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Operation Manual

knowledge

Action movie

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Contents

Read these instructions thoroughly before you use the **BÜCHI** Mini Spray Dryer **B-290**. Store these instructions in the immediate vicinity of the apparatus so that they may be consulted at any time.

Chapter 2 contains important safety information. Knowledge of this is imperative for the safe operation of the Mini Spray Dryer.

en, Version E (36 pages)	**Order code**
B-290 Instructions	**93001**

1 Scope of delivery

Figure 1.1: Spray Dryer, complete

Mini Spray Dryer B-290	**Order code**	
	200 V	230 V
Mini Spray Dryer B-290	**44781**	**44780**
Mini Spray Dryer B-290 Advanced	**44700**	**44699**

Enclosed Parts		
1	Instruction Manual:	
	German	**93000**
	English	**93001**
	French	**93002**
	Italian	**93003**
	Spanish	**93004**
1	Glass assembly complete	**44680**
1	5m Softaflex tube for compressed air	**04113**
1	Ico Quick Coupling	**04155**
1	4m Silicon tube for nozzle cooling	**04139**
1	2m Silicon tube	**04138**
1	Cleaning brush for nozzle	**44782**
1	3m Polypress tube for gas stream	**46341**
2	Hose clamps	**04236**

Additional with the B-290 Advanced		
1	Lamella Safety Curtain	**44783**
1	Feed Switch Valve	**44725**
1	Spray cylinder vertical outlet	**44697**
1	Screw cap 1.4mm	**44649**
1	2m Tygon tube MH2075	**46314**
1	2m Tygon tube F4040A	**46315**

Optional accessories	**Order code**	
	200 V	230 V
Inert Loop B-295 50Hz	**44779**	**44701**
Inert Loop B-295 60Hz	**46345**	**46344**

1	Brown glass assembly	**44758**
1	Nozzle complete	**44698**
1	Outlet filter, complete	**44754**
1	Inlet filter	**11235**
1	Compressed air maintenance unit	**04366**
1	Oil-free compressed air supply	**27907**

Table: Scope of delivery

2 Safety

The apparatus has been built in accordance with state-of-the art technology and recognized safety regulations.

Nonetheless risks and dangers can arise from the apparatus:

- If the apparatus is not used as directed.
- If the apparatus is operated by insufficiently trained personnel.

2.1 Symbols

Stop
Information on dangers which can lead to serious damage to property, to severe or life-threatening injuries.

Warning
Information on dangers which can lead to damage to health or property.

Please note
Information on technical requirements. Noncompliance can lead to faults, inefficiency and production losses.

2.2 Responsibilities of the Operator

The Mini Spray Dryer B-290 is only to be used by laboratory and specialist personnel and other persons, who, because of their training or professional experience, are able to assess the dangers which can result while operating the equipment.

Personnel without this training or persons who are in training require thorough instruction. These operating instructions are the basis.

2.3 Authorised use

The Mini Spray Dryer B-290 is conceived and designed as a laboratory instrument. Its regulation use is to dry aqueous solutions or suspensions in one operating process. In combination with the B-295, it is possible to work with organic solvents.

If the instrument is used with toxic or hazardous substances, it has to be installed into a closed fume cupboard.

Regulation use of the Mini Spray Dryer B-290 also includes its care and careful handling of the apparatus on the basis of the directions in these instructions.

2.4 Un-authorised use

Any uses other than those listed as well as any use which does not correspond to the technical data is a misuse. The operator bears sole risk for eventual damage related to such use. This laboratory apparatus is not intended for use in ex-protected areas.

In particular the following uses are not allowed:

- Spray Drying of organic solvent without the BÜCHI Inert Loop B-295 in open cycle
- Processing of explosive and deflagrating substances
- Working with oxygen-producing substances

2.5 Basic hazards

Fundamental dangers arise from:

- Equipment which shows visible damage
- Extended storage in unfavourable conditions
- Mixtures of unknown composition or with impurities
- Damaged glassware
- Hot surfaces
- Folded hoses
- Inflammable gases or vapours in the immediate vicinity of the apparatus
- Powdering, poisonous products while emptying the product vessel

2.6 Safety precautions

National and regional and local laws and regulations must be observed.

In order to arrest electrostatic charges away from the Mini Spray Dryer, the device is internally grounded. There is also the possibility of arresting electrostatic charges away from both the sample receiving vessel and the cyclone.

The wearing of personal safety equipment such as **safety glasses**, **protective clothing** is necessary. The glass parts are to be visually checked regularly for possible damage, stars, cracks. The grounded conductor (protective conductor) should never be disconnected. Otherwise their is the danger of electrical shock!

The operator is responsible for the instruction of his or her personnel. For this purpose these instructions can also be ordered in other languages (see Chapter 1, Scope of delivery). These instructions, as a component of the Mini Spray Dryer B-290, should be available to the operating personnel at any time at the location of use of the apparatus.

The operator is to inform the manufacturer immediately of all safety-related incidents which occur during use of the product.

2.7 Modifications

Modifications are only allowed after consultation and written permission from the manufacturer. Only glass parts recommended by the manufacturer may be used.

Only the specified parts to the Mini Spray Dryer meant for functional performance may be installed and removed. This is possible manually or with the supplied tool. The removal of protective fixtures and covers with the aid of the usual tools is forbidden, except by authorised repair and maintenance personnel.

2.8 Safety elements

Heating

- Excess temperature protection against uncontrolled overheating
- Automatic temperature control of the inlet temperature

Air

The system can be used in the suction mode or in the blowing mode. The suction mode is standard when working with aqueous solutions. The suction procedure used in the Mini Spray Dryer B-290 produces a light negative pressure in the apparatus and thus roles out, together with the intake filter and aspirator filter, a contamination of the environment through leaking from the apparatus. The blowing mode is used when working with the BÜCHI Inert Loop B-295 to have a small overpressure in the system. This pressure is a safety element and is indicated to detect a leakage.

Glass

- Use of inert 3.3 borosilicate glass
- Grounded coating of the inner surface of the cyclone to prevent any electrostatic charge of the powder
- Screwing Glass Connections to prevent glass breakage

3 Function

3.1 Functional principle of the drying air

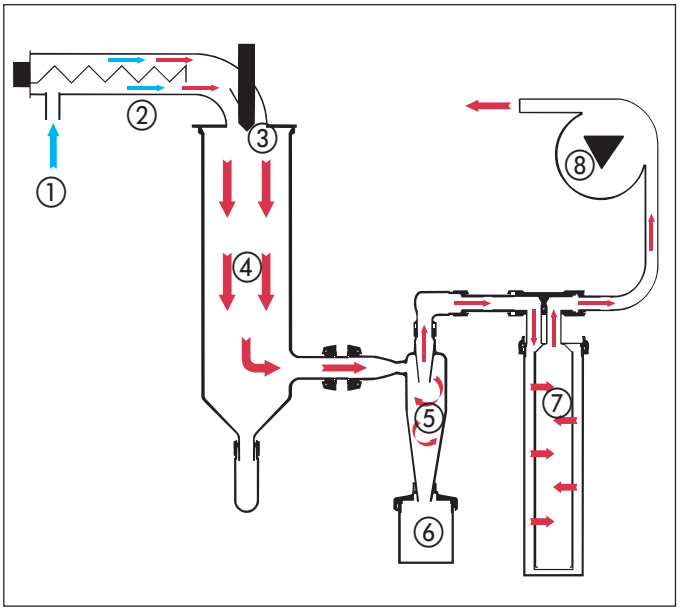


Figure 3.1: Functional principle 1

The Mini Spray Dryer B-290 operates according to a co-current air and product stream. This means that sprayed product and hot air have the same flow direction.

① Air inlet (optional with attached Inlet filter: see Chapter 8)
② Electric heater
③ Concentric inlet of the hot air around the spray nozzle
④ Spray Cylinder
⑤ Cyclone to separate particles from gas stream
⑥ Collecting vessel for dried product
⑦ Outlet filter
⑧ Aspirator to pump air through system

3.2 Functional Principle of the sample feed and dispersion

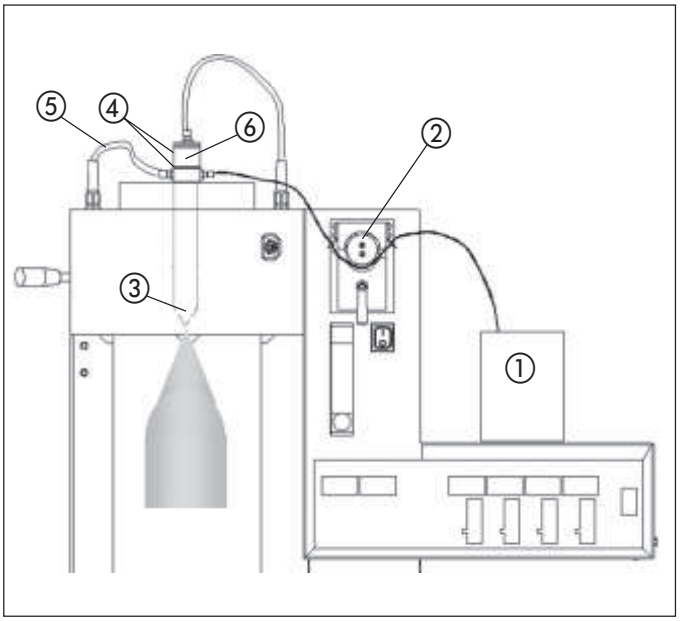


Figure 3.2: Functional principle 2

The Mini Spray Dryer has a integrated two-fluid nozzle: Compressed air is used to disperse the liquid body into fine droplets which are subsequently dried in the cylinder.

① Feed solution
② Peristaltic pump
③ Two fluid nozzle
④ Connection for cooling water
⑤ Connection for compressed air
⑥ Automatic nozzle cleaning system

4 Putting into operation

Check that the voltage of the socket is the same as that given on the apparatus plate.
The apparatus is supplied with an power cord which is directly plugged to the apparatus.

4.1 Installation location

The instrument must stand on a stable, horizontal base. The system is no ex-proof classification.

