NMR-MS-Bridge

User Manual

Version 002

Innovation with Integrity

NMR

This manual was written by

David Kilgour

P/N: H148345

DWG: 10000037827

For further technical assistance for this product, please do not hesitate to contact your nearest BRUKER dealer or contact us directly at:

Bruker Corporation
am Silberstreifen
76287 Rheinstetten
Germany
Phone: +49 721 5161 6155
FAX: +49 721 5171 01
E-mail: nmr-support@bruker.de
Internet: www.bruker.com

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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 understand 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.

1. This is the safety condition.

▶ 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.

1. This is the safety condition.

▶ 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.

1. This is the safety condition.

▶ This is the safety instruction.

NOTICE

NOTICE indicates a property damage message.

This is the consequence of not following the notice.

1. This is a safety condition.

▶ 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.

1. This is a safety condition.

▶ This is a safety instruction.

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

1.4 Font and Format Conventions

Type of Information	Font	Examples
Shell Command, Commands, "All what you can enter"	Arial bold	Type or enter **fromjdx zg**
Button, Tab, Pane and Menu Names "All what you can click"	Arial bold, initial letters capitalized	Use the **Export To File** button. Click **OK**. Click **Processing**…
Windows/ Dialog Windows	Arial, initial letters capitalized	The Stacked Plot Edit dialog will be displayed.
Path, File, Dataset and Experiment Names **Data Path Variables** **Bruker Trademarks**	Arial Italics	*$tshome/exp/stan/nmr/lists* *expno, procno,* *IconNMR™,* *TopSpin™,* *XWIN-NMR™*
Parameters	Arial in Capital Letters	VCLIST

Program Code
Pulse and AU Program Names
Macros
Functions
Arguments
Variables
	Courier	`go=2`
		`au_zgte`
		`edmac`
		`CalcExpTime()`
		`XAU(prog, arg)`
		`disk2, user2`

AU Macro	Courier in Capital Letters	`REX` `PNO`

Table 1.1: Font and Format Conventions

About

2 Introduction

2.1 Concept

The unit is designed to supply a simple make-up flow interface for Hyphenation experiments in the field of LC-NMR-MS.

Intended Use

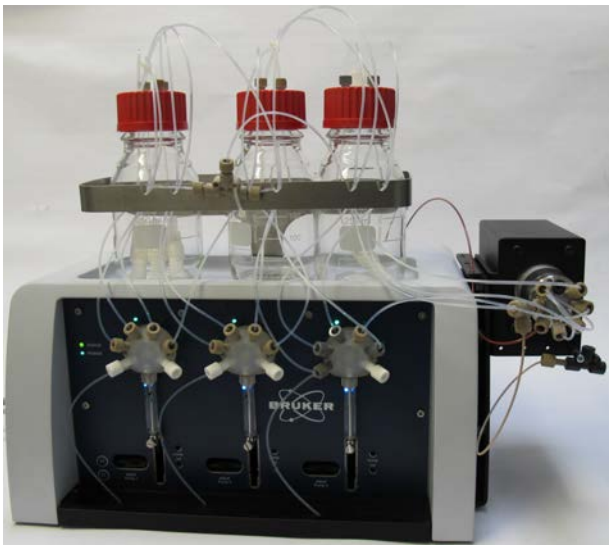


Figure 2.1: NMR-MS_Bridge

The unit consists of 3 syringe pumps each with its own selection valve mounted on a stainless steel chassis and covered with a shielded plastic housing. The 3 pumps are controlled from a control board. In addition there is an external multiposition valve which is also controlled from the control board. It is connected to the bottom left hand side of the unit. A single shielded cable carries both the power and the control data (RS485) to the valve actuator.

The pumps, the valve and the control board are all powered by a single wide input range 100W power supply.

A storage area for the liquids being pumped, together with a waste flask, is available at the top of the unit. The 3 flasks sit in a tray (a depression in the plastic housing) intended to catch any leaking fluid. A safety holder prevents the flasks from tipping over. The flasks have caps into which the supply tubing is screwed. This further reduces the likelihood of fluid spillage and also ensures that the contents are kept as uncontaminated as possible. The air intake on the solvent cap is populated with a filter to further reduce the contamination risk.

The liquid intakes to the valves is also filtered.

Beneath the syringes is a drip tray to catch any drips from the syringes or the valves. The base of the tray slopes down to the left. There is an outlet to a larger waste tube which must be connected to an external waste bottle. There is also an outlet from the base of the upper leak tray which is connected to this external waste tube. At the outlet from the unit a leak sensor detects if any significant leak is taking place. A leak is simply reported further to the external control system (normally the Bruker HyStarProgram), which must react appropriately.

There should be no leakage when the unit is working correctly!

The flowrates involved are so low (Max. 180µL / Min) that only a large leak will be detected. An occasional drop will simply evaporate long before it can even reach the leak sensor.

As the unit usually pumps organic solvents (usually Acetonitril, Methanol, Isopropanol) those components coming into contact with the liquids must be resistant to it. The syringe and valve components have been specially selected to be suitably resistant.

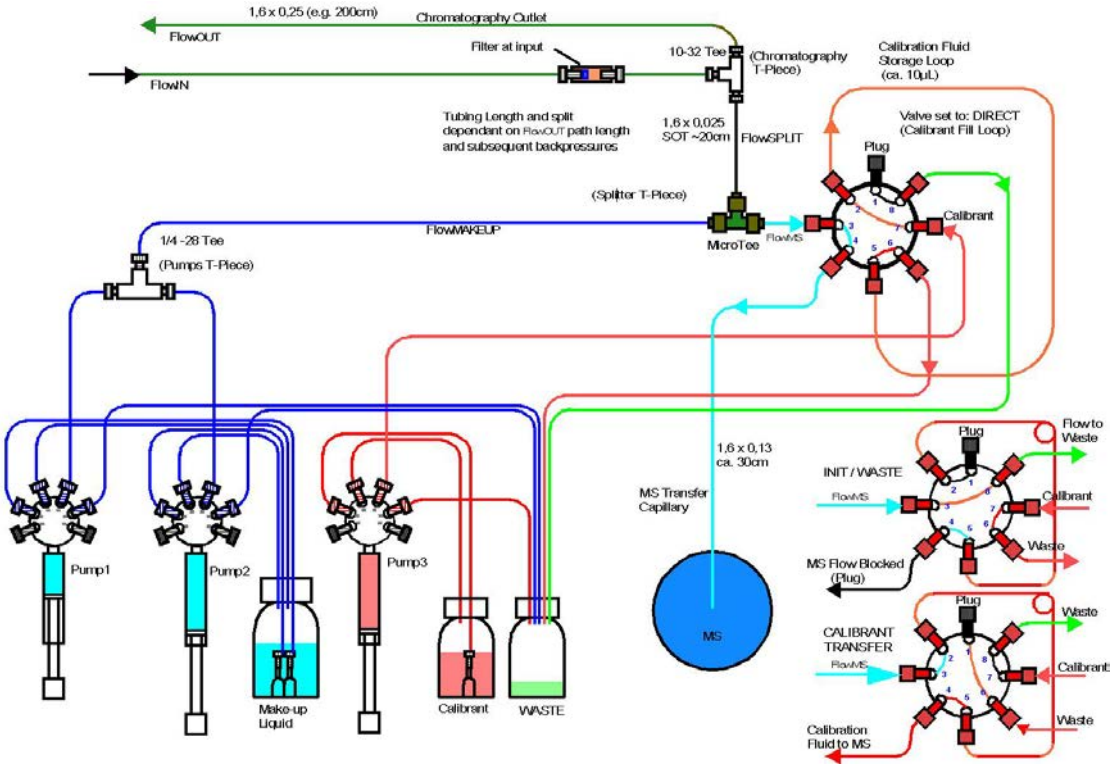


Figure 2.2: Tubing

The unit works as in interface between an NMR and an MS spectrometer during hyphenation experiments. (Hence the name NMR-MS-Bridge). The flow-in is split in an approx. 1:20 (to 1:40) ratio such that the low flow heads to the MS and the high flow out to chromatography (usually HPLC) and beyond. The splitter is simply a T-Piece with two outlets. The backpressure in the 2 outlets is the same but in the low flow direction very narrow capillaries are used to constrict the flow. The liquid in the high flow direction can be switched to a number of targets. It is important that the fluid reaches the MS (low flow direction) early enough for the fluid at the high flow side to be switched to these different paths as required. If the fluid arrives too late at the MS then any Mass peaks detected are also late and the fluid at the high side may already be past the valve due to switch it to a further measurement or storage path.

The NMR-MS-Bridge supplies a precise and consistent ‘make-up’ flow which is added to the low flow to push it faster to the MS. This flow is programmable and is set according to the precise system used and, of course, to the flowrate in use. The simply goal is for the MS peaks and the HPLC absorption peaks to be synchronous.

Pumps 1 and 2 operate sequentially to generate a constant flow. This flow is normally directed through the valve (in direct posn. See diagram) to the MS. Also in this position the calibration storage loop is filled with calibration fluid. In order to synchronise the system, at the beginning of each measurement the external valve is turned to the ‘calibrant transfer’ position. Here the contents of the calibrant storage loop are pumped into the MS. This generates a very specific MS peak. After the calibrant transfer the valve is restored to the ‘direct’ position and the measurements continue.

The external valve can either be mounted at the side of the unit (as shown in the picture) or mounted alone on its mounting bracket in close proximity to the MS input. As can be seen in the diagram, the tubing from the valve to the MS lies in the critical low flow path so depending on the space available near the MS system either the complete unit or just the external valve can be located close to the input.

2.2 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 is not covered by your warranty.

- The unit should only be installed in a suitably equiped laboratory where the personnel are trained in the various safety guidelines pertaining to working in the laboratory.
- Carefully unpack the unit. The NMR-MS-Bridge can be mounted differently according to the type of MS unit in use. It is important that the unit is mounted such that the output from the external multiposition valve to the MS is kept short. A nominal path length of 30cm should be assured. If there is insufficient space for the complete unit near enough to the MS, then the valve can be removed from the side of the unit [(see 'External Valve' on the Service Chapter)…… link benötigt] it alone can be placed near the MS input with the rest of the unit approx. 1m away. The tubing and cabling to the external valve are already set up to allow approx. 1m to 1.4m separation.
- With the unit mains switch (left, bottom rear) in the off position, connect the mains plug to the unit and plug it into a suitable mains socket (110-230V 50/60Hz AC). Connect your controlling PC (with HyStar already installed) to the unit using the Ethernet cable supplied or similar and via your Ethernet switch used to route Ethernet connections to the other systems under control of HyStar.
- You must use an adequate mains cable rated to at least 250V and which meets any local safety requirements. A suitable UL listed detachable mains cable is available in the accessory kit supplied with the unit.

Flush System Prior to Use

- Fill the Solvent and Calibrant flasks with your solvent and calibrant and mount them together with the waste flask in the secure area provided at the top of the unit (See the section *Refilling the Storage Flasks [▷ 73]*). Move the solvent return tubing from pumps 1 and 2 from the solvent flask to the waste flask. These tubes are connected to the pump valve port 'A' – the 3rd port from the left of the syringe going clockwise. Similarly move the calibrant return tube at pump 3 to the waste flask. This ensures that, during the flush operation, no 'contaminated' liquid is returned to the supply flasks. Additionally connect the tube from port 3 of the external valve, which would normally go to the MS, to a waste flask. Connect the Flow input (at the union) to the Chromatography output (at the T-Piece) to effectively block these 2 paths.
- Power on the unit, the front LEDs all blink until the pumps and the external valve are found. After ca. 20 secs the unit initializes the 3 pumps and then the external valve. On the PC start your browser off choice (screenshots are from Internet Explorer). Enter the default URL 192.168.254.42 for the unit, see chapter *Embedded Web Server [▷ 47]*. The following page is displayed:

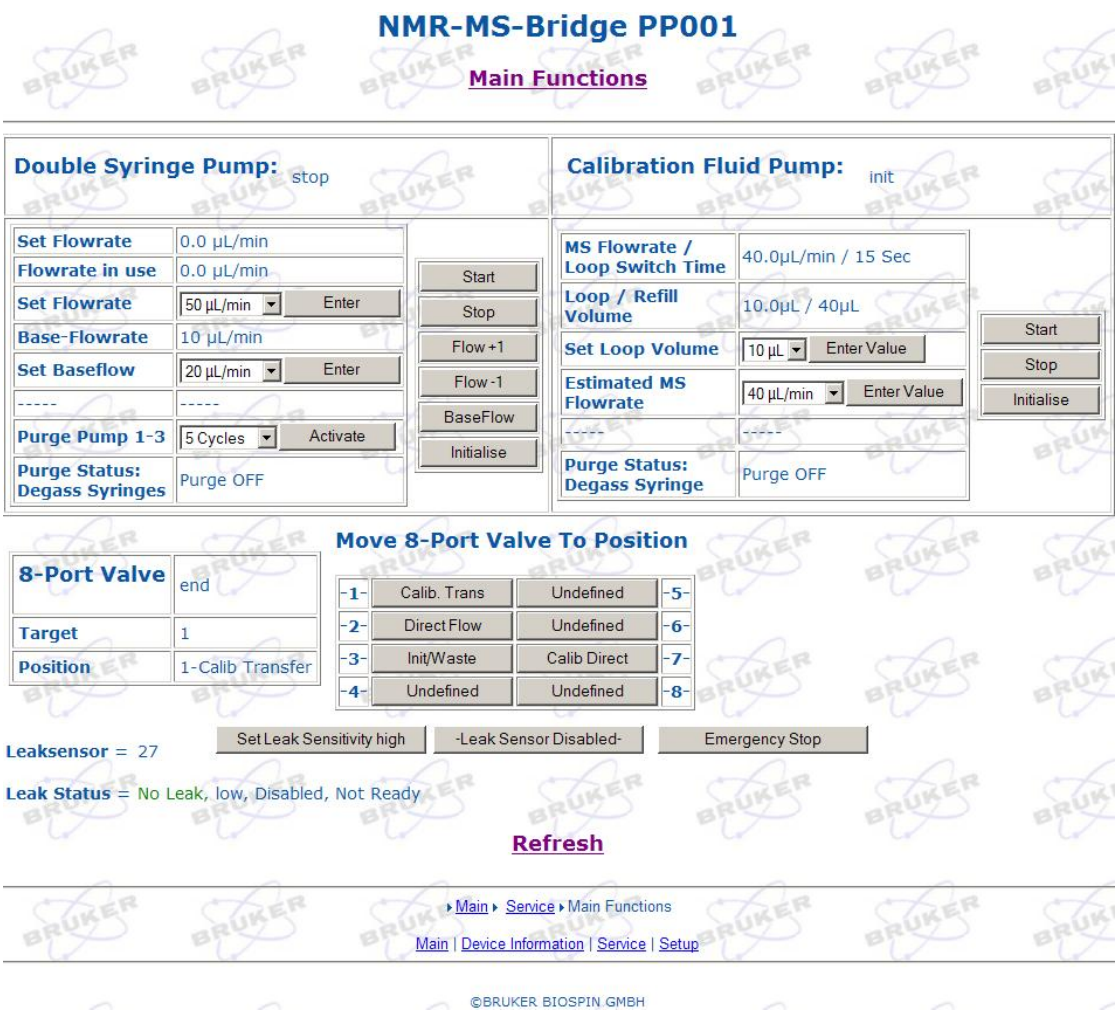


Figure 2.3: Service_Page

- Before running the unit with the MS it has to be purged of the test liquid and any gas which may have got into the system during storage or transport.
- At the left hand side, adjacent to the ‘Purge Pump1-3’ Text, select 10 Cycles then click on the ‘Activate’ button.
- Now in the fields for both the ‘Double Syringe Pump’ and the ‘Calibration Fluid Pump’ click on the ‘Start Purge’ buttons. At the ‘Double Syringe Pump’ field, at ‘Set Flowrate’ select 180µL /min and click on the adjacent ‘Enter’ button.
- All 3 pumps will now run the syringe purge operation for 10 syringe cycles. For each pump, this simply runs the following sequence: The pump valve ports from the left of the syringe clockwise: F,E,A,B,C,D.
  - Set the pump valve to position ‘F’ (connects syringe to the supply liquid).
  - Move the syringe down filling it with the contents of the supply flask.
  - Set the pump valve to position ‘A’ (connects the syringe to supply return –waste here)
  - Moves the syringe up forcing out gas at the top of the syringe
  - Once the syringe is near the top, the pump is re-initialised

