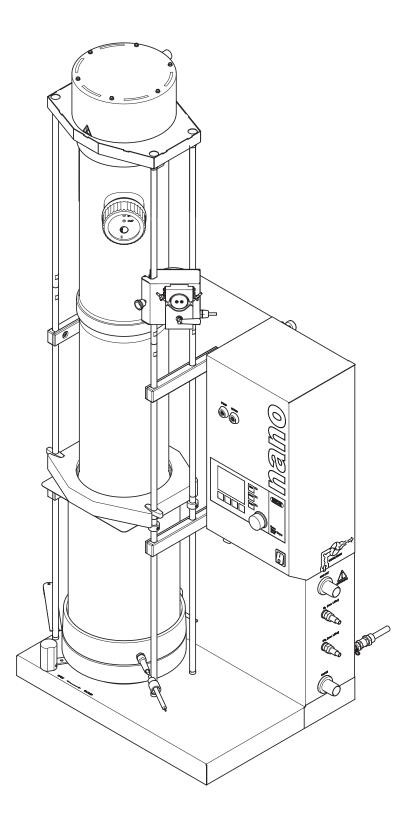


Nano Spray Dryer B-90 HP Operation manual





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1 About this manual

This manual describes the Nano Spray Dryer B-90 HP and provides all information required for its safe operation and to maintain it in good working order. It is an integral part of the product and is addressed to Isuitably qualified laboratory staff.

It is recommended to read this manual carefully and to note the safety precautions in chapter 2 before installing and running the system for the first time. The manual should be stored in the immediate vicinity of the instrument, so that it can be consulted at any time.

No technical modifications may be made to the instrument without the prior written agreement of BUCHI. Unauthorized modifications may affect the system safety or result in accidents.

BÜCHI Labortechnik AG accepts no liability whatsoever for any faults or damage that result from the failure to follow these manual.

1.1 Abbreviations

EPDM	Ethylene Propylene Dimonomer
FFKM	Perfluoroelastomers
FPM	Fluoroelastomer
PEEK	Polyetheretherketone
PTFE	Polytetrafluoroethylene (Teflon)
PE	Polyethylene
PPS	Polyphenylene sulfide
PVC	Polyvinylchloride
MMD	Medium Mass Diameter

1.2 Safety warnings and safety signals used in this manual

DANGER, WARNING, CAUTION and NOTICE are standardized signal words for identifying levels of hazard seriousness of risks related to personal injury and property damage. All signal words, which are related to personal injury are accompanied by the general safety sign.

For your safety it is important to read and fully understand the table below with the different signal words and their definitions!

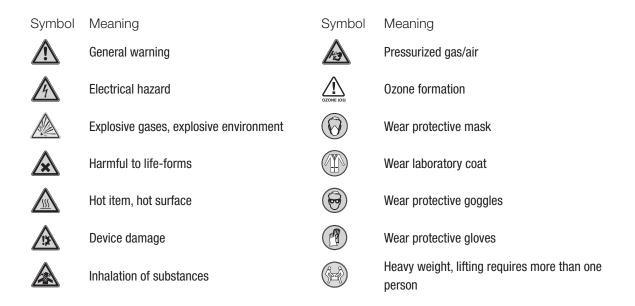
Sign	Signal word	Definition	Risk level
	DANGER	Indicates a hazardous situation which, if not avoided, will result in death or serious injury.	****
	WARNING	Indicates a hazardous situation which, if not avoided, could result in death or serious injury.	***☆
	CAUTION	Indicates a hazardous situation which, if not avoided, may result in minor or moderate injury.	****
no	NOTICE	Indicates possible property damage, but no practices related to personal injury.	★☆☆☆ (property damage only)

Supplementary safety information symbols may be placed in a rectangular panel on the left of the signal word and the supplementary text (see below example).

Space for	SIGNAL WORD
supplementary	Supplementary text, describing the kind and level of hazard / risk seriousness.
safety	List of measures to avoid the herein described, hazard or hazardous situation.
information	•
symbols.	•

1.3 Table of supplementary safety information symbols

The below reference list incorporates all safety information symbols used in this manual and their meaning.



1.4 Additional user information

Paragraphs starting with NOTE give helpful information for working with the device/software or its supplementaries. NOTEs are not related to any kind of hazard or damage (see example below).

NOTE

Useful tips for the easy operation of the instrument/software.

1.5 Copyright and trademarks

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Product names and registered or unregistered trademarks that are used in this instruction manual are

used only for identification and remain the property of the owner in each case. For example, Rotavapor® is a registered trademark of BÜCHI Labortechnik AG.

1.6 Available languages

If you need another language version of this manual, you can download available versions at www.buchi.com.

2 Safety

This chapter highlights the safety concept of the instrument and contains general rules of behavior and warnings from direct and indirect hazards concerning the use of the product.

For the users safety, all safety instructions and safety messages in the individual chapters shall be strictly observed and followed. Therefore, the manual must always be accessible to all persons performing the tasks described herein.

2.1 Proper use

The Nano Spray Dryer B-90 HP and the Nano Spray Dryer B-90 HP Advanced have been designed and built as laboratory instruments.

- The Nano Spray Dryer B-90 HP serves to spray-dry aqueous solutions, nanoemulsions or nanosuspensions in 'open loop'.
- The Nano Spray Dryer B-90 HP Advanced can additionally process organic-based solutions, nanosuspensions or nanoemulsions in 'closed loop' when combined with the Inert Loop B-295. Processing in "closed loop" requires the combined use of N₂ and CO₂.

If the instrument is used with potentially toxic or hazardous substances, it has to be installed inside a closed fume hood or glove box. In such cases, the complete processing and system handling has to be performed within the ventilated box to avoid toxication and other hazardous situations to the user and the environment.

In any case, all exhausts leaving the exhaust gas tubing have to be lead away instantly by a ventilation system, to remove possibly hazardous substances and fumes from the working area. The ventilation system has to be equipped with safety measures such as outlet filters to avoid contamination of the environment. When the Nano Spray Dryer B-90 HP is used in combination with other instruments all related manuals are to be fully observed.

2.2 Improper use

Applications not mentioned in section 2.2 are considered to be improper. Applications which do not comply with the technical data (see *section "3.4 Technical Data" on page 24* of this manual) are also considered to be improper.

The operator bears the sole risk for any damages or hazards caused by improper use!

The following uses are expressly forbidden:

- Use of gases that are not mentioned as proper drying gas in this operation manual.
- Spray drying of biohazard materials.
- Spray drying of organic solvents in 'open loop' and without the Inert Loop B-295.
- Installation or use of the instrument in rooms, which require ex-protected instruments.
- Spray drying of substances which might explode or ignite due to the processing, the selected parameters of the Nano Spray Dryer B-90 HP or the installed sub systems.
- Use of samples which might produce oxygen during the process.

NOTE

Spray drying liquids with a low conductivity can cause difficulties in terms of atomization.

2.3 Staff qualification

The instrument may only be used by laboratory personnel and other persons who based on training and professional experience know of the potential dangers that can develop when operating the instrument.

Unqualified persons are unable to identify risks and are therefore exposed to greater dangers. These operating instructions are aimed at the following target groups:

2.3.1 Users

Users are persons that meet the following criteria:

- They have been instructed in the use of the device.
- They are familiar with the contents of these operating instructions and the applicable safety regulations and apply them.
- They are able on the basis of their training or professional experience to assess the risks associated with the use of the device.

2.3.2 Operator

The operator (generally the laboratory manager) is responsible for the following aspects:

- The device must be correctly installed, commissioned, operated and serviced.
- Only suitably qualified staff may be assigned the task of performing the operations described in these operating instructions.
- The staff must comply with the locally applicable requirements and regulations for safe and hazard-conscious working practices.
- Safety-related incidents that occur while using the device should be reported to the manufacturer (quality@buchi.com).

2.3.3 BUCHI service technicians

Service technicians authorized by BUCHI have attended special training courses and are authorized by BÜCHI Labortechnik AG to carry out special servicing and repair measures.

Untrained personnel, or persons who are currently being trained, require careful supervision by a qualified person. The present Operation Manual can serve as a basis for training.

2.4 Product safety

Safety warnings in this manual (as described in section 2.4) serve to make the user alert and to avoid hazardous situations emanating from residual dangers by giving appropriate counter measures. Risks to users, property and the environment can however arise when the instrument is damaged, used carelessly or improperly.

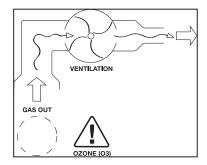
2.4.1 General hazards

The following safety messages show general hazards which may occur when handling the instrument. The user shall observe all the listed counter measures in order to achieve and maintain the lowest possible level of hazard.

Additional warning messages are indicated whenever actions and situations described in this manual are related to situational hazards.

2.4.2 Warning labels on housing

The warning label below can be found at the gas outlet connector on the right side of the Nano Spray Drver B-90.



Meaning:

Ozone formation in the electric field of the electrostatic particle collector when using oxygen containing gases as drying gas (like air). Withdraw gases via fume hood or equivalent ventilation system directly.

2.4.3 Safety measures

Always wear personal protective equipment such as protective eye goggles, protective clothing, inhalation protection and gloves when working with the instrument.

2.4.4 Built-in safety elements and measures

Heating:

- Overheating protection circuit
- Automatic temperature control of heater and inlet temperature

Spraying:

- Automatic spray control
- Overpressure leakage detection

High voltage and electrostatic charges:

- Safety current limitation
- Light barrier to detect the correct placement of the particle collector
- Internal grounding to remove electrostatic charges

Air/Gas:

- · Automatic processing interruption when system pressure is too low
- Outlet filter to protect the environment from the contamination by fine dust particles coming from the exhaust gas tubing

Glass:

• High temperature resistance and high transparency borosilicate glass

Pressure:

- Automatic pressure control at outlet filter
- Over pressure safety valve (opens at 300 mbar)

Optional system configuration with Inert Loop B-295:

- Overpressure leakage detection
- Safe gas condition (< 6 % O₂ content) is detected and enables the regulation of the drying gas mixture via a communication cable.

2.5 General safety rules

2.5.1 Responsibility of the operator

The head of the laboratory is responsible for training his/her personnel.

The operator shall inform the manufacturer without delay of any safety-related incidents which might occur during operation of the instrument. Legal regulations, such as local, state and federal laws applying to the instrument must be strictly followed.

2.5.2 Duty of maintenance and care

The operator is responsible for the proper condition of the instrument at use and that maintenance, service and repair jobs are performed with care and on schedule by authorized personnel only.

2.5.3 Spare parts to be used

Use only genuine consumables and genuine spare parts for maintenance to assure good system performance and reliability. Any modifications to the spare parts used are only allowed with the prior written permission of the manufacturer.

2.5.4 Modifications

Modifications to the instrument are only permitted with the written approval of the manufacturer. Modifications and upgrades shall only be carried out by an authorized BUCHI technical engineer. The manufacturer will decline any claim resulting from unauthorized modifications.

3 Product Description

The Nano Spray Dryer B-90 HP is available in two different set-ups:

Nano Spray Dryer B-90 HP Basic:

• For aqueous applications in 'open loop'

Nano Spray Dryer B-90 HP Advanced:

- For solvent based applications in combination with Inert Loop B-295
- Optional use with Dehumidifier B-296 Nano
- Equipped with additional oxygen safety circuit and gas regulation valves
- Accessories are resistant against most solvents.

This chapter explains the basic working principle of the Nano Spray Dryer B-90 HP and its accessories.

3.1 Functional principle

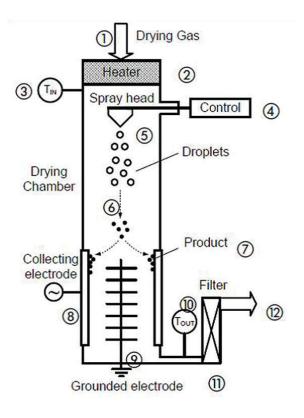
The Nano Spray-Dryer B-90 has been designed to produce particles from the submicron to the micrometer range, out of a solution, nanosuspension or nanoemulsion. High recovery yields can moreover be obtained thanks to its novel electrical particle collector.

3.1.1 Spray drying process

The spray drying process takes place inside the drying cylinder(s) (spray cylinder and optional extension cylinder) where tiny droplets of the sample material are ejected by the nebulizer into a hot pre-established drying gas flow, allowing the droplets to dry. Due to their small size, the produced particles can be collected from the drying gas flow by an electrode and an electrical field of sufficient power.

The spray drying process can be divided into three separated processes which need to be started in sequence. The first step is the establishment of a drying gas circuit to form the co-current gas flow accompanying the sprayed liquid; the particle formation process can then be started and will be accompanied by the particle collection process. The following draft offer a detailed overview of those processes.

3.1.2 Drying gas circuit



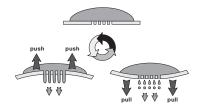
- The drying and transport gas enters the instrument via the "drying gas inlet" (1) and its flow is monitored by a flow meter influencing the gas regulator.
- The gas is then heated to the desired temperature (up to 120°C) (2) by a heating system consisting of an electrically heated porous metal foam which allows a minimized heat up time and the generation of a laminar gas flow towards the drying section.
- The heating control is based on a pluggable PT-1000 temperature sensor. The outlet temperature is measured by the outlet temperature sensor (10) which is located after the particle collector.
- Since a gas flow is established in the spray dryer, a pressure is built up and monitored by a relative pressure sensor that compares the internal pressure with the environmental pressure. In the event of a pressure loss, the sensor will cause a stop of the drying process.
- In order to retain possibly harmful particles towards the environment in open loop, an outlet filter (11) is fixed on the drying gas outlet (12) which works as an exhaust.
- In closed loop mode, the outlet filter is an essential part for cleaning the recycled gas from unwanted residues and impurities.
- An oxygen sensor is used in closed loop as a safety feature to monitor the percentage of oxygen in the drying gas. It will cause a stop of the process in case of emergency.

3.1.3 Particle formation and collection process

Once the gas flow is established, the spray can start and the particle formation process will take place in the spray cylinder and glass cylinder extension if the tall set up is used. The collection process will then happen on the collecting electrode.

• The feeding pump circulates the sample between the product vessel and the spray head, assuring a constant substance feeding for the spray generation.

• The nebulizer inside of the spray head (5) generates precisely controlled micro droplets using piezotechnology and ejects them into the spray cylinder.