If the Mini Spray Dryer is intended for use to work with organic solvents, the system must be used in a fume hood or with the safety curtain. Additionally, the Inert Loop B-295 has to be connected.

The dimension of the Mini Spray Dryer are:

Width:	60 cm
Depth:	50 cm
Height:	110 cm
Weight:	46 kg
Operating temperature:	5-40°C
Operating temperature with Inert Loop B-295:	5-30°C

4.2 Unpacking

Look for any damage after unpacking. It is important that any damage in transit be identified right when unpacking. If necessary, make an immediate assessment of the situation (notify the post office, the railroad or the shipping company involved).

Save the original packing for possible transport at a later date.

Due to the weight of the Mini Spray Dryer B-290, at least 2 persons with appropriate tools must be present when moving the instrument.

4.3 Installation of the compressed air maintenance unit

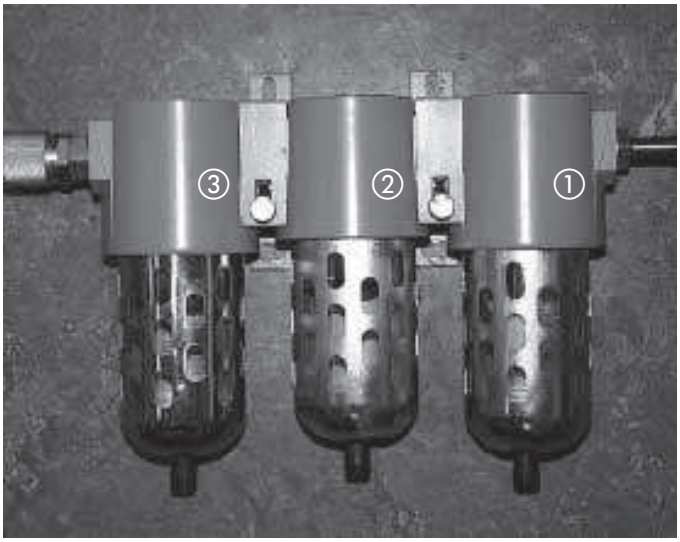


Figure 4.1: Installation of the compressed air maintenance unit

In order that the Mini Spray Dryer achieves an optimal atomisation performance, the supply of compressed air with a pressure of 5-8 bar is necessary. The compressed air must be dried and free from impurities (contamination of the product through impure air). If there is no air treatment plant available, we recommend the use of a WILKERSON maintenance unit (see Fig. 4.1), consisting of:

① Water separator

② Filter

③ Activated charcoal filter

If there is no compressed air plant available, the compressed air can be taken from compressed air bottles or produced with an oil-free air compressor with an output of 5-8 bar as required (see Chapter 8: Spare Parts and Accessories).

In case of compressed-air bottles care should be taken that they contain compressed air exclusively and no inflammable gases.

4.4 Installation of the glass assembly

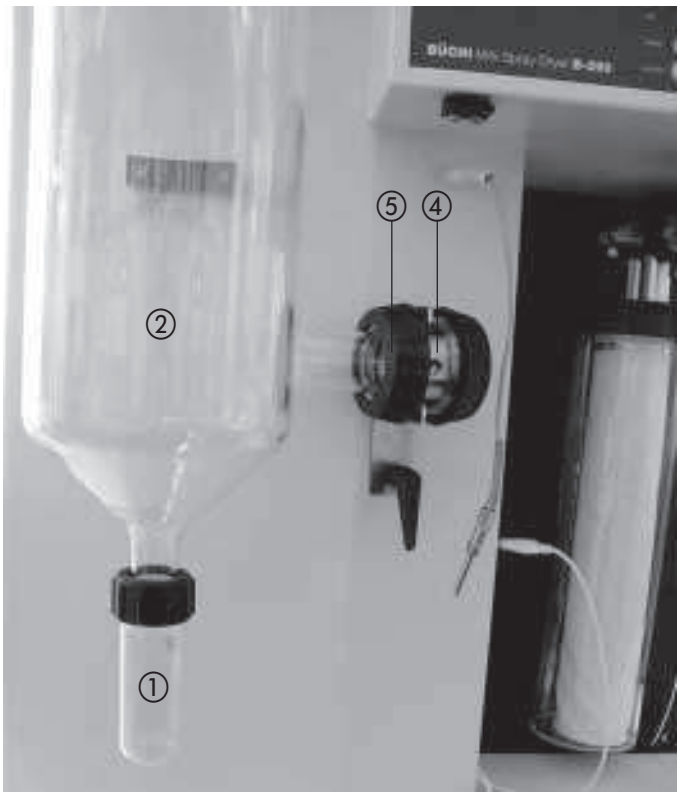


Figure 4.2: Pre-assembly of the spray cylinder

Care is to be taken that only glassware in perfect condition is used, which shows no cracks, stars or other damage.
The glassware is to be checked visually before the initial putting into operation of the apparatus.

- The separation flask ① is connected to the spray cylinder ② by closing the SVL 42 connection tightly.
- The connection piece ④ is screwed on the cylinder outlet flange with the flange screw joint ⑤. The connection is sealed with a Viton® O-Ring.

Figure 4.3: Installation of the spraying cylinder

- The Cylinder is mounted on the Mini Spray Dryer by holding the glass in the upper flange around the nozzle. At the same time, the connection piece is inserted into the support element ⑥. The fixation ⑦ must be opened. By thoroughly pushing the lever ③ on the left side, the flange of the cylinder is held firmly.
- The glass flange is pressed regulary on the above seal.
- The fixation ⑦ is closed to have it tightly fastened.

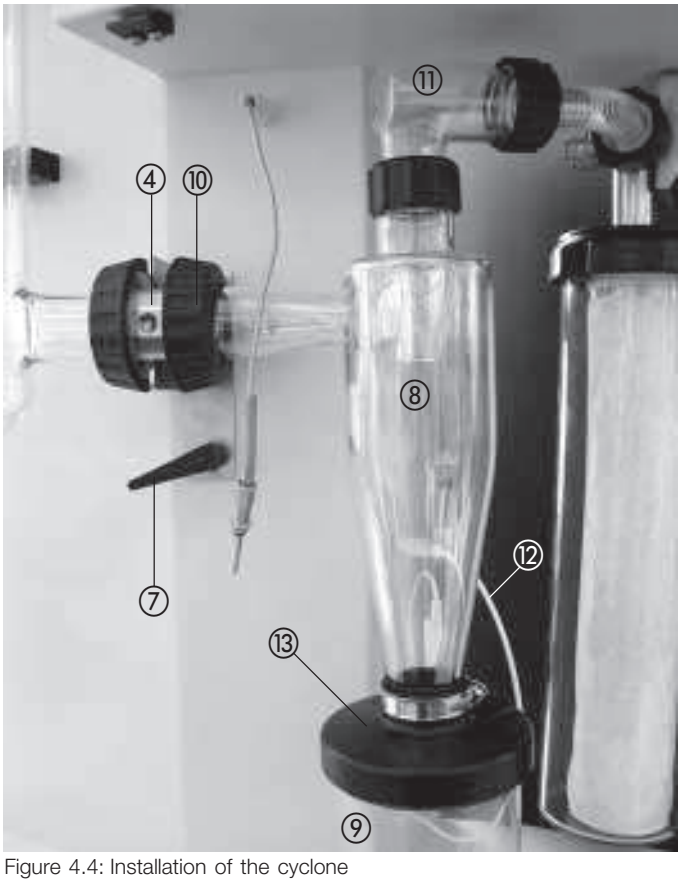


Figure 4.4: Installation of the cyclone

- Attach the cyclone ⑧ together with the collection vessel ⑨ to the connection piece ④ with a second flange screw joint ⑩.
- The glass elbow ⑪ is screwed on the top of the cyclone
- The grounding cable ⑫ connects the cover of collection vessel ⑬ with the housing to minimise electrostatic charge of the product.

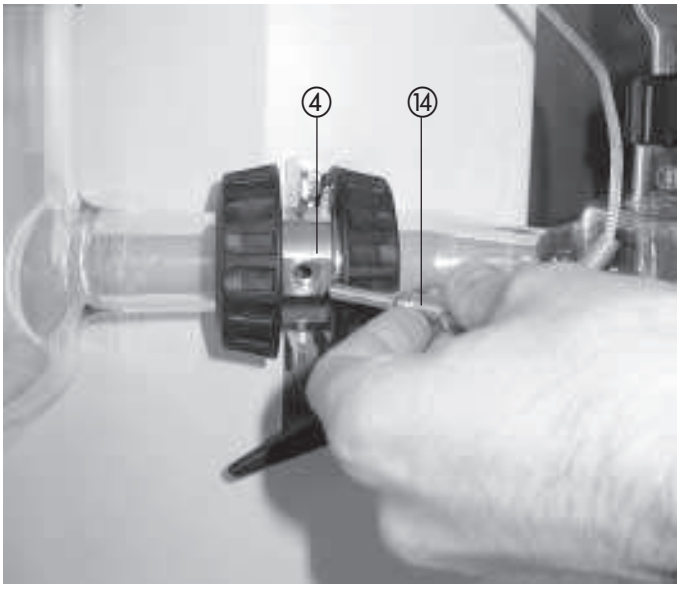


Figure 4.5: Mounting of the outlet temperature sensor

- Insert the outlet temperature sensor ⑭ into the connection piece ④ and screw it in. A metallic ring and O-Ring is inserted to seal the probe.
- Close all connections tightly.

4.5 Installation of the spraying nozzle

The nozzle tip consists of the nozzle cap with a 0.7 mm diameter hole and the screw cap (1.4 or 1.5 mm in diameter). This geometry results in a outer mixing of fluid body and gas. The tip of the screw cap has a inserted ruby stone with a precise opening and sharp edges to guarantee a precise and reproducible spray cone.

The smaller screw cap of 1.4 mm diameter results in a lower consumption of gas, as the concentric ring around the nozzle is smaller. This nozzle is recommended when nitrogen is used to minimise operating costs. The larger screw cap opening of 1.5 mm is typically used when air is the spraying gas, as this design is more robust to get a vertical and uniform spray cone.