- This sequence is repeated for 10 cycles and ensures the path from the supply flasks to the pumps and the syringes are properly washed and purged of air.

- When both operations are finished (about 10 minutes) once again, adjacent to the ‘Purge Pump1-3’, select 10 Cycles then click on the ‘Activate’ button. Click on ‘refresh’ to keep the display up-to-date.
- Click the Degass/Fill button to toggle between the purge syringe mode and the fill loop mode (to wash the system path). Here we want to wash the system path. On the bottom field in the ‘Calibration Fluid Pump’ area you will see ‘Purge Status: Fill Loop.
- At the ‘Move 8-Port Valve to Position’ field click on 3 ‘Direct Flow’ to move the external valve to position 3. In the ‘Calibration Liquid Pump’ field click on Start Purge. This runs similarly to before but now as the syringe moves up the calibration fluid fills the path from the pump to the external valve, then through the loop at the external valve and out to waste.
- Once this is done (again about 10 minutes) click on ‘Stop Purge’ at both the ‘Double Syringe Pump’ and ‘Calibration Fluid Pump’ Fields. At both the ‘Double Syringe Pump’ and ‘Calibration Fluid Pump’ fields, click on ‘Initialise’ to re-initialise all 3 pumps.
- This takes about 1 min. Then at the ‘Double Syringe Pump’ select a Flowrate of 100µl/min and enter then click on start to start the pumps.
  - One pump pumps out through the pump valve port ‘B’ through the T-Piece and on and through the external valve port 3, out port 4 to the Tubing to the MS (in a waste flask at present).
  - As it nears the end the other pump starts to pump out.
  - The first pump is re-initialised the refills from the supply flask.

The sequence repeats until stopped.

- This washes the path from the unit to the rotary valve. Allow this to run for about 10mins then click on **Stop**.
- Connect the tubing (see diagram) to your flow source and chromatography.

Compass/HyStar Software

The Bridge is fully compatible with the current software Compass 1.2/1.3 including HyStar 3.2 SR3

Differences towards previous model BNMI-HP

This differences are mainly of interest in the case of an instrument exchange.

- Exchange of BNMI-HP <-> NMR MS Bridge is possible without any changes in software or firmware
- Some rarely used features in the HyStar software are no longer supported with the NMR MS Bridge, some functionalities are implemented in a different way
  - The flow rate gradient of the MS makeup pump is not supported (entries in HyStar timetable are simply ignored, end flow rate is used over whole analysis time)
  - In the Flow Injection window in combination with the BPSU-36/2 it is not possible to run a flow-injection MS analysis in parallel to the NMR analysis.
  - A calibration process started from HyStar requires that HPLC pump is switched on (see below)
- Calibration is now performed by switching a loop with calibration liquid into the flow path rather than infusing the liquid into the source. For the setup in HyStar this means
  - The duration of the calibration process programmed in HyStar has the same meaning – No change.
  - The max. reasonable time for a calibration process now depends on the volume of the calibration loop and the flow rate (split + MS makeup flow) – check if loop volume is sufficient for the calibration time (nominal 10µL was good during validation)

- The flow rate of the calibrant as programmed in HyStar is ignored
- The HPLC pump must be switched on during the calibration process

Configuration and Calibration

- Start HyStar
- In the hardware setup
  - activate the BNMI-HP with the address 192.168.254.42

    Note the NMR MS Bridge is controlled as if it were a BNMI-HP
  - Define the offset for LC1 Detector and MS to 0sec
- Use the experiment Demonstrate NIP chromatography'
  - set the MS makeup flow to 0ul/min
  - Ensure that no peak trapping or loop storage is done
  - Set the SPE makeup flow to 3ml/min (or the highest value you are using)
  - Acquire UV and MS data
  - Run a chromatography
  - Determine the offset of the MS and UV peaks for LC-SPE-NMR
  - If the MS peaks appear before the UV peaks you must check the installation.
  - If the MS peaks are aligned with the UV peaks – o.k.!
  - If the MS peaks are **later** then the UV peaks – rerun the experiments with different MS makeup flow rates until the retention time matches.
  - **Note: Typically MS peaks appear 3sec earlier if the MS makeup flow is increased by 10ul/min!**
- Determine the offset of the MS and UV peaks for standard HPLC
  - Repeat the procedure as above, but change the SPE makeup flow in the method to 0.01ml/min
    Note both MS makeup flow rates are typically between 0 – 100µl/min

Usage

HyStar Method

In the setup menu of the method program… set…

- The required MS makeup flow to align the MS and UV chromatograms. This depends on the type of experiment (LC-MS only or LC-SPE-MS see Calibration above)
- Do not enter other flow rate values in the timetable (they would be ignored)
- The initial valve position set direct if you immediately want to switch the split flow to the MS when a method is loaded
- The time point and duration of the calibration, typically at 0.2min for 0.1min. The flow rate of the calibration is irrelevant (leave it at 10ul/min)

Acquisition Window

- Switch the HPLC pump on
- If not already done in the method, switch the unit to direct to check if the MS shows stable conditions
- For the first usage after power-up perform a calibration and check that calibrant arrives in the MS (HPLC pump must be on)

Analysis

- The unit switches the valve to the programmed position and the MS makeup pump starts with the programmed flow rate.
- At the defined time point the calibration loop is be switched into the flow path and the calibrant within is eluted into the MS source.

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

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 the chapter Contact 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.

2.7 EC Declaration of Conformity

EG Konformitätserklärung
EC-Declaration of Conformity
Declaration de Conformité – CE
Bruker BioSpin Group

Der Unterzeichner, der den nachstehenden Hersteller vertritt:
The undersigned, representing the following manufacturer:
Le signataire, qui représente le producteur suivant:

Hersteller / Manufacturer / Producteur: **Bruker BioSpin GmbH**
Anschrift / Address / Adresse: Silberstreifen 4, 76287 Rheinstetten, Germany

erklärt hiermit, dass die... / herewith declares that... / déclare par la présente que le ...

Produkt Serie / Product Series/ Série de Produit **NMR MS Bridge**
Produkt ID / Product ID / ID de Produit: **H139366**

...in Übereinstimmung mit den Bestimmungen der nachstehenden EG-Richtlinien (einschließlich aller zutreffenden Änderungen) ist.
...is in conformity with the provisions of the following EC directives (including all applicable amendments).
...est conforme aux dispositions des directives européennes suivantes (y compris tous les amendements applicables).

2006/95/EG Niederspannungsrichtlinie (NS-RL)	2006/95/EC Low Voltage Directive (LVD)	2006/95/CE Directive Basse Tension (DBT)
Europäische Richtlinie betreffend elektrische Betriebsmittel zur Verwendung innerhalb bestimmter Spannungsgrenzen European Directive to electrical equipment designed for use within certain voltage limits LVD - Low Voltage Directive Directive européen au matériel électrique destiné à être employé dans certaines limites de tension		(früher: 73/23/EWG) (former: 73/23/EEC) (avant: 73/23/CEE)
Angewandet harmonisierte / Applied harmonized standards / Suite à des normes harmonisées applicables:		
EN 61010-1:2010		

2004/108/EG Elektromagnetische Verträglichkeit (EMV)	2004/108/EC Electromagnetic Compatibility (EMC)	2004/108/CE Compatibilité Electromagnétique (CEM)
Europäische Richtlinie über die elektromagnetische Verträglichkeit Directive relating to electromagnetic compatibility Directive Européen concernant la Compatibilité Électromagnétique		(früher: 89/336/EWG) (former: 89/336/EEC) (avant: 89/336/CEE)
Angewandte harmonisierte Normen / Applied harmonized standards / Suite à des normes harmonisées applicables:		
EN 61326-1:2013	EN 61000-3-2:2010	EN 61000-3-3:2009

Rheinstetten , 16.12.2013
Deutschland / Germany / Allemagne

Dr. Ulrich Braumann
Produktverantwortlicher / Produkt Manager / Chef de Produit

Diether Maier
Technischer Direktor / Technical Director / Directeur Technique

2013-12-04 CE NMR MS Bridge.docxNMR MS Bridge Revision 00/BBIO Group

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 General

Before you start any repair inside of 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 check 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 in this manual. The NMR-MS-Bridge must only be used to supply a makeup flow in an LC-NMR-MS environment. Intended use also includes compliance with all specifications within this manual. Any use which exceeds or differs from the intended use and the instructions of this manual shall be considered improper use and may impair the safety of operation. No claims of any kind for damage will be entertained if such claims result from improper use.

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.

- ▶ Unauthorized persons must be kept away from the danger and working zone.
- ▶ If in doubt, address the persons in question and ask them to leave the danger and working zone.
- ▶ 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.

Handling of the Flasks

⚠ CAUTION

Spillage Risk When Refilling/Replacing Liquid Storage Flasks!

The storage flasks can contain dangerous solvent (acetonitrile /methanol). These flasks should be refilled/replaced as described in this manual.

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

Refer to the section *Refilling the Storage Flasks [▶ 73]* 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.

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

General Workplace Hazards

Electrical Current

⚠ WARNING

Danger of injury from electrical shock!

Life threatening shock may result when the housing is opened during operation.

- ▶ Only qualified personnel should open the housing.
- ▶ Disconnect the device from the electrical power supply before opening the device. Use a voltmeter to verify that the device is not under power!
- ▶ 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.

- ▶ Clean the device before maintenance work and/or returning to Bruker for repair.
- ▶ Prepare a list of materials in which the device came into contact.
- ▶ 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.

3.7.1 General Workplace Dangers

Unit Overheating

NOTICE

Material damage hazard from unit overheating.

Material damage is unlikely but may occur if the unit overheats as a result of a blocked air inlet and/or outlet. The unit has both a temperature sensor and a fan monitor. If the fan is stopped a warning is generated and if the temperature exceeds the upper limit (default 42°C) the pumps and valves are simply stopped.

- ▶ The unit must be mounted on a level and even surface.
- ▶ At the rear of the unit there must be at least a 1 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.

- ▶ The mains connector must be easily accessible at all times.

⚠ 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.

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

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.

- ▶ Before working on the specified components, ensure that they have been completely disconnected from the power supply.
- ▶ 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.

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

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.

- ▶ 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 Danger from Chemical Substances

WARNING

Danger of injury from contact with NMR solvents!

The solvents used are potentially hazardous.

- ▶ Only personnel trained in the handling of these solvents may work at or near the system.
- ▶ Always wear suitable safety clothing and safety glasses when working at or near the system.

⚠ DANGER

Danger of injury from glassware breakage!

Broken glassware may cause minor injuries or material damage, but may also result in a life threatening situation if hazardous substances are used.

- ▶ If glassware breaks, refer to the corresponding precautions and cleaning/disinfection instructions.
- ▶ Wear protective equipment.
- ▶ Perform all tasks with the glassware carefully.
- ▶ **The laboratory supervisor is responsible for:**
- ⇨ Establishing and enforcing standard sample handling and cleaning procedures.
- ⇨ Establishing and enforcing the use of protective clothing and equipment.
- ⇨ Training laboratory personnel.
- ⇨ Preparing an emergency plan.

⚠ WARNING

Danger of injury from vapor formation!

During the work process, highly flammable vapors may form. During the work process, vapors may form which cause serious injury if inhaled.

- ▶ Never have an open flame near the unit or any of your solvent flasks. Before handling the unit ensure you discharge any electrostatic buildup you have to a safe ground. This is to avoid electrostatic sparks in an area where solvents are used.
- ▶ Only install the device in a well-ventilated room or ensure that an extractor is fitted.

NOTICE

Material damage hazard from material contact with NMR solvents!

Material damage may result when the device comes in contact with NMR solvents.

- ▶ Follow instructions provided in the manual for correct handling of solvents.
- ▶ If surface damage should occur, contact Bruker for repair of damaged parts.

3.8 Environmental Protection

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.

- ▶ Always observe local environmental regulations regarding handling and disposal of pollutants.
- ▶ 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.

3.9 Spare Parts

Loss of Guarantee

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

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

4 Technical Data

4.1 General Information

Data	Value	Unit
Weight (Flasks empty)	11	kg
Width (excluding connector, incl. valve)	40	cm
Depth	22	cm
Height (unit alone)	23.5	cm
(Height) With Bottles and holder	32	cm

Table 4.1: Technical Data: General Information

4.2 Connection Values

Electrical

Data	Value	Unit
Voltage	110-230	V AC
Apparent power consumption, maximum	100	VA
Circuit protection	2 x 2.0AT 250V	A
Frequency	50/60	Hz
DC Out (to external Valve)	24	V
DC Out (to external Valve)	2	A
Peak DC Out (to external Valve)	2.5	A
DC Out (Protection) Multifuse	2.5	A

Table 4.2: Electrical Connection Values

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 35 °C.