- While being carried through the drying cylinder(s) (spray cylinder and optional extension cylinder) (6) by the laminar drying gas flow, the droplets will then gently be dried into solid particles (7).
- The collecting electrode (8) retrieves the dried particles using an electric field generated between the collecting electrode and the HV-electrode (9). The HV-electrode deflects the particles and the collecting electrode attracts them.
- The dried solid particles hence accumulate on the inside of the collecting electrode. The exhaust gas leaves the spray dryer via the "outlet filter" which retains free particles from the gas.

3.1.4 "Open loop" and "closed loop" modes

In "open loop" mode, the drying gas can either be supplied by an in-house compressed air, an oil-free compressor or the BUCHI Aspirator (see *section "3.7 Aspirator" on page 28*). When compressed air is being used, it is recommended to use the air maintenance unit to assure air of clean, dry and oil free quality. A pressure regulating valve is required to match the instruments input pressure value.

If no compressed air is available, the BUCHI Aspirator unit with inlet filter can be used to establish the required drying air flow rate. The aspirator can be connected directly to the inlet of the Nano Spray Dryer B-90 HP without a pressure regulating valve.

In "closed loop" mode, with the Nano Spray Dryer advanced, the gas stream is built up using the Aspirator. The drying gas outlet is then connected via the Dehumidifier B-296 Nano (optional), the Inert Loop B-295 and the Aspirator to the "Drying gas inlet". More information about those accessories can be found in *section "5.3.2 'Closed loop' gas supply configuration" on page 50*.

NOTE

If the air humidity is too high, the current in the particle collector could be too low. In that case, ambient humidity can be reduced by the use of the Dehumidifer B-296 Nano.

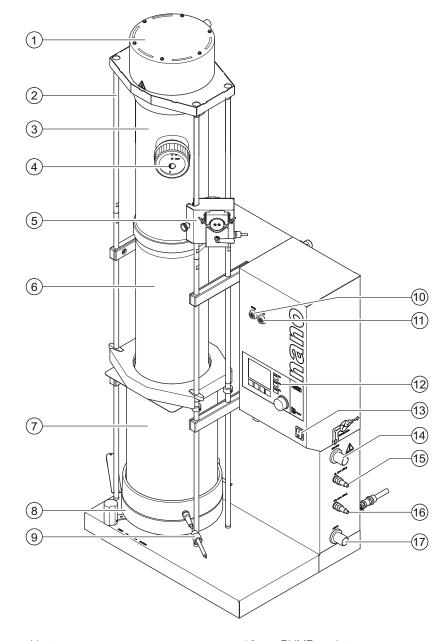
NOTE

It is usually observed that:

- the smaller the mesh size, the smaller the ejected droplets and thus the dried particles
- for a fixed vibration frequency, the smaller the mesh, the lower the throughput.

3.2 Configuration Nano Spray Dryer B-90 HP

3.2.1 Front View



1	Heater	10	PUMP socket
2	Metal bars	11	SPRAY socket
3	Spray cylinder	12	Display
4	Spray head	13	Main switch
5	Peristaltic pump	14	GAS OUT
6	Glass cylinder extension	15	N ₂
7	Insulation glass cylinder	16	AIR (CO ₂)
8	Lifting device	17	GAS IN

9

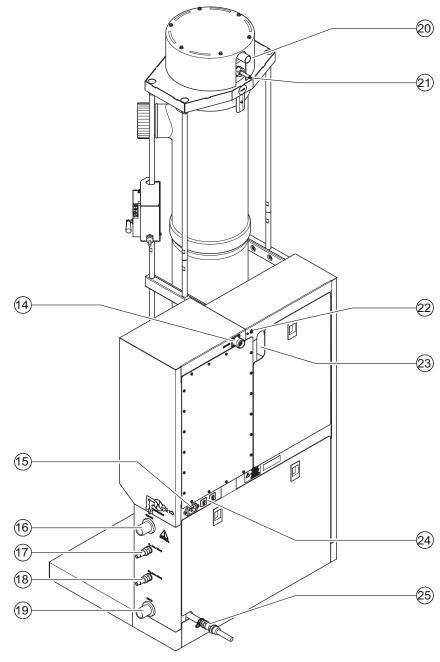
HIGH VOLTAGE plug

3.2.2 Front connections



Pos.	Socket label:	Description:
1 2 3	HIGH VOLTAGE T _{out} B-295	Socket for particle collector with plug thread for reliable plug locking Outlet temperature sensor socket Inert Loop B-295 communication socket
4	USB	Port for data records to PC

3.2.3 Rear and side connections

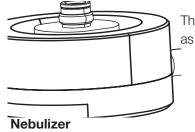


- 14 HEATER Socket for heater plug
- 15 Socket for mains plug
- 16 GAS OUT
- 17 N₂
- 18 AIR (CO₂)
- 19 Gas IN

- 20 Gas inlet
- 21 Inlet temperature sensor
- 22 T_{IN} Inlet socket for temperature sensor
- 23 Gas tube to heater
- 24 Fuses, resettable
- 25 Pressure relief valve

3.3 Components

3.3.1 Spray head



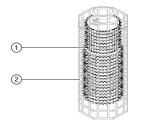
The spray head incorporates the nebulizer and provides electrical connection as well as bypass for spraying solution.

3.3.2



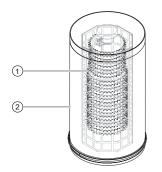
The nebulizer generates precisely controlled micro droplets from bulk liquids using the piezotechnology. It includes a piezoelectric actuator with a thin stainless steel membrane. The centre of the membrane contains an array of micron-sized holes and vibrates at ultrasonic frequencies, allowing the ejection of precisely sized droplets at high speed.

3.3.3 HV-Electrode



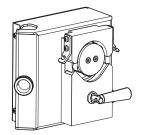
The HV-Electrode (1) creates an electric field enabling the collection of the sprayed particles on the collection electrode. A special protective grid (2) is supplied with the HV-Electrode so that it can be removed and cleaned safely by the user.

3.3.4 Collecting electrode



Solid particles are accumulated at the surface of the cylindrical particle collecting electrode (2) by a strong electrical field generated via high voltage by HV-Electrode (1).

3.3.5 Peristaltic pump

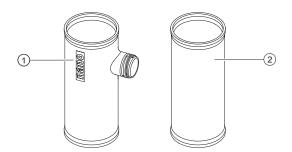


The peristaltic pump recirculates the solution to be sprayed between the containing vessel and the spray head.

NOTE

Dry run of the pump does not damage the instrument or parts of it in the medium term.

3.3.6 Spray Cylinder and Glass Cylinder Extension



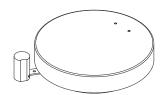
The drying process takes place inside the spray cylinder (1). In order to increase the residency time, an additional glass cylinder (2) (cylinder extension) can be added.

3.3.7 Heater



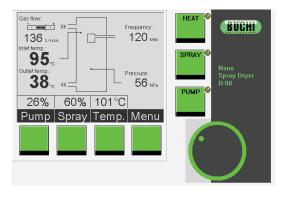
The heater provides optimal energy input to heat the drying gas.

3.3.8 Lifting device



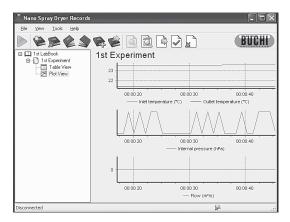
The lifting device enables the opening and the sealing of the nano spray-dryer.

3.3.9 Display



The display allows visualization and control of the process parameters.

3.3.10 "Nano spray dryer records" software



The PC-software enables online data monitoring and storage of experimental runs in a virtual library.

3.3.11 Outlet filter



The outlet filter recovers small particles so that they don't get released into the environment.

3.3.12 Oxygen analyzer and oxygen sensor



The oxygen sensor can be found in the advanced model. It is used to supervise the oxygen percentage in the drying gas in closed loop mode.

3.3.13 Type Plate



Each accessory has its own type plate at the rear. On the type plate the following information can be found:

- company name and address
- device name
- serial number
- input voltage
- frequency
- maximum power rating
- year of manufacture

The following additional information can usually be found on the type plate:

- country of manufacture
- approvals
- product code
- fuse rating
- how to dispose of the device

3.4 Technical Data

This chapter introduces the reader to the instrument and its accessories. It contains technical data and performance data.

The scope of delivery can only be checked according to the individual delivery note and the listed order numbers.

NOTE

For additional information about the listed products, see www.buchi.com or contact your local supplier.

3.4.1 Technical data Nano Spray Dryer B-90 HP

Technical data Nano Spray Dryer B-90 HP	
Dimensions (W x H x D)	58 x 110 x 55 cm (Short set-up) 58 x 150 x 55 cm (Tall set-up)
Weight	65 kg (Short set-up) 70 kg (Tall set-up)
Electricity	
Power consumption	max. 1500 W
Connection voltage	100–240 VAC \pm 10 %
Input fuse	12 A
Frequency	50/60 Hz
Heating capacity	max. 1400 W
Overvoltage category	ll
Drying process	
Max. inlet temperature	120 °C
Evaporating capacity	max. 0.2 L/h H_2^0
	(higher rates with organic solvents are possible)
Drying gas flow	80–160 L/min
N_2 or CO_2 inert gas input pressure	max. 2 bar
Nebulizer	small, medium, large
Mean droplet size range	3-15 μm MMD
Mean particle size range	200 nm – 5 μm
Spray frequency	80 – 140 kHz
Mean residence time	1 – 4 sec.
Interface	USB II
Environmental conditions	
Pollution degree	2 (for indoor use only)
Temperature	5 – 40 °C
Altitude (above sea level)	up to 2000 m
Humidity (curve parameter)	Maximum relative humidity 80 % up to 31 °C, then decreasing linearly to 50 % relative humidity at 40 °C

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3.5 Inert Loop B-295

3.5.1 Function

The Inert Loop B-295 is an accessory that enables the safe spray drying of organic solvents in a 'closed loop' configuration. It includes a cooling unit with pressure and oxygen monitoring elements and safety controls. Communication to the Nano Spray Dryer B-90 HP Advanced is established via a communication cable.



NOTE

If mixtures of organic solvents and water are used in 'closed loop' configuration, BUCHI strongly recommends to install the additional Dehumidifier B-296 Nano. The Dehumidifier B-296 Nano is condensing the water to prevent water from entering into the Inert Loop B-295. This is necessary to protect the heat exchanger in the refrigerator from water freezing and causing potential damage.

3.5.2 Type Plate

See section "3.3.13 Type Plate" on page 23.

3.5.3 Technical Data Inert Loop B-295

Technical data Inert Loop B-295	
Power consumption	max. 1.4 kW
Connection voltage	200 VAC ±10%
	230 VAC ±10%
Frequency	50 or 60 Hz
Min. outlet temperature	down to -20 °C
Rate of cooling	800 W at -10 °C
Overvoltage category	ll
Dimensions (W×H×D)	60×70×84.5 cm
Weight	110 kg
Environmental conditions	
Pollution degree	2 (for indoor use only)
Temperature	5 – 40 °C
Altitude (above sea level)	up to 2000 m
Humidity (curve parameter)	Maximum relative humidity 80 % up to 31 °C, then decreasing linearly to 50 % relative humidity at 40 °C

3.6 Dehumidifier B-296 Nano

3.6.1 Function



The Dehumidifier B-296 Nano is an accessory which can dry gases at constant and reproducible humidity conditions with a dew point of 3 to 5 °C. The humidity condensates while passing the refrigerator of the Dehumidifier B-296 Nano and is being collected in a closed condensate bottle.

3.6.2 Type Plate

See section "3.3.13 Type Plate" on page 23.

3.6.3 Technical Data Dehumidifier B-296 Nano

Technical data Dehumidifier B-296 Nano	
Power consumption	700 W
Connection voltage	200 VAC ±10%
	230 VAC ±10%
Frequency	50/60 Hz
Min. outlet temperature	+ 2 °C
Rate of cooling	400 W at 0 °C
Overvoltage category	ll
Dimensions (W×H×D)	35×40×60 cm
Weight	36 kg
Environmental conditions	
Pollution degree	2 (for indoor use only)
Temperature	5 – 40 °C
Altitude (above sea level)	up to 2000 m
Humidity (curve parameter)	Maximum relative humidity 80 % up to 31 °C, then
	decreasing linearly to 50 % relative humidity at 40 $^{\circ}\mathrm{C}$

3.7 Aspirator

3.7.1 Function



The Aspirator works as a pump to establish a constant gas flow. It has a bench top dimension of $47,5\times30\times32$ cm and can be placed beside the Nano Spray Dryer B-90 HP Advanced or on the tray of a trolley.

3.7.2 Type Plate

See section "3.0.20 Type Plate" on page 29.

3.7.3 Technical Data Aspirator

Power consumption	max. 1000 W	
Connection voltage	200–240 VAC ± 10 %	
Input fuse	FST 5×20 T8A L 250 V	
Frequency	50/60 Hz	
Dimensions (W×H×D)	47,5×30×32 cm	
Weight	20 kg	
Overvoltage category		

Pollution degree	2 (for indoor use only)
Temperature	5 – 40 °C
Altitude (above sea level)	up to 2000 m
Humidity (curve parameter)	Maximum relative humidity 80 % up to 31 °C, then
	decreasing linearly to 50 % relative humidity at 40 °C

4 Transportation and Storage

4.1 Transport

Attention	
Risł	c of breakage due to incorrect transportation
•	Make sure that all parts of the device are safely packed in such a way as to prevent breakage,
	ideally in the original box.
•	Avoid sharp movements during transit.

- After transportation, check the device for damage.
- Damage that has occurred in transit should be reported to the carrier.
- Keep the packaging for future transportation.

4.2 Storage

- Make sure to comply with the ambient conditions.
- Wherever possible, store the device in its original packaging.
- After storage, check the device for damage and replace damaged parts if necessary.

5 Putting into operation

This chapter describes how the instrument has to be installed. It also gives instructions for the initial startup.

NOTE

Inspect the instrument for damages during unpacking. If necessary, prepare a status report immediately to inform the postal company, railway company or transportation company. Keep the original packaging for future transportation.

5.1 Installation site

Put the instrument on a stable, horizontal surface. Consider the maximum product dimensions and weight. Installation on a mobile trolley is also possible. Obtain the environmental conditions as described in section "3.4.1 Technical data Nano Spray Dryer B-90 HP" on page 24.