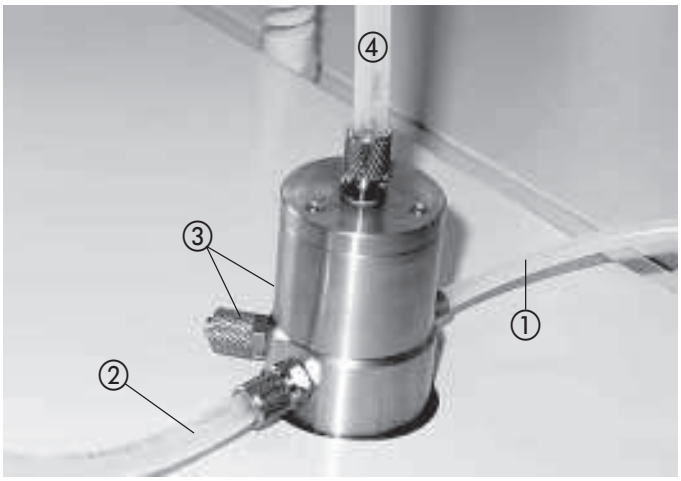


Figure 4.6: Installation of the spraying nozzle

- Insert the nozzle into the heater element at the top of the Mini Spray Dryer.
- The two-fluid spraying nozzle has a connection for the feeding tube ①, a connection for the pressurised air or nitrogen ② and two connections ③ for an optional cooling/heating with an external thermostat. The top connection ④ is for the implemented pneumatic nozzle cleaning: a needle is pushing through the nozzle and prevents a clogging.

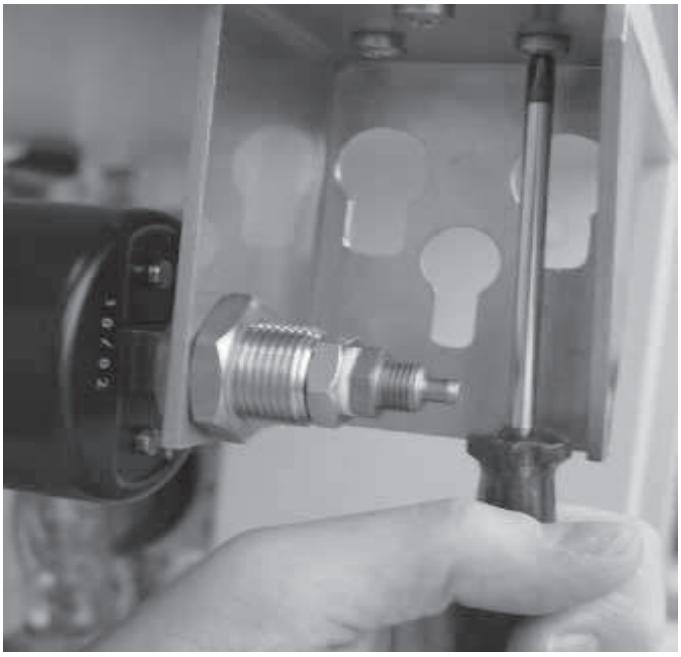


Figure 4.7: Installation of the support angle

4.6 Installation of the filters

Outlet filter

A support angle is screwed to the under side of the panel. The filter is hanged directly. The connecting tubing is installed with a SVL 42 between cyclone and filter. Please take care that the sealing of the SVL 42 in the screw cap.

Figure 4.8: Outlet filter / Aspirator filter

The aspirator filter, consisting of a woven polyester textile, prevents contamination of the environment and the possible corrosion of the aspirator by very fine particles which can not be separated in the cyclone. It can be washed by hand or machine. All needed connections are included.

The second filter included is made of a PTFE Membrane. By counter-pulsing the filter with pressurised air, some of the collected particles can be recovered resulting in a higher yield (see Chapter 5).

The manometer indicates the system pressure before the filter. With clean filter, the corresponding pressure drop is marked. If the pressure drop increases by more than 20 mbar relative to the clean filter, it has to be wasted or replaced.

Inlet filter

The Inlet filter is directly connected to the air inlet of the heater. The adapter is attached with a screw from the side. If the system is operated in the blowing mode, the adapter piece is turned and directly screw into the aspirator inlet connection.

4.7 Adjusting of peristatic pump bed

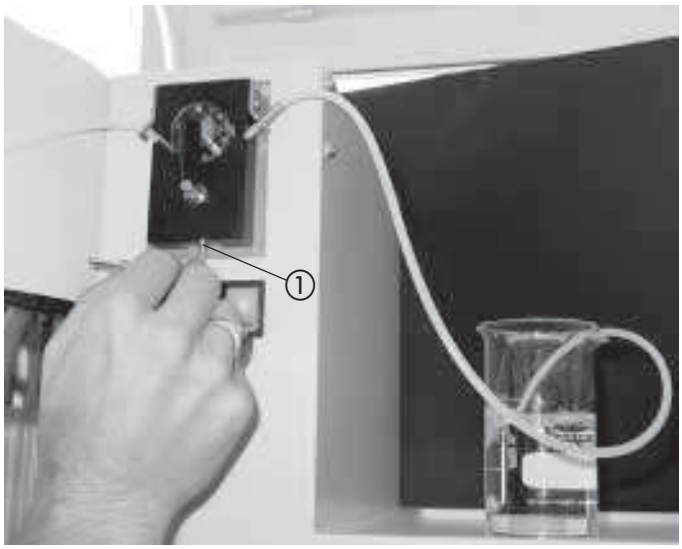


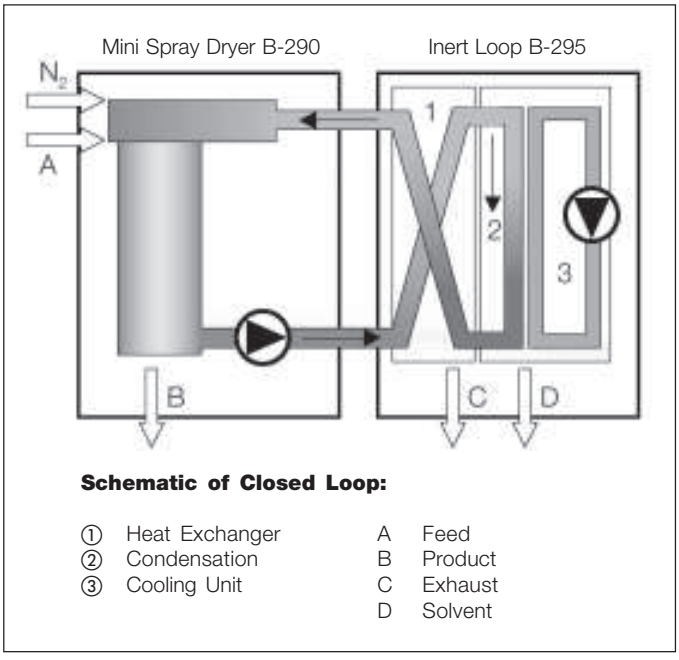
Figure 4.9: Adjusting of peristatic pump bed

The pump bed of the peristaltic pump is adjusted to the standard silicone tube.

If different tubes, e.g. the Tygon tubes for organic solvents, are used, the pump bed is adjusted with an Allan Wrench.

- Insert the tube and close the pump bed
- Put one tube end in water
- Smoothly blow by mouth air in the tube
- Turn the fixation screw ① with the Allan Wrench
- As long as bubbles rise, continue to tight the screw
- Exactly at the moment, when no bubbles appear anymore, the pump bed is correctly adjusted

4.8 Installation of the Inert Loop B-295



Schematic of Closed Loop:

①	Heat Exchanger	A	Feed
②	Condensation	B	Product
③	Cooling Unit	C	Exhaust
		D	Solvent

Schematic 1: Combined system of the B-290 and B-295

The Inert Loop B-295 is an accessory to enable the safe use of organic solvent in a closed loop.

The Inert Gas is loaded with solvent from the spray drying process. After pre-cooled in a heat exchanger, the solvent is condensed in a refrigerator and collected in a closed bottle. The cleaned gas stream is pre-heated in the heat exchanger and flows back to the Mini Spray Dryer.

Aqueous solutions may freeze in the heat exchanger of the refrigeration unit. Therefore, when working with water, the cooling temperature must be set above 10°C.
If a low end humidity must be obtained, a mixture of 20% ethanol/80% water is recommended.

Figure 4.10: Inert Loop B-295

The Inert Loop B-295 is on wheels an can be installed beside the Mini Spray Dryer B-290. The system is connected to the mains supply.

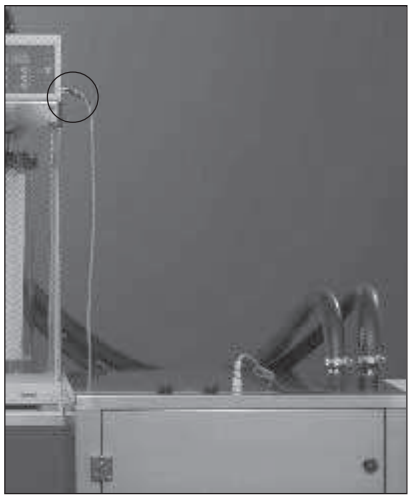


Figure 4.11: Communication cable for B-290 / B-295

The communication cable enables the safe operation between these two units.

4.9 Tubing

Bild 4.12: Quick coupling / rear side

The Büchi Mini Spray Dryer B-290 needs a pressurised air for the two-fluid nozzle and the nozzle cleaner between 5 and 8 bar. Therefore, the air or nitrogen is connected on the rear front of the instrument via a quick coupling.

The Mini Spray Dryer can be used in a open or closed cycle. The open cycle is in the sucking mode as a standard. However, if the exhaust is aggressive and could lead to a corrosion of the aspirator, the instrument can also be driven in a blowing mode. This mode leads to a higher noise emission.