- Maximum relative humidity 80% for temperatures up to 31 or decreasing linearly to 50% relative humidity at 40 °C.
- MAINS supply voltage fluctuations within the stated voltage range.
- TRANSIENT OVERVOLTAGES up to the levels of OVERVOLTAGE CATEGORY II.
- Pollution degree 2.
- Protection class IP20.

Environment

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

Table 4.3: Operating Environment

4.4 Rating Plate

The rating plate is located at the power input and includes 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

Figure 4.1: Rating Plate

4.5 Fluids Used

3 x 250ml storage flasks are kept in a leakage bath on the top of the unit. The flasks are secured against tipping and the majority of the tubing is screwed into the caps. The leakage bath has a liquid outlet which is connected to the drip tray. This in turn has a liquid outlet which you must connect to a large external waste container with the tubing supplied.

Flask 1 (mounted on the far left of the unit)

- Contains the solvent to be pumped by pumps 1 and 2. This is usually acetonitrile or methanol. Other solvents may be used, however, it is the user's responsibility to ensure that the solvent is compatible with materials used in the construction of the unit (see below).

Flask 2 (mounted in the centre)

- The waste outlets from the pumps and the valve are fed here.

Flask 3

- Contains the calibrant (an aqueous salt solution which gives very recognizable MS peaks)

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 pump operation.

Flasks	Glass
Flask Inserts	POM (Vapour and occasional Contact)
Tubing	Teflon, FEP, PEEK
Fitting Seals	Teflon
Valves	Kel-F, Teflon
Syringes	Glass, Teflon

Table 4.4: Materials used in the construction

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

Main Outer Housing	Polyurethane RIM SP-V0
Front Panel	Polyester on a Stainless Steel plate
Rear Panel	Stainless Steel
Inner Casing	Stainless Steel (painted)
Drip Tray	Black Anodized Aluminium

Table 4.5: Materials with occasional contact with the fluids

- 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, whereby the pressure in the syringe exceeds 5 bar, the pump 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.

4.6 Spare Parts

User Serviceable Spare Parts	Bruker PN	Description
Flask 250ml	86136	250ml GL45 Schott Lab. Flask
Inlet Filter (in flask)	86135	Upchurch A446
Syringe	1820587	Flexfluidics 100µL 300117TCV3
Pump Valve	88808	Kloehn 6 Port Valve 30207
External Valve	1814451	VICI 8-Port + Drive C5H-0008EUDFXY-BRU
Mains Fuses	2254	2A / 250V T (slow blow

Table 4.6: User Serviceable Spare Parts

5 Design and Function

5.1 Overview

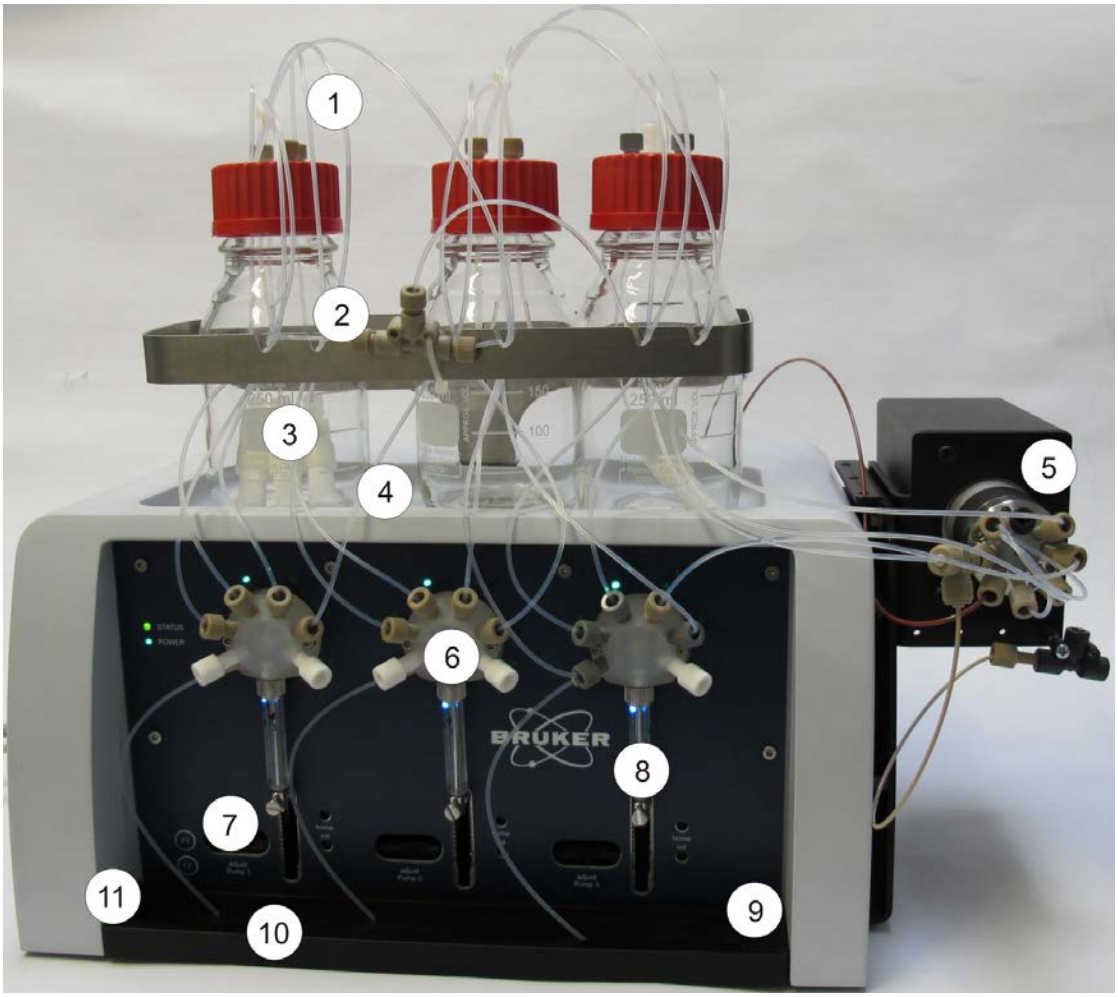


Figure 5.1: NMR-MS Bridge

1	Teflon FEP Capillary Tubing	7	Pumps (left to right: pump1, pump2, pump3)
2	Tilt protection for flasks	8	Syringes
3	Storage Flasks (left to right: make-up solvent, waste, calibrant liquid)	9	Front Panel
4	Leakage bath	10	Drip Tray
5	External Multiposition Valve	11	Leak Sensor at outlet
6	Pump selection valves		

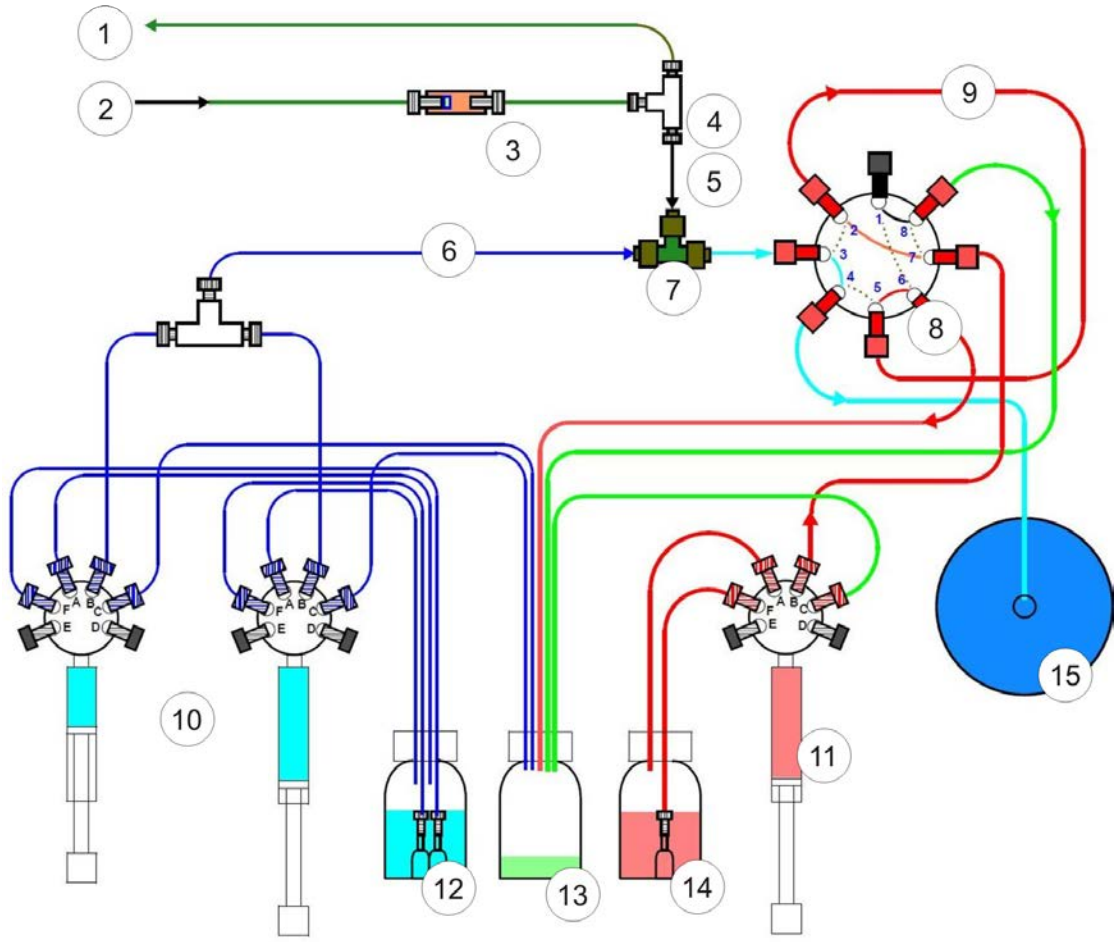


Figure 5.2: Flow Plan

1	Flow Out to Chromatography	9	Calibrant Loop
2	Flow In	10	Double Syringe Pump
3	Union with Input Filter	11	Calibrant Pump
4	Splitter T-Piece	12	Make-up Solvent
5	25µm ID Capillary Tubing	13	Waste
6	Make-up flow	14	Calibrant
7	Mixer T-Piece	15	Mass Spectrometer (MS)
8	External Rotary Valve		

5.2 Brief Description

The input flow is split at the splitter T-Piece. The flow towards the MS through the extremely narrow bore (25µm) is severely restricted. Depending mainly on the length of tubing here and the length of tubing in the chromatography direction the flows are split in an approx. 2% - 5% / 98 % to 95% ratio.

It is very important that the flow out to the chromatography and the flow to the MS arrive synchronously. This is achieved by having the flow to the MS, which would normally arrive later, increased by a programmable make-up flow.

In the NMR-MS-Bridge, Pumps 1 and 2 make up the double syringe pump. They operate sequentially to generate a constant make-up flow. This flow is mixed with the 2% to 5% flow from the splitter and directed through the valve (in direct posn. See diagram) to the MS.

Also in this position the calibrant loop is filled with calibration fluid. In order to synchronise the system, at the beginning of each measurement the external valve is turned to the ‘calibrant transfer’ position (the valve connections are shown as dotted lines). Here the contents of the calibrant storage loop are pumped into the MS. This generates a very specific MS peak. After the calibrant transfer the valve is restored to the ‘direct’ position and the measurements continue.

5.3 Unit Description

The unit consists of 3 pumps, a power supply and a controller board mounted on a stainless steel chassis. This, in turn, is covered by an EMI shielded plastic housing. The plastic housing has a secure storage area for the flasks, a leak bath and a drip tray. Any leak fluid is directed out past a leak sensor in the drip tray to an external waste container (not supplied).

An external valve directs either the sample + make-up flow or the calibrant liquid to the MS. The external valve can either be mounted on its bracket at the side of the unit or the bracket with the valve can be separately situated at a suitably close distance to the MS input.

The unit is intended to be controlled from within Bruker’s HyStar program. The unit is the successor to the BNMI-HP and is treated in HyStar exactly as if it were a BNMI-HP.

LEDs

On the front panel at each of the pumps are 2 sets of status LEDs. These give a quick visual feedback regarding the status of the unit even in daylight when viewed from a distance.

There are 6 green LEDs around the 6 Port valve. A lit LED adjacent to a port indicates that this port is connected to the syringe. Behind each syringe is a column of blue LEDs. When the syringe is moving up (pumping out fluid) the LEDs move upwards in a chain. When the syringe is moving down (sucking in fluid) the LEDs move downwards in a chain. When the syringe is stationary the lit LEDs show an approximate indication of the syringe position.

Top left on the front panel are a power (green when the unit is under power) and a red/green status LED. The status LED is normally green but turns red should an error occur.

5.4 Connections

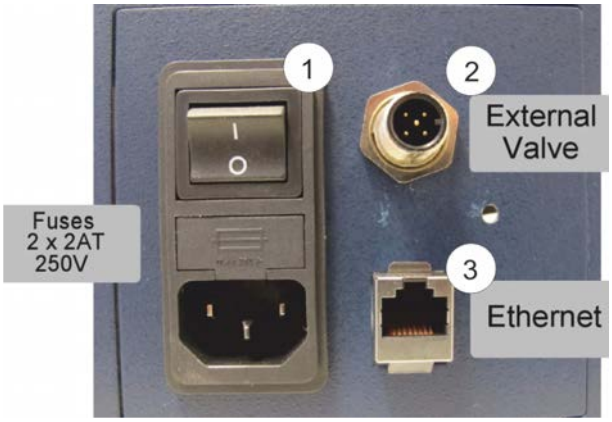


Figure 5.3: Connections

1	Mains Connector
2	External Valve
3	Ethernet Connector

Mains Connector

You must use an adequate mains cable rated to at least 250V and which meets any local safety requirements.

A suitable UL listed detachable mains cable is available in the accessory kit supplied with the unit.

External Valve

Use only the connector cable supplied to connect this output to the external valve.

Ethernet Connector

Connect this using a suitably certified ethernet cable (supplied in the accessory kit) to your controlling PC (over an ethernet switch).