NOTE

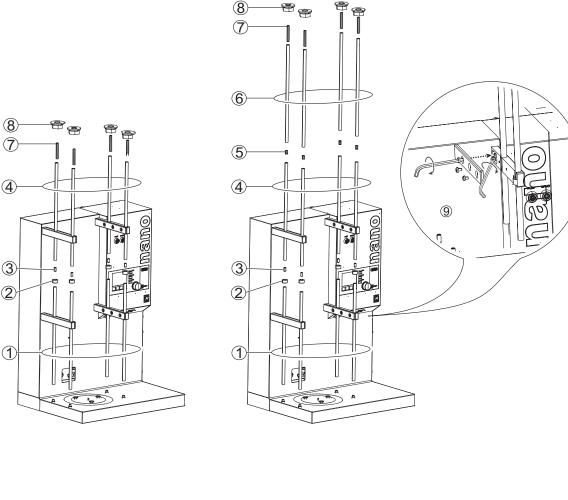
To cut the power in case of an emergency by unplugging, access to the mains plug must not be blocked by any instrument or item.

	A Warning			
/!\	Death or serious injuries by use in explosive environments			
	Do not operate the instrument in explosive environments.			
	Do not operate the instrument with explosive gas mixtures.			
	Before operation, check all gas connections for correct installation.			
	Directly withdraw released gases and gaseous substances by sufficient ventilation.			
	A Caution			
	Risk of minor or moderate injury by heavy weight of the instrument			
	• Due to heavy weight do not transport the instrument on your own.			

- Do not drop the instrument.
- Place the instrument on a stable, even and vibration-free surface.
- Keep limbs out of crushing zone.

5.2 Installing the Nano Spray Dryer B-90 HP

5.2.1 Installing in short / tall set-up with short / long metal bars



- 1 Lower metal bars
- 2 Adjusting rings
- 3 Short headless screws

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4 Middle metal bars

- 5 Upper metal bars
- 6 Long headless screws
- 7 Counter nuts
- 8 Bar holders



Caution

Risk of electrical hazard

• Switch off the instrument and remove the power cord before any work.

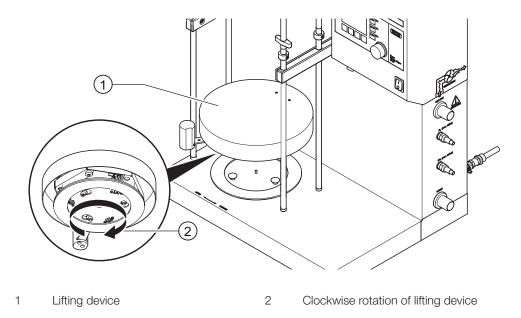
NOTE

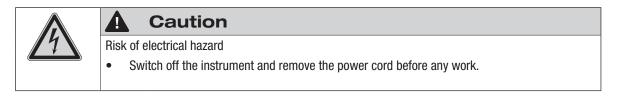
Parts (1) - (4) and the black holders (8) are factory pre-installed.

- Screw the four lower metal bars (1) into the housing basement with a flat spanner.
- Fix the four adjusting rings (2) onto the previously installed metal bars.

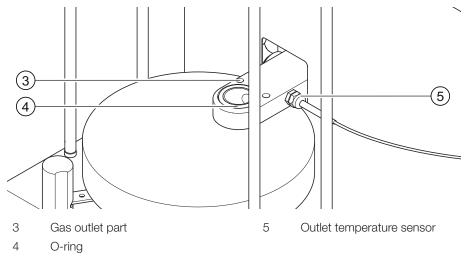
- Mount four short headless screws (3) onto the lower metal bars(1). Use a hex-wrench to screw them tightly in.
- Screw the middle metal bars (4) tightly onto the lower ones.
- Tall Set-up only: Mount four short headless screws (3) onto the middle metal bars. Use a hex-wrench to screw them tightly in.
- Tall Set-up only: Screw the four upper metal bars (5) onto the middle ones and screw them tightly together.
- Mount the four long headless screws (6) onto the upper / middle metal bars. Use a hex-wrench to screw them tightly in.
- Put the four counter nuts (7) upside-down and onto the long headless screws on top of the bars and turn them clockwise to the lowest possible position.
- To install the bar holders (8), first screw the holder base at the housing. Use the metal bars for alignment. Subsequently attach the side of the holders with the side caps and three screws.

5.2.2 Installing the lifting device





- Position the lifting device, using the three bottom sided timing holes, onto the matching knobs at the instrument's basement.
- Rotate the lifting device some degrees counterclockwise to fix it tightly by hand. The lever has to be positioned towards the front.

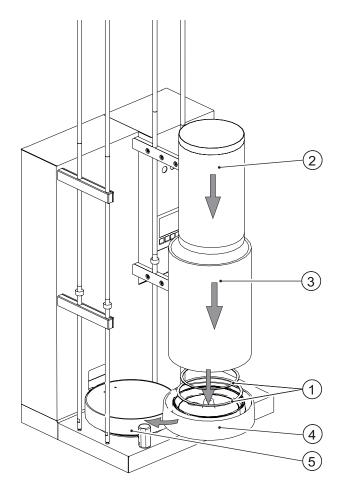


- Screw the gas outlet part onto the lifting device with two hex-wrench screws.
- Take the small o-ring from the set of sealings. Place it on the sealing seat of the gas outlet part.
- Install the outlet temperature sensor at the gas outlet part. For the correct installation depth

keep the sensor housing approximately 70 mm out of the gas outlet part. Use a flat spanner to fix the screw fitting of the sensor housing tightly

- Subsequently connect the outlet temperature sensor plug to socket.
- After installation place it on "open".

5.2.3 Installing the electrode assembly



- 1 Flat seal
- 2 Metal collecting tube electrode
- 3 Insulation glass cylinder
- 4 Particle collector basement
 - Lifting device



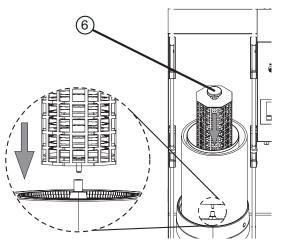
5

Caution
Risk of minor or moderate injury by electric shock.
• Switch off the instrument before connecting or disconnecting the high-voltage cable
Do not touch the high-voltage cable with wet hands
Use only damage free cabling

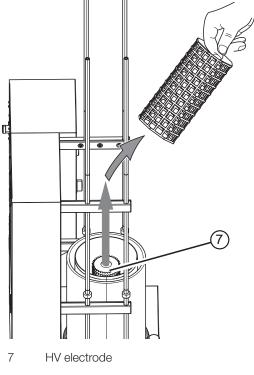
NOTE

Before installing the electrode, clean parts (1) to (5) thoroughly.

- Position the flat seal (1) at the particle collector basement (4).
- Position the metal collecting tube electrode (2) on the collector basement (4) and assure electrical contact.
- Install the insulation glass cylinder (3) on the collector basement (4).
- Place the assembly on the lifting device (5).

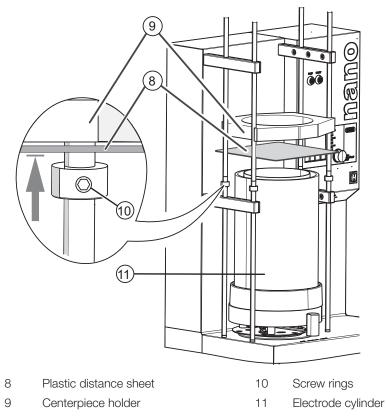


- 6 Protective grid holder
- Use the protective grid holder (6) to carefully insert the HV-electrode (7) into its seat at the bottom plate of the particle collector. Clean and dry this part before installation.



To unlock the protective grid (6), open the spring lock at the handling knob first. Carefully lift off the grid from the HV electrode (7) in a vertical upwards movement.

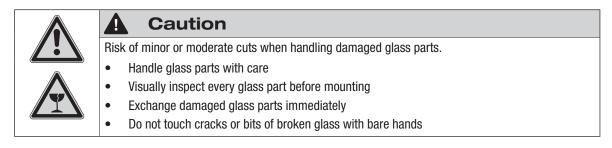
► Finally, connect the electrical cable of the particle collector to the high voltage socket at the frontside of the instrument (see section "3.2.2 Front connections" on page 18) and fix it hand-tight.



- Put the plastic distance sheet (8) on top of the electrode cylinder (11).
- Put the centerpiece holder (9) on the plastic distance sheet.
- Adjust the four screw rings (10) on the metal bars to touch the underside of the distance sheet. Fix them tightly with an hex-wrench.
 - Finally, remove the plastic distance sheet by pulling it out towards the front.

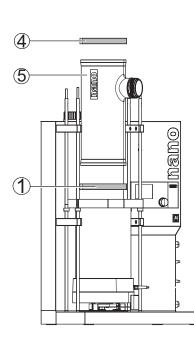
After correct installation it is possible to remove the complete electrode cylinder assembly towards the front.

5.2.4 Installing the glass cylinder assembly in short / tall set-up



NOTE

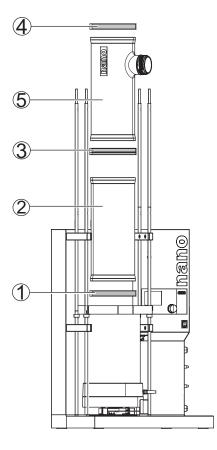
Clean every glass part before mounting to avoid cross contamination.



- 1 Flat sealing ring
- 2 Glass cylinder
- 3 Glass-to-glass sealing

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4 Second flat sealing ring

5 Spray cylinder



Caution

Risk of electrical hazard

Switch off the instrument and remove the power cord before any work.

Tall set-up

- Mount a flat sealing ring (1) on the bottom of the lower glass cylinder (2).
- Place the glass cylinder (2) on the centerpiece holder.
- Position the glass-to-glass sealing (3) on top of the lower glass cylinder. The smaller diameter has to be on top towards the spray cylinder.
- Position the second flat sealing ring(4) on top of the spray cylinder (5).
- Place the spray cylinder on top of the glass-to-glass sealing (3).

All sealings are part of the sealing set.

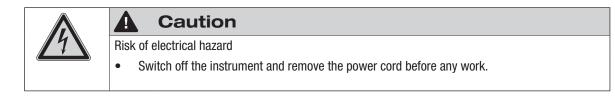
Short set-up

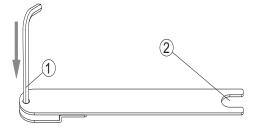
Mount a flat sealing ring (1) on the bottom of the spray cylinder (5).

- Position the second flat sealing ring (4) on the spray cylinder (5).
- Place the spray cylinder (5) on the centerpiece holder.

5.2.5 Installing the heater module

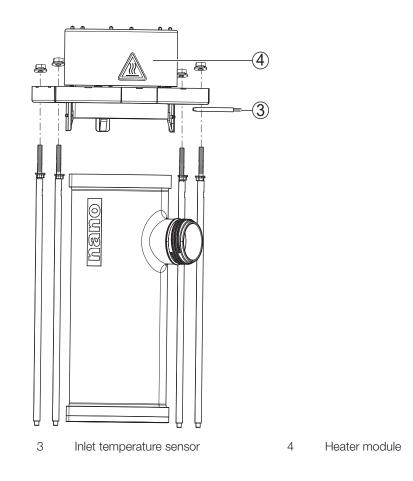
Caution
Risk of minor or moderate burning when handling hot parts.
Do not touch hot parts
Let the system cool down for some minutes after use





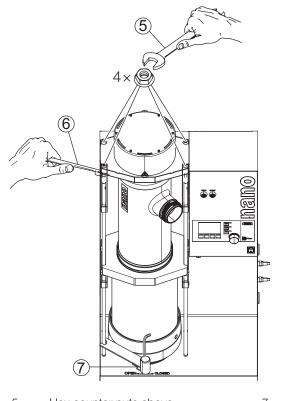
The special mounting tool serves as a mounting gauge for the correct distance of all mounted parts between lifting device and heater.

- Put the lever arm of the lifting device into the end of the tool first (1). The tool and the handle are equipped with holes. Line up all holes and insert a hex-wrench through the holes to stabilize the correct tool position.
- Use the left metal bar (2) to support the tool end.



The lifting device is now in mounting height for the heater module installation.

- Mount the inlet temperature sensor (3) at the heater module (4). Keep the temperature sensor end about 70 mm out of the screw connection. Use a flat spanner to tighten the screw fitting.
- Place the heater module on the metal screw bars.
- Fix 4 hex nuts on the metal bars, aligned with the spraying cylinder, flat end towards the top of the bars.
- Position the heater module on the metal bars.
- Fix 4 hex nuts on the metal bars, over the heater, flat end towards the heater.



5 Hex counter nuts above 7 Mounting tool

- 6 Hex counter nuts below
- Make sure the spray cylinder is in-line with the heater module and that the sealing is properly placed in between.
- Tighten the hex counter nuts above (5) and below (6) the heater crosswise. Do not overtighten the nuts.
- Close the system and remove the mounting tool (7) from the lifting device after installation.
- Connect the electrical cable of the heater to the back side of the instrument.
- Connect the inlet temperature sensor (3) to its socket at the back side of the instrument.
- Connect the drying gas tubing to the inlet of the heater module (4). Use a hose clamp for fixation.
- Open the back panel on the right.
- Connect the drying gas tubing between the heater inlet and the connector nipple inside the instrument (open the upper back panel of the instrument to get access).

5.2.6 Assembling the spray head

The spray head is a major part of the whole spray drying process. To ensure proper function and highest yield, dry and clean conditions of all parts during assembling and installation are required. For detailed cleaning information see *section "7.6 Cleaning" on page 84*.



Caution

Risk of minor or moderate burning when handling hot parts.

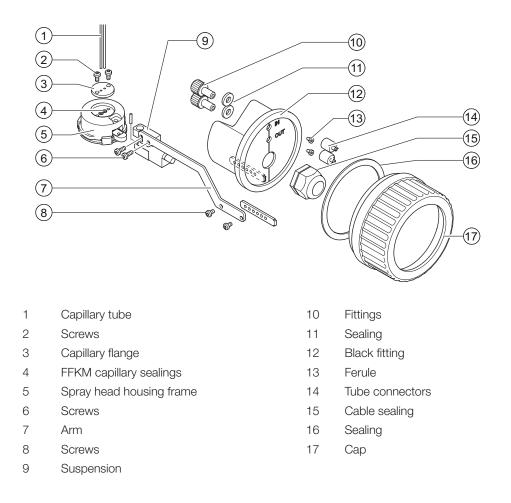
- Do not touch hot parts
- Let the system cool down for some minutes after use

NOTE

Only use original BUCHI parts for installation.

