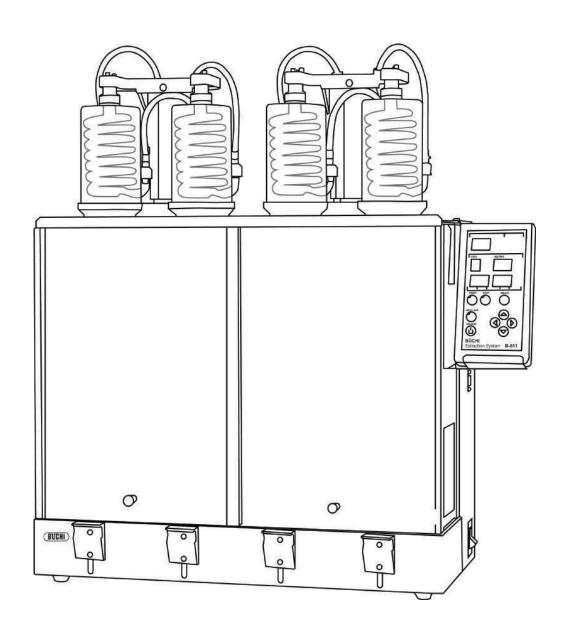


# **Extraction System B-811** Operation Manual





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## 1 About this manual

This manual describes the Extraction System B-811 and provides all information required for its safe operation and to maintain it in good working order.

It is addressed to laboratory personnel in particular.

Read this manual carefully before installing and running your system and note the safety precautions in section 2 in particular. Store the manual 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. Technical data are subject to change without notice.

#### NOTE

The symbols pertaining to safety (WARNINGS and ATTENTIONS) are explained in section 2.

This manual is copyright. Information from it may not be reproduced, distributed or used for competitive purposes, nor made available to third parties. The manufacture of any component with the aid of this manual without prior written agreement is also prohibited.

The English manual is the original language version and serves as basis for all translations into other languages. If you need another language version of this manual, you can download available versions at www.buchi.com.

#### 1.1 Reference documents

For information on the Hydrolysis Unit B-411 / E-416, the Recirculating Chiller B-740 and the Mixer B-400, please refer to the corresponding manuals available in English, German, French, Italian and Spanish

- Hydrolysis Unit B-411 / E-416, Operation Manual numbers 96684 96688
- Recirculating Chiller B-740, Operation Manual numbers 96690 96694
- Mixer B-400, Operation Manual numbers 96540 96544

#### 1.2 Abbreviations

LSV: Large sample volumes PTFE: Polytetrafluoroethylene

# 2 Safety

This chapter points out the safety concept of the instrument and contains general rules of behavior and warnings from hazards concerning the use of the product.

The safety of users and personnel can only be ensured if these safety instructions and the safety-related warnings in the individual chapters are strictly observed and followed. Therefore, the manual must always be available to all persons performing the tasks described herein.

## 2.1 User qualification

The instrument may only be used by laboratory personnel and other persons who on account of training or professional experience have an overview of the dangers which can develop when operating the instrument.

Personnel without this training or persons who are currently being trained require careful instruction. The present Operation Manual serves as the basis for this.

## 2.2 Proper use

The instrument has been designed and built for laboratories. It serves for the extraction (solid-liquid extraction) of samples with known solvents or solvent mixtures and the reduction or drying of the extract. Solvents with boiling points between 30 °C and 150 °C as well as inert gas may be used.

It is used for:

- Soxhlet Standard
- Soxhlet Warm
- Hot Extraction
- Continuous

## 2.3 Improper use

Applications not mentioned above are improper. Also, applications, which do not comply with the technical data, are considered improper. The operator bears the sole risk for any damages caused by such improper use.

The following uses are expressly forbidden:

- Use of the instrument in rooms which require ex-protected instruments.
- Determination of samples, which can explode or inflame (example: explosives, etc.) due to shock, friction, heat or spark formation.
- Use with samples that react with solvent.
- Use in conjunction with solvents containing peroxides.
- Use in overpressure situations.
- Use with other glassware than the original from Buchi.



#### WARNING

Generally, the triangular warning symbol indicates the possibility of personal injury or even loss of life if the instructions are not followed.



#### WARNING

Hot surface.



#### **WARNING**

Electrical hazard.



#### **ATTENTION**

With the general "Read this" symbol, ATTENTIONs indicate the possibility of equipment damage, malfunctions or incorrect process results, if instructions are not followed.

#### NOTE

Useful tips for the easy operation of the instrument.

## 2.5 Product safety

The Extraction System B-811 is designed and built in accordance with state-of-the-art technology. Nevertheless, risks to users, property, and the environment can arise when the instrument is used carelessly or improperly.

The manufacturer has determined residual dangers emanating from the instrument

- if the instrument is operated by insufficiently trained personnel.
- if the instrument is not operated according to its proper use.

Appropriate warnings in this manual serve to make the user alert to these residual dangers.

#### 2.5.1 Instrument-related hazards

Pay attention to the following safety notices:



#### **WARNING**

Potentially hot surfaces during operation, especially at the heating elements (up to 250 °C).

Always be aware of the risk of being burned.



#### **WARNING**

Potentially lethal voltage inside the instrument.

- Do not remove covers and other parts protecting from electricity.
- Always keep the areas of electric parts, such as power supply plug, mains switch, etc. dry.



#### WARNING

Potential explosion risk if solvent vapors accumulate within the instrument housing.

- Always use the instrument in a well ventilated area.
- · Beware of damaged or cracked glass parts.
- Beware of damaged seals.
- Beware of incorrect installed or damaged glass valve.
- Beware of the fire hazard.

#### 2.5.2 Other hazards



#### WARNING

Certain solvents in or in the vicinity of the Extraction System B-811 can form peroxides and/or are highly inflammable.

- Always be aware of the explosion risk when working with hazardous substances or with substances of unknown composition.
- Always provide a good ventilation within or in the vicinity of the system.

## 2.5.3 Safety measures

In case a safe operation of the instrument no longer appears possible, the instrument must be shut down and secured against unintentional operation.

It must be assumed that safe operation is no longer possible:

- if there is visible instrument damage,
- following a long period of storage under unfavorable conditions,
- following transport under difficult conditions.



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

#### 2.5.4 Safety elements

#### Electrics:

The main power switch automatically turns to the zero position (off) if the power supply is overloaded or a short circuit occurs.

#### **Electronics**

- The heating elements are equipped with an electronic over-temperature protection.
- If no cooling water is detected at the cooling water output (for example, due to a leak in the condenser), the electronic will stop all processes.
- The protective shield protects operators from broken glass in case of an accident or explosion.
- An extraction position is only activated if a beaker is in contact with the lower heating element. In
  order to start a program, at least one extraction position must have been activated. If one of the
  glass beakers break, the program closes the glass valves and terminates the processing of all
  activated heating elements.
- If one of the solvent beakers gets dry (e.g. due to a defective seal or an open cooling unit), the software turns off the corresponding heating element.
- It is possible to release solvent and/or open the gas valve at any time during operation (only applicable for activated extraction positions).
- Only high quality borosilicate 3.3 glass is used. In order to prevent glass breakage, the cooling supply connections are threaded and can therefore be screwed on and off.

#### 2.6 **General safety rules**

#### Responsibility of the operator

The head of laboratory is responsible for training his 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.

#### Duty of maintenance and care

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

#### 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.

#### **Modifications**

Modifications to the instrument are only permitted after prior consultation with and 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 Technical data

This chapter introduces the reader to the instrument specifications. It contains the scope of delivery, technical data, requirements and performance data.

## 3.1 Scope of delivery

Check the scope of delivery according to the order number.

#### NOTE

For detailed information on the listed products, see www.buchi.com or contact your local dealer.