6 Transport, Packaging and Storage

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 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 Operation

7.1 Function of Unit

The unit consists of a *Splitter* set up to split the incoming sample flow into two paths: a high flow out for further chromatography analysis and a low flow (2 - 5% of total flow) to the MS.

Splitter

The splitter consists of the Splitter T-Piece and the tubing leading to chromatography (high flow) and to the MS (low flow). The chromatography tubing has a much larger inner diameter (ID = 130µm) than the tubing to the MS (ID = 25µm). The backpressure over a piece of tubing is proportional to the flow through the tubing and to $1/(ID)^4$. By adjusting the tubing lengths in the two paths, the split ratio can be set. This is set in house to a nominal 3%. In practice this will vary depending on the final tubing lengths to the connected instrumentation and also on the back pressure built up within the connected devices - an increased back pressure (for example from SPE) decreases the split ratio. This does not usually matter very much: The conditions will not change during an experiment.

The unit has three syringe pumps. Pumps 1 and 2 work together to supply a constant 'makeup' flow to be added to the sample flow to the MS. This pushes the sample faster to the MS and allows an MS peak to be identified early enough to be used as a trigger for further LC-NMR-MS operations. Pump 3 supplies a calibration fluid to the MS to flag the start of an experiment. An 8 port rotary valve is used to switch between the functions.

The flow rate used for the makeup pumps is in the same order of magnitude as the MS flow rate (after the splitter) and is usually around 50µl/min.

See the **Tubing Connection Plan** on the following page.

The makeup flow is generated by two syringe pumps (Pump1 and Pump2) which pump sequentially to generate a constant flow. The solvent mixture used is normally the same as the solvent used by the LC pump.

Syringe Valve Connections.

Mounted on top of each syringe is a six position valve. The 6 ports are labelled A...F and correspond to valve positions 1...6. When the valve is set to a position, the corresponding port is connected to the syringe.

When a syringe is filling with solvent it is set to position 6 (port F), when pumping out it is set to position 2 (port B). Position 3 (port C) is connected to the waste flask and position 1 (port A) is used for pumping recycled solvent back to the solvent flask.

Syringe Pump Initialization (all 3 syringe pumps)

- Home operation
  - Syringe valve set to position 1 (solvent recycle to solvent flask)
  - The syringe moves up until the home sensor is found and stops. (the contents of the syringe are pumped back in to the solvent flask)
- The syringe pump controller loads the internally saved home position. The syringe is moved to position 0 (This is as far up as the syringe can move just before the syringe piston reaches the end of the syringe - this position is calibrated in the syringe pump hardware setup and must be re-calibrated whenever a syringe or syringe valve is replaced - see Syringe Pump Home Calibration in chapter *Preventive Maintenance [▶ 74]*).
- The syringe valve is set to position 6 (solvent flask - filter).

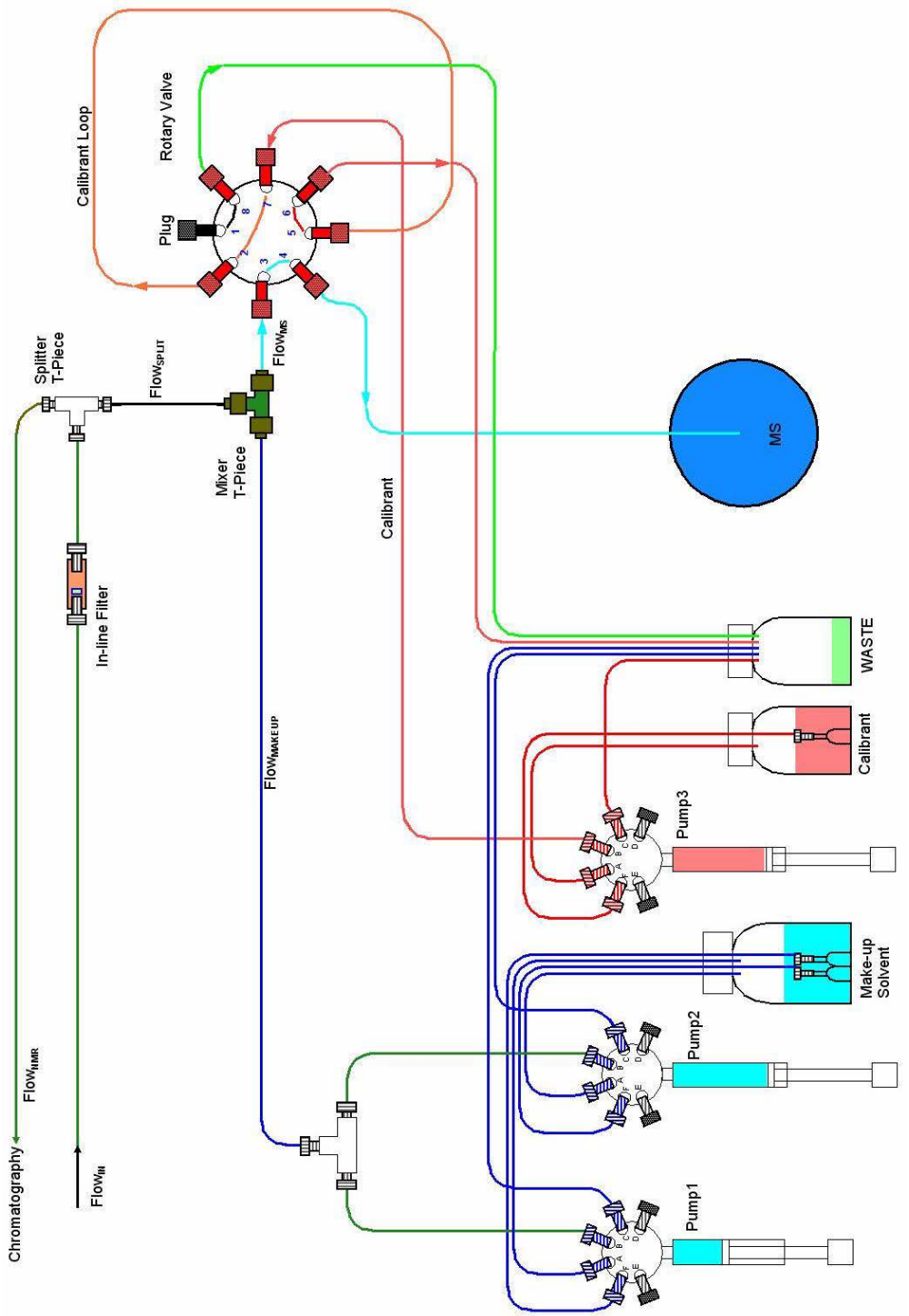


Figure 7.1: Tubing Connection Plan

- The syringe sucks in fully (step position 12000 = 100µl) and waits a few seconds.
- The syringe valve is set to position 1 (solvent flask - recycle)
- The syringe moves back up (step position 11800) pumping out approx 1.5µl.
  - This helps to clear any gas bubbles which may have collected at the top of the syringe.

A Pump Cycle - Syringe Pump1 and Pump2

In the following description Pump1 and Pump2 are interchangeable.

- Pump1 and Pump2 have both been initialized.
- On operation start, Pump 1 accelerates up to its programmed speed (= flow rate)
- The flow rate can be changed at any time.
  - A flow rate change just before pump changeover is first implemented as the 2nd pump starts to pump.
- Pump1 heads up towards position 1000 (this is before the home sensor) and its position is monitored.
- When Pump1 is within 5 seconds of the end (position 1000), the Pump2 operation is started.
  - Pump2 syringe valve moves to position 6 (output) and the syringe pump waits for a start signal.
- When Pump1 is very close to the end (dynamic, speed-dependent and defined by the pcxx value - see the embedded web server - setup) the start signal is sent to Pump2.
- Pump2 accelerates to the programmed speed as Pump1 brakes to a halt at position 1000. (Pump swap over period)
- Pump1 runs an initialization operation (described above) while Pump2 pumps solvent at the correct flow rate.
- This cycle continues until stopped.

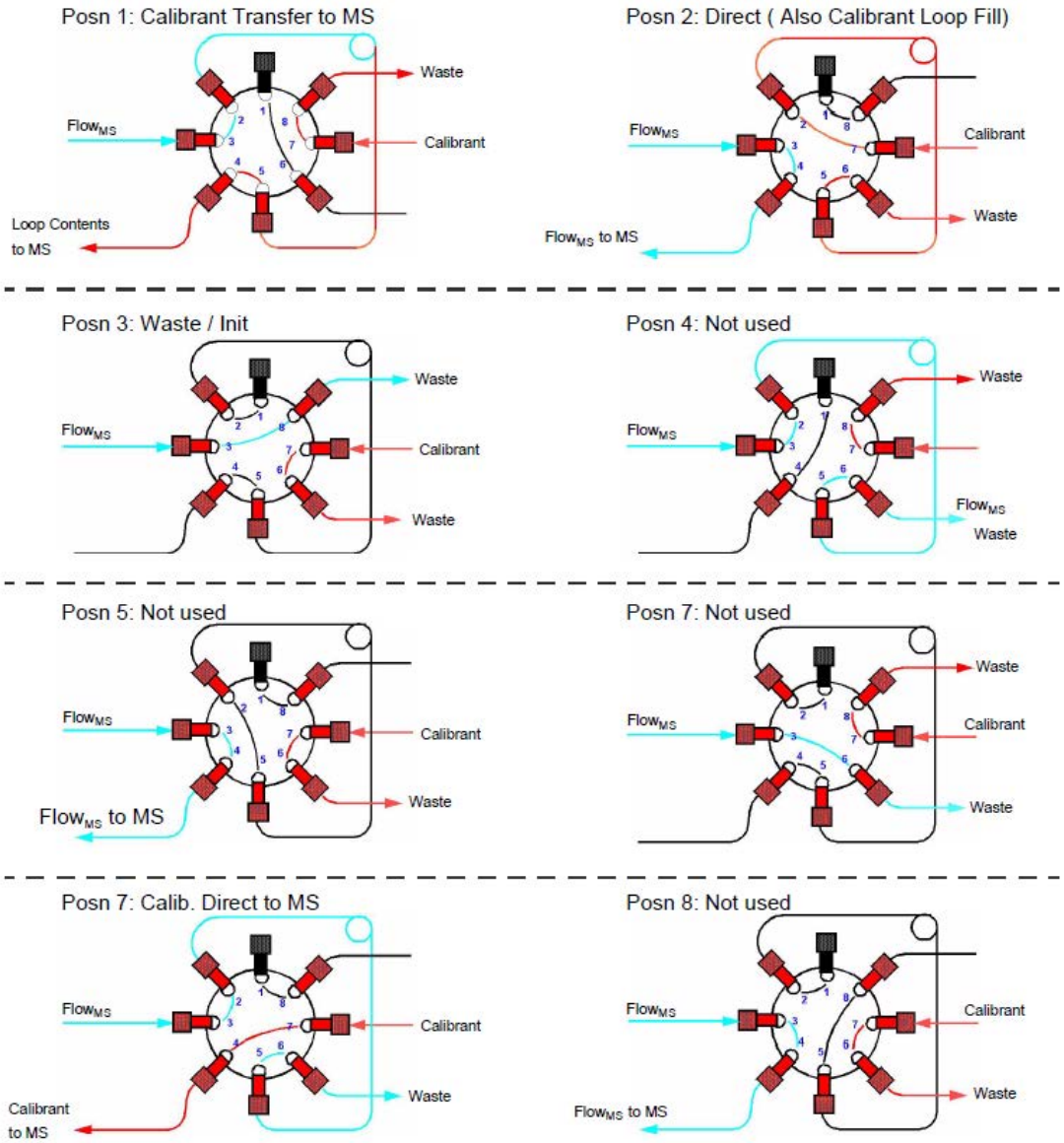
Calibration Fluid Pump Operation

In order to identify the start of an experiment (MS measurements) a small volume of Calibration liquid is pumped into the MS. This is a salt solution which can be readily identified in the MS. It gives unique MS peaks well removed from those of interest in the analysis itself.

During initial setup the path from Pump3 to the rotary valve and into the calibrant loop had been filled with calibration liquid (see Embedded Web Server - Calibration Pump) and Pump 3 has been initialized.

- • The calibration operation is started (HyStar or manual command)
- • The rotary valve switches to the Calibration Transfer position (1).
  - Here FlowMS flows into port 3 and out of port 2 into the calibrant loop.
  - The contents of the loop are forced into port 5, out port 4 and on to the MS.
  - This requires an active FlowMS. The calibration pump does not pump the calibration fluid into the MS.
- Once the timeout period is over (see Embedded Web Server - Calibration Pump) the rotary valve is returned to the Direct position (2).
- Pump3 pumps four times the loop volume (i.e. 40µl for a standard loop) to the rotary valve port 7, through and out of port 2.
- This replaces the contents of the calibrant loop with fresh calibrant.
- If there is now less than 4 times the loop volume remaining in the Pump3 syringe, then Pump3 is re-initialized.

8-Port Rotary Valve



The valve used is a Vici 8 Port Selection Valve with Bruker Etch on the Rotor, 1/16” 10-32 Ports with 0.15mm ID hole rated to 50C/300psi liquid, type N60/H mounted on a VICI Universal Actuator.

The fluid flow paths for each of the 8 positions are pictured above.

7.2 Safety

Improper Operation

⚠ WARNING

Danger of injury from improper operation!

Improper operation can result in serious injury and significant damage to property.

- ▶ Carry out all operating steps in accordance with the specifications and instructions in this manual.
- ▶ Before starting work, ensure that
  - All covers and safety devices are installed and functioning properly.
  - No persons are in the danger zone.
- ▶ Never disable or bypass safety devices during operation.

⚠ WARNING

Dangers when the unit is left to run unattended. (for example - overnight)

If any of the tubing paths from the pumps is blocked this can result in leakage onto the drip dray at the front of the unit releasing potentially hazardous solvent into the lab.

Ensure there are no blockages:

- ▶ If the unit is simply in purge mode check that you can see liquid dripping in to the waste flask (shows no blockages).
- ▶ If an analysis is running check that the calibration fluid is detected correctly (indicates no blockage in path from calibration pump)
- ▶ Check also that your MS peaks are shifted the expected amount. This indicates no blockage on the make-up pump path.
- ▶ Examine the drip tray at the front of the unit. There should be no drips.
- ▶ Ensure the waste flask has sufficient space left for the expected waste liquid volume.
- ▶ Ensure the solvent and calibrant flasks have sufficient liquid for the unit to operate correctly unattended. - Too little fluid will not cause any damage but some of your analysis could be lost.

