section Cryocooler Coolant Circuit [▶58]).
	All other system check failures.	Contact Bruker service.
No coolant flow detected.	Coolant pump failure.	Contact Bruker service.
	Coolant flow sensor failure.	Contact Bruker service.
Cryocooler does not start.	Cryocooler fuse blown.	Check or replace the cryocooler fuse (see Warm Up [▶53]).
	Cryocooler power supply failure.	Contact Bruker service.
Cannot reach the target conversion temperature.	Poor vacuum in cryostat.	Execute a warm-up (section Warm Up [▶53]), then a purge (Purge Function [▶48]). If the problem persists, contact Bruker service.
	Conversion chamber temperature sensor failure.	Contact Bruker service.
	Cryocooler failure.	Contact Bruker service.
Coolant temperature is too high.	Cryocooler overheating.	Contact Bruker service.
	Low coolant level.	Refill coolant reservoir (see section Cryocooler Coolant Circuit [▶58]).
	Radiator dirty.	Clean the radiator with a vacuum cleaner (see section Cleaning Instructions [▶58]).
	Incorrect fan speed.	Contact Bruker service.

Problem	Cause	Remedy
	Defective water pump.	Contact Bruker service.
	Coolant temperature sensor failure.	Contact Bruker service.
When ready, cannot deliver parahydrogen in continuous flow mode.	Conversion chamber blockage.	Execute a warm-up (section Warm Up [▶53]), then a purge (Purge Function [▶48]).
	Outlet valve malfunction.	Contact Bruker service.
Batch delivery container evacuation fails.	Leak in container connections.	Check / remake all connections to the container.
Time out while filling container.	Conversion chamber is blocked.	Execute a warm-up (section Warm Up [▶53]), then a purge (Purge Function [▶48]).
Repeated conversion chamber blockages.	H_2 gas purity problem.	Use high purity H_2 gas: 99.999% or better.
	Purge not executed prior to cool-down.	Execute a warm-up (section Warm Up [▶53]), then a purge (Purge Function [▶48]).
	Purifier cartridge saturated.	Contact Bruker service.

9 Maintenance

The BPHG 90 is a low maintenance instrument and the critical components have been selected to ensure a long operating life time. Periodic maintenance should be carried out on a yearly basis by trained service personnel. The periodic maintenance actions are summarized in the section Periodic Maintenance [▸57].

User maintenance is limited to cleaning the instrument, ensuring an adequate level of coolant for the closed loop cooling system is maintained, and replacing fuses when needed. A full description of these actions is given in section Hydrogen Gas Sensor [▸58].

9.1 Periodic Maintenance

Several system components of the generator (H_2 gas sensor, coolant pump, fans, H_2 purifier cartridge) may need a periodic maintenance. The instrument monitors the operating time of the active components and, when their service time interval has elapsed, maintenance warnings are shown (see section Operating Information [▸46]).

A first (non-critical) maintenance warning occurs when the operating time has reached 90% of the service interval of a component. After such a warning the instrument will continue to operate normally but maintenance warnings are repeated at preset intervals until the operating time has reached 100% of the service interval. At that point the instrument can no longer be operated until service has been carried out.

The periodic maintenance of the instruments should be carried out by Bruker Service. It is recommended to arrange a service visit immediately following the first maintenance warning.

9.1.1 Hydrogen Gas Sensor

The internal hydrogen gas sensor must be tested every 12 months by a trained service technician. It is located inside the cabinet on the rear panel over the vacuum pump.

9.1.2 Vacuum Pump

The vacuum (diaphragm) pump needs to be checked for wear and tear every 10000 operating hours (approximately 1 year of full time operation) and replaced if necessary.

9.1.3 Coolant Circuit

9.1.3.1 Coolant Replacement

The coolant level needs to be maintained by the user (see section Cryocooler Coolant Circuit [▸58]). The cooling circuit of the cryocooler will be cleaned and the coolant replaced during the annual maintenance.

9.1.3.2 Fans and Radiator

The coolant radiator fins need to be cleaned and the fans be checked for wear and tear during the annual maintenance. They will be replaced if necessary.

9.1.3.3 Coolant Pump

The coolant pump needs to be checked for wear and tear every 10000 operating hours (approximately 1 year full time operation) and replaced if necessary.

9.1.4 Gas Purifier Cartridge Replacement

The gas purifier cartridge must be replaced every 8000 hours (approximately 12 months of operation) or if the conversion chamber is often blocked by frozen contaminants. The BPHG 90 monitors the operating time of the instrument and issues a maintenance warning if the cartridge approaches its maximum life time.