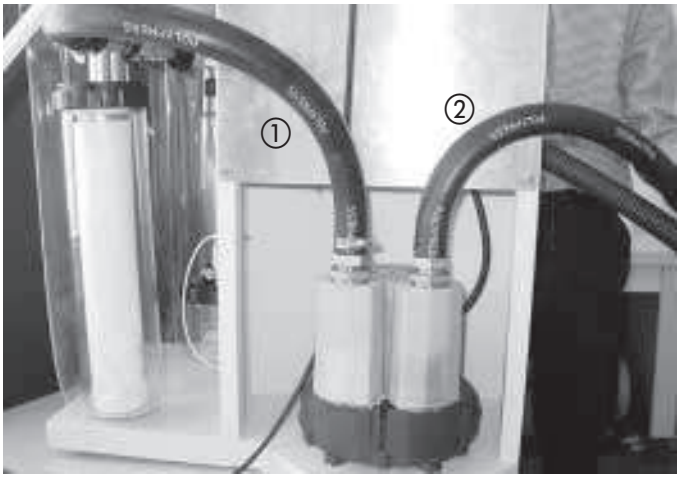


Figure 4.13: Open cycle; sucking mode

Open cycle, sucking mode

① Tubing cyclone or filter, respectively to aspirator

② Exhaust tube

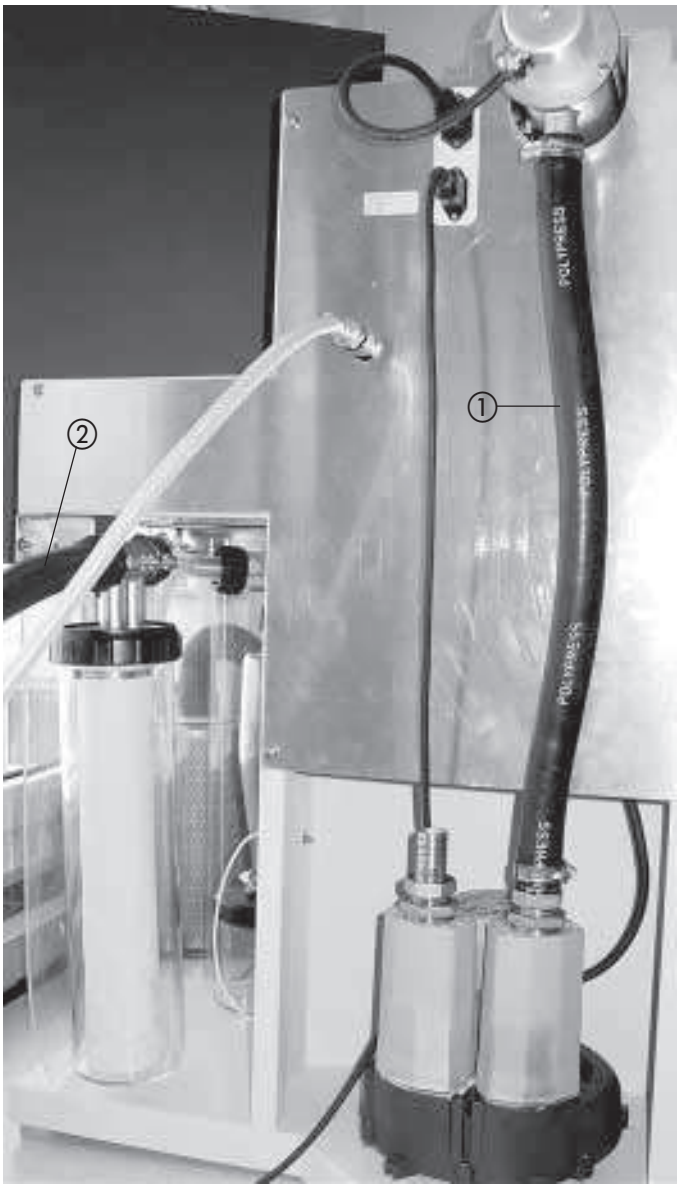


Figure 4.14: Open cycle; blowing mode

Open cycle, blowing mode

① Tubing aspirator to heater

② Exhaust tube from cyclone or filter, respectively

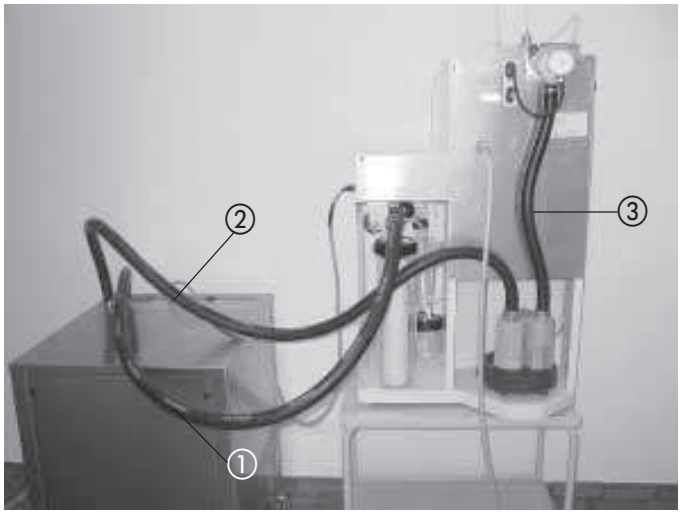


Figure 4.15: Tubing B-290 / B-295

Closed cycle mode with Inert Loop B-295

① Tubing between filter and refrigeration unit inlet

② Tubing refrigeration outlet and aspirator

③ Tubing between aspirator and heater


Figure 4.16: Exhaust tube

Exhaust tube

The Inert Loop B-295 has a sideward hose connection for the exhaust gas. Due to the constant feeding with nitrogen, a volume is permanenty leaving the system through this outlet tube. If air is sucked in through the tube, this is an indication that the closed loop is not dense.

The exhaust tube must be lead to a fume cupboard or outside to prevent a contamination of the room with solvents.

4.10 Installation check

An installation check is to take place after successful installation and before the first spray drying.

- Inspect the glass visually for possible damage.
- Check the electrical connections.
- The cover of the receiving vessel must be connected with the apparatus by means of the cable for arresting electrostatic charges.
- The outlet temperature probe must be inserted in the coupling.

5 Operation

Make sure that the apparatus has been put into operation in accordance with Chapter 4, Putting into operation.

5.1 Layout of the operating and display elements

Figure 5.1: Layout of the operating and display elements

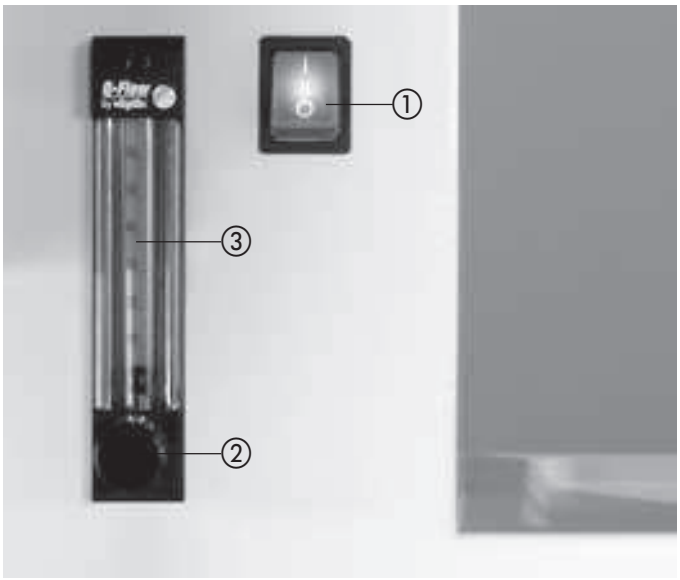


Figure 5.2: Layout of the operating and display elements

① Main switch
② Rotary switch for selection of air flow
③ Rotameter for air quantity (spraying air)
④ Main switch, pump
⑤ Main switch, aspirator
⑥ Main switch, heating
⑦ manual operation pneumatic nozzle cleaner
⑧ Regulating push-button, pump
⑨ Regulating push-button, aspirator
⑩ Regulating push-button, heating
⑪ Nozzle cleaner interval adjustment
⑫ LED display pump output in % of max. pump rate
⑬ LED display aspirator output in % of max. aspirator rate
⑭ LED display set value, inlet air temperature
⑮ LED display actual value, inlet air temperature
⑯ LED display actual value, outlet air temperature
⑰ Button feed switch valve
⑱ LED display for nozzle cleaning

5.2 Conversion tables for the parameters

A) Flow meter spraying air (Rotameter)

The Rotameter is a indicator for the spray gas flow. The table gives a correlation between indicated height and volume throughput. The nozzle has a certain pressure drop which increases with higher gas flow. As the gas volume strongly corresponds to the actual pressure, the table also contains a row for the effective volume flow, determined in a spray process with water.

Height (mm)	Normlitre/ hour	Pressure drop	Volume flow (real)
5	84		
10	138		
15	192		
20	246	0.15	282.9
25	301	0.18	355.18
30	357	0.23	439.11
35	414	0.3	538.2
40	473	0.41	666.93
45	536	0.55	830.8
50	601	0.75	1051.75
55	670	1.05	1373.5
60	742	1.35	1743.7
65	819	1.8	2293.2

Table 1: Conversion table of the spray flow (cm at the glass in liters per hour)

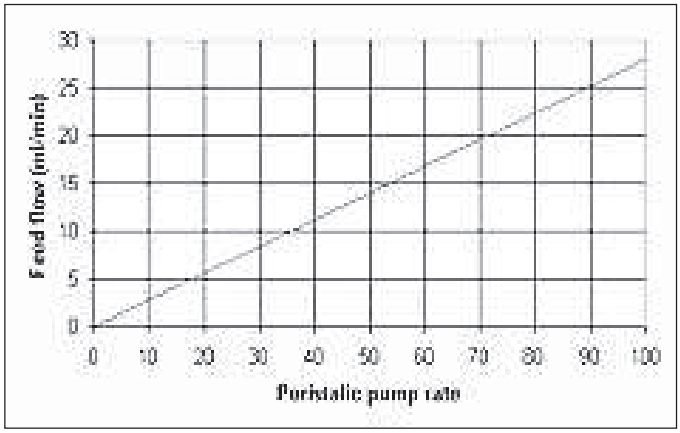


Diagram 2: Pump settings vs. throughput

B) Peristaltic pump

The peristaltic pump can be adjusted to different tubes according to the inner and outer diameters. With different tube diameter, the absolute flow is varying. The diagram shows the correlation for the standard 2/4 silicone tube.