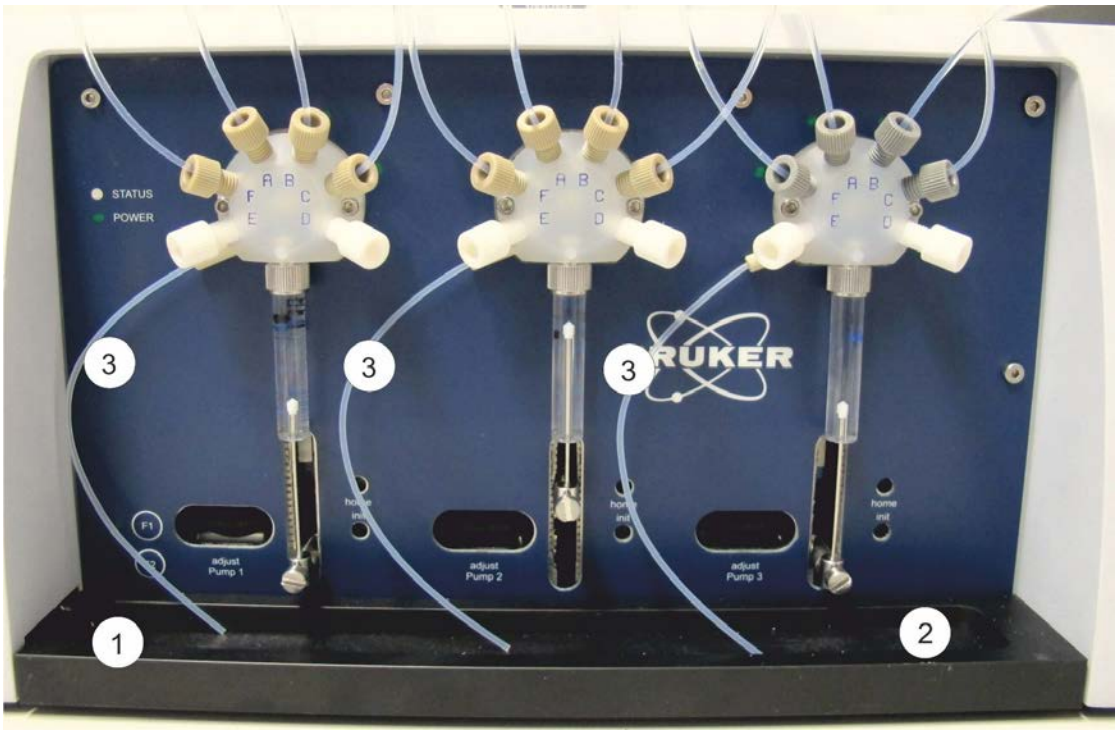


Figure 7.2: Valve overpressure leakage output tubing

1	Leak sensor
2	Leak tray with slope from right to left
3	Valve overpressure leakage output tubing

Liquid pressure is determined by the speed of the syringes (faster -> higher pressure) and by the fluid paths (long small-bore paths require more pressure to push the liquid through) and also by the viscosity of liquid being transferred. The system is designed such that under standard operating conditions the maximum pressure built up when operating at maximum flow is < 4 bar. The highest pressure in the system is at the point where the pressure is generated, i.e. at the syringes.

The fittings and tubing are all rated to > 20 bar. The selection valve connected to the syringe is rated to 5 bar. Should an overpressure condition occur, for example, due to a blockage in the output path, fluid will leak out at the point where the system has the lowest pressure rating: at the rotary valves above the syringes. The valves are designed to have a defined output path in the event of an overpressure condition - liquid runs out the tubing to the left of the valve and into the leak tray. Excessive leakage runs out the leak tray past the leak sensor and into the customer's waste flask. Only a large leak volume is detected by the leak sensor.

Overpressure conditions cause the syringe step motor to lose steps - this flags an error to the controller. As the maximum flow rate is 180µL per minute, the maximum leakage here is also 180µL min.

At these rates of loss, the gas concentration (due to evaporation) is very unlikely to reach an explosive concentration.

⚠ WARNING

Danger of fire/explosion due to local concentrations of solvent vapor

This can result in injury or death and damage to the laboratory instruments.

- ▶ Never have an open flame near the unit.
- ▶ When working directly at the unit always first ensure you discharge any potential static buildup to a suitable earth (e.g. the mounting screws at the right hand side of the unit) to reduce the likelihood of static electricity sparking onto the unit near where solvent may have leaked.
- ▶ The operator must always be trained in the handling of any solvent used especially with respect to the dangers inherent to the solvent. (flammability, poisonous etc.).

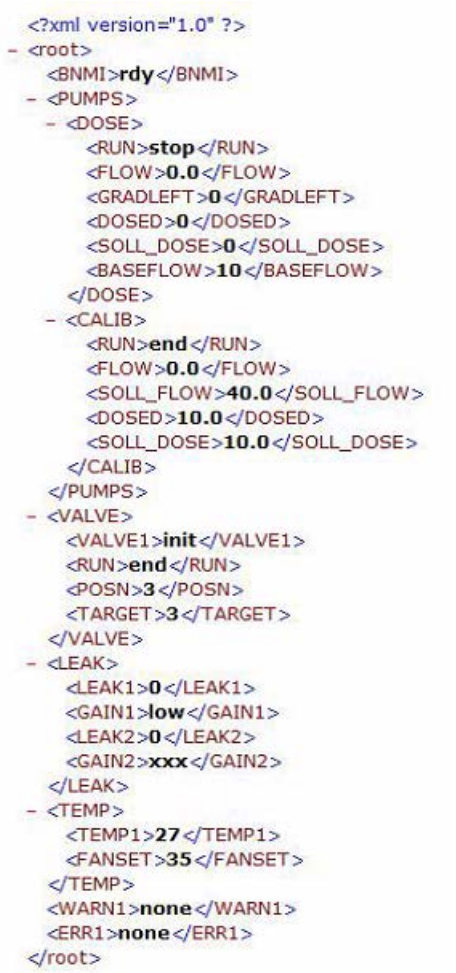
8 Embedded Web Server

The NMR-MS-Bridge is controlled over its Ethernet connection. This is implemented over a number of embedded web pages.

The xml Status Page

This page is found at the url: http://192.168.254.42/status.xml

This is the xml status page read by HyStar.



The entries have the following meanings:

LABEL	VALUES	COMMENT
<BNMI>	xxx	Unknown - (Controller board error)
	start	At startup
	init	initialization running
	err	an error has occurred

LABEL	VALUES	COMMENT
	rdy	Device is ready for use
<DOSE><RUN>		- main pump status -
	Init	pump initialization running
		pump operation running
		pump running in *baseflow* mode
		pump stopped (needs init)
		pump stopped - ready to go
		pump paused - ready to go
	err	a pump error has occurred
	test	test mode - purge running
<DOSE><FLOW>	xx.x	The flowrate when running
<DOSE><BASEFLOW>	xx.x	The *set* value of the base flow
<DOSE><GRADLEFT>, <DOSED>, <SOLL_DOSE>	0	always 0, not used
<CALIB><RUN>		- calibration pump status -
	init	pump initialization running
	run	pump operation running
	end	pump stopped - ready to go
	err	a pump error has occurred
	test	test mode - purge running
<VALVE><VALVE1>		- rotary valve functional position - (see 'Valve Positions')
	undefined	unknown or position not normally used for LC-NMR-MS
	direct	at direct flow position
	waste	at pump to waste position
	calib	at calibration position
	init	at initialized position
	sample, reverse, transfer	- are not used in the present hardware setup.
<VALVE><RUN>		- rotary valve status -
	xxx	unknown - initialization not yet done
	init	valve initialization running
	run	valve running
	end	valve stopped - at valid position
	err	a valve error has occurred
<VALVE><POSN>		- rotary valve numerical position
	xx	1 to 8 for position 1 to 8

LABEL	VALUES	COMMENT
<VALVE><TARGET>		- rotary valve target numerical position
	xx	1 to 8 for position 1 to 8
<LEAK><LEAK1>		- Leak Sensor Status -
	0	No leak detected
	1	Leak detected
<LEAK><LEAK2>		- There is no 2nd leak sensor in this hardware version -
<TEMP><TEMP1>		- Temperature status
	xx	15 - 45 is the temperature in °C within the unit
<TEMP><FANSET>		
	xx	30 - 40 is the temperature in °C above which the fan runs faster.
<WARNx>		x is the 'Warning number' - incremented on each error
When a minor error occurs a warning is generated and displayed here. The warnings are displayed as a list. The last warning is always 'none'		
<ERRx>		x is the 'Error number'
When a major error occurs an error is generated and displayed here. The errors are displayed as a list. The last error is always 'none'.		

The errors and warnings are for debug use only. Should an error condition repeatable occur then the unit probably has some hardware problem and you should contact Bruker service. HyStar will not operate the pump while an error condition exists. An occasional error can be cleared by entering the following into the Browser url:

http://192.168.254.42/$ERR=ack

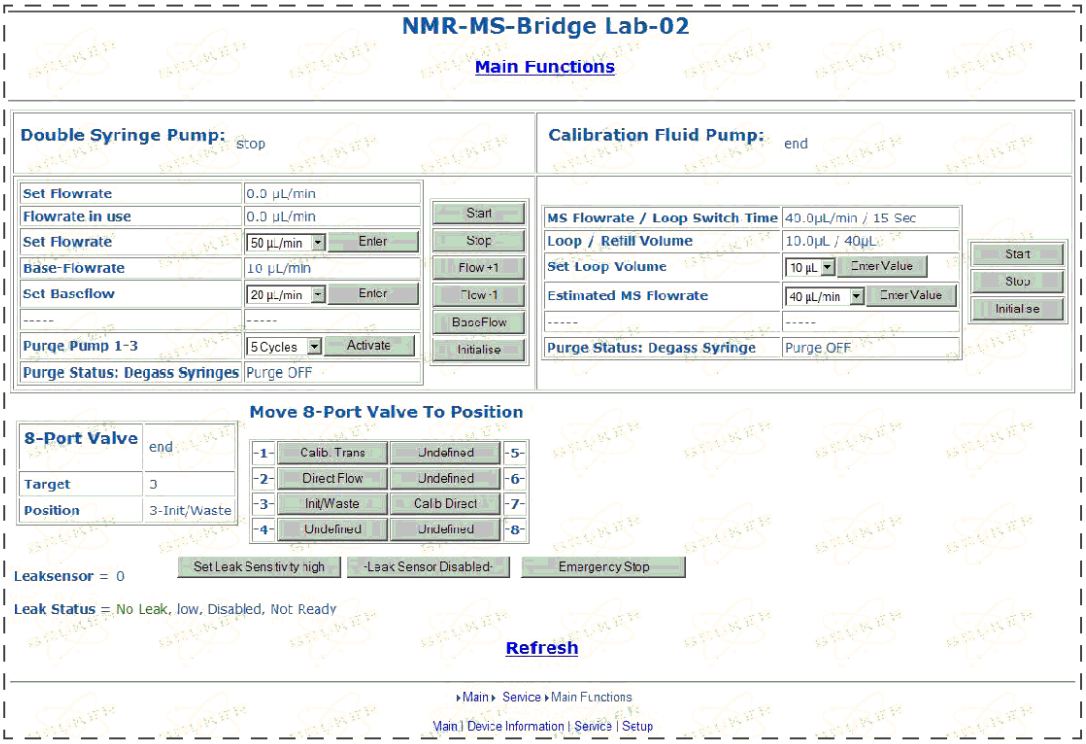
This simply acknowledges the errors but does not clear the error condition. The unit may also have to be re-initialized!

Main Functions Page: http://192.168.254.42/bnmihp.html

This page is used primarily during initial setup and test. All the operational functions of the unit can be started from here.

This is a simple static web page. To refresh the display click on either **Main Functions** or **Refresh**.

Do not use F5 or the browser refresh.



Meaning or Function

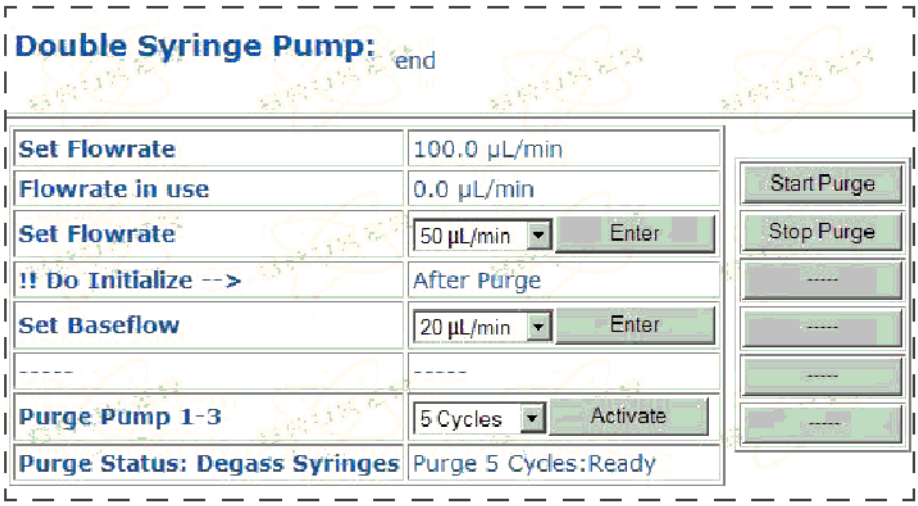
Double Syringe Pump	- refers to the standard pump operations	
	xxx, start, stop, init, err	- exactly the same as in <DOSE><RUN> in the status page.
Set Flowrate	Shows the target flowrate	
Flowrate in Use	Shows the present pump flowrate	
Set Flowrate	- with dropdown menu	Select your desired target flowrate and click on **Enter**.
Base Flowrate	- is a ‘default’ background flowrate.	Shows the value of the target base flowrate.
Set Baseflow	- with dropdown menu	Select your desired target base flowrate and click on **Enter**.
Purge Pump 1-3	- with dropdown menu: (see purge operation)	Select the number of purge cycles and click on ‘Activate’. - applies also to Calib pump
Purge Status: Degass Syringes	- only for the purge operation -	Purge OFF - purge has not been activated

Buttons

Start	Starts the pump operation with the target flowrate (also from Baseflow)
Stop	Stops the pump
Flow+1	increments the target flowrate by 1 (limits to max of 180)
Flow-1	decrements the target flowrate by 1 (limits to min of 1)
Baseflow	switches immediately to the Baseflow target value
Initialise	both syringe pumps are initialised

Purge Operation:

If the Purge mode has been activated then the display changes to:



Purge Mode Buttons:

Start Purge	- starts the purge/degass operation - default purge flowrate is 100µl/min
Stop Purge	- stops the purge/degass operation

After the purge operation has been started you can use the Set Flowrate dropdown menu to change the flowrate used. The flowrate is updated on the display immediately but the new flowrate is actually only used from the start of each purge cycle.