9.1.5 Cryocooler

The cryocooler needs no special maintenance. It needs to be replaced every 50000 operating hours (more than 5 years of full time operation).

9.2 User Maintenance

9.2.1 Cleaning Instructions

Clean the cabinet and screen using a mild soap and water mixture. Use a soft cloth or sponge slightly moistened with the mixture. Note that the enclosure of the BPHG is not water tight and avoid using excessive amounts of cleaning fluid.

Do not use harsh cleaners, solvents, or detergents. Never spray the cleaning fluid directly onto the touch-screen.

Ensure the radiator on the rear panel does not become clogged with dust. It can be cleaned using a vacuum cleaner.

9.2.2 Cryocooler Coolant Circuit

The recommended coolant for the closed-loop cooling system of the BPHG 90 consists of distilled water with the addition of a small amount of algaecide to avoid organic contamination by algae.

The coolant system requires regular topping up. The instrument monitors the coolant level in the reservoir and issues a warning if it drops below a pre-set level. If the level is too low, the operation of the cryocooler is stopped. It is recommended to completely replace the coolant every **6 months**.

9.2.2.1 How to Drain the Cooling System

To drain the cooling system, proceed as follows:

1. Stop the generator operation and power off the device. Disconnect the power cord.
2. Remove the cover on the rear panel to gain access to the coolant reservoir.
3. Remove the cap of the coolant reservoir.

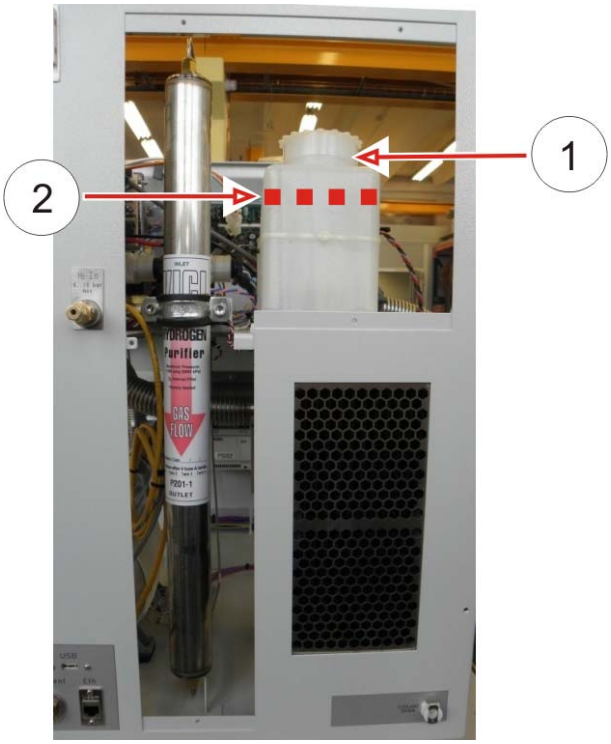

Figure 9.1: Coolant Reservoir

1	Coolant reservoir
2	Coolant high level

4. To collect the liquid, place a container of at least 1 liter volume under the drain and plug the drain tube with its fitting in the plug labelled “Coolant drain” (see the figure below).
   ⇨ The flow of coolant will start automatically.

Figure 9.2: Coolant Drain

See also

How to Drain the Cooling System [▶59]

9.2.2.2 How to Fill the Cooling Circuit

The procedure is similar to the drainage procedure:

1. Stop the generator operation and power off the device. Disconnect the power cord.
2. Remove the cover on the rear panel to gain access to the coolant reservoir (use a Philips screwdriver).
3. Remove the cap of the coolant reservoir.
4. Fill the reservoir with coolant up to high level (approx. 2 cm from top). If some coolant is spilled, clean and dry thoroughly the inner surfaces of the generator before switching on the generator.
5. Power up the unit and check if the coolant returns into the reservoir when the pump is on. The coolant pump is on whenever the cryocooler is in operation.
6. If the coolant flow is not visible in the reservoir, stop and restart the pump until the coolant return jet can be seen is the reservoir. Repeat this operation several times if necessary. After a complete drainage of the cooling system, the tubing may contain air bubbles and coolant may not circulate immediately. If this occurs add slightly more water in the reservoir.

9.2.3 Fuses Replacement

⚠ 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.

⚠ CAUTION

Risk of fire due to incorrect fuses usage

Replace the fuses only with the same type and electrical rating as indicated on the fuse replacement label.

9.2.4 Replace Power Line Fuses

Figure 9.3: Line Fuses Holder

1. Power off the generator and remove power cable from plug.
2. With a small flat screwdriver open the fuse holder.
3. Remove both fuses.
4. Install new fuses (P/N 2257).
5. Close the fuse holder.