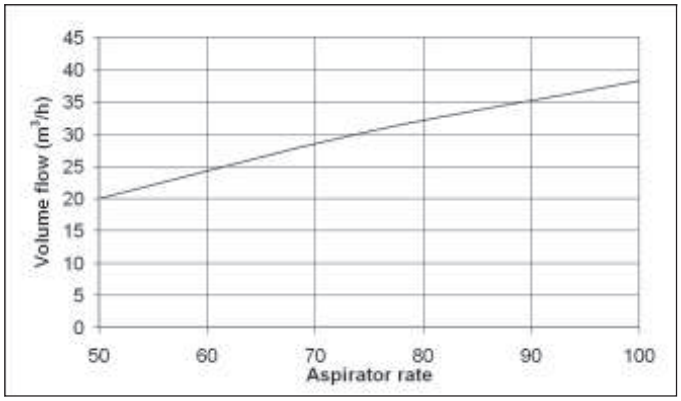


Diagram 3: Apirator settings vs. throughput

C) Aspirator

The Aspirator has a maximum gas flow of 40 m^3/h. The flow is depending upon the pressure drop of the overall system.

To determine the exact volume flow for steady and reproducible operating conditions, a measurement tube is supplied as an accessory (see Chap. 8). A Testo handheld flowmeter (hot-wire flowmeter) is required for the measurement.

5.3 Spray process

1. Turn apparatus on. The apparatus now carries out an automatic self check and then goes automatically into operating mode.

2. Set air flow at about 30 mm with the adjustment knob.

3. Pre-select the desired inlet temperature with the adjustment button.

4. Turn on the aspirator. As a standard, the aspirator rate should be 100% to maximize the separation rate of the cyclone. If a small powder humidity is required, the aspirator rate can be decreased.

5. Turn on heating and wait until the system has reached steady conditions.

6. If necessary, the nozzle cooling is now turned on.

7. Pure solvent, e.g. distilled water is case of aqueous solutions, is now sprayed in by means of the peristaltic pump. The spray cone is symmetrical and in the axis of the spray cylinder. If this is not the case the nozzle may be dirty or defective.

8. The spray quantity of pure solvent can be changed with the adjustment button of the peristaltic pump. The spray flow strongly influences the output temperature, since the water draws energy from the air by evaporation. In this way the output temperature can be set to the desired value with the quantity of sprayed solution via the pump speed of the peristaltic pump.

   The outlet temperature can be regarded as the upper thermal load of the product, so care is to be taken that the product is not destroyed by a too high output temperature.

9. As soon as the desired operating conditions have been achieved and are stable, the pure solvent which is sprayed in is replaced by the prepared product. Somewhat less solvent is now sprayed into the system by the concentration of the product in the water, which results in slightly higher output temperature (the pump rate can be slightly increased to adjust again).

5.4 Optimizing parameters

The Parameter of the spraying process are all correlated with each other and in different dependence. Therefore, special training papers can be downloaded directly from the Internet.

Please visit our homepage www.buchi.com. In the section Solutions/Drying/More Information, you find a document to download.

5.5 End of spray process

1. After the solution is completely spray dryed, pure solvent should continue to be sprayed for a short time, in order to remove product deposits from the hoses and the nozzle. Air and solvent should alternate to have a efficient cleaning.

2. Pump the hoses empty, turn off the peristaltic pump and lower the pump bed.

3. Turn off nozzle cleaner.

4. Turn off heating.

5. The system now cools down. As soon as the temperature in the apparatus sinks below 70°C, the aspirator can be turned off.

6. The receiving vessel with the product can now be removed. This should not be done when the aspirator is on since there is the possibility that the air stream could blow the product out of the receiving vessel into the surrounding area.

7. The glass assembly, hoses and the nozzle should be cleaned after every spray process.

5.6 Operation with the Inert loop B-295

The Inert Loop B-295 is connected according to Chapter 4, Installation. During operation, the communication cable between Mini Spray Dryer B-290 and Inert Loop B-295 must not be plugged out or in. The Mini Spray dryer has first to be switched off.

1. The Mini Spray Dryer is switched on. The unit automatically detects the connected periphery. **Both Signal lamps on the Inert Loop B-295 are burning**. If the Oxygen signal lamp is off, this could mean that from a previous operation, there is still enough inert gas in the loop. To calibrate the oxygen sensor before every process, it has to be ambient oxygen concentration. Thus, please open one tube connection wherever suitable and let the aspirator run for 2 minutes.
2. The Aspirator is switched on. The heater is switched on.
3. The spray gas source is checked to be an inert gas, normally nitrogen. The needle valve on the flow meter is opened and the flow is adjusted to the requested level. The closed system starts now to be inertised.
4. As soon as a certain pressure drop threshold is built by the gas flow and sprayign gas, **the signal lamp of the pressure monitoring is switched off**. If the pressure is dropping under the threshold (due to glass breakage, stop of gas inlet, taking away of a glass component etc.) the peristaltic pump and the heater is blocked. This is indicated by lightening the signal lamp and the Message PR LO on the display.
5. The overpressure in the system due to the constant flow inlet is limited by the exhaust outlet on the B-295, which is permanently open. Therefore, no overpressure can be built. The gas mixture from the exhaust outlet is slightly contaminated with solvents. This gas flow must be aftertreated any carried away according to regional standards and laws.
6. The oxygen concentration is decreasing due to the constant inlet of inert gas. As soon as the concentration is below 6%, **the signal lamp of the oxygen control is switched off**. If the oxygen threshold of 6% is exceeded (e.g. by having pressurized air instead of inert gas) the peristaltic pump and the heater is blocked. This is indicated by lightening the signal lamp and the Message O2 HI on the display.
7. As soon as both signal lamps are switched off, the peristaltic pump and the heater is deblocked and the spray process can be started according Chap. 5. The blocking and signals are reversible. If the pressure increases or the oxygen decreases, respectively, the pump and heater is deblocked again und must be started manually.

5.7 Emptying of the solvent recovery flask

At the right bottom side, a flask is positioned to receive the condensed solvent. To empty the flask, the valve at the top is closed and the flask is carefully taken out.

After the spraying process, the receiving bottle must be emptied regularly or the valve closed, respectively, to prevent a re-evaporation.

5.8 Calibration of the oxygen sensor

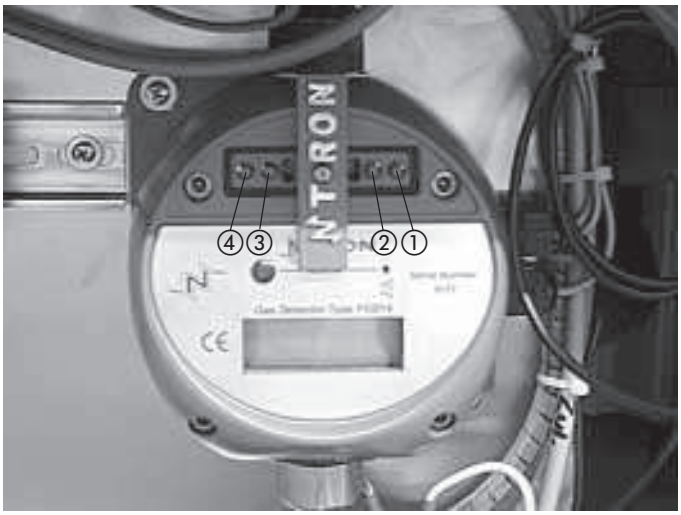


Figure 5.3: Oxygen analyser

The oxygen sensor is already calibrated ex work. However, due to shipping and other influences, the sensor may be calibrated again. At atmospheric condition, it has to monitor 21.0 ± 1.5%.

1. Remove the front plate with a screwdriver
2. Open the cover over the buttons on the oxygen analyser by turning it in vertical position.
3. Press menu ①, C:01 will appear.
4. Press UP ③ / DOWN ④ to change to C:2.
5. Press Enter ②, the oxygen concentration is shown on the display.
6. Press Enter ② again to calibrate the sensor.
7. Press Menu ① to quit the menu mode.

5.9 Particle Recovery of the Outlet filter


Figure 5.4: Outlet filter

If the system is run with a PTFE filter, the fine particles can partially be recovered by counter-pulsating the filter membrane and blowing the particles away.

- The PTFE membrane is attached instead of the polyester filter. The bottom is closed with a polypropylene plug.
- The manometer tube is separated from the filter and the filter is removed from the nozzle and attached to the connection of the manometer.
- The filter is put on the product feed table. The tube of the nozzle cleaning is taken away from the nozzle and attached to the connection of the manometer.
- The outlet gas connection is closed with a attached SVL 42 plug.
- With the nozzle cleaning set on level 1, a pressure blow is generated every 5 seconds on the inner side of the filter leading to a separation of the fines from the membrane. The particles are falling in the glass and can be recovered.

5.10 Description of the Connections

Figure 5.5: Connections

The Mini Spray Dryer has several connection on the right side under the product feed table. Fig. 5.3 shows the arrangement.


Figure 5.6: Remote Control

A) Remote Control

The Remote Control is directly connected and enables the easy operation even with closed fume hood. The flow meter for the spraying air is the only parameter which cannot be adjusted via the RC panel.