Status Display

The status display is also modified:

Double Syringe Pump	- refers to the standard pump operations -	
	Purge: Degas Syringe	- appears once the purge is started -

Purge Status: Degass Syringes	- only for the purge operation -	
	Purge x Cycles: Ready	- purge has been activated for x cycles but not started -
Status: Degass Syringes	- only for the purge operation -	
	Purge x Cycles: Ready	- purge has been activated for x cycles but not started -
	x Cycles Remaining	- purge has been started, there are x cycles still to do -

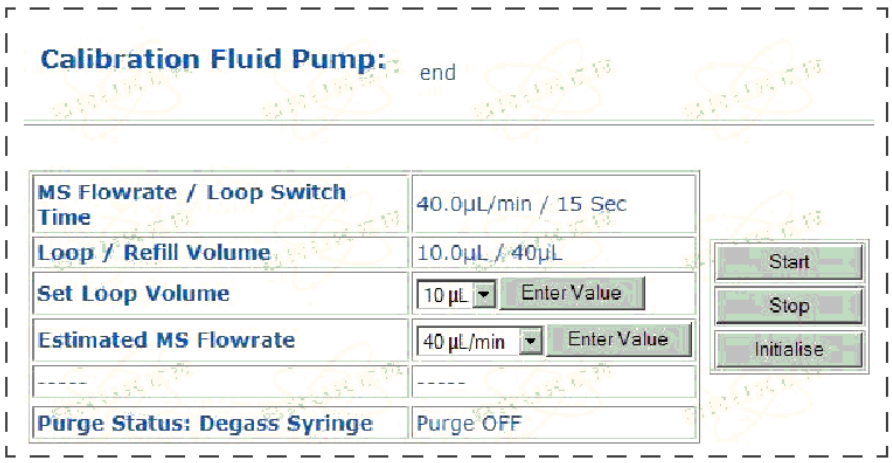
After a purge operation the syringe pumps must be initialised before using normal mode.

This is indicated by the status message:

!! Do Initialize --> After Purge

After setup or if you have changed the solvent you will also have to fill the path from the syringe pumps to the rotary valve. In normal pump mode (after initializing):

- Set the rotary valve to **Init/Waste** and the Flowrate to **100µl/min**
- Click **Start** and let the pump run for 2-3 minutes



Calibration Fluid Pump:	- refers to the calibration pump operations	xxx, start, stop, init, err, test

MS Flowrate / Loop Switch Time - in the example here: **40.0µl/min / 15 Sec**.

With a flowrate of 40.0µl/min the 10µl loop is emptied in 15 seconds. In HyStar the volume must be set to the loop volume (default 10µl) and the calibration flowrate set to the same as (or less than) the LC pump flowrate. The time for which the loop is switched into the MS flowpath is calculated accordingly. Setting the calibration flowrate lower ensures that at least the loop volume is pumped out. Setting it higher risks too small a calibration volume being pumped into the MS. The lower and upper limits for the switch time are set to 6 and 60 secs.

Loop / Refill Volume	- in the example here: **10.0µL / 40µL** see explanation above
Set Loop Volume	allows to enter a different volume from the drop down menu.
Estimated MS Flowrate	allows to enter a different MS flowrate from the drop down menu.
Purge Status: Degass Syringes	- only for the purge operation -
Purge Status: Fill Loop	- alternative purge operation for calibration pump – Purge OFF - purge has not been activated

The purge **Activate** button for the double syringe pump also applies here.

Buttons

Start	Starts the calibration pump operation
Stop	Stops the calibration pump operation
Initialise	Initialises the calibration pump

Calibration Pump Purge

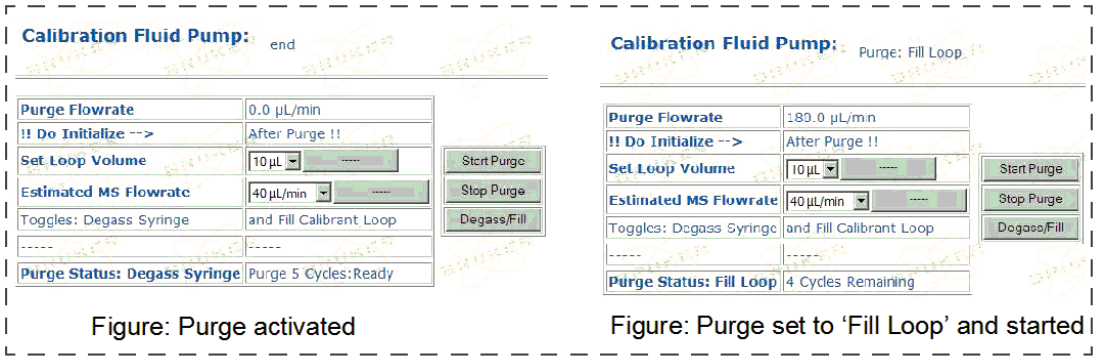


Figure: Purge activated

Figure: Purge set to 'Fill Loop' and started

The Calibration Fluid Pump display changes slightly when in *Purge* mode.

Purge Mode Buttons:

Start Purge	starts the purge/degass operation - default purge flowrate is 100µl/min
Stop Purge	stops the purge/degass operation
Degass/Fill toggles between standard purge (Degass) and Fill Loop mode: The rotary valve is set to 'calib' and the syringe valve is switched to pump out to the valve. This fills the path from the calibration pump to the rotary valve and through the calibration fluid loop. This must be done once at system setup otherwise the first calibration operations will not operate correctly.	

Once the purge operation has been started you can use the **Set Flowrate** dropdown menu on the Double Syringe Pump field to change the flowrate used. The flowrate is updated on the display immediately but the new flowrate is actually only used from the start of each purge cycle.

The status display is also modified:

Calibration Fluid Pump:	- refers to the calibration pump operations	
	Purge: Fill Loop	The syringe fills the path through the loop with calibrant.
	Purge: Degass	The syringe runs a degas cycle
Purge Status: Degass Syringes	- only for the purge operation -	
Purge Status: Fill Loop	- alternative purge operation for calibration pump	
	x Cycles Remaining -	purge has x cycles left to complete

After a purge cycle the syringe pump must be initialised before using normal mode.

This is indicated by the status message:

!! Do Initialize --> After Purge

8 Port Rotary Valve

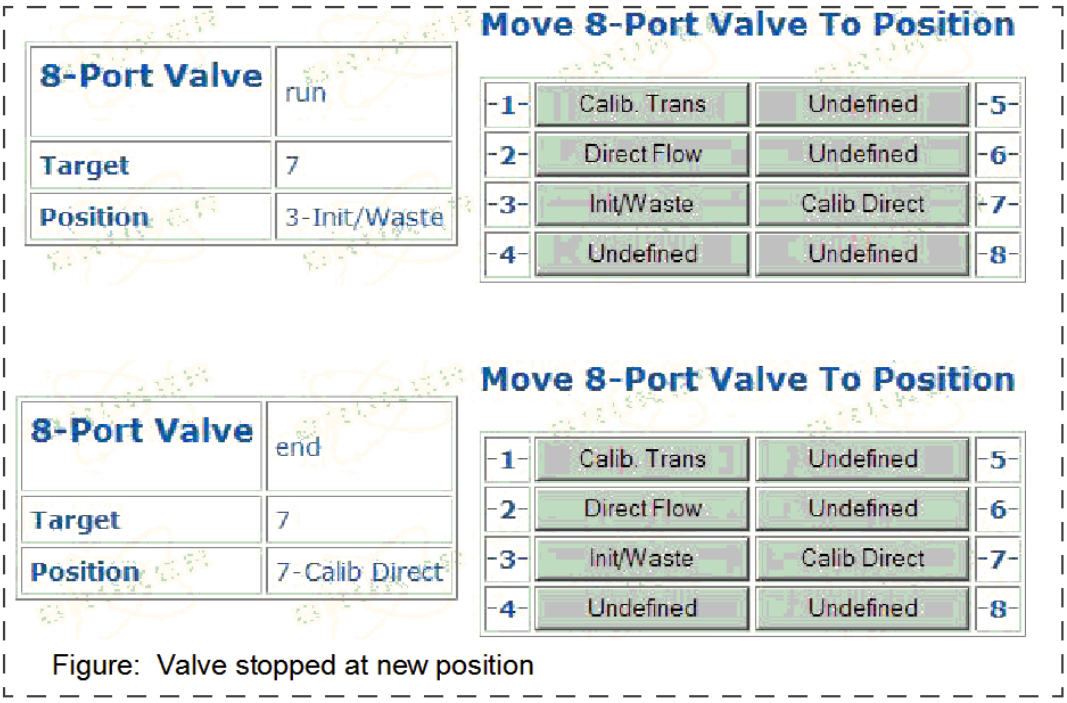


Figure: Valve stopped at new position

Move 8-Port Valve to Position

The rotary valve can be moved to any of 8 positions (See Valve Positions).

In the present version, four of these positions are used.

Pressing the button corresponding to the position (number / name) causes this position to be loaded as the new target position. The valve then moves to this position.

8-Port Valve

init	The valve is being initialised (very first move after power up)
err	An error has occurred.
run	The valve is moving to the target position.
end	The valve has stopped at the target position.

Target

x	1.....8 is the target position

Position

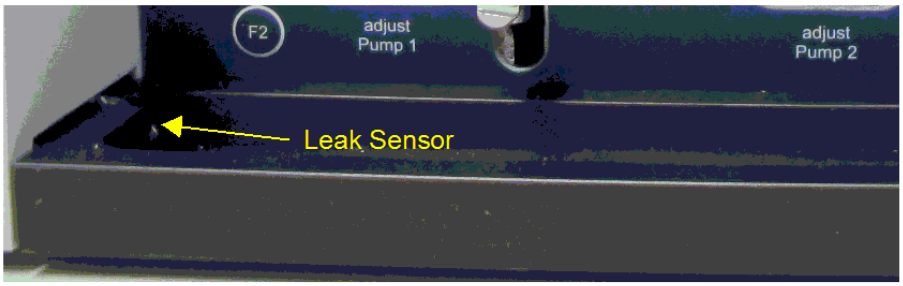
x / Posn Name	1.....8 + Name is the present (or last if moving) position.

As the web page is static you have to refresh the page after setting a new target position in order to see the updated status.

The position **Calib Direct (7)** is not supported by HyStar. This is used only in service mode when you wish to pump fluid from the calibration pump directly into the MS:

Set the calibration pump to **Purge - Fill Calibrant Loop** mode and start. Then set the rotary valve to position 7.

Leaksensor



Leaksensor = 0 Set Leak Sensitivity high -Leak Sensor Disabled-

Leak Status = No Leak, low, Disabled, Not Ready

Leaksensor = 0 Set Leak Sensitivity low Disable Leak Sensor

Leak Status = No Leak, high, Enabled, Not Ready

Leaksensor = 0 Set Leak Sensitivity low Disable Leak Sensor

Leak Status = No Leak, high, Enabled, READY

Leaksensor = 17 Set Leak Sensitivity low Disable Leak Sensor

Leak Status = Leak Detected, high, Enabled, ready

Leaksensor = 2 Set Leak Sensitivity low -Leak Sensor Disabled-

Leak Status = No Leak, high, Disabled, ready

The leak sensor is located in the outlet path at the left of the drip tray on the front of the unit. It will only react to a relatively large leak: enough liquid gathering in the drip tray to run out of the outlet and past the sensor. A few µl dripping on the tray will not be detected.

It works by detecting the sudden temperature differences caused by liquid coming into contact with the sensor. As such it needs time after power up (and being enabled) for the monitoring firmware to determine the background 'steady state' condition. Slow temperature changes are not identified as a leak. These are simply used to move the reference point at which a leak is detected.

Display:

Leaksensor = x	- intended mostly for debug - x climbs when a temperature drop (wet sensor) is detected

Leak Status	- displays the leak status -	
	No Leak, Leak Detected	- description of leak status
	high, low	- sensitivity: should normally be high
	Enabled,Disabled	- sensor enabled or not
	Not Ready	- when first enabled needs a short time to adjust
	ready	- sensor will work but at reduced sensitivity
	READY	- sensor fully operational.

Buttons

Set Leak Sensitivity high	- click here to enable leak sensor in high sensitivity mode.
Disable Leak Sensor	- click here to disable leak sensor.
Set Leak Sensitivity low	- click here to enable leak sensor in low sensitivity mode

(the high/low sensitivity button toggles when clicked: high->low->high)

If a leak is detected (**Leak Detected**) this is also reported as an error in the status.xml web page.

To clear the condition:

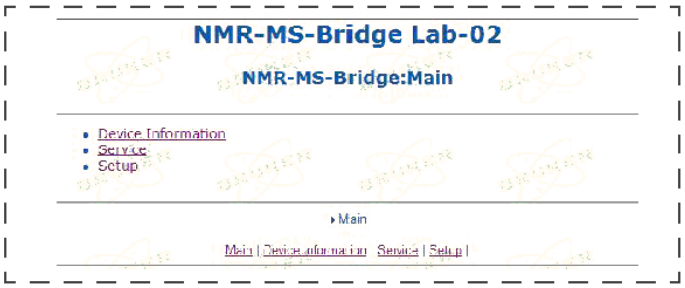
Identify and repair the leak.

- Thoroughly dry off the leaked liquid, including any liquid in the vicinity of the sensor-use a rolled up Kleenex or similar to wipe the inside of the exit path at the sensor.
- On the web page click on Disable Leak Sensor.
- In the url enter http://192.168.254.42/$ERR=ack to clear the error list (see page xx)
- Click on Set 'Leak Sensor high to re-enable the leak sensor (or ...low if ..high is not displayed - click on ... high afterwards)

- If the Leaksensor value immediately climbs and a leak is flagged again then you have not completely dried off the leak sensor. - repeat the above steps - you may have to wait a further 5 - 10 minutes before re-enabling the leak sensor to allow any remaining traces of liquid to evaporate.
- The leak sensor can also be enabled/disabled and set to high/low from HyStar.