#### 3.1.1 Standard instrument



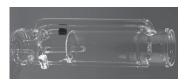
Table 3-1: Standard instrument	
Product	Order number
Extraction System B-811, 230 V	36680
Extraction System B-811, 115 V	36681
Extraction System B-811, 100 V	40550



Table 3-2: LSV instrument	
Product	Order number
Extraction System B-811 LSV, 230 V	37900
Extraction System B-811 LSV, 115 V	37901
Extraction System B-811 LSV, 100 V	40549

Table 3-3: Mixed instrument	
Product	Order number
Extraction System B-811 Standard/LSV (two positions of each), 230 V	11056621

## 3.1.2 Standard accessories





















Product	Order number	Order number
	B-811 Standard	B-811 LSV
4 Extraction chambers	36710	37902
4 Condensation tubes	37482	37903
4 Sample holders	36559	37904
1 Set of solvent beakers (4 pieces)	37276	38597

(4 pieces)		
Pack of paper thimbles 33 x 94 mm	41883	
(6 pieces)		
Pack of paper thimbles 43 x 123 mm		41884
(6 pieces)		

37281

37563

Set thimble holders 33 x 94 mm	37279	-
(4 pieces)		

8 Holder rings (black ring already mounted to the extraction chambers with the seals)

1 Set of glass sample tubes

36709

2 Sets of seals for extraction chambers (4 pieces per set, already mounted to the extraction chambers with the holder rings) 37388

37496











Product	Order number	Order numbe
	B-811 Standard	B-811 LSV
4 Condensers	367	11
1 Set of blank plugs (4 pieces)	373	68
4 Magnetic valves complete	366	87
Membrane with anchor for valve unit (included in magnetic valve complete (36687))	375	34

1 Inert gas supply

04113
37780
22352
04134
36530
08607
04133

Table 3-5: Mixed instrument accessories		
Product	Order number	
4 Condensers	36711	
2 Extraction chambers	36710	
2 Extraction chambers LSV	37902	
2 Condensation tubes	37482	
2 Condensation tubes LSV	37903	
2 Sample holders	36559	
2 Sample holders LSV	37904	
1 Set of solvent beakers, 4 pieces	37276	
1 Set of solvent beakers LSV, 4 pieces	38597	
1 Set of glass sample tubes	37281	
1 Set of glass sample tubes LSV	37563	
2 Thimble holders 33 x 94 mm (part of	-	
set thimble holders 37279)		
2 Holder baskets for thimbles 43 x 123 mm	37905	
1 Pack of paper thimbles 33 x 94 mm,	41883	
6 pieces		
1 Pack of paper thimbles	41884	
43 x 123 mm, 6 pieces		
8 Holder rings	36709	
2 Sets of seals	37388	
1 Set of blank plugs, 4 pieces	37368	
4 Magnetic valves complete	36687	
1 Inert gas supply	37496	
Cooling water hose	04113	
Water connection complete	37780	
2 Hose clamps	22352	
1 Silicon hose water drain	04134	
4 Conducts	36530	
PTFE band	08607	
1 Silicon hose for condenser, 250 mm	04133	

Table 3-6: Standard/LSV/mixed instrument accessories			
Product	Order number		
1 Power cable			
Type CH	10010		
Type Schuko	10016		
Type GB	17835		
Type USA	10020		
Type AUS	17836		
1 Operation Manual B-811			
English	96675		
German	96674		
French	96676		
Italian	96677		
Spanish	96678		

## 3.1.3 Optional accessories







Table 3-7: Optional accessories			
Product	Order number	Order number	
	B-811 Standard	B-811 LSV	
Set of solvent beakers (4 pieces)	37276	38597	
Set of glass sample tubes	37281	37563	
(4 pieces)	_		
Holder basket for thimbles; to be used	-	37905	
with holder, order no. 37904, LSV			
(1 piece)			



Holder for glass sample tubes, PTFE,	51903	-
microwavable (6 pos.)		













Table 3-7: Optional accessories (cont.)			
Product	Order number	Order number	
	B-811 Standard	B-811 LSV	
Holder for glass sample tubes	37462	-	
Set thimble holders 43 x 123 mm (4 pieces)	37280	-	
Set thimble holders 33 x 94 mm (4 pieces)	37279	-	
Set thimble holders 22 x 80 mm (4 pieces)	37278	-	
Set thimble holders 25 x 100 mm (4 pieces)	37277	-	
Pack of paper thimbles 33 x 94 mm (6 pieces)	41883	-	
Pack of paper thimbles 43 x 123 mm (6 pieces)	418	84	
Pack of paper thimbles 25 x 100 mm (6 pieces)	41882	-	
Pack of paper thimbles 22 x 80 mm (6 pieces)	41881	-	
Star printer 512 serial, complete	482	:58	

Printer cable IDP-460	28468

Adapter for printer cable IDP-460	31411











Ink ribbon for printer IDP-460	38683

Ink ribbon for Star printer 512	44306

Table 3-7: Optional accessories (cont.)			
Order number	Order number		
B-811 Standard	B-811 LSV		
38684			
42654			
	Order number B-811 Standard 386		

Quartz sand (0.3 - 0.9 mm), 2.5 kg 37689	
--	--

Paper thimbles 25 x 100 mm	18105
(25 pieces)	
Paper thimbles 43 x 123 mm	18106
(25 pieces)	
Silicone tubes 6/9	04133
Silicone tube 12/14, 2 cm	04134
Angle	01992
LSV upgrade kit (Standard to LSV)	37910 -

## 3.2 Technical data overview

Table 3-8: Technical data		
	Extraction System B-811	Extraction System B-811 LSV variant
Dimensions (L x H x D)	600 x 980 x 290 mm with extended condenser holder 600 x 700 x 290 mm during opera- tion	600 x 980 x 290 mm with extended condenser holder 600 x 700 x 290 mm during opera- tion
Connection voltage	100 - 120 VAC / 200 - 240 VAC ± 10%, 50/60 Hz	100 - 120 VAC / 200 - 240 VAC ± 10%, 50/60 Hz
Power consumption	max. 1250 W	max. 1250 W

Weight	32 kg	32.5 kg
Overvoltage category	II	II
Pollution degree	2	2
Environmental conditions	for indoor use only	for indoor use only
Temperature	5 – 40 °C	5 – 40 °C
Altitude	up to 2000 m above sea level	up to 2000 m above sea level
Humidity	maximum relative humidity 80% for	maximum relative humidity 80% for
	temperatures up to 31 °C, and then	temperatures up to 31 °C, and then
	linearly decreasing to 50% at 40 °C	linearly decreasing to 50% at 40 $^{\circ}\text{C}$

Table 3-8: Technical data (cont.)			
	Extraction System B-811	Extraction System B-811 LSV variant	
Cooling water consumption	60 I / h	60 I / h	
Max. water pressure	6 bar	6 bar	
Interface	RS 232	RS 232	

## 3.3 Materials used

Table 3-9: Materials used		
Component	Material designation Material code	
Housing	Coated tin, stainless steel	
Remote controller	Acrylonitrile-butadiene-styrol	ABS
Hoses	Polytetrafluorethylene, Silicone	PTFE, Si
Protective shield	Polycarbonate	PC
Cable	Polyurethane	PUR
Valve body	Aluminium	Al
Valve threads	Polyoxymethylene POM	
Valve membrane	Fluorez	
Seals	Polytetrafluorethylene,	PTFE
	glass fiber reinforced polyether- etherketon, fluororubber	PEEK 30 GF
Seal holders	Polyethylenterephtalate	PETP 30 GF
Holder ring for extraction chamber	Glass fiber reinforced polyamid 6.6	PA 66 GF
Materials in contact with media	Borosilicate glass 3.3, PTFE	
Sample, condenser holders and extraction chamber holder	PTFE, Glass fiber reinforced synthetics	UP-GFK

# 4 Description of function

This chapter explains the basic principle of the instrument, shows how it is structured and gives a functional description of the assemblies.