9.2.5 Replace Cryocooler Controller Fuse

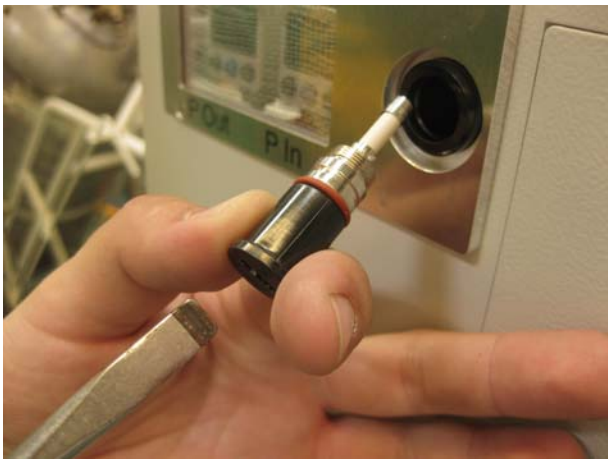

Figure 9.4: Cryocooler Controller Fuse

The power supply (red wire= +48V) of the cryocooler controller board is protected by a fuse located on the rear side of the cabinet.

1. Turn off the generator.
2. The fuse holder is located on the rear side of the generator.
3. Unscrew the fuse holder with a flat screwdriver, remove the fuse.
4. Install new fuse (P/N 1808869).
5. Close fuse holder.

9.2.6 Replace Cryostat Vacuum Gauge Fuse

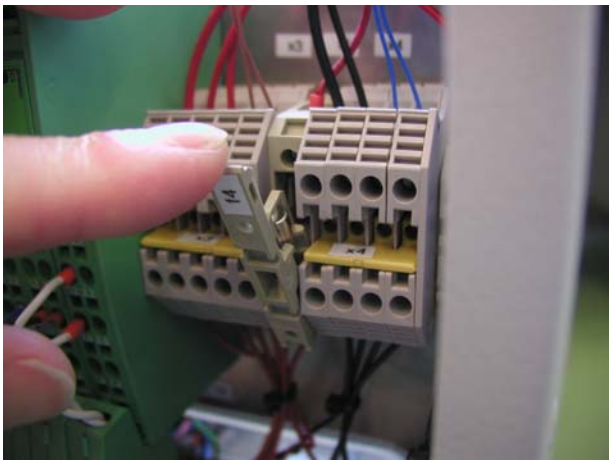

Figure 9.5: Vacuum Gauge Fuse

The vacuum gauge is protected by a fuse (F4) located on the connecting block beside the system green electronic modules.

1. Turn off the generator.
2. Pull open the fuse holder.

9.3 Spare Parts

Part number	Description	Comments
2257	Fuse 5 x 20 mm 4AT/250 V.	Power line fuse.
1808869	Fuse 5x20 mm 10.0AT/250 V.	Cryocooler controller fuse.
2271	Fuse 5x20 mm 1A F/250 V.	Vacuum gauge fuse (F4).
1802701	Hydrogen gas purifier cartridge.	Removes contaminants from inlet H_2 gas (moisture, oxygen, etc…).

Table 9.1: Spare Parts

10 Dismantling and Disposal

Following the end of its operational life, the BPHG must be dismantled and disposed of in accordance with the environmental regulations.

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 Biospin is not covered by your warranty.

10.1 Dismantling

Before dismantling:

1. Shut down the device and secure to prevent restarting.
2. Physically disconnect the power supply from the device.
3. Disconnect the gas supply line.
4. Remove hydrogen purifier cartridge, drain cooling circuit and dispose of in accordance with the environmental regulations.
5. Clean assemblies and parts properly and dismantle in compliance with applicable local occupational safety and environmental protection regulations.

10.2 Disposal

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

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

P/N	Description	Qty
3000	Power cable 3m 3x1.0 mm².	1
2257	Fuse 5 x 20 mm 4.0 AT.	2
1803847	Stainless steel gasket.	2
W131932	drain hose with nipple, tubing d=8mm black l=1m.	1
1806787	Personal H_2 gas detector with power supply.	1
W127310	Cd manual H_2 gas detector English.	1
W131933	Tubing l=10 meter with pump silencer.	1
1803111	Brass “Swagelok” tube fitting 1/8 inch.	2
1803121	Stainless steel tubing 1/8”, l=6m.	1
1807578	Stainless steel hose DN6 f1/8bsp-f1/8bsp l=4m.	1
1806418	Union fitting G1/8- G1/8” stainless steel conical.	1
39930	PTFE tape 12 mm.	1
W127477	Label "Danger Hydrogen".	2
1807106	Wrench 7/16”.	1
1808576	Wrench 1/2 x 9/16”.	1
1904	Straight male pipe coupling 8 mm-1/8” cylindrical.	2
W131498	BPHG technical manual Z33015.	1
1808869	Fuse 5 x 20 mm 10.0 AT.	1