Other Web Pages Main

http://192.168.254.42/ews.html

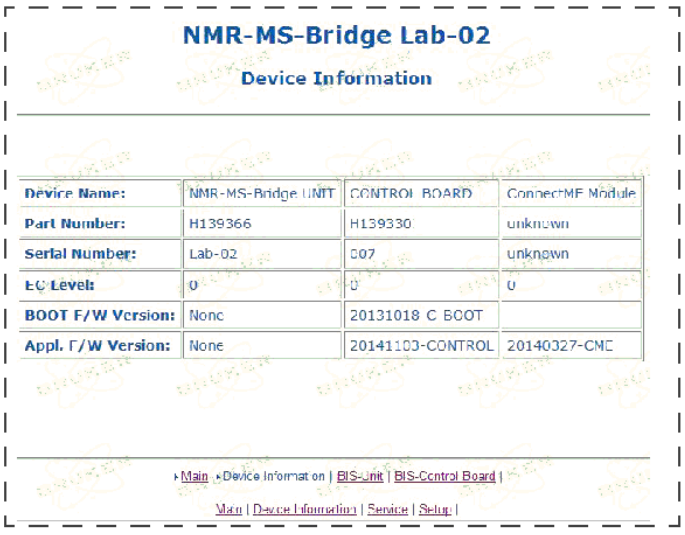


The Main Page (ews = embedded web server) simply lists the main links to other pages

At the bottom of all of the web pages there are quick links to most of the other pages.

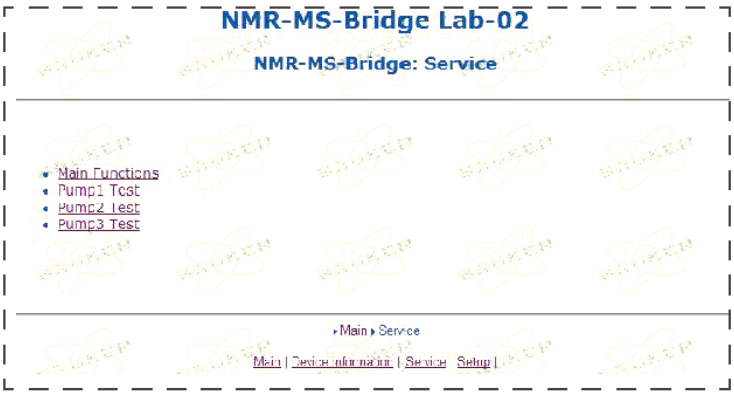
Click on the link to open the web page.

Device Information



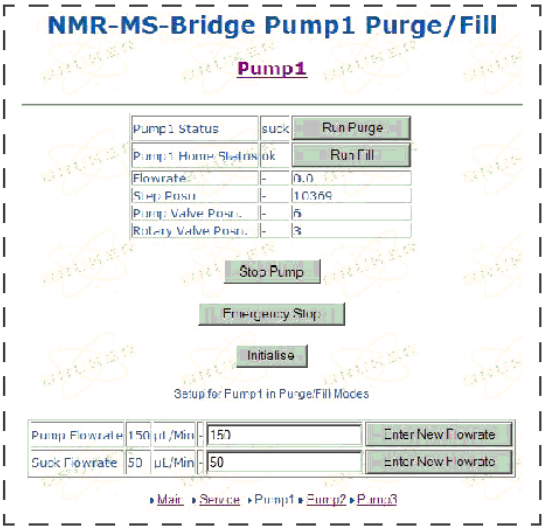
This displays information on the hardware engineering levels and firmware levels in the unit. This information is important if you have are having problems an need to contact Bruker Service. The ‘Bis’ Pages are for service use only.

Service Pages



This provides links to further service options.

Pump1 Test



Pump2 and Pump3 Test are identical but apply to pumps 2 and 3.

The operations started from here can now mostly be started from the ‘Main Functions’ page. These pages are primarily intended for debug use and will be rarely used.

Do not use these operations while HyStar is running and connected to the system.

Pump1 Status	- shows the state of the pump in this mode only -	
	suck, pump, init, end	- filling (suck), dispensing (pump), initialising, stopped
Pump1 Home Seek Status	ok, no	- pump home seek has been done (or not done)
Flowrate	xx	- xx = flowrate used in this mode only

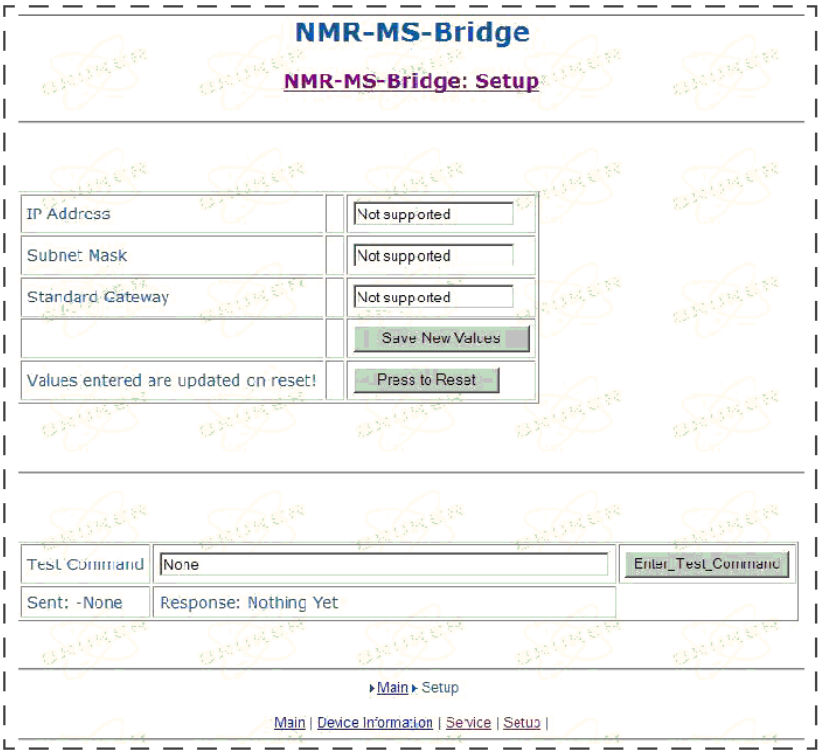
Step Position	- NOTE 12000 steps for 100µl syringe, higher is fuller.	
	xxxx	- present step (motor) position.
Pump Valve Position	- valve at top of syringe	
	x	- 1 to 6 corresponding to the valve positions A - F
Rotary Valve Position	- position as in ‘Main Functions’ page	
	x	- 1 to 8

Buttons

Run Purge	- starts the purge operation for this pump only
Run Fill	- starts the fill operation for this pump only
Stop Pump	- stops this pump
Emergency Stop	- Stops all pump and valve operations
Initialise	- initialises this pump only
Enter New Flowrate	- Flowrate entered in adjacent field is used for pumping operations in this mode only.
Suck Flowrate	- Flowrate entered in adjacent field is used for sucking operations in this mode only.

After any pump test operation here the pump must be initialised before running any standard operation.

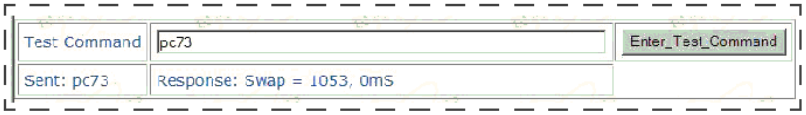
Setup



IP Address	- not supported, no function
Subnet Mask	- not supported, no function
Standard Gateway	- not supported, no function

Buttons

Save New Values	- not supported, no function
Press to Reset	- Resets the Unit
Enter_Test_Command	- for low level debug. Commands entered here are routed directly to the pump controller. The command itself appears after Sent: and the reply after response: The only command which a user should use is ‘pcxx’ (xx = 0 to 250). See example below



The meaning of the reply is again only useful for debug purposes.

This command **pcxx** can be used to adjust the changeover characteristic of the double syringe pump. As one syringe pump has almost completely emptied it decelerates to a halt and the other syringe pump is accelerated to its pump speed (= flowrate). The overlap timing can be changed with this value. Higher means more overlap. The default value is 60. Any value entered here is lost on reset. If your system shows small spikes (+ve or -ve) in the flowrate at the pump changeover you can try to fine-tune it here. The default value works very well in the validation systems.

9 Maintenance

9.1 Safety

Electrical System

⚠ WARNING

Danger of injury from electrical shock!

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

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

Improperly Performed Maintenance

⚠ WARNING

Danger of injury due to improperly performed maintenance!

Improperly performed maintenance may lead to serious injury and significant material damage.

- ▶ Provide for sufficient mounting clearance before starting to work.
- ▶ Keep the assembly area tidy and clean! Loose components and tools lying around or on top of each other may lead to accidents.
- ▶ When reinstalling previously removed components, make sure that the components are mounted properly, all fixing elements are reinstalled, and all screws are tightened to torque.
- ▶ Before putting the device back into operation:

⇨ Make sure that all maintenance work has been performed and completed following the instructions and information provided in this manual.

⇨ Make sure that no persons are still in the danger zone of the device.

⇨ Make sure that all covers and safety devices have been installed and function properly.

Securing to Prevent Restart

⚠ WARNING

Danger to life from an unauthorized restart!

In the event of an unauthorized restart of the power supply during maintenance, there is a danger of serious injuries or death for persons in the danger zone.

- ▶ Switch off all power supplies before starting work and make sure they cannot be switched on again.

Moving Parts

⚠ CAUTION

Accident hazard from movement of mechanical parts!

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

▶ Shut off the device before accessing.

Environmental protection

Observe the following environmental protection instructions during maintenance work:

- In respect to all lubrication points supplied manually with lubricant, remove any escaping, used or surplus grease and dispose of in accordance with applicable local regulations.

9.2 Replacement Parts

Part Number	Description
1820587	Syringe
88808	Valve
88807	Pump
1804561	Inline Filter Frit (2µm)
86135	Solvent Filter 10µm
1814451	External Rotary Valve
H146537	Cable to External Valve

Table 9.1: Bruker Replacement Parts

- Fittings (standard PEEK/TEFLON 1/4 -28 and 10-32 HPLC Fittings)
- Tubing 1/16" (1.6mm) OD FEP/PTFE tubing - see plan for ID and lengths.
- Mains cable - Standard IEC 60320 Style C13 compliant cable, must be rated to at least 250V, 5A and meet any local safety requirements.

Replacement parts must be exchanged by Bruker Service staff!

The only exceptions are: the tubing and fittings, the syringes, pump valves, the external rotary valve and its connection cable.

All these parts are easily accessible. See replacement instructions in chapter *Customer Serviceable Parts [▷ 75]*. Only original parts from Bruker are to be used for the device. Use of any parts other than from Bruker invalidates all warranty.

Parts which are returned to Bruker for repair or disposal must be accompanied by a repair declaration (see *Safety and Repair Declaration Form [▷ 79]*).

9.3 Assembling and Mounting the Fittings

Figure 9.1: Tube Cutting Tool (Shown is the A-327 from Upchurch) - always use a tube cutter.

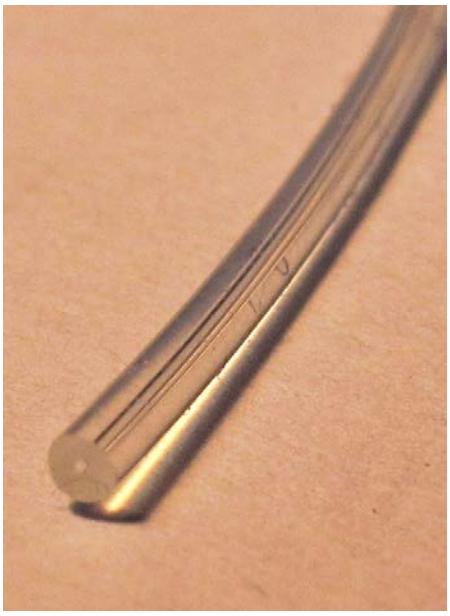


*Figure 9.2: The tubing must be cut as near as possible to 90° **(extremely important)***

Figure 9.3: 10-32 One piece Fitting

Figure 9.4: 10-32 Union (used to connect two lengths of tubing - High Pressure)

Figure 9.5: 1/4”-28 Union (used to connect two lengths of tubing - Not High Pressure)

Figure 9.6: 10-32 T-Piece (used to connect three lengths of tubing - High Pressure)

Figure 9.7: 1/4”-28 T-Piece (used to connect Three lengths of tubing - Not High Pressure)

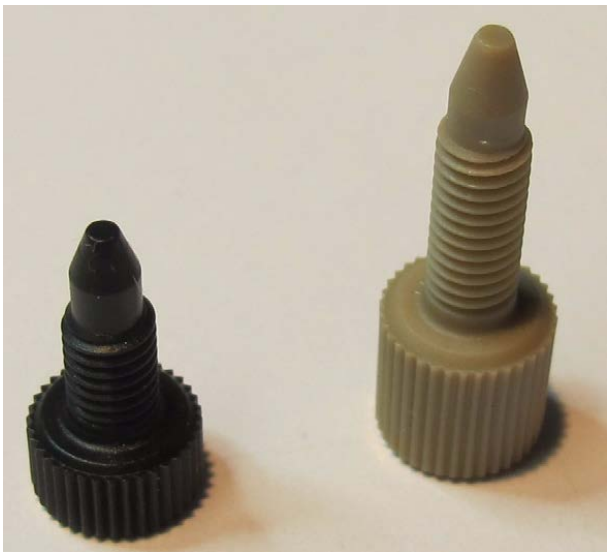


Figure 9.8: 10-32 Plugs (used to seal an unused 10-32 port)

Figure 9.9: 1/4”-28 Plug (used to seal an unused 1/4”-28 port)

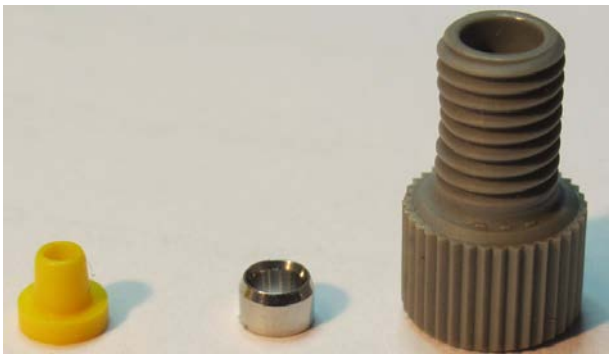
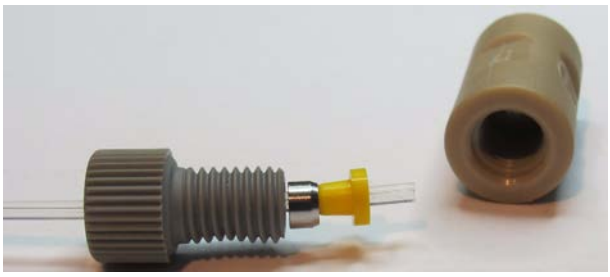


Figure 9.10: 1/4”-28 hand tight fitting

with ferulle  and clamp ring  .