#### 4.1 Instrument function

There are several important components integrated into the B-811 extraction system which allow you to use the instrument for many kinds of applications in the area of solid-liquid extraction.

- 1. The solvent and extract only come in contact with glass and PTFE. This allows the instrument to be used for applications in the area of residue analysis (e.g. for environmental analysis applications).
- 2. The extraction and drying steps can be carried out under inert gas. This prevents the oxidation of the extract and the efficient and soft reduction of the extract.
- 3. With the adjustable optical sensor the amount of solvent can be minimized depending on the sample volume.
- 4. Each extraction position has 2 heating elements. Therefore, all 4 integrated extraction methods can be carried out without having to exchange or disassemble any glass part.

The operation panel makes programming and storing the extraction parameters easy. Up to 50 programs with up to 9 steps each can be stored. An extraction method can be selected for each program. The integrated front shield protects from glass breakage. It reduces simultaneously external heat loss and therefore increases the evaporating capacity.

#### NOTE

All positions are run with the same extraction type.

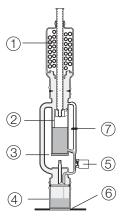
## 4.2 Overview of the instrument



- ① Operating panel
- ② Protective shield
- 3 Main power switch
- 4 Heating element

Fig. 4.1: Overview of the instrument

- ⑤ Beakers
- 6 Extraction chamber
- 7 Condenser



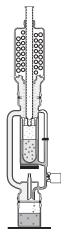
- ① Condenser
- ② Glass sample tube
- 3 Upper heating element
- 4 Solvent beaker
- (5) Glass valve
- **6** Lower heating element
- 7 Optical sensor

Fig. 4.2: Overview of a single extraction position

## 4.3 The three steps in general

The complete extraction process is set with the operating panel and follows a defined program. The process consists of three individual steps - Extraction, Rinsing and Drying – which runs automatically according to the parameters set in the program.

#### 4.3.1 Extraction



The sample is placed into the glass sample tube or thimble and put into the extraction chamber. One of four available extraction methods can be chosen and set on the operation panel to be defined for the program.

- Soxhlet Standard
- Soxhlet Warm
- Hot Extraction
- Continuous Flow

During the entire process (step 1 to 9) inert gas such as nitrogen can be supplied to prevent the oxidation of the analyte. In case of heat-sensitive analytes a keeping solvent can be added in the beginning of the extraction to avoid a concentration to dryness.

For detailed information on the extraction methods, please refer to chapter 4.4.1 - 4.4.3.

Fig. 4.3: Extraction

#### 4.3.2 Rinsing

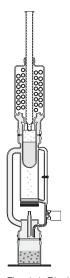
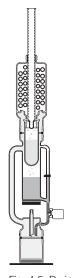


Fig. 4.4: Rinsing

Once the extraction is completed, the glass valve opens and an efficient rinsing is performed; finally the glass sample tube is lifted up automatically. During the rinsing process, the condensed solvent washes final traces of soluble matter from the sample and from the interior of the extraction chamber.

#### 4.3.3 Drying



After the rinsing process is completed, the glass valve closes while the lower heating is activated again. The solvent recovery is carried out. The solvent is evaporated, condensed at the condenser, and collected in the extraction chamber. At the end of drying the entire solvent is evaporated and collected in the extraction chamber to be eventually re-used.

Fig. 4.5: Drying

## 4.4 The three steps in detail

#### 4.4.1 Extraction - Step 1

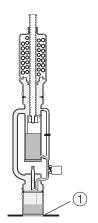
The extraction process takes place in step 1. The sample is placed into the glass sample tube or thimble. The solvent is added from the top of the condenser or directly into the beaker. The extraction procedure depends on the selected extraction method. One of the four possible extraction methods is applied:

#### Soxhlet Standard

The method "Soxhlet Standard" follows a real Soxhlet extraction known from the glass apparatus.

- The solvent is heated up by the lower heating element and evaporated. The upper heating element
  is deactivated. The vapour rises up to the condenser. The condensed solvent flows down along the
  condensation tube into the extraction chamber with the sample inside. The condensed solvent is
  collected in the extraction chamber while the glass valve is closed. The sample is extracted.
- The solvent level rises up to the optical sensor.
- Each time the solvent level reaches the optical sensor, the solvent containing the extracted compounds is released into the beaker by opening the magnetic valve. The glass valve remains open, until the extraction chamber is empty. One cycle is finished.

The number of cycles and/or time defines the length of the extraction process. If the programmed number of cycles is reached and/or the extraction time is up, all valves are opened and the solvent is released into the beaker. Then the program switches to the next step.



1) Lower heating element activated

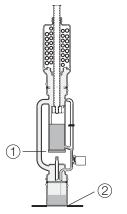
Fig. 4.6: Procedure of Soxhlet Standard

#### Soxhlet Warm

The method "Soxhlet Warm" follows a real Soxhlet extraction known from the glass apparatus. The extraction is carried out under enhanced conditions because the Soxhlet extraction chamber is additionally heated up.

- The solvent is heated up by the lower heating element and evaporated. The vapor rises up to the
  condenser. The condensed solvent flows down along the condensation tube into the extraction
  chamber with the sample inside. The condensed solvent is collected in the extraction chamber
  while the glass valve is closed. The sample is extracted.
- The solvent level rises up to the optical sensor.
- Each time the solvent level reaches the optical sensor, the solvent containing the extracted compounds is released into the beaker by opening the magnetic valve. The glass valve remains open, until the extraction chamber is empty. One cycle is finished. After the first cycle is finished (1st solvent level detection at the optical sensor), additionally the upper heating element is activated and warms up the solvent in the extraction chamber.

The number of cycles and/or time defines the length of the extraction process. If the programmed number of cycles is reached and/or the extraction time is up, all valves are opened and the solvent is released into the beaker. The upper heating element is turned off. Then the program switches to the next step.



- (1) Upper heating element activated
- 2 Lower heating element activated

Fig. 4.7: Procedure of Soxhlet Warm

#### Hot Extraction

In case of a "Hot Extraction" the sample is placed in the boiling solvent and extracted. This method is also know as Randall or Goldfisch extraction.

- The solvent is heated up by the lower heating element and evaporated. The vapor rises up to the
  condenser. The condensed solvent flows down along the condensation tube into the extraction
  chamber with the sample inside. The condensed solvent is collected in the extraction chamber
  while the glass valve is closed. The sample is extracted.
- The solvent level rises up to the optical sensor.
- Each time the solvent level reaches the optical sensor, the glass valves opens for a short time and
  a few milliliters of hot solvent containing the extracted compounds are drained into the beaker.
  After the first drain happened the upper heating element is activated and the solvent in the extraction chamber is warmed up. The extraction efficiency is improved because an exchange of fresh
  and used solvent is constantly given.
- During the extraction process the solvent level remains close to the detection line of the optical sensor.

When the programmed extraction time is up, all valves are opened and the solvent is released into the beaker. The upper heating element is turned off. Then the program switches to the next step.

- ① Upper heating element activated
- 2 Lower heating element activated
- 3 Solvent level remains at the height of the optical sensor

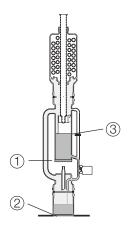


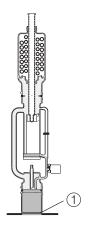
Fig. 4.8: Procedure of Hot Extraction

#### Continuous Flow

The method "Continuous Flow" is also known as Twisselmann extraction. The method is a washing up under controlled conditions.