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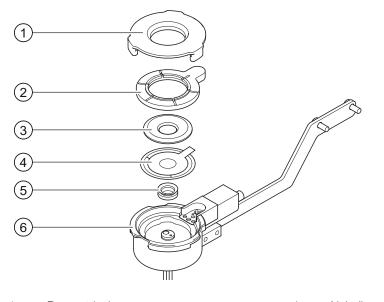


Put the sealing onto the two fittings and screw them into the head holder hand-tight.

- Put the black fitting around the head holder.
- Mount the spray head on the head holder.
- Insert the electrical cable by pulling it through the cable hole of the head holder.

- Fit the rubber sealing on the electrical cable and tighten the cable using a flat spanner.
- Put the small FFKM sealings around the capillary tubes and insert them into the spray head housing frame.
- Fix the capillaries by screwing the capillary flange onto the head housing frame.
- Introduce the feed capillaries into the fittings and attach the ferules to the capillaries.
- Screw the tube connector onto the fittings.

5.2.7 Mounting the nebulizer on the spray head



1	Bayonet lock	4	Nebulizer
2	Lock washer	5	Reservoir gasket

- 3 Visor gasket 6 Spray head housing frame
- Fix the reservoir gasket (5) in the spray head. The lip of the gasket has to point towards the nebulizer.
- Carefully insert the nebulizer (4) into the spray head (6), metallic part at the bottom, electrical circuit on top (dome targeting to the outside). Make sure the pin of the spray head is fixed through the hole of the nebulizer to stabilize it.
- Fit the visor gasket (3) into the PEEK lock washer (2). The shiny part of the visor gasket should be against the lock washer. Fit this part on top of the nebulizer. The slots must be outside of the head.
- Close the spray head with the metallic bayonet lock (1) by rotating it clockwise.
- Install the spray head in the spray cylinder.
- Align the "Tube connectors" and the electrical cable connection vertically, so that the head is spraying downwards.
- Connect the spray head cable to the socket labelled with "SPRAY" at the housing front.

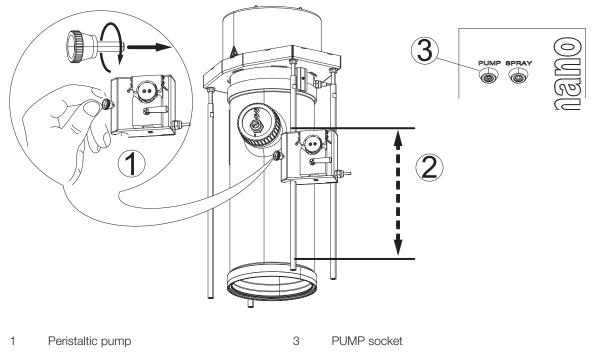
5.2.8 Adjusting and fixing the peristaltic pump height

The peristaltic pump uses a pulley drive to transport liquids through the tubing. The pump design is dry run safe. During the spray drying process it constantly feeds the spray head with sample liquid.

Notice

Risk of instrument short-circuits and damage by liquids

- Do not spill liquids over the instrument or parts of it
- Wipe off any liquids instantly
- · Place the sample vessel onto the designated reservoir-plate on top of the instrument
- Ensure a safe positioning of the sample vessel
- Do not move the instrument when it is loaded with liquid
 - Keep external vibrations away from the instrument



- 2 Height of the peristaltic pump
- Mount the peristaltic pump on a metal bar and fix it with the thumb screw.
- To adjust the height (2), unfasten the thumb screw and move the pump to the optimal height. Fasten the thumb screw again to fixate the pumps position.
- Connect the power cable of the pump to the socket labelled with "PUMP" (3) at the housing front.

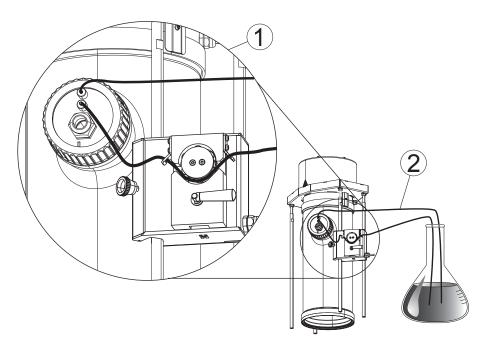
5.2.8.1 Adjusting the pump bed

- Turn the lever horizontally to the right. Pre-adjust the pump bed to working position for liquid transport with a hex-wrench. The correct position depends on the thickness of the transport tube material.
- Place the tube in the pump mechanism. Swivel the lever to the left. The pump is set up correctly when the tube is squeezed between the pulleys without damaging the tube.

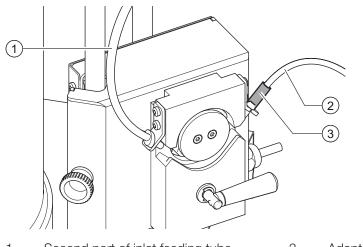
5.2.8.2 Installing the feeding tube

Steps before installation:

- Switch off the Nano Spray Dryer B-90 HP.
- Place the sample vessel on the top cover of the instrument or on the stand plate.
- Choose the appropriate tube depending on the solvent being used (see section "Choosing the feeding tube material" on page 47).



- 1 Feeding tube connected with spray head 2 Feeding tube in the sample vessel
- Install the feeding tube in the peristaltic pump first. Make sure the plastic manifold is seated in the right holder of the pump mechanism (see example photo below).
- Connect the tube on the inlet of the spray head (1). The inlet and outlet positions depend on the internal tubing of the spray head.
- Place the free end of the tube in the sample vessel (2).
- Connect a second tube on the outlet of the spray head and place its free end in the sample vessel.



Second part of inlet feeding tube
 First part of inlet feeding tube
 Adapter piece

Illustration with attached feeding tube and adapter piece (transparent adapter on the pump inlet at the right side).

Choosing the feeding tube material			
Solvent	Silicone tube	Tygon MH 2375	Tygon F 4040 A
Methanol	suitable	suitable	suitable
Ethanol	suitable	suitable	suitable
Acetone	unsuitable	suitable	unsuitable
Toluene	unsuitable	unsuitable	suitable
Isopropanol	suitable	suitable	suitable
Chloroform	unsuitable	unsuitable	unsuitable
Dichloromethane	unsuitable	unsuitable	unsuitable
THF	unsuitable	unsuitable	unsuitable
Ethylacetate	unsuitable	suitable	unsuitable
Hexane	unsuitable	unsuitable	suitable
Water	suitable	suitable	suitable

5.3 Drying gas supply configuration

The choice of drying gas supply (air or inert gases) depends on the processed sample. There are significant differences between 'open loop' and 'closed loop' configuration of supply and exhaust gases. Strictly follow the configuration descriptions in this section as well as the defined proper and improper use in section 2 for safe operation.

A Danger
Death by suffocation or serious poisoning by inhalation of inert gases.
Do not inhale inert gases
Directly withdraw released gases and gaseous substances by sufficient ventilation
Only operate the instrument in ventilated environments
• Before operation, check all parts, connections and sealings involved in the gasflow for proper
sealing
Exchange worn out or defective parts immediately

A Warning
Death or serious poisoning by contact or incorporation of harmful substances at use.
Before operation, check the instrument for correct assembling
Before operation, inspect sealings and tubes for good condition
Exchange worn out or defective parts immediately
Exchange clogged filters immediately
Only operate the instrument in ventilated environments
Directly withdraw released gases and gaseous substances by sufficient ventilation
Perform a dry-run without sample material and check for gas leakages

Warning
Death or serious injuries by compressed gas.
Depressurize gas circuit before any modification
Wear safety goggles

	Notice
! \$	Risk of instrument damage by wrong gas supply.
	All labels at gas connections must be clearly legible
	Before operation, check all gas connections for correct installation
	External supply pressure must meet the system specifications

5.3.1 'Open loop' air supply configuration

In 'open loop', compressed air or the Aspirator with inlet filter in blowing mode is required to establish the necessary drying gas overpressure. To ensure the optimal drying air performance in this operation mode, the supplied air has to be dry and free of impurities. If this can not be guaranteed, BUCHI recommends the use of a Wilkerson air maintenance unit.

Danger

Death by suffocation or serious poisoning by inhalation of exhaust gases.

- Do not inhale exhaust gases
- Directly withdraw released gases and gaseous substances by sufficient ventilation
- Only operate the instrument in ventilated environments
- Before operation, check all parts, connections and sealings involved in the gasflow for proper sealing
- Exchange worn out or defective parts immediately

A Danger
Risk of minor poisoning by inhalation of ozone.
Directly withdraw released gases and gaseous substances by sufficient ventilation

When working with pressurized gases:

- Turn off the pressure source and remove the power cord before installation work.
- Depressurize every part of the system and the supply tubes.

NOTE

If there is no compressed air available, the compressed air can also be supplied by an oil-free compressor.



The optional Wilkerson air unit is to be installed between a compressed air source and the Nano Spray Dryer B-90 HP air inlet. For installation, see documentation of the Wilkerson air maintenance unit!



GAS OUT (Polypress with clamp fitting) 3 Flow

- Flow regulation valve (only necessary with direct compressed air supply)
- 2 GAS IN (Polypress/Nylflex with clamp fitting)

The drying air connection is located on the right side of the instrument (see *section "3.2.3 Rear and side connections" on page 19* for details). The connections are made via Polypress and Solaflex tubing in combination with clamp fittings. Any exhaust gases must be safely withdrawn (e.g. through a fume hood).

5.3.2 'Closed loop' gas supply configuration

1

The Nano Spray Dryer B-90 HP Advanced has been designed to be used in 'closed loop'. This mode is universal and can be used to spray dry aqueous or solvent based samples with air or inert gas under safe conditions. To ensure the optimal drying gas performance in this operation mode, the supplied gas has to be dry and free of impurities.



1 GAS OUT (Polypress with clamp fitting)

2 N₂ (quick coupling)

- AIR (CO₂) (quick coupling)
- 4 GAS IN (Polypress with clamp fitting)

The drying gas connections are located on the right side of the instrument (see *section "3.2.3 Rear and side connections" on page 19* for details). The connections are made via Polypress and Nylflex tubing in combination with clamp fittings and via quick coupling connection. The choice of connection depends on the type socket and on the transported gas. The output gas is recycled by an Inert Loop B-295/Dehumidifier B-296 Nano. In 'closed loop' mode, the Aspirator works as a recirculating pump to establish a constant gas flow.

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5.4 Installing / replacing the outlet filter

The Nano Spray Dryer B-90 HP is equipped with a safety outlet filter. The filter should be replaced when clogged. This is indicated by the appropriate error message. Switch off the instrument, remove the power cord and depressurize the system and the supply tubes bevore starting to disassemble.

Danger

Death or serious poisoning by gases or particles at 0₂-sensor or filter malfunction

- Exchange defective 0,-sensor immediately
- Exchange 0₂-sensor regularly within the specified maintenance intervals
- Exchange clogged filters immediately
- Exchange filters regularly within the specified maintenance intervals
- Safely dispose filter immediately



Notice

Risk of instrument damage by internal overpressure.

- External supply pressure must meet the system specifications
- Exchange clogged filters immediately
- Safely dispose filter immediately



- Disconnect the power cord from the mains.
- Gently pull off the back panel of the housing and put it aside. The outlet filter is now directly accessible.
- Loosen the hose clamps to remove the filter element.
- Put in the new filter and fasten the hose clamps. Reinstall the back panel.
- Connect the power cord to the mains. The device can now be switched on.

NOTE

The filter has a designated gas flow direction. To ensure maximum throughput and filter lifetime it must be installed correctly. The designated flow direction is indicated by an arrow on the filter housing. For correct alignment, the arrow has to point towards the gas outlet connector of the Nano Spray Dryer B-90 HP when installing the filter. After exchange, dispose the used filter according to local regulations immediately.

5.5 Aspirator installation modes

To establish the required pressure for the drying gas flow, the Aspirator can be used in blowing mode. The Aspirator can be part of an 'open loop' configuration. In 'closed loop' the use of an Aspirator is required.

The Aspirator has a bench top dimension of 47,5×30×32 cm and can be placed beside the Nano Spray Dryer B-90 HP or on the tray of the trolley.

NOTE

The Aspirator can not be used in combination with the flow regulation valve. Remove the flow regulation valve if mounted.

5.5.1 Installing the Aspirator in 'open loop' configuration



1 Air inlet with filter

- 2 Air outlet towards the gas inlet connector of the Nano Spray Dryer B-90 HP in Polypress design
- 3 Output flow-through selector (frequency based regulation)
- Mains socket, switch and fuse box Air inlet filter

Disconnect the power cord from the mains and depressurize all system parts.

4 5

- Connect the air inlet filter to the aspirators air inlet.
- Connect the air outlet to the Nano Spray Dryer B-90 HP gas inlet.
- Connect the power cord to the mains. The device can now be switched on.

5.5.2 Installing the Aspirator in 'closed loop' configuration

This operation mode is only applicable in combination with the Nano Spray Dryer B-90 HP Advanced model.



- 1 Gas inlet (connection to the outlet of the Inert 3 Loop B-295 or Dehumidifier B-296 Nano)
- 2 Gas outlet towards the gas inlet connector of 4 the Nano Spray Dryer B-90 HP in Polypress design
- Output flow-through selector (frequency based regulation)
- Mains socket, switch and fuse box
- Disconnect the power cord from the mains and depressurize every part of the system.
- Connect the gas inlet to the outlet of the Inert Loop B-295 or Dehumidifier B-296 Nano.
- Connect the gas outlet to the GAS IN connection of the Nano Spray Dryer B-90 HP Advanced.
- Connect the power cord to the mains. The device can now be switched on.

5.6 Inert Loop B-295 installation

In 'closed loop' the Nano Spray Dryer B-90 HP Advanced can be combined with the Inert Loop B-295 upgrade set and the Aspirator. This enables spray drying of organic solvent based solutions under inert conditions in a fume hood or equivalent. Additionally the Dehumidifier B-296 Nano can be installed to dry the recirculating gas.