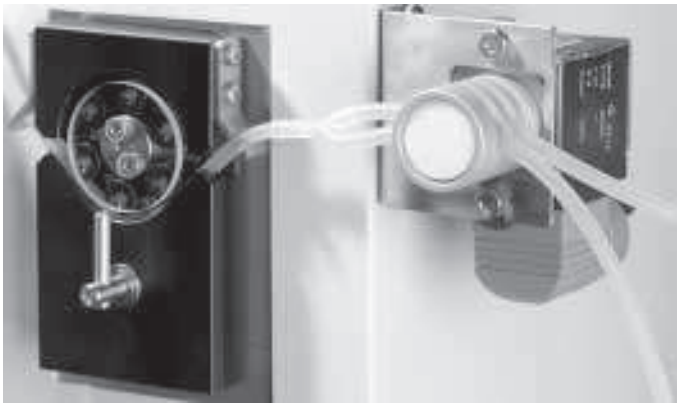


Figure 5.7: Feed Switch Valve

B) Feed Switch Valve

The Feed Switch Valve is a useful tool together with the RC panel. During the start and end of the spray process, the feeding tube has to be moved from pure solvent to product solution and vice versa. This can be automated with the feed switch valve. A Y piece is inserted between peristallic pump and feed switch value.

C) Communication to Inert Loop B-295

As already described in the above subchapter, the communication cable for the Inert Loop B-295 is inserted here.

D) RS-232 serial port Data Output

The Mini Spray Dryer is supplied with a standard protocol for data readout. This is of special importance if the system is qualified and underlies special regulations.

The transmission rate is 2400 Baud/s, Parity No.

The parameters are sent out every 30 seconds as an ASCII-Code, separated by a TAB (ASCII 09) and ended with a RETURN (ASCII 0D).

The source is defined as follows:

No.	Information	type	
1	Time since mains on	integer	s
2	Actual Temp. Inlet	integer	°C
3	Actual Temp. Outlet	integer	°C
4	Heater on/off	0/1	
5	Set Temp. Inlet	integer	°C
6	Aspirator on/off	0/1	
7	Aspirator rotation speed	integer	%
8	Pump on/off	0/1	
9	Pumpe rotation speed	integer	%
10	Feed switch valve	1/2	
11	Connection Inert Loop B-295 no/yes	0/1	
12	Oxygen high	0/1	
13	Pressure low	0/1	
14	Error message	integer	

Table: Documentation protocol

The RC-232 can be directly connected to a PC with a standard serial cable. With the 'hyperlink terminal' included in the Microsoft package. A standard RC-232 cable (9 pin, not crossed) is used.

5.11 Error and status messages

Error messages

Code	Meaning	Possible Cause	Remedial Action
001	Entry temperature sensor interruption	Defective sensor, sensor cable or internal wiring	Contact Customer Service
002	Entry temperature sensor short circuit	Defective sensor, sensor cable or internal wiring	Contact Customer Service
003	Entry temperature excessively high (< 230°C)	Defective heating control or heating relay	Contact Customer Service
004	Exit temperature sensor interruption	Defective sensor, sensor cable or internal wiring	Contact Customer Service
005	Exit temperature sensorshort circuit	Defective sensor, sensor cable or internal wiring	Contact Customer Service
006	Exit temperature excessively high	Defective sensor, sensor cable or internal wiring	Contact Customer Service
010	FU-malfunctionFU=Data Transmission?	Defective FU or wiringFU=Data Transmission?	Contact Customer Service
011	Heating malfunction	See under "System does not heat up"	Follow prescribed procedures
Only for operation with the Remote Control Panel			
100	Connection to Remote Control Panel interrupted or disconnected	Defective connection cable or not plugged in	Check connection cable for damage and insert plug if necessary
Only for operation with the B-295 Inert Loop			
200	Connection to B-295 interrupted or disconnected	Defective connection cable or not plugged in	Check connection cable for damage and insert plug if necessary
201	B-295 plugged in while operation in process	Connection cable plugged in after system switched on	Restart the system
202	O2 Alarm not given during start up	Oxygen sensor "spent"	Replace oxygen sensor
		Oxygen measuring equipment defective	Contact Customer Service
203	Pressure Alarm not given during start up	Pressure switch defective or contaminated	Contact Customer Service
204	Malfunction in cooling aggregate B-295		Contact Customer Service

Table 5.1: Error messages

Status messages

Only when operating with the Inert Loop B-295			
O2 HI	O2-sensor indicates excessively high O2-content	Oxygen connected instead of Nitrogen	Connect Nitrogen supply
		System not yet sufficiently inert (Display on oxygen measurement equipment still above threshold level but sinking)	Wait until threshold level is reached
		Entry of oxygen resulting from a leak	Check pipe system(pipe system in order, seals inserted and undamaged, cover firmly closed., seal in spray cylinder properly in place)
PR LO	Pressure Monitor shows operation taking place at too low a pressure	Leakage in gas circulation system	Check pipe system(pipe system in order, seals inserted and undamaged, cover firmly closed., seal in spray cylinder properly in place)

Table 5.2: Status messages

5.12 Fault elimination

Malfunction	Possible Cause	Corrective Action
Equipment cannot be switched on	No voltage	Insert mains plug /examine plug for damage
	Fuse has blown	Replace the fuse (3.15 A)
Peristaltic pump does not deliver	The rollers are not in contact with the running surface	Elevate the running surface with the lever
		Adjust the lifting height of the running surface with the hexagonal Allen Key from beneath
Product is delivered after the spray flow is switched on although the pump is switched off	The pressure of the rollers on the running surface is too weak	Adjust the lifting height of the running surface with the hexagonal Allen Key from beneath
Aspirator is noisy	The aspirator is dirty	Have the aspirator cleaned by a qualified person
		If no discharge filter is fitted, this should be inserted to avoid further contamination of the aspirator
	The operating mode is set to "Blow"	Reset to "Suction" mode if possible
System does not heat up	Heating circuit not plugged in	Plug in heating circuit
	Heating is not switched on	Switch on heating circuit
	Nominal entry temperature is below room temperature	Pre-select a new input temperature
	Fuse has blown	Replace fuse (12.5 A)
	Heater defective	Contact Customer Service
	Faulty pipe system (Faulty flow direction or no flow in the heating system)	Check pipe system
Nozzle blocked up	Product is too concentrated	Use a lower concentrationin pump
		Increase number of pulses for nozzle cleaning
	Encrustation on nozzle exit	Cool nozzle with cooling water
Product drips in spray chamber	No spray flow	Open compressed air valve
	Insufficient spray flow	Inspect pressure of air in the supply pipe (5 - 8 bar)
Insufficient aspirator performance	Discharge filter blocked	Dismantle filter and clean
Deposits on the spray cylinder	Nozzle is not clean	Completely dismantle the nozzle and clean with water
	Nozzle is defective (Bentnozzle needle)	Replace nozzle or defective element
	Product will not dry	Reduce the temperature difference between entry and exit
		Increase rate of spray flow(>600 l / Hr)
		Reduce peristaltic pump performance
	Entry temperature is above the melting point of the product	Reduce entry temperature
	Product related deposits	No action possible

Table 5.3: Fault elimination

Malfunction	Possible Cause	Corrective Action
Glass elements become wet	Peristaltic pump lever has become loose	Tighten lever
Irregular or pulsed spraying	Leaks in the spray nozzle	Check seals in the spray nozzle and replace if necessary
Deposits in the cyclone	Product-related deposits	No action possible
	Static charge build-up	Insert earthing cable
	Product too moist	Increase exit temperature to dry the product
	Temperature too high	Reduce aspirator performance to increase dwell time of product
Exit temperature does not rise	Sensor not inserted	Place probe in coupling element
	Fault in pipe system	Check pipe system
Entry temperature falls	Heating is switched off	Switch on heating
	Heater plug out	Insert heater plug
	Fuse has blown	Replace fuse (12.5 A)
Exit temperature falls	No heating	Follow measures under "Fall of entry temperature"
	Spraying too powerful	Reduce production rate of peristaltic pump
Rise in exit temperature	Nozzle blocked	Clean the nozzle by actuating the Cleaning Button or by switching on Nozzle Cleaning
		Increase number of pulses for the nozzle cleaning activity
	Hose not dipped into stock solution	Dip hose into product
	Change of concentration in stock solution	Agitate product (magnetic agitator) to obtain uniform concentration
	No feed of product	Switch on peristaltic pump

Table 5.3: Fault elimination (cont.)

6 Maintenance

Follow all instructions aimed at keeping the Mini Spray Dryer in a reliable operational state. This includes periodic cleaning and inspection for noticeable damage.

Check that the plug has been pulled out and that the compressed air and cooling water are both switched off before maintenance work on the apparatus.

6.1 Clearing

The glassware is to be cleaned with the usual cleaning agents (e.g. mild soap solution). The housing is to be cleaned with a damp cloth without the use of organic solvents and to be visually inspected for defects (operating elements, plug).

Defective apparatus components are to be replaced. Tubes can be dismounted are washed with usual cleaning agents. The system can also be fooded for cleanly. The exhaust tube must be raised. Then, the system can be filled. After filling, the drain value of the solvent is opened and the exhaust tube lowered for the complate emptying of the washing solution.

6.2 Customer service

Repairs to the apparatus may only be carried out by authorized service technicians. These are persons with a sound technical training and knowledge of the dangers which result from not observing safety regulations.

Authorized Büchi service centers have a special service manual available to them.

6.3 Changing the oxygen sensor

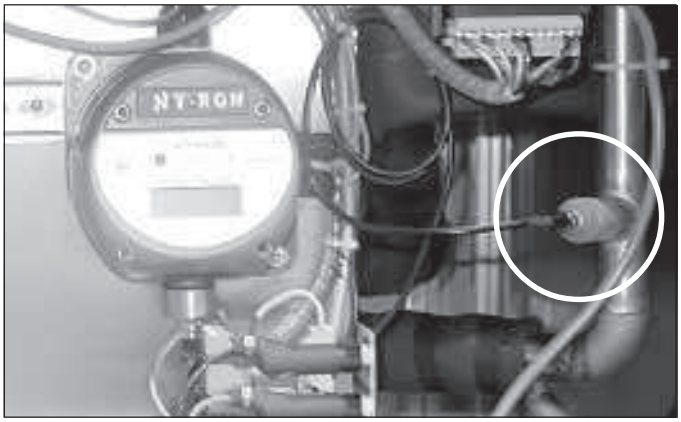


Figure 6.1: Changing the oxygen sensor

After approx. 1.5 year of lifetime, the oxygen sensor will strongly decrease the shown oxygen concentration. This is an indication that the sensor has to be replaced.