- The solvent is heated up by the lower heating element and evaporated.
- The vapor rises up to the condenser. The condensed solvent flows down along the condensation tube through the sample into the beaker. During the entire extraction process the glass valve is open. The optical sensor is deactivated. The sample is rinsed efficiently.

When the programmed time is up, the solvent is in the beaker and the program switches to the next step.



1) Lower heating element activated

Fig. 4.9: Procedure of Continuous Flow

#### 4.4.2 Rinsing - Step 2

Once the extraction (step 1) is completed (time and/or number of cycles are reached), the glass valve opens on all positions and the solvent is released into the beaker. The solvent evaporates, rises up to the condenser, condenses and flows down along the condensation tube through the sample into the beaker. The condensed solvent washes final traces of soluble matter from the sample and from the interior of the extraction chamber (cleaning glass parts).

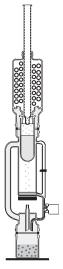


Fig. 4.10: Procedure of Rinsing - Step 2

#### Step 2 without following steps (step 3 to 9); step 2a is inactive

Do not enter any parameters in step 3 to 9, if the program should stop after step 2 (without drying). Only in this case the solvent containing the extract remains in the beaker at the end of the rinsing step. The glass valve is open for the entire rinsing time. This is operation is recommended for volatile and/or heat-sensitive analytes.

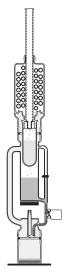
#### Step 2 with following steps (step 3 to 9); step 2a is active

The step 2a cannot be programmed (hidden in step 2), but prepares the drying process and supports the solvent recovery.

When the rinsing time (step 2) is expired the program switches to step 2a. The samples are lifted up automatically. Then the valve closes and the solvent evaporates, rises up to the condenser and condenses. The freshly condensed solvent is collected in the extraction chamber. When the solvent is

completely evaporated, a few milliliters of solvent are automatically released into the beaker. Again, this solvent evaporates, a temperature increase on the heating element's sensor is detected and again a few milliliters of solvent are released. This occurs three times. After the third solvent drain is happened, several milliliter of solvent remain in the beaker. Then the heating element is automatically turned off and the position stands in the wait mode. Within 30 minutes all positions should have reached this status, before the program switches to step 3. Otherwise an error 5 is displayed individually on the relevant position.

#### 4.4.3 Drying - Step 3 (to 9)



When the program switches to step 3, the heating elements are powered on again according to the level set in the program. Usually the heating level is reduced in order to prevent the extract from an overheating. The solvent is evaporated while the valve is closed and collected in the extraction chamber. The following steps (3 to 9) allow the heating power to be reduced levelwise, which provides a mild drying.

A drying under inert gas is available using the inert gas option. It is activated by pressing the INERT GAS key in the relevant step and remains active until the end of the step or the entire program is finished. In case of oxygen-sensitive analytes inert gas is supplied during processing. Heat-sensitive analytes should not be extracted to complete dryness; add a keeping solvent (high-boiling point solvent) prior to extraction to prevent overheating of the analyte.

Fig. 4.11: Procedure of Drying - Step 3-9

## 4.5 Programming

## 4.5.1 Extractions without rinsing and without drying

Step 1: Enter parameters of step 1 and switch to step 2.

Step 2 - 9: Set parameters to 0. Store program.

Table with parameters to be entered:

	Soxhlet Standard	Soxhlet Warm	Hot Extraction	Continuous
12:39 Step 1	lower heating level	upper and lower heating level	upper and lower heating level	lower heating level
	Extraction time and / or number of cycles	Extraction time and / or number of cycles	Extraction time	Extraction time
8 Step 2			ing level = 0 time = 0	
8tep 3 - 9			ing level = 0 time = 0	

At the end of the program the solvent containing the extract remains in the beaker.

#### 4.5.2 Extractions with rinsing and without drying

Step 1: Enter the parameters of step 1 according to table.

Step 2: Enter lower heating level and rinsing time.

Step 3 - 9: Set parameters to 0. Store program.

Table with parameters to be entered:

	Soxhlet Standard	Soxhlet Warm	Hot Extraction	Continuous
☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐	lower heating level	upper and lower heating level	upper and lower heating level	lower heating level
	Extraction time and / or number of cycles	Extraction time and / or number of cycles	Extraction time	Extraction time
8 Step 2	lower heating level rinsing time			
18:38 Step 3 - 9	lower heating level = 0 drying time = 0			

At the end of the program the solvent containing the extract/analyte remains in the beaker. The step 2a is not activated (no concentration to dryness!).

#### 4.5.3 Extractions without rinsing and with drying

Step 1: Enter parameters of step 1 according to table.

Step 2: Set parameters to 0.

Step 3-9: Enter lower heating and drying time for every further step. Store program.

Table with parameters to be entered:

	Soxhlet Standard	Soxhlet Warm	Hot Extraction	Continuous
☐ <i>日</i> 日 日 Step 1	lower heating level	upper and lower heating level	upper and lower heating level	lower heating level
	Extraction time and / or number of cycles	Extraction time and / or number of cycles	Extraction time	Extraction time
18:33 Step 2	lower heating level = 0 rinsing time = 0			
18:39 Step 3 - 9	lower heating level drying time			

The rinsing of the sample is skipped. The drying can be performed by decreasing the lower heating level from step to step (3 - 9). The sample is dried slowly. This is recommended for volatile and/or heat-sensitive analytes.

A mild drying is performed by using the inert gas option to prevent oxidation. It is activated by pressing the INERT GAS key and remains activated until the step is finished. When the program is expired the solvent is collected in the extraction chamber (automated solvent recovery) and the analyte/extract is in the beaker.

#### 4.5.4 Extractions with rinsing and with drying

Step 1: Enter parameters of step 1 according to table.

Step 2: Enter lower heating level and rinsing time.

Step 3-9: Enter lower heating and drying time for every further step. Store program.

Table with parameters to be entered:

		Soxhlet Standard	Soxhlet Warm	Hot Extraction	Continuous
08:88	Step 1	lower heating level	upper and lower heating level	upper and lower heating level	lower heating level
		Extraction time and / or number	Extraction time and / or number of	Extraction time	Extraction time
		of cycles	cycles		
B:B	Step 2		lower heat	ing level	
		rinsing time			
08:33 S	Step 3 - 9		lower heat	ing level	
		drying time			

The drying can be performed by decreasing the lower heating level from step to step (3-9). The sample is dried slowly. This is recommended for volatile and/or heat-sensitive analytes.

A mild drying is performed by using the inert gas option to prevent oxidation. It is activated by pressing the INERT GAS key and remains activated until the step is finished. When the program is expired the solvent is collected in the extraction chamber (automated solvent recovery) and the analyte/extract is in the beaker.

## 4.6 Control panel of the Extraction System B-811



- Fig. 4.12: Control panel of the Extraction System B-811
- A detailed description of the function keys is given in chapter 6.1.

- (1) Extraction method
- 2 PROGRAM number
- 3 Step in progress
- **4** HEATING

Displays the heating level programmed for the upper heating element

(5) HEATING

Displays the heating level programmed for the lower heating element (active on the picture)

- 6 Remaining time
- 7 POSITION

Shows which extraction position corresponds to the parameters being displayed

- START
- 9 STOP
- 10 SELECT
- 1) INERT GAS
- Navigation
- SOLVENT drain

# 5 Putting into operation

This chapter describes how the instrument is installed and gives instructions on the initial start-up.

#### 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

#### NOTE

We recommend to operate the instrument under a fume hood if e.g. diethyl ether or chloroform are used. Do not operate the Extraction System B-811 under the same fume hood as the hydrolysis unit.



#### **ATTENTION**

To ensure the instrument safety do not place any object on top of it.



#### **ATTENTION**

For safety reasons, make sure that the distance between the instrument and another object or the instrument and a wall is at least 30 cm.