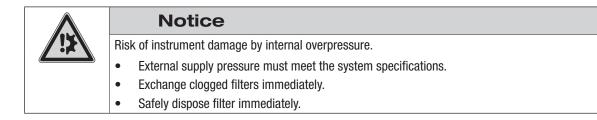
A Danger
Death by suffocation or serious poisoning by inhalation of inert gases.
Do not inhale inert gases.
Directly withdraw released gases and gaseous substances by sufficient ventilation.
Only operate the instrument in ventilated environments.
• Before operation, check all parts, connections and sealings involved in the gasflow for proper sealing.
Exchange worn out or defective parts immediately.

A Danger
Death or serious poisoning by gases or particles at 0_2 -sensor or filter malfunction.
• Exchange defective 0 ₂ -sensor immediately.
• Exchange 0 ₂ -sensor regularly within the specified maintenance intervals.
Exchange clogged filters immediately.
Exchange filters regularly within the specified maintenance intervals.
Safely dispose filter immediately.

A Warning
Death or serious injuries by compressed gas.
Depressurize gas circuit before any modification work.
Wear safety goggles and safety googles.

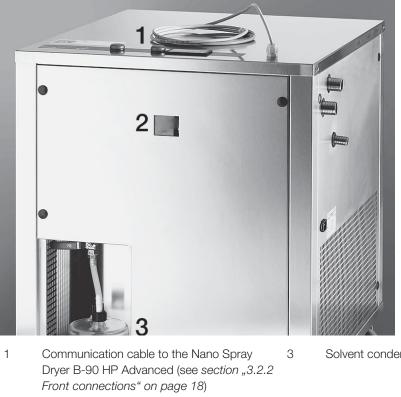
A Warning
Death or serious poisoning by contact or incorporation of condensed liquid.
Dispose of condensate safely after use.
Wear safety gloves and safety googles.

	Notice				
! ¥	Risk of instrument damage by wrong gas supply.				
	All labels at gas connections must be clearly legible.				
	Before operation, check all gas connections for correct installation.				
	• External supply pressure must meet the system specifications.				



5.6.1 Installing the Inert Loop B-295

During the process, a constant inert gas flow (N₂) is fed into the gas loop to refresh the used drying gas. Some CO, is injected in order to maintain gas proper conductivity and helps to efficiently retrieve the particles. This constant gas feeding requires an exhaust gas output to balance the pressure inside the gas circuit, therefore the Inert Loop B-295 is equiped with a hose connection for the exhaust gas (4). The use of inert gas requires additional safety measures.



2 Oxygen concentration display window Solvent condensate collector

4 Hose connection

The Inert Loop B-295 is mounted on transport wheels and can be placed next to the Nano Spray Dryer B-90 HP Advanced in the lab.

The communication cable is part of the oxygen safety loop. When the oxygen content is too high, the spray drying process will be interrupted and an error message will appear on the display of the Nano Spray Dryer B-90 HP Advanced.





- Gas outlet towards gas inlet of the AspiratorGas inlet connected to the gas outlet of the
- 5 Gas inlet connected to the gas outlet of the Nano Spray Dryer B-90 HP Advanced
- Exhaust gas outlet towards check valve (7)
- Check valve with exhaust gas tube (to connection 6)
- Disconnect the power cord from the mains and depressurize each part of the system.

6

7

- Install the check valve at the exhaust gas tubing outlet and lead it to a fume hood or ventilation system.
- Connect all the tubes (see pictures).
- Connect the communication cable to the Nano Spray Dryer B-90 HP Advanced.
- Connect the power cord to the mains. The device can now be switched on.

NOTE

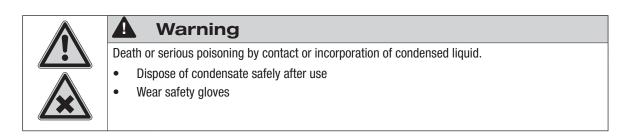
If air is sucked in through the tube during operation, this is an indication that the 'closed loop' is not tight. Switch off the assembly and re-check each connection and sealing immediately. Take out the necessary corrective actions to obtain a sealed configuration.

NOTE

It is forbidden to use another gas composition to inertise the system in closed loop.

5.7 Dehumidifier B-296 Nano installation

The Dehumidifier B-296 Nano can be used for reproducible air inlet conditioning by condensation of water/solvent residues in gases. It works at a fixed cooling temperature of 0 °C.



5.7.1 Installing Dehumidifier B-296 Nano in 'open loop' configuration

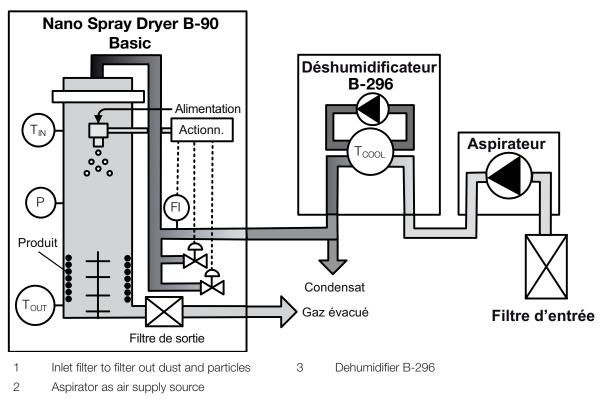
The use of the Dehumidifier B-296 Nano is recommended to reduce air humidity when aqueous samples are being processed in open-mode using the Aspirator with inlet filter.



- 1 Input/Output gas connectors (Polypress 3 Power switch style)
- 2 System status display
- 4 Attached condensate vessel
- Disconnect the power cord from the mains and depressurize each part of the system.
- Place the Dehumidifier B-296 Nano next to the Nano Spray Dryer B-90 HP and the Aspirator.
- Connect the tubing from the outlet (labelled on the enclosure) of the Dehumidifier B-296 Nano to the gas inlet of the Nano Spray Dryer B-90 HP (see section "3.2.3 Rear and side connections" on page 19). Use hose clamps to fix the tubing.
- Connect the tubing from the Aspirator outlet (see *section "3.7 Aspirator" on page 28*) to the inlet of the Dehumidifier B-296 Nano (labelled on the enclosure). Use hose clamps to fix the tubing.
- Connect the inlet filter to the gas inlet of the Aspirator. Use a hose clamp to fix the tubing.
- Pull the display out of its slot from the front side and insert a LR44 battery in it.
- Connect the power cord to the mains. The device can now be switched on.

5.7.1.1 <u>'Open loop' inlet air conditioning</u>

In 'open loop' configuration with Aspirator and Inlet filter the Dehumidifier B-296 Nano has to be connected to the gas inlet of the Nano Spray Dryer B-90 HP for inlet air conditioning. The dried, cooled air enters the spray dryer subsequently.



5.7.1.2 Use in 'closed loop'

The Dehumidifier B-296 Nano in 'closed loop' set-up allows spray drying of water-organic solvent mixtures, oxygen sensitive substances or flammable powders under safe conditions. Suitable spray drying conditions:

- Water-organic solvent mixture with up to 20 % organic solvent content
- For high boiling solvents mixtures (bp > 70 °C) with up to 50 % organic solvent content

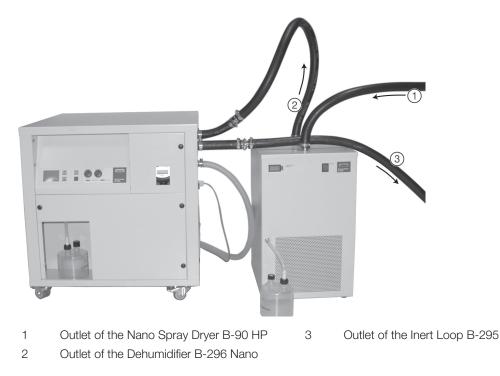
In this configuration the used inert gas (N_2 or CO_2) is loaded with water-solvent mixture residues from the spray drying process. After the cooling and drying process the gas flows back to the spray dryer.

5.7.2 Installing the Dehumidifier B-296 Nano in closed loop configuration with the Inert Loop B-295

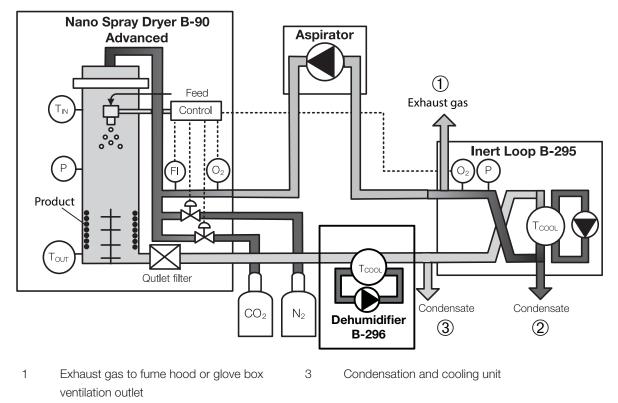
If mixtures of organic solvents and water are used, the Dehumidifier B-296 Nano is strongly recommended for a 'closed loop' configuration. The Dehumidifier B-296 Nano removes the water from the drying gas before entering the Inert Loop B-295. This separation prevents condensed water from freezing in the Inert Loop B-295 and potential damage of the heat exchanger.

NOTE

The upgrade kit for the Inert Loop B-295 has to be installed according to the specifications given in section "5.6.1 Installing the main device of Inert Loop B-295" on page 55.



- Disconnect the power cord from the mains and depressurize every part of the system.
- Connect the gas outlet of the Nano Spray Dryer B-90 HP (1) to the inlet of the Dehumidifier B-296 Nano.
- Connect the Dehumidifier B-296 Nano outlet tube (2) to the Inert Loop B-295 inlet. Use hose clamps to fix the tubing.
- Connect the outlet of the Inert Loop B-295 (3) to the inlet of the Aspirator. Use hose clamps to fix the tubing.
- Connect the gas outlet of the Aspirator to the GAS IN of the Nano Spray Dryer B-90 HP Advanced. Use hose clamps to fix the tubing.
- Plug the Inert Loop B-295 communication cable into the Nano Spray Dryer B-90 HP.
- Connect the power cord to mains. The devices can now be switched on.



2 Solvent condensate

5.8 Final installation check

This check has to be carried out after each installation and prior to the first spray drying process. All connected devices (e.g. mains voltage and gas pressure) must match the technical data of the installed system or system set-up.

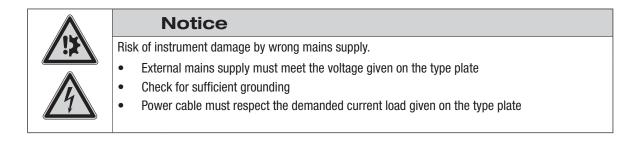
- Switch off the Nano Spray Dryer B-90 HP.
- Inspect each glass component for damage.
- Check the gas tubing connections and gas supply for tightness and good condition.
- Check that the complete set-up is sealed tightly.
- Check the high voltage cable of the electrical particle collector for proper seating.
- Check each electrical connection such as optional or external components for proper connection: temperature sensors, heater, peristaltic pump cabling. The number of sensors to be checked depends on the actual configuration.

NOTE

At 100 L/min gas flow, the internal pressure reading should be in the range of 30 to 60 mbar for a well sealed system in 'open loop'.

5.9 Electrical connections

After the installation procedure and the final check has been completed successfully, the power plug of the Nano Spray Dryer B-90 HP and optional instruments must be connected to the mains for the spray drying process.



The used mains circuit has to:

- provide the voltage that is given on the type plate of the instrument.
- be able to handle the load consisting of the connected instruments.
- be equipped with adequate fusage and electrical safety measures, in particular proper earthing.

See also technical data of all components regarding the different minimum system requirements!

NOTE

Additional electrical safety measures such as residual current brakers may be necessary to meet local laws and regulations! External connections and extension lines must be provided with an earthed conductor lead (3-pole couplings, cord or plug equipment). All used power cords shall be equipped with molded plugs only to avoid risks due to unobservant defective wiring.

6 Operation

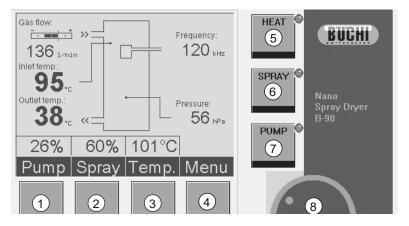
This chapter gives examples of typical instrument applications and instructions on how to operate the instrument properly and safely.

6.1 Starting up the instrument

- Make sure the Nano Spray Dryer B-90 HP is properly connected to the mains supply.
- Carry out a final installation check prior to every spray drying process.
- Switch on the Nano Spray Dryer B-90 HP at the main switch.
- Switch on all other electrical devices of the actual configuration at their main switch.

6.2 Main screen

At boot-up, an internal system check will be performed automatically. The main screen below will then appear on the Nano Spray Dryer B-90 HP, the display showing the current instrument status.



Pump button to adjust the pump rate 5 Heat button to switch the heater ON/OFF 1 2 Spray button to change the relative spray 6 Spray button to switch spray function ON/OFF rate З Temp button to change the inlet temperature 7 Pump button to switch the pump ON/OFF 4 Menu button to enter the menu structure 8 Selection turning knob to change process parameters

6.2.2.1 (Peristaltic) Pump selection

Pump rate from 3% to 100% can be selected

6.2.2.2 Spray selection

Spray head output between 10 % and 100 % spray rate can be selected.

For a spray output above 80 %, "!" will appear on display, meaning that the power of the spray may reduce the spray mesh life quicker than for a normal use.

6.2.2.3 <u>Temperature selection</u>

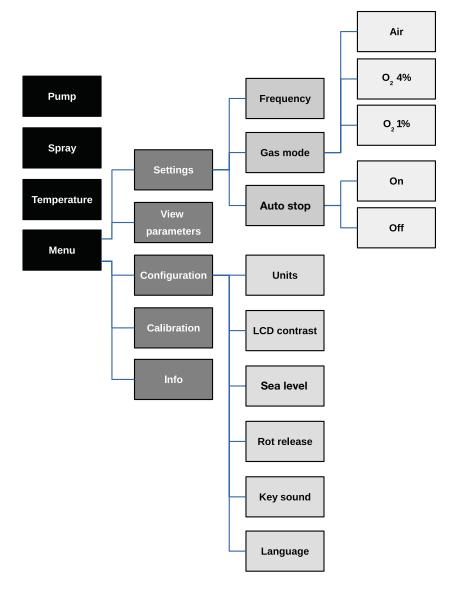
The drying gas inlet temperature can be set between 18 °C to 120 °C.