Table 10.1: Accessories Set

Figure 10.1: Straight Male Pipe Coupling 8 mm-1/8” Cylindrical P/N 1904

Figure 10.2: Fuse 5x20 mm 4.0AT P/N 2257 2:

Figure 10.3: Fuse 5x20 mm 10AT P/N 1808869

Figure 10.4: Power Supply Cable 3m 3x1.0 mm^2 P/N 3000

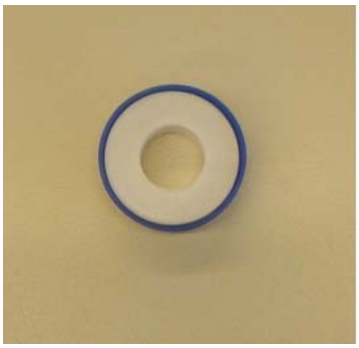

Figure 10.5: PTFE Tape 12 mm P/N 39930

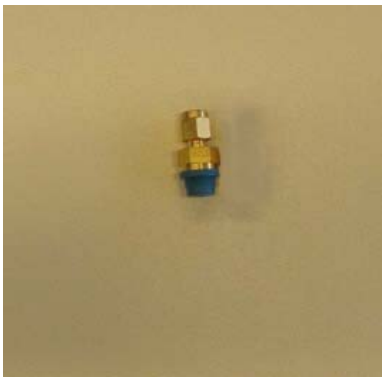

Figure 10.6: Brass Swagelok Tube Fitting 1/8“ P/N 1803111

Figure 10.7: Stainless Steel Tubing 1/8“ l=6m P/N 1803121

Figure 10.8: Stainless Steel Gasket P/N 1803847

Figure 10.9: Union Fitting G1/8“- G/8“ Conical P/N 1806418

Figure 10.10: Personal H_2 Gas Detector P/N 1806787

Figure 10.11: CD Manual of H_2 Gas Detector P/N W127310

Figure 10.12: Wrench 7/16“ P/N 1807106

Figure 10.13: Wrench 9/16“ P/N 1808576

Figure 10.14: Stainless steel hose DN6 F 1/8“ BSP F 1/8“ BSP l=4m P/N 1807578

Figure 10.15: Label Danger Hydrogen P/N W127477

Figure 10.16: CD Technical Manual BPHG Z33015 P/N W131498

Figure 10.17: Drain Hose with Nipple d=8 mm P/N W131932

Figure 10.18: Tubing l=10 m with Vacuum Pump Silencer P/N W131933

11 Contact

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Figures

Figure 2.1: BPHG 90 Front View 9
Figure 2.2: BPHG 90 Rear View 10
Figure 4.1: Rating Plate Example 28
Figure 4.2: H2 Gas Supply Panel P/N W126956 30
Figure 4.3: 2 p-H2 Storage Kit P/N W126957 30
Figure 6.1: H2 Gas Inlet Port 37
Figure 6.2: H2 Gas Vent Outlet 38
Figure 7.1: Touch Screen for Instrument Operation 41
Figure 7.2: Parahydrogen Fraction Function of Conversion Temperature 41
Figure 7.3: Power Switch and Panel LED's 42
Figure 7.4: System Check with Low Coolant Level Warning 43
Figure 7.5: Hydrogen Gas Sensor Warming Up 43
Figure 7.6: Exhaust Gas Warning Message 44
Figure 7.7: Stand-by Screen with Warm-up Conversion Chamber 44
Figure 7.8: System Settings and User Settings Interface 45
Figure 7.9: User Settings Menu with Adjustable Parameters 46
Figure 7.10: Operation Information Screen 46
Figure 7.11: Example of Non-critical Warnings 47
Figure 7.12: Critical Maintenance Warning 47
Figure 7.13: Purge in Progress Screen 48
Figure 7.14: Cool Down Screen 49
Figure 7.15: Ready Screen with Access to Main Operating Functions 50
Figure 7.16: Continuous Flow Progress Screen 50
Figure 7.17: Container Connection Dialogue Window 51
Figure 7.18: Container High Pressure Warning Screen 52
Figure 7.19: Batch Delivery Progress Screen 52
Figure 7.20: End of Batch Delivery Process 53
Figure 7.21: Warm-up Dialogue and Information Screen 54
Figure 7.22: Warm-up Progress Screen with Abort Function 54
Figure 9.1: Coolant Reservoir 59
Figure 9.2: Coolant Drain 59
Figure 9.3: Line Fuses Holder 61
Figure 9.4: Cryocooler Controller Fuse 61
Figure 9.5: Vacuum Gauge Fuse 62
Figure 10.1: Straight Male Pipe Coupling 8 mm-1/8” Cylindrical P/N 1904 64
Figure 10.2: Fuse 5x20 mm 4.0AT P/N 2257 2: 65
Figure 10.3: Fuse 5x20 mm 10AT P/N 1808869 65
Figure 10.4: Power Supply Cable 3m 3x1.0 mm2 P/N 3000 65
Figure 10.5: PTFE Tape 12 mm P/N 39930 66
Figure 10.6: Brass Swagelok Tube Fitting 1/8“ P/N 1803111 66
Figure 10.7: Stainless Steel Tubing 1/8“ l=6m P/N 1803121 66
Figure 10.8: Stainless Steel Gasket P/N 1803847 67
Figure 10.9: Union Fitting G1/8“- G/8“ Conical P/N 1806418 67
Figure 10.10: Personal H2 Gas Detector P/N 1806787 67
Figure 10.11: CD Manual of H2 Gas Detector P/N W127310 67