#### **ATTENTION**

To ensure the proper functioning of the optical sensor do not expose the instrument to direct sunlight.

Place the instrument on a stable, horizontal and clean surface.

#### 5.2 Electrical connections



#### **ATTENTION**

Make sure that the voltage on the socket corresponds to the voltage given on the type plate of the instrument.

Always connect the instrument to an earthed socket. External connections and extension cables must be provided with an earthed conductor lead (3-pole couplings, cable or plug equipment) as the mains lead has a molded plug, thus avoiding risks due to inadvertent defective wiring.

Make sure that no electric sparks form in the instrument or its surroundings as they might damage the instrument.

Electrical connections have to be made to the:

- Power supply
- Operating panel
- Printer

## 5.3 Mounting the glass parts

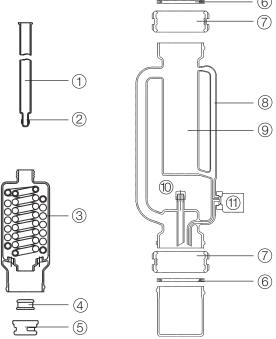


Fig. 5.1: Mounting the glass parts

- Place the PTFE seal (a) into the holder ring
   so that it clicks into place on both sides.
- Screw both (upper and lower) holder rings

   onto the extraction chamber (9) (1/4 turn with slight pressure) until they click into place. Make sure that the Büchi logo is aligned to the vapor duct.
- 3. Screw the magnetic valve (1) onto the extraction chamber (9).
- 4. Insert plug of magnetic valve unit and open the adjustment for the optical sensor at the instrument. Place the assembled extraction chamber in the instrument. Start with the rightmost unit and continue to the left.
- 5. Fasten the optical sensor to the side arm tube (8).
- 6 6. Screw the inert gas hose (1) or screw cap onto the extraction chamber.
  - Place the PTFE insert (4) into the condenser
     (3).
  - 8. Place the condenser ③ into the guide ring of the instrument.

To keep the tube holder (5) from falling off the condensation tube (1) wrap threads of the condensation tube (2) with PTFE band. Replace band when necessary. Insert condensation tube (1) from top into condenser (3) and fix on top. Screw tube holder (5) onto condensation tube (1).

 Screw the cooling water hose tight (connections facing towards the back; see chapter 5.5).

## 5.4 Fastening the optical sensor

To guarantee optimal functioning of the optical sensor, the glass part must be attached using the strap.

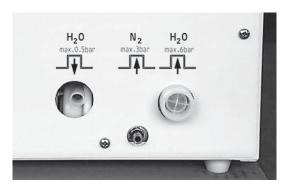
You do not have to loosen the strap to adjust the solvent level. The optical sensor can easily be adjusted up and down.

Make sure that there is enough solvent in the extraction chamber so that the solvent level you have set will be reached.

#### NOTE

Some samples absorb a lot of solvent. You may have to add more solvent so that the required solvent level is reached in the extraction chamber.

## 5.5 Connecting to tap water



- Cooling water outlet
- ② Nitrogen connection
- 3 Cooling water inlet

Fig. 5.2: Rear view of the B-811

The water pressure may have a maximum of 6 bar (operating pressure 2 - 6 bar).

The screw cap nut of the water connection has an R 1/2" standard thread.

The counterpressure at the cooling water outlet may be at a maximum of 0.5 bar.

Connect the cooling water hose (order number 04113) to the cooling water inlet ③ and the cooling water hose (order number 04134) to the outlet ①.

#### NOTE

- The temperature of the cooling water must be at least 20 25 °C below the boiling point of the solvent. If this is not possible, you will need to use a refrigerated circulation.
- When using a refrigerated circulation, you should note that the coolant pump will have to work against a closed valve.

Check the cooling water hoses for bends.

The cooling water valve is closed by the software 15 minutes after the program has been completed.

## 5.6 Inert gas connections



#### **ATTENTION**

The applied pressure should not exceed 3 bar. A larger flow of the inert gas causes exessive loss of solvent.

The inert gas can be connected using the hose provided. The connection is removed by pressing the black ring and pulling out at the same time.

Typical pressure to supply four positions in operation is a maximum of 3 bar at the beginning, which may be reduced to 1 - 2 bar during operation.

## 5.7 Connecting to a recirculating chiller

#### 5.7.1 When is it recommended to work with a recirculating chiller?

The temperature difference between the boiling point of the solvent and the cooling water temperature should be 20 - 25 °C to achieve a good condensation of the solvent in the condenser and to keep a good solvent recovery throughput during the extraction. When using solvents like dichloromethane or petroleum ether, a recirculating chiller (e.g. B-740) should be used if the tap water is warmer than 15 °C.

The cooling water tubes pass mainly inside the B-811 to facilitate the instrument handling and to protect the tubes from damages. When using tap water usually there are no extreme temperature differences between cooling water and surrounding. Using a recirculating chiller the temperature difference between the cooling medium and the surrounding is usually higher and thereby condensation water can be formed outside the tubes. This condensation water can accumulate inside the B-811 and affect the optimal use. To prevent an accumulation we recommend to connect the tubes outside the housing.

The graphic below shows the connection to a chiller.

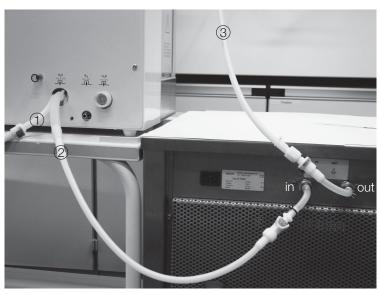


Fig. 5.3: Connections to a chiller

- Connection from left condenser
- ② Outlet from B-811 to chiller inlet
- ③ Connection from right condenser to chiller outlet (see also Fig. 5.6)

## 5.7.2 Connection to the cooling water sensor

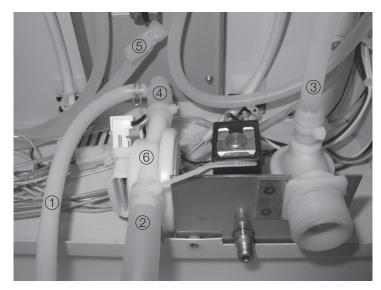


Fig. 5.4: Connection to the cooling water sensor

- ① Silicone tube from the left condenser (see Fig. 5.6)
- (2) Outlet to chiller inlet
- (3) Not used
- 4) Angle
- (5) Closed internal tube
- (a) Impeller water meter with cooling water sensor

To connect the cooling water sensor proceed as follows:

- Turn off the B-811 and disconnect it from the power supply.
- Disconnect the back board.
- Disconnect the internal tube (5) from the sensor and close it with a cable binder.
- Connect the silicone tube (1) (about 90 cm) to the angle.
- The silicone tube ② will be connected to the chiller (see 5.7.4) via the back board (see 5.7.3).

#### 5.7.3 Rear connections

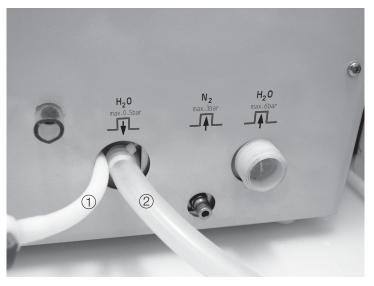


Fig. 5.5: Rear connections

To establish the rear connections proceed as follows:

- Thread the silicone tube 1 through the back board.
- Connect the tube ② to the chiller inlet, use an adapter if necessary.
- Connect the tube 1) to the left condenser (see 5.7.4).