NOTE

The lowest possible drying gas inlet temperature in 'open loop' mode depends on the local room temperature.

6.3 Menu structure overview

The table below shows the menu structure of the Nano Spray Dryer B-90 HP.

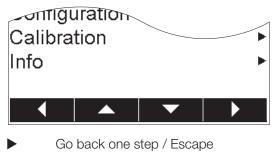


6.4 Display elements and menu functions

6.4.1 Operating controls

6.4.1.1 <u>Multifunctional buttons</u>

The multifunctional buttons below the screen are programmable. Their actual use is displayed on the screen directly above the buttons.



- Move cursor upwards
- Move cursor downwards
- Enter next menu level / OK (confirm selection)

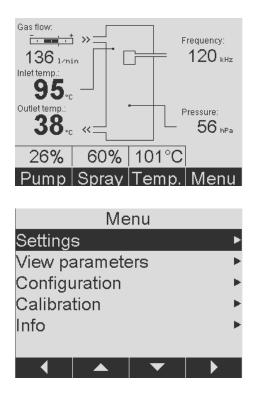
6.4.2 Rotary knob

The rotary knob is used to select values out of lists within sub menus.



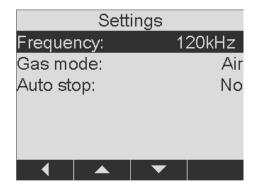
- Turning the knob clockwise moves the cursor downwards.
- Turning the knob counterclockwise moves the cursor upwards.

6.5 Menu



The menu can be selected from the main display by pressing the menu button. From the menu display, several options can be selected as shown in the picture below.

6.5.1 Settings menu



In the settings menu, the frequency, the gas mode and the auto stop can be modified.

Selection of the spraying frequency:

To select the spray frequency, select the frequency menu with the multifunctional button below the screen and use the rotary knob to select the frequency. Frequencies from 80 to 140 kHz can be selected.

NOTE

After setting a new spraying frequency, a few seconds are required to be able to observe the influence of this change on the spray cone.

Gas mode:

- To select the gas mode, enter the gas mode menu with the multifunctional button below the screen and use the rotary knob to select the gas mode.
- Set to "Air" for 'open loop' mode
- Set to "~4 % O_2 " for 'closed loop' mode with inert gas N_2 and CO_2 (only Nano Spray Dryer B-90 HP Advanced)
- Set to "~1 % O_2 " for 'closed loop' mode with increased gas N_2 and CO_2 consumption (for oxygen and ozone sensitive material only Nano Spray Dryer B-90 HP Advanced)

Auto Stop:

To turn "auto stop" on or off, select "auto stop" with the multifunctional button below the screen and use the rotary knob to select the parameter.

When auto stop is on, the process will be automatically stopped when the spray head is detected empty.

6.5.2 View parameter menu

Parameters		Parameters		Parameters	
Inlet temperature:	95 °C	Gas flow:	0 l/min	Net voltage:	>159 VAC
Outlet temperature:	38 °C	N₂-Valve:	closed	HV-Voltage:	0 V
Glass detector:	Off	CO2-Valve:	closed	HV-Current:	0 uA
Auto stop value:	1207	Pressure:	0 hPa	Voltage sprayhead:	9.257 V
O ₂ concentr.:	4.0 Vol%	Rel. pressure 2:	0 hPa	Current sprayhead:	0.455 A
				Power sprayhead:	1.166 W

To enter the "Menu" press the corresponding multifunctional button below the screen. Inside the menu, use the multifunctional buttons for navigation.

The "View parameters" menu and its sub menus provide information about the actual system status. It is accessible at any time and can supply the user with useful information about ongoing processes. No changes can be made here.

NOTE

In 'closed loop' mode the N_2 and O_2 value open and close depending on the O_2 content in the gas loop to maintain the selected O_2 concentration limit (< 4 % or <1 %).

6.5.3 Configuration menu

Configuration			
Units			►
LCD-co	ontrast:		60%
Sea level:		I	600m
Rot. release:		3	000ms
Key sou	und:		Yes
Language:			English
•		•	

- To enter the "Menu" press the corresponding multifunctional button below the screen.
- Inside the menu, use the multifunctional buttons for navigation to "Configuration" and confirm the selection.

Items in menu "Configuration": Description/available parameters:

Units	 Temperature in °C, K or °F Volume flow in L/min, m³/h, ft³/h Pressure in mbar, Torr, hPa
LCD-Contrast	Select a screen contrast between 0 and 100 %.
Sea level	Select the sea level in meters over sea level.
Rot. release	Defines the time interval in which the rotary knob is active after pressing the selection buttons for "Pump", "Spray" and "Temp.". The default time is 3 seconds. This is a feature to avoid unintended inputs.
Key sound	If "YES" is selected a sound is audible when pressing a function key. To suppress the acoustic confirmation select "NO".
Language	Choose the language of the user interface between Chinese, English, French, German, Italian, Japanese or Spanish.

6.5.4 Calibration menu

To enter the "Menu" press the corresponding multifunctional button below the screen. Inside the menu, use the multifunctional buttons for navigation to "Calibration". Stop the gas flow and confirm by pressing the "OK" button to recalibrate the internal flow sensor of the Nano Spray Dryer B-90 HP.

Calibration	Flow calibration
Flow calibration	
	Be sure that flow is zero.
	▲ ▲ ▼ OK

NOTE

The calibration should be performed periodically to ensure best spray drying results.

6.5.5 Info menu

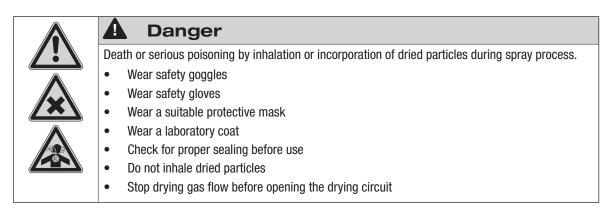
This menu displays the current system software version in eight digits. It appears after every boot up for an instant and can be reviewed via the "Info" menu entry. The firmware version can be useful during service.



6.6 Starting a spray drying process

6.6.1 System preconditions

Each item of the system must be correctly installed according to the chapter of the operation manual related to the specific set-up, see section 5 for more information. Carry out a final installation check (see section "5.8 Final installation check" on page 60) before every spray drying process. See also section "2.3 Staff qualification" on page 11 for general warnings.



6.6.2 Starting a spray drying process

- Switch on the instrument. After the automatic self-check, the operating mode is activated.
- Switch on each device installed in the current configuration.
- Set the drying gas rate to a reasonable flow of about 80–150 L/min with the external gas regulator (when using compressed air) or adjust the frequency controller when using the Aspirator (set a rotational frequency between 30–40 Hz).
- Read the relative inside pressure (typical pressure range 30 mbar to 60 mbar).
- Select the desired inlet temperature (typical setting between 18 °C and 120 °C).
- Select the appropriate feeding rate and switch on the heater by pressing the "Heat" button and wait until the system has reached stationary temperature conditions (typically after 5 to 10 min.).
- Switch on the peristaltic pump by pressing the "Pump" button.
- Select the relative spray rate (varying the spray power might improve throughput).
- Press the "Spray" button to start the spray process.

Note

Spray power above 80 % are labeled with a "!". This means that even though it is possible to spray with power between 80 % and 100 %, the nebulizer might be damaged or worn out quicker than advertised depending on the process.

The spray process starts and the electric field in the particle collector is turned on.

Select the most appropriate frequency for the sprayed solvent in the frequency menu to improve spray quality and throughput.

6.7 Optimizing parameters

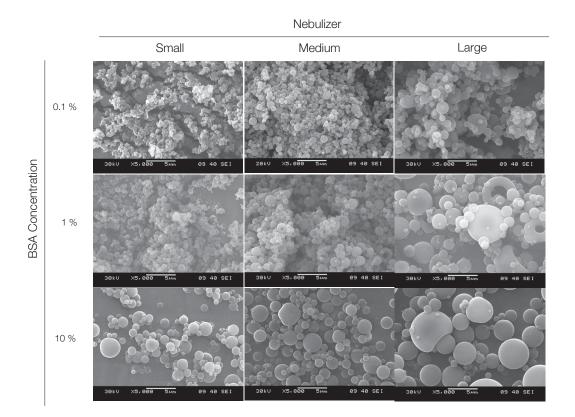
The relevant parameters for the spray drying process depend on each other.

6.7.1 List of spray drying parameters

- Drying gas flow rate
- Relative inside pressure
- Inlet temperature
- Feed rate

The properties of the spray dried solution determines the maximum process temperature. The maximum thermal load of the solid particles is caused by the spray head temperature or by the maximum inlet drying gas temperature. To reduce the thermal load on the feed, the sample vessel can be cooled down.

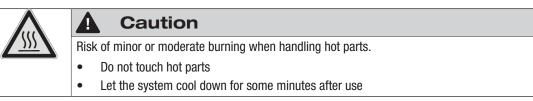
The achievable droplet sizes depend on the installed nebulizer and sample concentration. The following figure shows the influence of the sample concentration and the nebulizer size on particle sizes for the example of BSA.



6.7.2 Application information

For an overview of the possible applications and processes, please refer to our webpage (www.buchi. com) where training papers and application notes can be downloaded directly. For special applications please contact us.

6.8 Finishing a spray drying process



- Switch off the spray head by pressing the "Spray" button.
- Switch off the heater by pressing the "Heat" button and let the system cool down. Depending on the environmental temperature, this may take several minutes.
- Stop the gas flow at an inlet temperature below 70 °C.
- Read the internal pressure of the Nano Spray Dryer B-90 HP. No overpressure should be present.
- Increase the pump rate to 100% and take out the pump inlet tube of the sample solution. The pump will transport air, emptying the product from the tubing and the spray head. Turn OFF the peristaltic pump afterwards and lower the pump bed to release the mechanical load of the pulleys on the tube.

Before opening the glass assembly for particle recovery, see *section "6.7 Optimizing parameters" on page 70* to learn about the safest way to proceed.

6.9 Collecting the particles from the collecting electrode

A Danger
Death or serious poisoning by inhalation or incorporation of dried particles at recovery.
Wear safety goggles
Wear safety gloves
Wear a suitable protective mask
Wear a laboratory coat
Do not inhale dried particles
Stop drying gas flow before opening the drying circuit
Only recover particles in sufficiently ventilated flue or glove-box
Do not disperse the dried particles
Do not clean dusty parts with compressed air

6.9.1 General information about particle recovery

To recover the particles from the surface of the collecting electrode a particle scraper and a sheet of collecting paper can be used. It allows a manual recovery of the particles by depositing them on a weighing paper.

The electrostatic particle collector has a very high separation efficiency. Nevertheless some particles may be deposited on the glass cylinder wall and the inner star electrode.

Recommended set of tools for particle recovery:

- BUCHI particle scraper
- BUCHI weighing paper in A4 format

6.9.2 Particle recovery procedure

- Place a blank sheet of weighing paper on a free area inside a fume hood.
- Remove the collecting electrode from the bottom basement of the particle collector.
- Place the collecting electrode on the sheet of paper.
- Use a particle scraper to detach the particles from the cylinder.
- Lift the cylinder from the weighing paper and put it aside.
- Transfer the powder from the sheet of paper into a storage vessel. Depending on the substance, a particle recovery rate of up to 90 % can be achieved.

NOTE

Act carefully (e.g. avoid shaking movements) in order to prevent a loss of particles. Clean all contaminated parts thoroughly to avoid hazardous situations and cross contamination. For information about cleaning, see section "7.6 Cleaning" on page 84.

6.10 Operating Inert Loop B-295

System preconditions

Each item of the system must be correctly installed according to the set-up configuration. See section 5 for more information. Carry out a final installation check (see section 5.9) before starting a spray drying process.

	A Danger
	Death by suffocation or serious poisoning by inhalation of inert gases.
	Do not inhale inert gases
	Directly withdraw released gases and gaseous substances by sufficient ventilation
	Only operate the instrument in ventilated environments
	Before operation, check all parts, connections and sealings involved in the gasflow for proper sealing
	Exchange worn out or defective parts immediately
	A _
	A Danger
	Death or serious poisoning by gases or particles at 0_2 -sensor or filter malfunction
	 Exchange defective 0₂-sensor immediately
	• Exchange 0 ₂ -sensor regularly within the specified maintenance intervals
	Exchange clogged filters immediately
$\mathbf{\Lambda}$	Exchange filters regularly within the specified maintenance intervals
	Safely dispose filter immediately
	A
	A Warning

A Warning
Death or serious injuries by compressed gas.
Depressurize gas circuit before any modification workWear safety goggles

	A Warning
	Death or serious poisoning by contact or incorporation of condensed liquid.
	Dispose condensate after use safely
×	Wear safety gloves

- Make sure the dongle is properly plugged into the Nano Spray Dryer B-90 HP. The instrument automatically detects the connected periphery.
- Switch on the Nano Spray Dryer B-90 HP.
- Select the gas mode (~4 % O₂ or ~1 % O₂).
- Check the oxygen sensor, the Inert Loop B-295 will be automatically switched on by the Nano Spray Dryer. The Ambient oxygen concentration should be around 21 %.

- Switch on the Aspirator.
- Turn on the inert gases N_2 and CO_2 . Set the CO_2 pressure to maximal 2 bar and the absolute N_2 pressure below 1.3 bar.
- Switch on the heater by pressing the "Heat" button and wait until the system has reached stationary temperature conditions (typically after 5 to 10 min.).
- Once the set oxygen concentration has been reached (either 4 % or 1 % according to the gas mode selected) turn on the peristaltic pump to supply the spray head with the sample solution.
- When the sample solution has reached the spray head, press the "Spray" button, the system switches on the spray and the high voltage at the collecting electrode. The inert gas flow starts working together with the spray head.
- Select the most appropriate spray power and spraying frequency for the sprayed solvent to improve spray quality and throughput.

If a setup with the Dehumidifier B-296 Nano is used, switch on the Dehumidifier B-296 Nano at the same time as the Inert Loop B-295.

NOTE

Inert gas pressure larger than the values reported above will activate the safety pressure relief valve, causing inert gas loss.