List of Figures

Figure 10.12: Wrench 7/16“ P/N 1807106 68
Figure 10.13: Wrench 9/16“ P/N 1808576 68
Figure 10.14: Stainless steel hose DN6 F 1/8“ BSP F 1/8“ BSP l=4m P/N 1807578 69
Figure 10.15: Label Danger Hydrogen P/N W127477 69
Figure 10.16: CD Technical Manual BPHG Z33015 P/N W131498 69
Figure 10.17: Drain Hose with Nipple d=8 mm P/N W131932 69
Figure 10.18: Tubing l=10 m with Vacuum Pump Silencer P/N W131933 70

Tables

Table 4.1: Technical Data: General Information 27
Table 4.2: Electrical Connection Values 27
Table 4.3: Inlet Gas Values 27
Table 4.4: Outlet Gas Values 27
Table 4.5: Operating Environment 28
Table 4.6: Cooling Down 28
Table 4.7: Warm Up 28
Table 5.1: Symbols on the Packaging 32
Table 9.1: Spare Parts 62
Table 10.1: Accessories Set 64

List of Tables

Index

B

Batch Delivery 49
Blockage 48

C

Capillary Tube 37, 37
Continuous Flow 49
Conversion Chamber 10, 53, 53
Conversion Chamber Blockage 56
Conversion Temperature 41
Coolant drain 59
Coolant Radiator 58
Coolant Reservoir 60
Coolant Temperature 55
Cool-down Process 49
Cooling Circuit
    Filling the cooling circuit 60
Cooling Pump 60
Copyright 11
Cryocooler 9
Cryocooler Controller Fuse 61
Cryostat Vacuum 48

D

Diaphragm Pump 57
Disposal 63
Drain Hose with Nipple 64, 69

E

Error LED 42
Ethernet 39

F

Fume Cupboard 43

G

Gas Delivery 51
Gas Generator 36
Gas Purifier Cartridge 58, 62

H

H2 Gas Detector 64
H2 Supply Panel 29
Hydrogen Gas
    Source 36
Hydrogen Gas Sensor 57
Hydrogen Gas Supply 9
Hydrogen Sensor Test 43

I

inlet pressure 50

L

Laboratory Personnel 19

M

magnetic field 21
Main display 55
Maintenance Warning 45, 57

N

Noise 49

P

Parahydrogen 10
Periodic Maintenance 57
Personal H2 gas Detector 67
Personal Protective Equipment 20
Personnel Qualifications 19
PH2 Storage Kit 30
Plastic Tubing 43
Power Switch 39
Product Liability 17
Purge 48, 56
Purge Procedure 48

R

Ready LED 42
Risk Assessment 17

Index

S

Safety Valves 38
seals 37
Service
    Menu 45
Service settings 45
Solenoid Valves 9
SOP 18
Spare Parts 62
Standard Operating Procedure 18
Swagelok Fittings 37
System Settings 45

T

Terms and Conditions 11
Terms of Delivery 11
Timeout 46
Troubleshooting 55

U

Union Fitting 67
Untrained Personnel 10
USB port 39
User settings 45

V

Vacuum Chamber 48
Vacuum Gauge 62
Vacuum Gauge Fuse 62, 62
Vacuum Pump 9, 57

W

Warm-up 53
Warning Signs 35
wrench 64

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