Changing the oxygen sensor:

1. Open the front plate using a allen wrench.
2. Disconnect the cable by turning the plug.
3. Screw the sensor out and replace it by a new one.
4. Connect the cable again.
5. Calibrate the sensor according chapter 5.8

7 Taking out of operation

Dangerous substances must be removed and the apparatus must be thoroughly cleaned. In this way any risk that persons will be injured through contact with dangerous substances will be eliminated.

7.1 Storage

The apparatus and accessories must be stored in a dry location.

7.2 Packing/transport

The original packaging is designed for the transportation of the apparatus as well as the glass parts. Only the original packaging is to be used for any possible further transport.

7.3 Disposal

So that the Mini Spray Dryer can be disposed of in an environmentally-correct manner, there is a list of the materials of the most important parts, which are also provided with material codes, in Chapter 9, Appendix. This guarantees that the parts can be separated and recycled. We refer you to the corresponding guidelines for the disposal of electrical parts. In addition, regional and local laws are to be observed by disposal.

8 Replacement parts / Accessories

Only original Büchi accessories and replacement parts guarantee safe use and reliable operation of the apparatus. The use of other than Büchi replacement parts and accessories is only allowed with the permission of the manufacturer. The replacement parts catalog may be used for purposes of assembly and disassembly only in connection with the corresponding Chapters 5 and 7 of the instructions. Inspection by and surrender to third parties as well as production in accordance with this manual are forbidden. Copyright remains with Büchi Labortechnik AG.

8.1 Spray nozzle

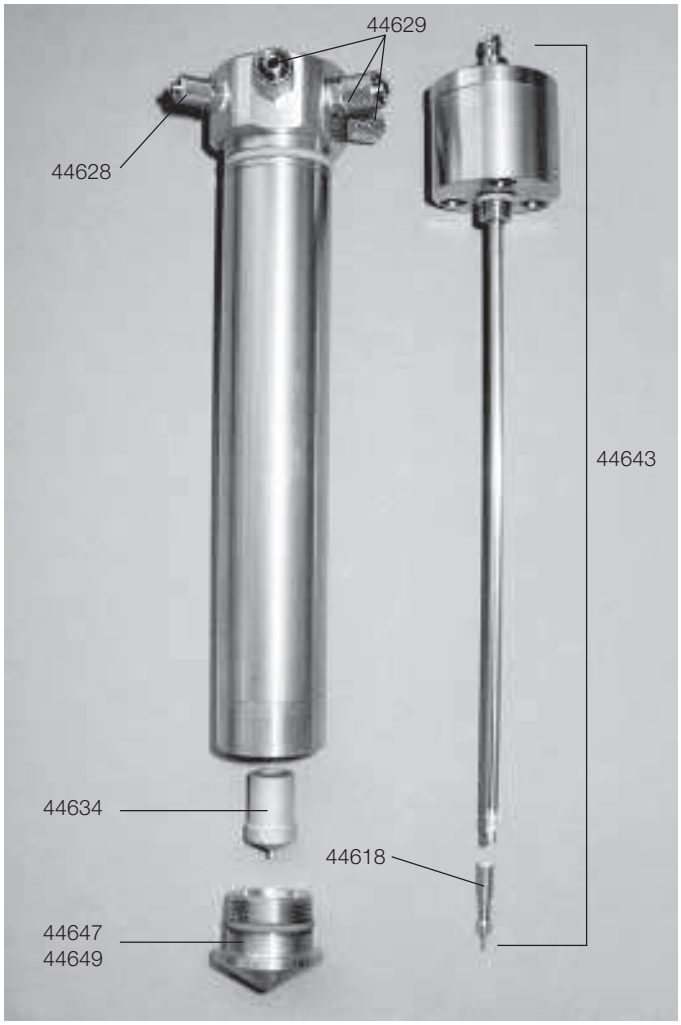


Figure 8.1: Spray nozzle

	Description	Order code
1	Needle for nozzle cleaning	**44618**
1	Screw connection product tube	**44628**
1	Screw connection air and cooling	**44629**
1	Nozzle tip	**44634**
1	Nozzle cleaning cpl.	**44643**
1	Nozzle cap Ø 1,5 mm	**44647**
1	Nozzle cap Ø 1,4 mm	**44649**
1	Set of O-rings	**44759**
1	O-ring in Kalrez for nozzle tip	**46361**

Table: Spray nozzle

8.2 Glass parts

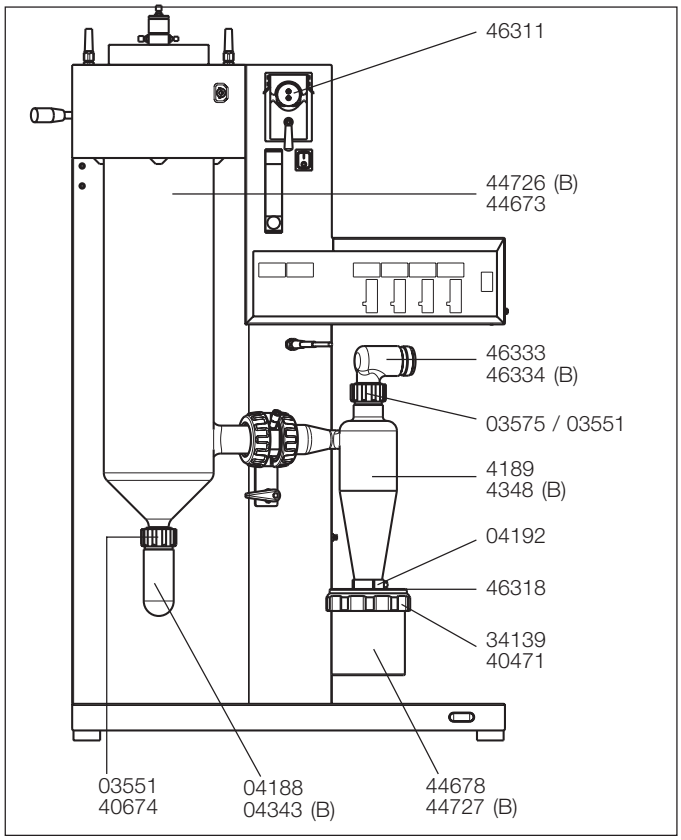


Figure 8.2: Glass parts 1

	Description	Order code
1	Screw cap SVL 42	**03551**
1	Seal SVL 42, PTFE	**03575**
1	Separation flask	**04188**
1	Cyclone	**04189**
1	Seal to cyclone	**04192**
1	Separation flask, brown glass	**04343**
1	Cyclone, brown glass	**04348**
1	Flange screw coupling	**34139**
1	Seal to product vessel	**40471**
1	Silicon seal SVL 42,	**40674**
1	Angle piece	**46333**
1	Angle piece, brown glass	**46334**
1	Cylinder, sideways outlet	**44673**
1	Product collection vessel	**44678**
1	Cylinder, sideways outlet, brown glass	**44726**
1	Product collection vessel, brown glass	**44727**
1	Cover to collection vessel	**46318**
1	Resistant O-Ring Set in Kalrez	**46364**
1	Plastic closure for product collection vessel	**46358**

Table: Glass parts 1

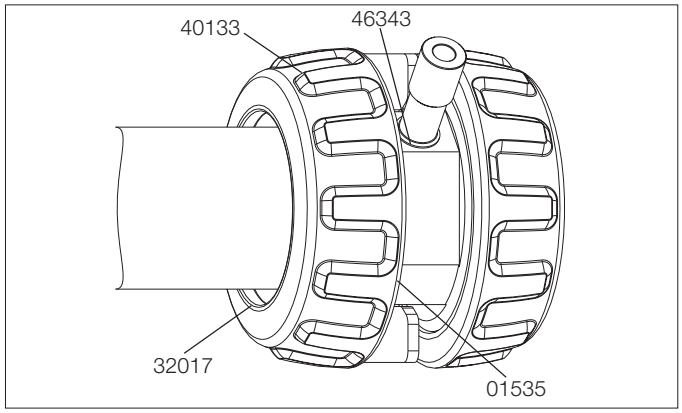


Figure 8.3: Glass parts 2

1	O-Ring	**01535**
1	Tension spring	**32017**
1	Set Flange screw coupling (2 units)	**40133**
1	Set O-Ring and clamping ring (5 units)	**46343**
1	O-Ring Kalrez for screw coupling	**46363**

Table: Glass parts 2

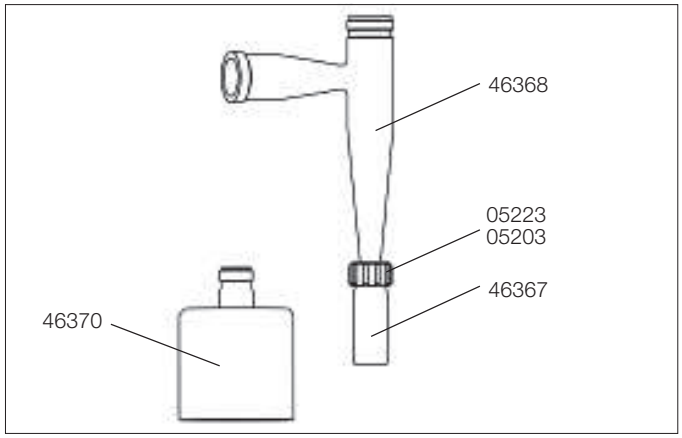


Figure 8.4: Cyclone

	High-performance Cyclone	**46368**
	Small product collection vessel	**46367**
	Screw cap SVL 30	**03223**
	PTFE seal SVL 30 x 20	**05203**
	Screw cover SVL 30	**05223**
	Cyclone complete (all above numbers)	**46369**
	Large product collection vessel	**46370**