Aqueous solutions or mixtures of organic solutions and water may freeze in the heat exchanger of the Inert Loop B-295. Therefore, when working with water- or organic based solvents, the cooling temperature must be set above +10 °C. The use of the Dehumidifier B-296 Nano is highly recommended when working with water/organic solvent mixtures.

A leaking Aspirator can cause high O_2 concentrations in 'closed loop'. If the concentration is considerably high, stop spray drying process and check the Aspirator for leakages immediately.

In case of a limited available sample amount, it is possible to set up the process using a pure organic solvent and then change to the sample production while the system is spraying. Once the sample is being sprayed, minor changes in frequency might be necessary in order to achieve an optimal throughput.

6.11 Operating the Dehumidifier B-296 Nano

The Dehumidifier B-296 Nano can be used in 'open loop' or in 'closed loop' configuration in combination with the Nano Spray Dryer B-90 HP.



Warning

Death or serious poisoning by contact or incorporation of condensed liquid.

- Dispose of condensate safely after use
- Wear safety gloves

Each item of the system must be properly installed according to the chosen set-up, see section 5 for more information. Carry out a final installation check (see section 5.9) before every spray drying process!

- Switch on the Nano Spray Dryer B-90 HP Advanced.
- In 'closed loop' configuration only, plug the dongle into the Nano Spray Dryer B-90 HP.
- For "close loop" configuration, select the inert gas mode (~4 % O_2 or ~1 % O_2) in "submenu 1" and turn on the inert gases N_2 and CO_2 . The instrument automatically detects the connected dongle.

The CO2 pressure must be set to maximal 2 bar and the absolute $\rm N_{\rm 2}$ pressure below 1.3 bar.

- Check the oxygen sensor with ambient air. The Ambient oxygen concentration should be around 21 %.
- Switch on the Dehumidifier B-296 Nano.
- Switch on the Aspirator.
- Switch on the heater by pressing the "Heat" button and wait until the system has reached stationary temperature conditions (typically after 5 to 10 min.).
- Once the set oxygen concentration has been reached (either 4 % or 1 % according to the gas mode selected) turn on the peristaltic pump to supply the spray head with the target sample solution.
- When the sample solution has reached the spray head, press the "Spray" button, the system switches on the spray and the high voltage at the collecting electrode. The inert gas flow starts working together with the spray head.
- Select the most appropriate spray power and spraying frequency for the sprayed solvent to improve spray quality and throughput.

NOTE

Before switching from one organic solvent solution to another it is highly recommended to dry the working 'closed loop' by running the aspirator with dry air for half an hour. This will significantly lower the possibility of cross contamination.

6.12 Using the Nano Spray Dryer Records software on a PC

The PC-software enables the online monitoring of the data and the storage of experimental runs in a virtual library.

Functionality:

- Documentation of the process data of the experimental runs
- Export of the process parameters for further data analysis

6.12.1 Connecting the instrument to the PC

Connect the Nano Spray Dryer B-90 HP to the PC using the USB cable. Insert the CD into the PC and follow the instructions of the program for system requirements and software installation.

6.12.2 Starting the program





Double click on the program icon to start Nano Spray Dryer Records.

6.12.3 Initial view and program functions

The menu bar entries and accessible sub-entries are listed below:

File:	New LabBook
	Open LabBook
	Close LabBook
	Exit
View:	Show tree
Tools:	Selftest
	Options
Help:	Contents
	Import License
	Software registration
	About

6.12.4 Tool bar functionality

1	2	3	4	(5)	6	7	8	9	(10)	(11)	(12)	
	0					B	Q	Ċ,				BUCHI

Tool bar items: Tool functionality:

1	Start/Stop recording
2	New LabBook
3	Delete LabBook
4	Open existing LabBook
5	Close LabBook
6	Import LabBook
\bigcirc	Export LabBook
8	Open Experiment
9	Show Report (Create a pdf-file with a choosen data set)
0	Export to CSV (Character-Separated Values)
1	Close Experiment
(2)	Delete Experiment

6.12.5 LabBook entries

Each experiment is stored as an entry in a LabBook. In order to record an experiment a LabBook must already be available or has to be created first.



- Click the button "New LabBook", a small pop-up window appears (see screenshot above). Name the LabBook and click "create". The new LabBook will appear in the left tree area of the program screen.
- Press the "Start/Stop recording" button to acquire online process data while the Nano Spray Dryer B-90 HP is switched on.

6.12.6 Program options

The program can be configured in the "Options" menu.

Options		
Recording Sample Rate [sec]	1 🗊	Report customizing Company name
Units		Logo 🔣
Temperature	°C 🗸	Startup
Pressure	hPa 🗸	Remember opened LabBooks
Flow Rate	m²/s 🗸	Remember opened Experiments
Use machine settin	gs when connected	Caution: Enabling this options will affect application startup time
Language	English (United State	OK Cancel

- Select the appropriate data recording rate and storage interval.
- Select the units of temperature, pressure and gas flow.
- Upload a company logo and change displayed company name on PDF reports.
- Activate check boxes to set startup view.

NOTE

Any changes made under "Options" will be applied after a restart of the program.

6.12.7 Experiment metadata

LabBooks can be equipped with additional metadata for each experiment. These metadata can hold comments, unique attributes for identification and information about the drying conditions and system settings. This information will show up in PDF reports.

🗟 Nano Spray Dryer Records		
<u>File View T</u> ools <u>H</u> elp	Image: Constraint of the system of the sy	Configuration Mesh Type Glas assembly Spray head angle Spray head radial position Operation Mode
Dicconnected		<u> </u>
Disconnected		🧣

- Select a LabBook and one or more experiment entries. Activated experiments will appear in the tree area on the left.
- Click on a single experiment entry at the tree area. Metadata can now be manipulated.

6.12.8 Access recorded data

Nano Spray Dryer Records ⊻iew <u>T</u>ools <u>H</u>elp Eile BUCHI 🖃 🛄 1st LabBook 1st Experiment 1st Experiment Table View 23 22 00:00:30 00:00:40 00:00:20 Inlet temperature (°C) ature (°C) Outlet termine 00:00:20 00:00:30 00:00:40 Internal pressure (hPa) 00:00:20 00:00:30 00:00:40 - Flow (m³/s) sconnected 8

Recorded spray drying data can be displayed in plot or table view.

All main spray drying process parameters like inlet and outlet temperatures, pressure and gas flow are recorded and visualized in the "Plot View". The plots can be zoomed-in via mouse action. Use the computer mouse to mark an area of interest on a plot. Release the mouse button to zoom into the area.

🗟 Nano Spray Dryer Records				-				
<u>Eile View Tools H</u> elp								
□ □ 1st LabBook □ □ 1st Experiment	1st Exper	iment						
Table View Plot View	Time 🔺	Heat	Pump	Spray	Terr 📥 (°C)			
	00:00:33.297				60			
	00:00:34.297				60			
	00:00:35.297				60			

All other actual process data can be read out in the time based "Table View".

Click on the column headers to sort the table.

6.12.9 Data export

Experiments can be exported into a CSV file.

To export an experiment click the "Export to CSV" button. Subsequently name and save the file on a file system.

PDF reports can be generated from experiments via the "Show Report" button. This file can be named and saved on a file system.

6.13 Run a self-test on the Nano Spray Dryer B-90 HP

The software provides a self-test function to report the actual condition of the Nano Spray Dryer B-90 HP. The self-test instructions and procedures are displayed on the screen of the Nano Spray Dryer B-90 HP. At the end of the self-test, a PDF report is generated. It can be used as a support tool by BUCHI representatives for service and trouble shooting.

NOTE:

The serial number of the Nano Spray Dryer B-90 HP is requested during the self-test. It can be found on the name plate at the back side of the instruments housing.

6.13.1 Software licensing

The software can be used for 60 days. At the end of this period the software will request a license file to remain functional.

Fill in the Software Registration Form under Help.

The form generates an XML file. Send this file to your local BUCHI representative to receive a validated license file (*.lic) in return.

Import the license file via "Help">"Import License"

7 Maintenance and repair

This chapter gives instructions on maintenance work to be performed in order to keep the instrument in a good and safe working conditions. All maintenance and repair work requiring the opening or removal of the instrument housing must be carried out by trained personnel and only with the tools provided for this purpose.

NOTE

Use only genuine consumables and spare parts for any maintenance and repair work in order to assure warranty and continued system performance. Any modifications of the Nano Spray Dryer B-90 HP or parts of it need prior written permission of the manufacturer.

	Death or serious poisoning by inhalation or incorporation of dried particles during maintenance.
\mathbf{A}	Wear safety goggles
	Wear safety gloves
	Wear a suitable protective mask
$\mathbf{\Lambda}$	Wear a laboratory coat
	Clean all parts thoroughly
	Only maintain the instrument in ventilated environments
	Do not inhale dried particles
	 Stop drying gas flow before opening the drying circuit

	A Warning						
	Death or serious burning by electric current.						
	• Switch off the instrument, disconnect the power cord and prevent unintentional restart before						
	removing housing or parts of it						
17	Do not touch parts inside the instrument with wet hands						
	Do not spill liquids over electronic components						
	Do not squeeze cables, tubes or other items at reassembling						
	Exchange defective cabling or tubing before reassembling						

Warning

A

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Death or serious injuries by compressed air and gases.

- Depressurize air and gas circuit first
 - Wear safety goggles

	Notice
	Risk of instrument and instrument damage by liquids and detergents.
Do not spill liquids over the instrument or parts of it	
	Wipe off any liquids instantly
	Use ethanol or soapy water as detergent only

7.1 Customer service

The authorized service personnel only is allowed to perform repair work on the instrument. Authorization requires a comprehensive technical training and knowledge of the possible dangers which might arise when working at the instrument. Such training and knowledge can only be provided by BUCHI.

Addresses of official BUCHI customer service offices are given on the BUCHI website under:

www.buchi.com. If malfunctions occur on your instrument, ifyou have technical questions or application problems, contact one of these offices.

The customer service offers the following:

- Spare part delivery
- Repairs
- Technical advice

7.2 Housing condition

Check the housing for visible defects (switches, plugs, cracks) and clean it regularly with a damp cloth.

7.3 Glass component conditions

Clean the glass components after each spray process to prolong the lifetime. The glass assemblies can be taken out and cleaned manually with water and a commercial cleaning agent (e.g. mild soap solution) or in an ultrasonic bath. Visibly check for damages at all the glass components subsequently.

NOTE

It is recommended to clean all glass components.

A

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Regularly check the glassware components for damage and use only glassware in perfect condition, *i.e. glassware without cracks or stars.*



Caution

Risk of minor or moderate cuts when handling damaged glass parts.

- Handle glass parts with care
 - Visually inspect every glass part before mounting
- Exchange damaged glass parts immediately
 - Do not touch cracks or bits of broken glass with bare hands

7.4 Oxygen sensor

The live time of the oxygen sensor in the Nano Spray Dryer B-90 HP Advanced or Inert Loop B-295 is approx. 1.5 years. Replace the sensor after this period or in case of malfunction. To remove the sensor disconnect the sensor cabling and unscrew it by turning it anticlockwise. For installation proceed in reverse order.

7.5 Sealing conditions

All sealings are recommended to be checked yearly. When replacing the seals, take care not to damage them.

- To prevent damaging the seals never apply grease and never touch them with sharp objects.
- To prolong the lifetime of the seals, rinse them routinely with water or ethanol to avoid unwanted sample contamination to occur.
- Dry the cleaned seals with a soft, lint-free cloth.

7.6 Cleaning

The following subsections describe possible cleaning actions. All actions should be performed regularly and thoroughly.

7.6.1 Spray head and tube connections

Clean the feed tubing and the spray head after each spray drying process.

Recommended: Cleaning of the spray head and nebulizer in an ultrasonic bath for 1 - 2 minutes.



Caution

A

Risk of minor or moderate burning when handling hot parts.

- Do not touch hot parts
- Let the system cool down for some minutes after use

NOTE

For maximum throughput and to avoid cross contamination of the spray drying product it is essential to work with a clean spray head, nebulizer and tubing assembly. The cleaning process should not exceed 3 minutes. If the nebulizer is fully immersed in water, prolonged cleaning process can damage the gold connection. It is recommended not to exceed the mentioned cleaning time and to keep the gold connection out of water using the cleaning tool supplied as shown in the following picture.



Maximum water level

7.6.2 Outlet filter

A clogged outlet filter results in a pressure drop between the inside and the outside of the Nano Spray Dryer B-90 HP.

	Notice
!	Risk of instrument damage by internal overpressure.
	External supply pressure must meet the system specifications
	Exchange clogged filters immediately
	Safely dispose filter immediately

• Replace the outlet filter annually or if damaged.

7.6.3 Inert Loop B-295 and Dehumidifier B-296 Nano

The sealed working circuits of the devices and the optional heat exchanger in combination with the Inert Loop B-295 can be flooded completely for cleaning purpose.

	Notice	
!!	Risk of instrument and instrument damage by liquids and detergents.	
	Do not spill liquids over the instrument or parts of it	
	Wipe off any liquids instantly	
	Use ethanol or soapy water as detergent only	

- Switch off all relevant devices and unplug the respective power cords.
- Remove the molecular sieve bag for the duration of cleaning if installed.
- Check every sealing and tube for good condition and proper sealing.
- Raise the exhaust tube and fill the system through the inlet with cleaning medium (e.g. ethanol).
- Provide a collection vessel to collect the cleaning medium flowing from the system. Open the drain valve and lower the exhaust tube, so that the cleaning medium flows out.
- Rinse out and dry the condensate vessel.
- Reinstall the condensate vessel.
- To dry the working circuit connect the Nano Spray Dryer B-90 HP and dry it by running the Aspirator with dry air.

8 Troubleshooting

This chapter helps to resume operation after a problem has occurred with the instrument which does not require special technical training. It lists possible occurrences, their probable cause and suggests how to remedy the problem.

The troubleshooting table below lists possible malfunctions and errors of the instrument. The operator is enabled to correct some of those problems or errors by him/herself. For this, appropriate corrective measures are listed in the column "Remedy".

Malfunctions or errors which are not listed in the table are to be corrected by a BUCHI trained technical engineer who has access to the official service manuals. In this case, please refer to your local BUCHI customer service agent.