① Silicone tube from left condenser (see Fig. 5.6)

2) Outlet to chiller inlet

#### 5.7.4 Connection to the condensers



Fig. 5.6: Connection to the condensers (rear view)

- ① Left condenser connection to impeller water meter and then to chiller inlet
- (2) Not used
- ③ Water connection between the condensers
- 4 Right condenser connection to chiller outlet

To connect the condensers proceed as follows:

- Untie the tubes 2 and fix them with cable binders.
- Connect a silicone tube ③ (approx. 35 cm) by means of 2 tube adapters to the condensers in the middle.
- For water inlet connect a silicone tube with adapters to the right condenser ④ and connect the adapters to the chiller outlet.
- Connect the silicone tube (approx. 90 cm) from the rear side of the instrument ① (in Fig. 5.5) to the tube adapter of the left condenser ① (in Fig. 5.6).

# 6 Operation

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



## WARNING

Risk of injury

• Never operate the instrument when glassware is damaged or when a heating element is broken.

## 6.1 Description of the function keys

Description		
Starts the program currently in the program window.		
1x: The extraction parameters can be changed.		
The cooling water valve is opened		
2x: The cooling water valve is closed.		
1x: Switches to standby mode.		
The extraction parameters can now be changed without having to stop the		
program.		
2x: Interrupts/stops the program currently running.		
The cooling water valve is closed. The samples are removed from the		
extraction chambers.		
3x: The cooling water valve is opened.		
1 x Access to storage/programming modes.		
Ends programming mode.		
Stores the program.		
Prints the program.		
ON/OFF (possible at any time)		
Activate in the appropriate programming step (the LED lights up).		
Set the heating level, number of cycles and the extraction time for the process.		
Move to the next display segment within a program.		
Opens the solvent valve manually.		
Solvent may be released at any time as long as a solvent beaker is in place		
and the heating element (position) is activated.		

An overview of the function key arrangement is given in chapter 4.7.

## 6.2 Preparing for an extraction

To prepare the instrument for an extraction, proceed as follows:

- 1. Turn the condenser to the right and release it.
- 2. Press the stop key, the condensers move out.
- 3. Insert the glass sample tubes/thimbles into the sample holders.
- 4. Pull the condensers down with both hands (see Fig. 6.5) until they clicks into place.
- 5. Close the condenser by turning it to the left.
- 6. Set the height of the optical sensor (solvent must cover the sample).
- 7. Fill the solvent beaker and assemble it.
- 8. Activate the lower heating element by pressing it up.
- 9. Open the cooling water (tap water/chiller).
- 10. Select a stored program or program a new one.
- 11.Start the program.

## 6.3 Soxhlet Standard Mode - programming example

#### Program description:

A standard extraction according to the original Soxhlet extraction is carried out. The solvent level is detected by the optical sensor, the magnetic valve opens and the solvent is released into the beaker. Solvent: petroleum ether 40/60, for other heating levels see chapter 6.11.

Function key	Display	Description
SELECT	The <b>Mode</b> display blinks	Select the <b>Soxhlet Standard</b> Mode using the
	Display STEP 1 blinks	(STEP 1 = Extraction)
	Display <b>HEATING</b> blinks	Select the heating level (1 - 20) using the ☐ / ☐ keys Select level 9 for petroleum ether 40/60
	Display <b>CYCLES</b>	Select the number of cycles using the / keys
	blinks	If the number of cycles = 0 is entered, then the extraction is finished when the extraction time has expired
	Display <b>H: MIN</b> blinks	Select the extraction time (0 - 99:99 h) using the / keys
		If no time is entered, then the extraction is finished when all positions have completed the set number of cycles
		If both parameters are entered (time and number of cycles), then both criteria must be fulfilled before the instrument switches to the next step
	Display STEP 2 blinks	(STEP 2 = Rinsing)
	Display <b>HEATING</b> blinks	Select the heating level (1 - 20) using the    ✓ keys Select level 9 for petroleum ether 40/60
		In case of oxygen-sensitive analytes, the gas supply must be activated by pressing the INERT GAS key
		The inert gas valve is automatically opened up while the step starts

Function key	Display	Description
	Display <b>H: MIN</b> blinks	Select the rinse time (0 to 99:99 h) using the / keys
		The only stop criterion used for <b>STEP 2</b> is the rinse time
	Display <b>STEP 3</b> blinks	(STEP 3 = Drying)
	Display <b>HEATING</b>	Select the heating level (1 - 20) using the _ / _ keys
	blinks	Select level 4 for petroleum ether 40/60
	Display <b>H</b> : <b>MIN</b> blinks	Select the drying time (0 to 99:99 h) using the / keys
		The only stop criterion used for STEP 3 is the drying time
		In case of oxygen-sensitive analytes, the gas supply must be
		activated by pressing the INERT GAS key
		The inert gas valve is automatically opened up while the step
		starts
SELECT	No display blinks	The entered program is stored as program 0
		To store this program under a different program number, please
		see chapter 6.7

# 6.4 Soxhlet Warm Mode - programming example

## Program description:

This program is the same as the program for the Soxhlet Standard mode, except that the extraction chambers are heated up. This makes it possible to reduce the extraction time, especially when using solvents with high boiling points. Solvent: toluene, for other heating levels see chapter 6.11.

Function key	Display	Description
SELECT	The <b>Mode</b> display blinks	Select the <b>Soxhlet Warm</b> Mode using the  keys
	Display STEP 1 blinks	(STEP 1 = Extraction)
	Display <b>HEATING</b>	(The symbol for the <b>upper heating</b> element lights up)
	blinks	Select the heating level (1 - 10) using the
	Display <b>HEATING</b>	(The symbol for the <b>lower heating</b> element lights up)
	blinks	Select the heating level (1 - 20) using the / keys Select level 13 for toluene
	Display <b>CYCLES</b> blinks	Select the number of cycles using the 2 / 2 keys
		If the number of cycles = 0 is entered, then the extraction is finished when the extraction time has expired
	Display <b>H: MIN</b> blinks	Select the extraction time (0 - 99:99 h) using the 🖾 / 🖸 keys
		If no time is entered, then the extraction is finished when all positions have completed the set number of cycles
		If both parameters are entered (time and number of cycles), then both criteria must be fulfilled before the instrument switches to the next step

Function key	Display	Description
	Display <b>STEP 2</b> blinks	(STEP 2 = Rinsing) Only the lower heating elements are activated
	Display <b>HEATING</b> blinks	Select the heating level (1 - 20) using the  keys Select level 13 for toluene
		In case of oxygen-sensitive analytes, the gas supply must be activated by pressing the INERT GAS key
		The inert gas valve is automatically opened up while the step starts
	Display <b>H: MIN</b> blinks	Select the rinse time (0 to 99:99 h) using the / keys  The only stop criterion used for <b>STEP 2</b> is the rinse time
	Display STEP 3 blinks	(STEP 3 = Drying)
	Display <b>HEATING</b> blinks	Select the heating level (1 - 20) using the  keys Select level 4 for toluene
	Display H: MIN blinks	Select the drying time (0 to 99:99 h) using the / keys
		The only stop criterion used for <b>STEP 3</b> is the drying time
		In case of oxygen-sensitive analytes, the gas supply must be activated by pressing the INERT GAS key
		The inert gas valve is automatically opened up while the step starts
SELECT	No display blinks	The entered program is stored as program 0 To store this program under a different program number, please see chapter 6.7

# 6.5 Hot Extraction Mode - programming example

# Program description:

This program carries out a Hot Extraction according to Randall and Goldfisch. In case of a Hot Extraction the sample is placed into the boiling solvent and extracted. The optical sensor detects the solvent level in the extraction chamber, the magnetic valve is opened and a few milimeter of solvent are drained. At the same time fresh solvent is constantly condensing at the condenser and collected in the chamber. Solvent: toluene, for other heating levels see chapter 6.11.