Table: High-performance Cyclone

8.3 Filters

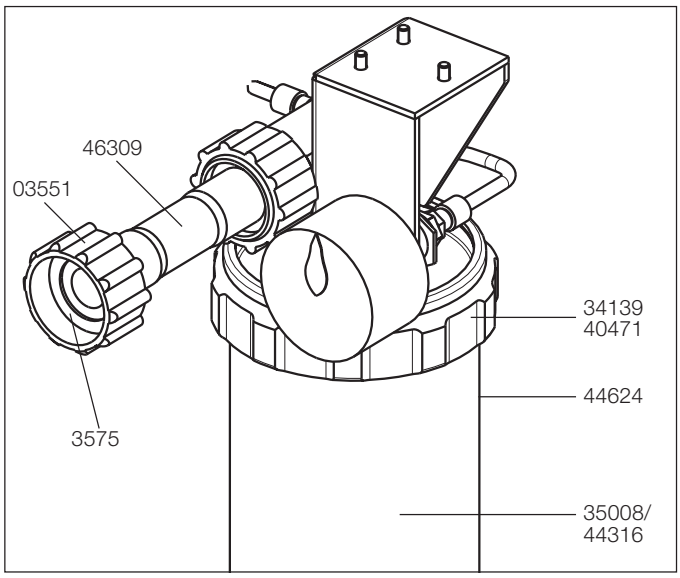


Figure 8.4: Outlet filter

Description		Order code
1	Screw cap SVL 42	**03551**
1	Seal SVL 42, PTFE	**03575**
1	Flange screw coupling	**34139**
1	Seal to product vessel	**40471**
6	Polyester filter bags	**35004**
1	Polyester filter bag	**35008**
1	Filter glass	**44624**
1	Filter tube, complete	**46309**
1	PTFE membrane filter	**46316**

Table: Filtes

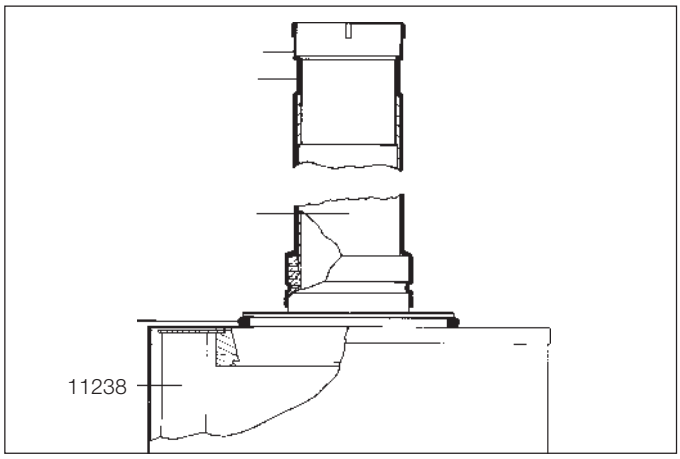


Figure 8.5: Inlet filter

1	Inlet filter, complete	**11235**
1	Replacement filter with seal	**11238**

8.4 Tubing

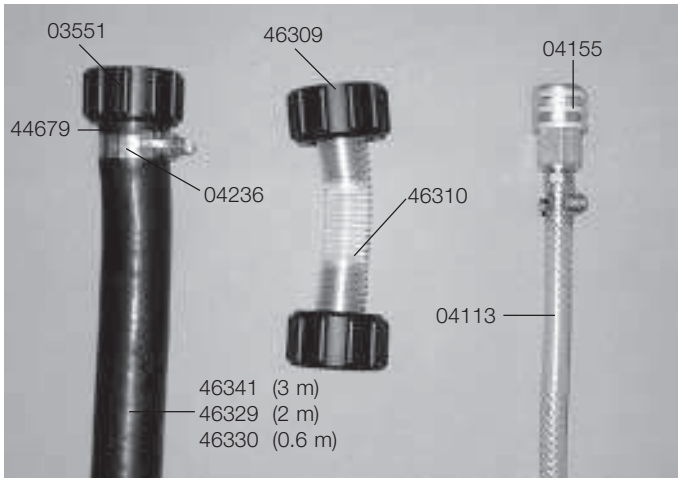


Figure 8.6: Tubing

Description	Order code
Softaflex tube	**04113**
2 m Silicone tube	**04138**
ICO quick coupling	**04155**
Polypress tube (3m)	**46341**
Polypress tube (2 m)	**46329**
Polypress tube (0.6 m)	**46330**
Filter tube, complete	**46309**
2 m Tygon tube MH 2075	**46314**
2 m Tygon tube F 4040A	**46315**
0.1 m Flextube PFA	**46310**

Table: Tubing

8.5 Trolley

Figure 8.7: Trolley

Trolley in stainless steel, powder coated
LxBxH: 850 x 420 x 560 mm

Description	Order code
Trolley	**41257**

8.6 Measurement tube for the gas throughput

Figure 8.8: Measurement tube for the gas throughput

For the precise determination of the gas throughput in absolute measures. A pipe generates a laminar flow for the determination with a hot-wire instrument. The appropriate instrument can be supplied by Testo (www.testo.com) type Test 435 with sensor No. 0635 1044.

Description	Order code
Measurement tube for gas flow	**44790**

8.7 Feed Switch Valve

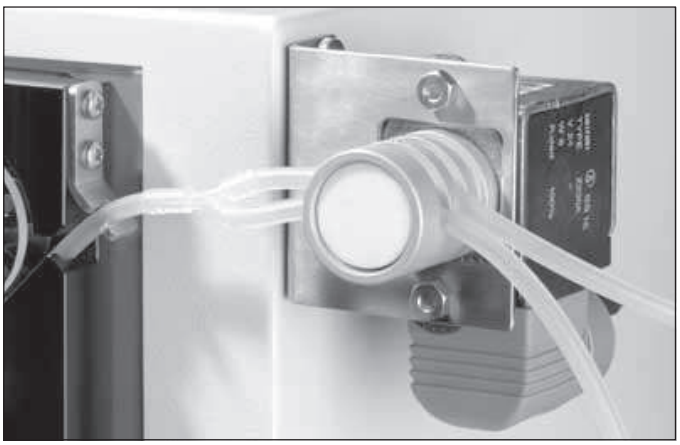


Figure 8.9: Feed Switch Valve

For the safe and automated switch between pure solvent and product when starting and ending the spray drying process.

Description	Order code
Feed Switch Valve	**44725**

9 Appendix

9.1 Technical data

BÜCHI Mini Spray Dryer **B-290**	
Power Rating	2900 W
Voltage	200/230 V ± 10%, 50/60 Hz
Ambient conditions	inside, below 2000 m.s.l., 5-40° maximum relative humidity 80% for temperatures up to 31° C decreasing linearly to 50% relative humidity at 40° C
Evaporating Capacity	1.0 l/h H_20, higher for organic solvents
Air Flow	max of 35 m^2/hr
Motor Control	Frequency Converter
Max. Temperature Input	220°C
Heating Capacity	2300 W
Heating Control	PT-100, Fuzzy Logic Control Accuracy +/- 2°C
Interface	Serial port RS-232 for all parameters
Spray Gas	Compressed air or nitrogen / 200 - 800 l/h, 5 - 8 bar
Nozzle Diameter	0.7 mm standard, other sizes on request
Nozzle Cap	1.4 mm and 1.5 mm diameter
Mean Dwell Time	1.0 - 1.5 seconds
Materials in contact with product	Acid-resistant stainless steel / 3.3 Borosilicate glass
Degree of pollution	2
Overvoltage category	II
Dimensions	60 x 50 x 110 cm
Weight	46 kg

BÜCHI Inert Loop **B-295**	
Power Rating	1 kW
Voltage	200/230 V ± 10%, 50 and 60 Hz
Min. Outlet Temperature	down to -20°C
Rate of Cooling	800 W at -10°C
Dimensions	60 x 70 x 84.5 cm
Weight	95 kg

Table: Technical data

9.2 Materials used

Description	**Material**	**Material code**
Glass assembly	3.3 Borosilicate glass	
Nozzle / Heater / Connection piece	Stainless steel	1.4301 / 1.4305
Seal cylinder	PTFE	
Seal cyclone / cylinder	Silicone	
Heat exchanger Inert Loop B-295	Stainless steel	1.4301
Polypress tube	EPDM	
Product feed tube	Silicone and Tygone	

Table: Materials used

9.3 FCC requirements (for USA and Canada)

English:

This equipment has been tested and found to comply with the limits for a Class A digital device, pusuant 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 like to cause harmful interference in which case the user will be required to correct the interference at his own expense.

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 règlementation FCC à la règlementation des radio-interférences du Canadian Department of communications. Ces limites sont destinées à fournir une protection odéquate contre les interférences nétastes lorsque l'appareil est utilisé dans un environnement commercial.

Cet appareil génère, utilise et peut radier une énergie à fréquence radioélectrique, il est en outre susceprible d'engendrer des interferences 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 les zones résidentielles peut causer des interférences nèfastes, auquel cas l'exploitant sera amené à prendre les dispositions utiles pour polier aux interférences à ses propres frais.

9.4 CE declaration of conformity

We **BÜCHI** Labortechnik AG
P.O.Box, CH-9230 Flawil, Switzerland

declare in general responsibility that the products:
BÜCHI Mini Spray Dryer **B-290**
BÜCHI Inert Loop **B-295**

to which this declaration refers is in conformity with the following standards:

EN 60204-1:1997 (~IEC 60204-1)
Safety of machinery - electrical equipment of machines; Part 1: General requirements

EN 61010-1:2002 (~IEC 1010-1)
Safety Rules for Electrical Measurement, Control and Laboratory Equipment: General requirements

EN 61326-1:1999
EN 61326/A1:1999
Electrical Equipment for Measurement, Control and Laboratory Use. EMC requirements

EN 61000-3-2: 1995/1996
Limits for harmonic current emissions

EN 61000-3-3: 2002
Limitation of voltage fluctuations and flicker

EN 55014: 200
Discondinuous emissions on power supply 150 kHz – 30 MHz

In accordance with the provisions of the EU guideline:
89/392/EEC (Guidelines for machines)
73/23/EEC (Guidelines for low voltage electrical equipement)
89/336/EEC (Electromagnetical compatibility)

Flawil, 6th march 2003

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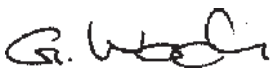

Guido Worch
Qualitymanager