8.1 Error messages and their remedy

Error mess	sages and their remedy		
Error number	Error reading	Possible cause	Remedy
007	Temperature sensor	Defective sensor, sensor cable or	Switch off the device and try again.
	outlet not connected	internal wiring	Check if the outlet temperature sensor is plugged in correctly.
			If the problem still occurs replace the outlet temperature sensor or contact the BUCHI customer service.
800	Temperature sensor	Defective sensor, sensor cable or	Switch off the device and try again.
	inlet not connected	internal wiring	Check if the inlet temperature sensor is plugged in correctly.
			► If the problem still occurs replace the inlet temperature sensor or contact the BUCHI customer service.
011	Inlet temperature exceeded	Heating control or heating relais defective	Switch off the device and try again.
			If the error still occurs replace the heater module or contact the BUCHI customer service.
014		HV-module defect or internal	Switch off the device and try again.
		wiring interruption or quality fall-off voltage because of a short circuit	If the error still occurs contact the BUCHI customer service.
015	Current in HV-Module too high	HV-module defect or internal wiring	Switch off the device and try again.
		interruption	If the error still occurs contact the BUCHI customer service.
016	Current in HV-Module too low	HV-connector not plugged in or defective wiring error	Switch off the device and try again.
			► Check the HV-module, connection.
			► If the error still occurs, contact the
			BUCHI customer service.

Error mess	ages and their remedy	Possible cause	Remedy
018	Spray head empty	The spray head is empty	Check if the sample has been fully sprayed.
			Check for the presence of air bubbles. If there are air bubbles, make sure your nebulizer is properly mounted, especially the gasket. Check if the flow rate is high enough to match the throughput
			Check if the inlet tube is in the solution - place the inlet tube in the sample solution.
			Check if the inlet tube is damaged or leaking -replace the tube.
019	No connection to IIC board. Check IIC Bus	Hardware defect	Contact BUCHI customer service.
020	Relative pressure sensor 1 not connected	Sensor not connected or defective internal wiring	Contact BUCHI customer service.
021	Relative pressure sensor 2 not connected	Sensor not connected or defective internal wiring	Contact BUCHI customer service.
023	Difference pressure sensor not connected	Sensor not connected or defective internal wiring	Contact BUCHI customer service.
024	Inside pressure low	System not leakproof	► Test for leaks.
025	Filter pressure drop low	Filter not installed or defective internal wiring	 Check if outlet filter is blocked. Replace the filter.
026	Flow rate low	No flow rate, system not leakproof	► Generate gas flow, test for leaks.
027	Inside pressure high	Gas pressure connection too high	Regulate gas pressure.
028	Flow rate high	Sensor is at stop position	Reduce gas flow rate.
029	Relative pressure sensor 1 defective	Sensor or internal wiring defective	Contact BUCHI customer service.
030	Relative pressure sensor 2 defective	Sensor or internal wiring defective	Contact BUCHI customer service.
032	Difference pressure sensor defective	Sensor or internal wiring defective	Close gas flow rate and restart the system, contact BUCHI customer service.

Error mess	ages and their remedy		
Error number	Error reading	Possible cause	Remedy
033	No connection to the spray head. Check the connection cable of	Connection problem with the spray head	Control if the cable is properly plugged into the socket – Plug the cable.
	the spray head		Control if the cable is damaged - replace the cable.
			► Control if the gold connection of the nebulizer are damage or dirty – clean the connections or replace the nebulizer.
			► Control if the spring contacts in the spray head are clean – clean the contacts and make sure they are properly dried.
			► If the error still occurs , contact the BUCHI customer service.
035	No glass insulation cylinder	Position switch	Install glass insulation cylinder
040	Valve 1 CO ₂ not connected	Internal wiring defective or missing	Contact BUCHI customer service
041	Valve 2 N ₂ not connected	Internal wiring defective or missing	Contact BUCHI customer service.
042	0 ₂ sensor not connected	Defective sensor, sensor cable or internal wiring	Contact BUCHI customer service.
043	0_2 sensor short-circuit	Defective sensor, sensor cable or internal wiring	Contact BUCHI customer service.
046	0_2 concentration high	System not leakproof, N ₂ -bottle empty	► Test for leakages. Check system sealings and N ₂ -supply.
047	0 ₂ concentration low	Valve defective	Contact BUCHI customer service.
050	B-295 0 ₂ concentration too high	System not leakproof, N ₂ -bottle empty	Test for leakages. Check system sealings and N_2 -supply.
051	Change gas mode	Electrical communication cable B-290/B-295 or dongle not connected	Check cabling or dongle.
058	EEPROM		► Contact BUCHI customer service.

For operation with the Inert Loop B-295 only

200	Connection to	Defective connection cable or	Check connection cable for damage
	B-295 interrupted or	instrument is not plugged in	and insert plug.
	disconnected		
201	B-295 plugged in	Connection cable plugged in after	Restart the system.
	while operation in	switching on the instrument	
	process		

Error messages and their remedy				
Error number	Error reading	Possible cause	Remedy	
203	Pressure alarm not given during start-up	Pressure switch defective or contaminated	 Contact BUCHI customer service. 	
204	Failure cooling unit B-295		Contact BUCHI customer service.	

8.2 Malfunctions and their remedy

Malfunctions and their remedy Malfunction	Possible cause	Remedy
Equipment cannot be switched on	Electrical power	► Insert mains plug, examine plug
	Blown fuse	for damage. ► Reset fuse manually.
Peristaltic pump does not deliver	The rollers are not in contact with the running surface	 Reset fuse manually. Elevate the running surface with the lever.
		► Adjust the lifting height of the running surface with the hexagonal hex-wrench from beneath.
System does not heat up	Heater cable not plugged in	Plug in heating circuit.
	Heating is not switched on	Switch on heat.
	Nominal entry temperature is below	Re-select a new inlet
	room temperature	temperature.
	Blown fuse	Reset fuse manually.
	Heater defective	Contact the BUCHI customer service.
	Faulty gas flow direction or no flow in the heating system	Check pipe system.
Spray head blocked	Product is too concentrated	Use a lower sample
		concentration.
		Change to a bigger nebulizer.
		Clean the spray head and nebulizer.
Product drips in spray cylinder	No spray flow	► Turn on spray.
	Insufficient spray flow	 Inspect inside gas pressure (20-50 mbar) and gas supply.
Deposits on the spray cylinder	Spray head is not clean	Completely dismantle the spray head and clean with water.
	Spray head is defective (bent PEEK tubing, washer or mesh)	Replace spray cap or defective element.
	Product will not dry	Increase inlet temperature.
	Inlet temperature is above the melting point of the product	► Reduce inlet temperature.
	Product related deposits	No action possible.

Malfunctions and their remedy		
Malfunction	Possible cause	Remedy
Irregular or pulsed spraying	Leaks in the spray head	! Caution: The auto stop function checks every 20 seconds the presence of product in the nebu- lizer, interrupting the spray in the corresponding interval.
		Check all sealings in the spray head and replace if necessary.
Deposits in the particle collector	Product too moist	Increase inlet temperature to dry the product.
		Increase residence time of product in drying cylinders.
		Increase gas flow rate to increase the energy input for drying.
Inlet temperature falls	Heating is switched off	Switch on heating.
	Heater plug is unplugged	Insert heater plug.
Outlet temperature falls	No heating	► Follow measures under "Fall of entry temperature".
	Spraying too powerful	Reduce production rate of spray.
Outlet temperature rises	System has not reached stationary temperature conditions	Warm up the system with hot drying gas for about 30 minutes.
	Spray head blocked	Clean the spray head.
	Feed tubing not dipped into sample solution	► Dip feed tubing into product.
	Change of concentration in sample solution	 Agitate product (magnetic stirrer) to obtain uniform concentration.
	No feed of product	Switch on peristaltic pump.

9 Shutdown, storage, transport and disposal

This chapter instructs how to shut down and to pack the instrument for storage or transport. Specifications for storage and shipping conditions can also be found listed here.

9.1 Storage and transport

Switch off the instrument and remove the power cord. To disassemble the Nano Spray Dryer B-90 HP follow the installation instructions in section 5 in reverse order. Clean the instrument and remove all liquids and dusty residues before packing the instrument.

	A Warning
1	Death or serious poisoning by contact or incorporation of harmful substances.
	Wear safety goggles
	Wear safety gloves
	Wear a suitable protective mask
	Wear a laboratory coat
	Clean the instrument and all accessories thoroughly to remove possibly dangerous substances
	Do not clean dusty parts with compressed air
	Store the instrument and its accessories at a dry place in its original packaging
	Caution
F	Risk of minor or moderate injury by heavy weight of the instrument.
	Consult a second person to transport the instrument
	Do not drop the instrument
	Place the instrument on a stable, even and vibration-free surface

9.2 Disposal

For instrument disposal in an environmentally friendly manner, a list of materials is given in chapter 3. This helps to ensure that the components can be separated and recycled correctly. Regional and local laws concerning disposal must be followed. For help, please contact your local authorities!

NOTE

When returning the instrument to the manufacturer for repair work, please copy and complete the health and safety clearance form on the following page and enclose it with the instrument.

10 Spare parts

This chapter lists spare parts, accessories and options including their ordering information. Only order spare parts and consumables from BUCHI to maintain the warranty status and to assure best performance and reliability of the system and affected components. Any modifications to the spare parts used are only allowed with the prior written permission of the manufacturer.

Always state the product designation, instrument serial and part numbers for warranty clearance when ordering spare parts!

10.1 Spray head

Spray head	
Description	Order no
Spray head holder	11065359
Set Nebulizer small (3 pcs)	11064560
Set Nebulizer medium (3 pcs)	11064561
Set Nebulizer large (3 pcs)	11064562
Set Reservoir gasket (2 pcs)	11064742
Set visor gasket (2 pcs)	11064741
Set feed capillary	11064740
Fittings D1/16" gray (25 pcs)	044816
Ferrules D1/16" gray (25 pcs)	044269
Set of silicone seals (20 pcs)	040023
Connecting screw cap GLS 80	051518
Closing screw cap GLS 80	051529

10.2 Glass parts and mounting items









Glass parts and mounting parts	
Description	Order no.
Spray cylinder	051511

Glass cylinder

051549

Sealing set complete

051778

Tool box B-90

051767

Centerpiece hold	er	051630
Support metal ba	ar	051575

10.3 Particle collector, heater and lifting device items











Electrical particle collector, heater and lifting		
device		
Description	Order no.	
Particle collecting tube	051662	
Glass insulation tube	051663	
Collector base complete	051669	

Nano particle scraper	11055338
Particle collection paper (100 pages)	11055339

Heater complete

051504

Set of 3 polyethylene frits with sealing ring	051777
Temperature sensor PT-1000	051766

High voltage star electrode protected	11055174
Protective grid	051680

Plastic distance sheet	051530
Lifting device	051607

10.4 Filters



Outlet filter	
Description	Order no.
Outlet filter	051656



Inlet filter	
Description	Order no.
Inlet filter complete	011235
Replacement filter with seal	011238
Tube Flexflyte D51mm (0.8 m)	011240

10.5 Tubing and small parts



Tubing	
Description	Order no.
Gas inlet connection	051781

046329
004236
11055829
045656
046356
044407
004113
022352
004138
046314
046315



Small parts	
Upgr. set B-90 Advanced for closed cycle	11055748
(service engineer required for installation)	
Oxygen Sensor	046348
Peristaltic pump complete	051735
Silicone feed tubing (per m)	004138
Tygon tubing MH 2375 transp. (per m)	046314
Tygon tubing F 4040 A yellow (per m)	046315
Tube Cutter	019830
Nozzle cleaning tool	11065352
PC software Nano Spray Dryer records	051776
USB Cable 2.0 A-B (2.0 m)	11055310

10.6 Inert Loop B-295 and Dehumidifier B-296 Nano



Inert Loop B-295, Dehumidifier B-296 Nano			
Description	Order no.		
Receiving vessel for solvent, complete	040398		
PTFE tube to receiving vessel	004105		
PTFE hose connection SVL 22	027338		
Oxygen sensor	046348		

11 Declarations and requirements

11.1 FCC requirements (for USA and Canada)

11.1.3.1 English:

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to both Part 15 of the FCC Rules and the radio interference regulations of the Canadian Department of Communications. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment.

This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

11.1.3.2 Français:

Cet appareil a été testé et s'est avéré conforme aux limites prévues pour les appareils numériques de classe A et à la partie 15 des réglementations FCC ainsi qu'à la réglementation des interférences radio du Département canadien des communications. Ces limites sont destinées à fournir une protection adéquate contre les interférences néfastes lorsque l'appareil est utilisé dans un environnement commercial.

Cet appareil génère, utilise et peut irradier une énergie à fréquence radioélectrique. Il est en outre susceptible d'engendrer des interférences avec les communications radio, s'il n'est pas installé et utilisé conformément aux instructions du mode d'emploi. L'utilisation de cet appareil dans des zones résidentielles peut causer des interférences néfastes, auquel cas l'exploitant sera amené à prendre les dispositions necessaires pour palier aux interférences à ses propres frais.

Health and Safety Clearance

Declaration concerning safety, potential hazards and safe disposal of waste.

For the safety and health of our staff, laws and regulations regarding the handling of dangerous goods, occupational health and safety regulations, safety at work laws and regulations regarding safe disposal of waste, e.g. chemical waste, chemical residue or solvent, require that this form must be duly completed and signed when equipment or defective parts were delivered to our premises.

Instruments or parts will not be accepted if this declaration is not present.

Equipment

Model:

KUL

Part/Instrument no.:

1.A Declaration for non dangerous goods

We assure that the returned equipment

- has not been used in the laboratory and is new
- was not in contact with toxic, corrosive, biologically active, explosive, radioactive or other dangerous matters.
 is free of contamination. The solvents or residues of pumped media have been
 - is free of contamination. The solvents or residues of pumped media have been drained.

1.B Declaration for dangerous goods

List of dangerous substances in contact with the equipment:

Chemical, substance	Danger classification

We assure for the returned equipment that

- all substances, toxic, corrosive, biologically active, explosive, radioactive or dangerous in any way which have pumped or been in contact with the equipment are listed above.
- the equipment has been cleaned, decontaminated, sterilized inside and outside and all inlet and outlet ports of the equipment have been sealed.

2. Final Declaration

We hereby declare that

- we know all about the substances which have been in contact with the equipment and all questions have been answered correctly
- we have taken all measures to prevent any potential risks with the delivered equipment.

Company name or stamp:			
Place, date:			
Name (print), job title (print):			
Signature:			
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			i ago il i

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