1. 1/4-28 fitting with tubing ready for connection: Push the tubing through the fitting, put the clamp ring onto the tubing with the conical side towards the tubing end (**very important**), then push the ferulle onto the tubing with the coned end towards the clamp ring. The ferulle sits loosely on the tubing which should protrude a few mm through the ferulle.

2. Lightly screw the fitting into the union while pushing the tubing into the union. Unscrew the fitting and examine the end.

3. Incorrectly mounted fitting: the tubing end lies within the ferulle. If you have not tightened the fitting too firmly you may simply be able to twist the ferulle and clamp ring further onto the tubing until it sits correctly.

4. Incorrectly mounted fitting: the tubing end was pushed through the ferulle.

If the fitting has been mounted incorrectly you will have to seperate the clamp ring from the ferulle (push a finger nail firmly onto the coned end of the clamp ring or very carefully nip the coned end of the clamp ring with a pair of wire cutters ensuring the the angled edge of the blade is at the clamp ring cone.) then remove the ferulle and clamp ring from the tubing. If the tubing end has been damaged cut off the last few mm. Replace the ferulle if it has been damaged.

5. Correctly mounted fitting: the tubing end is flush with the base of the ferulle. You can now screw the fitting firmly into its intended port.

6. 10-32 one piece fitting with tubing ready for connection. The tubing is pushed 8 - 15mm through the end of the fitting.

7. Firmly screw the fitting directly into the intended port while pushing the tubing into the port.

8. Fitting screwed (hand tight, do not use tools!) firmly into port.

1. The 10-32 Unions and T-Pieces often come with a set of 2 piece fittings. Here push the tubing through the fitting the push on the ferulle (1a) with the coned end towards the tubing end.

2. 10-32 2 piece fitting with the tubing protruding 8-10mm ready for mounting.

3. Screw the fitting firmly into its intended port while pushing the tubing into the port.

4. MicroTee with integral fitting (4b) disassembled.

5. Assemble the tubing as shown.

6. Screw the the fitting firmly onto its intended port while pushing the tubing into the port.

ALWAYS ENSURE THAT THE TUBING HAS BEEN CUT AS NEAR AS POSSIBLE AT 90°

9.4 Before Refilling

Correctly Assembling a 1/4"-28 Fitting

The tubing end must always be cut as near as possible to 90°. Push the fitting, then the stainless steel ferrule ring over the tubing. The tapered end of the ring MUST always point towards the tubing end and the Flat end towards the fitting, see the second figure in chapter *Assembling and Mounting the Fittings [▶ 65]*.

Push the ferrule on to the end of the tubing with the coned end towards the steel ring and fitting. Push the fitting, ring and ferrule towards the end of the tubing such that only a few mm of tubing protrude through the ferrule. Holding the fitting with tubing in one hand place a 1/4"-28 Union on to the tubing end and screw the union onto the fitting. As you do this lightly push the tubing through the fitting to ensure it is held against the base of the port. Screw the fitting lightly (not yet firmly) *hand tight* then unscrew it and examine it. The tubing end must be flush with the ferrule base as shown. Adjust as required. If you require to remove the steel ring (for instance to allow the ferrule to be move slightly on the tubing) use a pair of wire cutters and with the flat side of the tool set towards the ferrule VERY CAREFULLY nip the tapered end of the steel ring to force it away from the ferrule. Be careful you do not damage the ferrule! With the tubing now flush with the base of the ferrule, you can now firmly (but still hand tight) screw the fitting into the union. Remove and examine it again. If it looks good you can now screw it firmly into the correct port. Once the fitting is fully screwed in to the correct port, pull lightly on the tubing to ensure it is securely in place.

Correctly Assembling a 10-32 Fitting

The tubing end must always be cut as near as possible to 90°. In this case always screw the tubing and fitting directly in to the intended port. Push the tubing through the fitting such that it protrudes ca. 5-6mm. (as pictured). Now screw the fitting into the port while pushing the tubing firmly through the fitting. Once the fitting is fully screwed in to the correct port, pull lightly on the tubing to ensure it is securely in place.

Correctly connecting tubing to a MicroTee.

The tubing end must always be cut as near as possible to 90°. Remove the cap to the MicroTee port. Remove the fitting from the port. Put the cap and the fitting on to the tubing as shown. Put the fitting with the tubing into the MicroTee port. Firmly the tubing into the port while firmly tightening the cap. Once the fitting is fully screwed in to the correct port, pull lightly on the tubing to ensure it is securely in place.

9.5 Refilling the Storage Flasks

⚠ WARNING

Danger of injury from electrical shock!

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

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

⚠ DANGER

Danger of fluid spillage on the unit!

the storage flasks must never be refilled *in situ*. They must only be filled in the laboratory fume chamber intended for this purpose.

- ▶ Always wear protective gloves and suitable eye protection.

⚠ DANGER

Danger of injury from glassware breakage!

Broken glassware may cause minor injuries or material damage, but may also result in a life threatening situation if hazardous substances are used.

- ▶ If glassware breaks, refer to the corresponding precautions and cleaning/disinfection instructions.
- ▶ Wear protective equipment.
- ▶ Perform all tasks with the glassware carefully.
- ▶ **The laboratory supervisor is responsible for:**

⇨ Establishing and enforcing standard sample handling and cleaning procedures.

⇨ Establishing and enforcing the use of protective clothing and equipment.

⇨ Training laboratory personnel.

⇨ Preparing an emergency plan.

- Power off the unit and remove the mains plug.
- Unscrew the lid from the flask and remove it together with the tubing, fittings filters etc. Simply place these over an adjacent flask.
- Secure a standard cap on the flask.
- Take the flask to the area intended for refill operations (usually a fume cupboard).
- If you are using the same solvent as before you can simply remove the cap, fill the flask with fresh solvent and replace the cap.
- If you are using a different solvent you must either first thoroughly clean and dry the flask as defined in your own laboratory procedures or simply replace the flask with one of the same type filled with the new solvent. In any case ONLY carry the solvent flask back to the unit with a cap securely in place.
- Do not drop the flask! Place the flask back in the place provided at the top of the unit.
- Remove the cap.
- Replace the filters, tubing, cap insert and cap back on the flask and screw the cap in place.
- The waste flask must be handled in a similar manner. In this case the contents must be disposed of safely as defined in your own laboratory SOPs.
- After refilling you should run a purge operation – new solvent – purge the double pump, new calibration liquid – purge the calibration pump. A purge is not required if you have simply disposed of the waste fluid.

9.6 Preventive Maintenance

All parts in the device have been designed to work reliably with only a minimum of routine preventative maintenance.

- ALWAYS use in input filter in each pump's supply liquid. This prevents any particulate contamination coming into contact with the wetted moving parts of the pump components. These parts can otherwise be scratched and leak.

Wash the Calibration Pump Path

If the unit is likely to be unused for more than a few days flush the calibration pump and its flow paths of any calibration fluid. The calibration fluid is a strong salt solution and with time it can evaporate leaving a hard salty precipitate which can damage the moving surfaces of the syringe and the valves.

- Simply remove the cap and tubing to the calibration liquid flask. Put a standard lid on this flask then remove it from the system and store it somewhere safe.
- Replace this flask with an identical flask containing a ca. 0.004% Sodium Azid solution (to prevent fouling during storage) in demineralized water. Remove the connection from the external valve to the MS at the MS and put the tubing end into the waste flask. For the calibration pump only: run the initialize procedure followed by a 5 cycles **Degas Syringe** purge and finish with a 10 Cycle **Fill Loop** purge. Set the 8-Port valve to **Direct Flow** at the start of the **Fill Loop** purge and change it to **Calib. Trans** once you have only 4 - 5 cycles remaining. This will be sufficient to remove the calibration fluid from the syringe, the valve pump and the external valve.

9.7 Firmware Update

Should the instrument lose power or the communication be inadvertently interrupted during a firmware update the unit may no longer operate correctly. In this case get in touch with the Bruker Service dept. and you will receive guidelines on how to rescue the system. This can usually be done without having to return the system to Bruker.

The firmware update is done from within HyStar at the Hardware Configuration.

See Hystar Manual for further details.

9.8 Cleaning

Before cleaning, turn off the unit and remove the mains plug. Use a soft damp but not soaking cloth to gently wipe down any accessible surfaces EXCEPT at or immediately adjacent to the mains input. Use only demineralized water, isopropanol or a mixture of the 2 as a cleaning fluid. Wipe dry with a soft dry cloth. Do not reconnect the mains plug until the unit is completely dry.

See also

- Embedded Web Server [▶ 47]
- Safety and Repair Declaration Form [▶ 79]

9.9 Customer Serviceable Parts

Replacing a Pump Valve

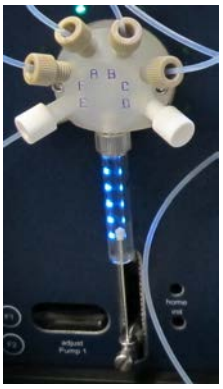


Figure 9.11: Pump Valve

Figure 9.12: Valve Rear Marking

Figure 9.13: Valve Drive

- Power the unit **Off** – **On** - **Off**: The pumps set the pump valves to the initialize position.
- Remove the screw securing the syringe to the pump at the bottom of the syringe.
- Remove the 2 screws (3/32” Alan Key supplied) securing the valve to the pump.
- Pull the valve together with the syringe and the connected fittings forward and away from the pump.
- Put a label on each of the tubing to the fittings (A to F to indicate which port it belongs to) and unscrew all of the fittings and the 2 plugs.
- Loosen (1 turn anticlockwise) the small lock screw on the valve at the front of the syringe port.
- Unscrew and remove the syringe from the valve.

On the New valve:

- Hold the valve such that the port for the syringe faces upward and place a Teflon sealing ring (spares supplied with the system) in the base of the port.
- Loosen (1 turn anticlockwise) the small lock screw on the valve at the front of the syringe port.
- Screw the syringe fully into the port
- Re-tighten the lock screw.
- Screw the fittings and the plugs back into the correct ports.
- Ensure that the marking on both the valve drive shaft on the pump and the shaft on the valve are both in the initialize position – marking at top, see pictures above.
- Screw the valve onto the pump on the unit.
- Pull/push the syringe to the correct position to allow you to screw it back on to the pump.
- Reset the home position for the pump (see below)

Figure 9.14: Syringe

- Power the unit Off – On - Off: The pumps set the pump valves to the initialize position.
- Remove the screw securing the syringe to the pump at the bottom of the syringe.
- Remove the 2 screws (3/32” Alan Key supplied) securing the valve to the pump.
- Pull the valve together with the syringe and the connected fittings forward and away from the pump.
- Leave the fittings and tubing on the valve.
- Loosen (1 turn anticlockwise) the small lock screw on the valve at the front of the syringe port.
- Unscrew and remove the syringe from the valve.
- Screw the new syringe fully into the valve port
- Re-tighten the lock screw.
- Ensure that the marking on both the valve drive shaft on the pump and the shaft on the valve are both in the initialize position – marking at top, see pictures above.
- Screw the valve onto the pump on the unit.
- Pull/push the syringe to the correct position to allow you to screw it back on to the pump.
- Reset the home position for the pump (see below)

Reset a Pump Home Position

- The pumps have a Home sensor near the top of the syringe movement. This sensor is detected several mm before the syringe piston reaches the end of the syringe. For correct operation the pump must *know* how far the home position is from the end of the syringe.
- Power up the system and wait until it has completed all of its internal initialize options (ca. 40secs)
- On the pump to be reset, using a small screwdriver (or Alan Key, pencil etc.) press the recessed **init** button.
- The pump valve moves to its **init** position (A) and the syringe is moved up until the home sensor is detected.
- Now turn the recessed **adjust** wheel from right to left. This moves the piston up towards the end of the syringe.
- Repeat until the piston reaches the end of the syringe and can be moved no further.
- Now place your finger on the center of the **adjust** wheel and move the wheel to the right.
- This moves the piston very slightly down from the end.
- Finally press the recessed **home** button. The pump now saves this position as its reference **0**.
- On the Service Web Page, see also *Embedded Web Server [▶ 47]* click on initialize for this pump (either double pump to initialize pumps 1 and 2 or Calibration pump to initialize pump 3).

Repair Declaration Form

- Use the Repair Declaration Form, whenever a device might be exposed to hazardous substances by customers, when it is to be returned to Bruker, see also *Safety and Repair Declaration Form [▶ 79]*.

10 Safety and Repair Declaration Form

Safety and Repair Declaration

Equipment Clearance Form for Service, Repair, Disposal or Transfer

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 ZFQS0083

Safety and Repair Declaration Form

11 Dismantling and Disposal

Following the end of its operational life, the device 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 is not covered by your warranty.

11.1 Safety

⚠ WARNING

Danger of injury from electrical shock!

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

- ▶ Only qualified personnel should open the housing.
- ▶ Disconnect the device from the electrical power supply before opening the device. Use a voltmeter to verify that the device is not under power!
- ▶ 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.

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

11.2 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.

11.3 Disposal

After the lifespan of the product, Bruker takes responsibility for disassembly and disposal in accordance with the European directive 2012/19/EC WEEE. Bruker BioSpin GmbH offers to take back the components free of charge after usage at the customer site upon request by the customer. If the customer wants to arrange disposal on their own, then this has also to be stated when the product is ordered.

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.

- ▶ Always observe local environmental regulations regarding handling and disposal of pollutants.
- ▶ 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.

12 Contact

Manufacturer:

Bruker BioSpin NMR
Silberstreifen
D-76287 Rheinstetten
Germany
Phone: +49 721-5161-6155
http://www.bruker-biospin.com
WEEE DE43181702

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

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Bruker Corporation

info@bruker.com
www.bruker.com

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