Function key	Display	Description
SELECT	The <b>Mode</b> display blinks	Select the <b>Hot Extraction</b> Mode using the  / keys
	Display STEP 1 blinks	(STEP 1 = Extraction)
	Display <b>HEATING</b> blinks	(The symbol for the <b>upper heating</b> element lights up)
		Select the heating level (1 - 10) using the  keys Select level 6 for toluene
	Display <b>HEATING</b>	(The symbol for the <b>lower heating</b> element lights up)
	blinks	Select the heating level (1 - 20) using the / keys Select level 13 for toluene

Function key	Display	Description
	Display <b>H: MIN</b> blinks	Select the extraction time (0 - 99:99 h) using the / keys
		In the <b>Hot Extraction</b> Mode the only stop criterion used is
		the extraction time. When the extraction time has expired the
		program switches to the next step
	Display <b>STEP 2</b> blinks	(STEP 2 = Rinsing)
	Display <b>HEATING</b>	Select the heating level (1 - 20) using the A / keys
	blinks	Select level 13 for toluene
		In case of oxygen-sensitive analytes, the gas supply must be activated by pressing the INERT GAS key
		The inert gas valve is automatically opened up while the step
		starts
	Display <b>H: MIN</b> blinks	Select the rinse time (0 to 99:99 h) using the / keys
		Display STEP 3 blinks (STEP 3 = Drying)
	Display <b>HEATING</b>	Select the heating level (1 - 20) using the 🖾 / 🔯 keys
	blinks	Select level 4 for toluene
	Display <b>H: MIN</b> blinks	Select the drying time (0 to 99:99 h) using the 🖾 / 🖸 keys
		In case of oxygen-sensitive analytes, the gas supply must be
		activated by pressing the INERT GAS key
		The inert gas valve is automatically opened up while the step starts
SELECT	No display blinks	The entered program is stored as program 0.
		To store this program under a different program number, please see chapter 6.7
		see chapter 6.7

# 6.6 Continuous Mode - programming example

## Program description:

In the continuous mode, known as Twisselmann Extraction, the magnetic valve is opened during the entire extraction step.

The sample is always extracted with fresh solvent. Solvent: petroleum ether 40/60, for other heating levels see chapter 5.12.

Function key	Display	Description
SELECT	The <b>Mode</b> display	Select the <b>Continuous</b> Mode using the  keys
	blinks	
	Display <b>STEP 1</b> blinks	(STEP 1 = Extraction)
	Display <b>HEATING</b>	Select the heating level (1 - 20) using the  keys
	blinks	Select level 9 for petroleum ether 40/60
	Display <b>H: MIN</b> blinks	Select the extraction time (0 - 99:99 h) using the 7 keys
		The only stop criterion used for <b>STEP 1</b> is the extraction time
	Display STEP 2 blinks	(STEP 2 = Rinsing)

Function key	Display	Description
	Display <b>HEATING</b> blinks	Select the heating level (1 - 20) using the  keys Select level 9 for petroleum ether 40/60
		In case of oxygen-sensitive analytes, the gas supply must be activated by pressing the INERT GAS key
		The inert gas valve is automatically opened up while the step starts
	Display <b>H: MIN</b> blinks	Select the rinse time (0 to 99:99 h) using the 4 / 4 keys
	Display <b>STEP 3</b> blinks	(STEP 3 = Drying)
	Display <b>HEATING</b> blinks	Select the heating level (1 - 20) using the  keys Select level 4 for petroleum ether 40/60
	Display <b>H: MIN</b> blinks	Select the drying time (0 to 99:99 h) using the 🗖 / 🗖 keys
		In case of oxygen-sensitive analytes, the gas supply must be activated by pressing the INERT GAS key
		The inert gas valve is automatically opened up while the step starts
SELECT	No display blinks	The entered program is stored as program 0.  To copy this program under a different program number, please see chapter 6.7

# 6.7 Storing / deleting a program

As soon as a new program has been created or an existing program has been modified, the number 0 is shown in the "PROGRAM" display. After the parameters have been entered, the program can be stored under a new or existing program number. 50 program numbers are provided.

### NOTE

Every time you shut down the instrument, the program 0 will be deleted.

- Close the program with "SELECT" so that no display on the control panel is blinking anymore.
- Press "SELECT". The mode display blinks now.
- Press now the program number switches between 0 and 1 (0/1).
- Select the desired program number (50 numbers available) using and In the "PROGRAM" switches between 0 and the desired program number and the "CLEAR" is displayed, then this program number is already in use.
- In order to overwrite an existing program, press "SELECT" again.
- "CLEAR" blinks to reconfirm if the program number can be used for the new program. Press "SELECT" once again to confirm and definitely overwrite the existing program number.

At any time the storage of a program can be exit by pressing any other key without changing anything.

- If "CLEAR" is not displayed, the program number is not used yet.
- Store the program by pressing the "SELECT" key.

# 6.8 Printing

### Online documentation

To keep an online log of an extraction, the B-811 must be connected to a printer. The extraction data are then printed out accordingly for each event that occurs.

SOXHLET WARM PROGRAM 1								
SOLVENT SAMPLE								
DAY	STEP	HE	ATER	CY	CLE	S		GAS
TME	INFO	UP	LOW	P1	P2	РЗ	P4	1
0 02:00 1	START	8	15	25	25	25	25	N
0 02:00 1	END	8	15	32	30	30	30	Ν
0 00:10 2	BEGIN	-	15	+	+	+	+	Υ
0 00:19 2	END	-	15	+	+	+	+	Υ
0 00:05 3	BEGIN	-	5	+	+	+	+	Υ
0 00:05 3	END	-	5	+	+	+	+	Υ
DATE:			VISUM:					

Fig. 6.1: Online printout with captions

# SOXHLET warm: Mode

PROGRAM: Currently selected program Solvent: (solvent being used)

Sample: (sample)

Day: Displays the day, changes for

extractions taking longer than

24 hours

Time: Extraction time remaining
Step: Currently active extraction step
Info: Result, such as Start/Stop,

Error messages

Heater: Up: upper heating stage Low: lower heating stage

Cycles: Number of cycles for heating

elements 1 - 4

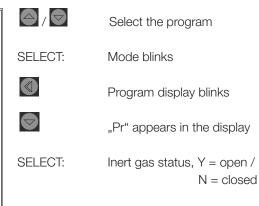
Gas: Inert gas status: Y = open

N = closed

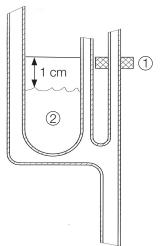
## Program documentation

SOXHLE	T WARM				F	PRO	GRA	M 1
SOLVEN' SAMPLE	T :							
DAY	STEP	HEA	ATER	CY	CLE	S		GAS
TIME	INFO	UP	LOW	P1	P2	Р3	P4	
0 02:00	1	8	15	25	25	25	25	Ν
0 00:10	2	-	15	-	-	-	-	Υ
0 00:05	3	-	5	-	-	-	-	Υ
DATE: _			VISUM:					

Fig. 6.2: Program printout without captions



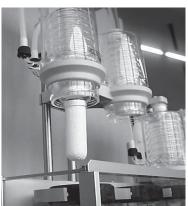
# 6.9 Optical sensor



The optical sensor ① detects the solvent level. In order to achieve the optimum extraction efficiency, the solvent level should be about 1 cm above the sample ②. The solvent should cover the sample during the extraction process.

Fig. 6.3: Adjustment of the optical sensor

# 6.10 Handling the sample/sample holder



The sample holder can handle paper thimbles of various sizes. The corresponding glass holders are listed in the accessories and spare parts list.

To fasten the sample tube, push the paper thimble into the glass holder. The notches must be at the top.

By turning the glass holder one half turn on the PTFE holder, the sample is connected.

The glass sample tubes are connected the same way.

Fig. 6.4: Sample handling



Fig. 6.5: Closing the system for extraction

The condensers can be lowered by pushing them down with both hands as shown on the picture.

The system can be closed by turning the condensers clockwise.

# 6.11 Choosing the heating parameters

The following heating parameters are recommended:

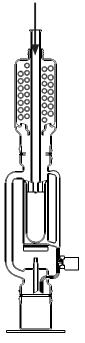
		Heating setting	Heating setting
Solvent	BP / °C	lower	upper
Acetone	56.1	11	3
Ethanol	78.4	16	4
Methanol	64.6	14	4
Xylol	139.5	15	10
Dichloromethane	39.9	9	2
Petroleum ether 40/60	40-60	9	2
n-Hexane	68.8	10	4
Toluene	110.6	13	6
Methylethylketone (MEK)	79.6	11	4
Water	100	20	10

If further heating parameters are required, please refer to the Application Note Nr. 811049 en. All instrument components in contact with the solvent/sample/analyte are inert. Residual effects (memory effects) are fully eliminated.

### NOTE

The choice of higher heating settings can lead to solvent loss because the cooling capacity is exceeded. Solvent loss can also be caused by damaged PTFE seals (order number 037388) and beakers (order number 037276 and 038597). If solvent loss appears the chemical resistance of some instrument components can be remarkably reduced.

# 6.12 Solvent addition during extraction



During the extraction an addition of solvent is possible. The solvent is added through the condensation tube directly into the sample using a pipette, rinsing bottles, a dispenser or other solvent delivery devices.

Fig. 6.6: Solvent addition during extraction

# 7 Maintenance

This chapter gives instructions on all maintenance work to be performed in order to keep the instrument in good working condition.



## **WARNING**

All maintenance and repair work requiring the opening or removal of instrument covers may only be carried out by trained personnel and with the tools provided for this purpose.



### **WARNING**

Electrical hazard:

 Prior to all maintenance work on the instrument switch off the power supply and remove all sources of flammable vapor.



### **ATTENTION**

Always wear personal protective equipment such as protective eye goggles, protective clothing and gloves when maintaining the instrument.



Check the instrument for proper operation after any repair work.

# 7.1 Housing

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



## ATTENTION

Never use solvents (acetone) as cleaning agents as these might damage the instrument.

## 7.2 Hoses and hose connections

Check the hoses and hose connections for defects (cracks, brittle areas) and replace damaged hoses immediately.

# 7.3 Glass parts

Visually inspect all glass parts for defects (cracks, stars, and splintering) regularly.

The glass parts should be cleaned using common commercial cleaners (for example, mild soap solutions). Dirt that is stuck to the condenser spirals (for example, algae build-up) should be removed using an appropriate cleaner (you may eventually need to let the parts soak for a while).

After the parts have been cleaned and are completely dry, you should visually inspect the glass parts for cracks and chipping.

# 7.4 Magnetic glass valve

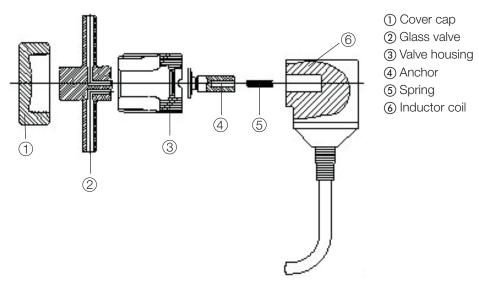


Fig. 7.1: Exploded view of the glass valve

- Insert the spring (5) into the anchor (4).
- Screw the valve housing (3) and the magnetic valve together (6).
- Push the valve onto the glass part 2).
- Screw the cover ① on and tighten slightly.

# 7.5 Membrane assembly

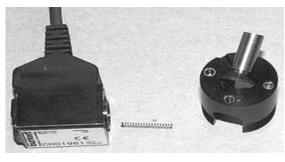


Fig. 7.2: Membrane assembly

- Insert anchor with membrane at an angle into valve housing
- Guide entirely through opening by pressing slightly with circular movements.

# 7.6 Heating elements

The heating elements should be cleaned regularly after they have cooled down. Remove dirt adhering to the plate with e.g. ethanol. Then clean the plate with a moist cloth and dry it.



## **ATTENTION**

Never use scratching sponges, cleaning pads or cleaner containing abrasives to clean the heating elements as they might damage the surface.

# 7.7 Holder ring with seal

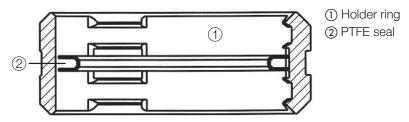


Fig. 7.3: Holder ring with seal



## **ATTENTION**

When removing and reinstalling the seals, make sure not to damage them.

Never apply grease to the seals and never touch them with sharp objects, otherwise they will get damaged and cause solvent loss.

To prolong the lifetime of the seals, rinse them regularly with distilled water or alcohol. Afterwards, dry them with a soft cloth.

# 7.8 Glass sample tubes

The glass sample tubes can be cleaned using commercial cleaning agent or in a dishwasher.

### 7.9 Solvent beakers

The roughened area at the bottom of the solvent beakers prevents solvents from bumping. In order to maintain this area in active state the beakers must be cleaned properly after every extraction.

# 7.10 Emergency release mechanism

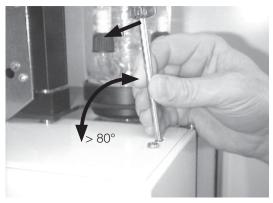


Fig. 7.4: Emergency release mechanism

In case of power outage the samples can be removed by unlocking the lift with a screwdriver. The screwdriver is inserted at an angle of about 80° into the square slit on top of the instrument. The mechanism is released by slightly tipping the screwdriver downwards.

## 7.11 Customer service

Only authorised service personnel are allowed to perform repair work on the instrument. These persons have a comprehensive technical training and knowledge of possible dangers which might arise from the instrument.

Addresses of official Buchi customer service offices are given on the Buchi website under: www.buchi.com. If malfunctions occur on your instrument or you have technical questions or application problems, contact one of these offices.

The customer service offers the following:

- Spare part delivery
- Repairs
- Technical advice

# 8 Troubleshooting

This chapter helps to resume operation after a minor problem has occurred with the instrument. 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 "Corrective measure".

The elimination of more complicated malfunctions or errors is usually performed by a Buchi technical engineer who has access to the official service manuals. In this case, please refer to your local Buchi customer service agent.

# 8.1 Malfunctions and their remedy

Table 8-1: General mal	functions and their remedy	
Error number	Possible cause	Corrective measure
Error 1	Empty beaker	Stop the program and refill the solvent beaker
Error 2	Beaker missing	Insert the beaker and turn on the heating element
Error 3	Optical sensor error	Contact the Buchi customer service
Error 4	Draining not possible	Clean the glass valve
Error 5	Evaporating time > 30 min between the heating elements	Heating power too low, volume differences are too large
Error 10	No cooling water	Open the cooling water flow
Error 11	No location active	Insert a beaker and turn on the heating element
Technical errors:		
Error 30	Temperatures too low on 1. heating	
	element	
Error 31	Temperatures too high, lower	
	heating	
Error 32	Lower heating circle broken	
Error 33	Temperatures too low on upper	
	heating element	
Error 34	Temperatures too high on upper	
	heating element	
Error 35	Upper heating circle broken	
Other error messages	Technical error	Contact the Buchi customer service

### 9 Shutdown, storage, transport and disposal

This chapter instructs how to shut down the instrument, how to pack it for storage or transport, and specifies the storage and shipping conditions.

### 9.1 Preparing the instrument for storage and transport

To prepare the instrument for storage and transport, unplug the power cord, remove the cooling water, and remove all glass parts from the instrument.

## Emptying the condenser

Place the cooling water out hose into a drain. Disconnect the silicon hose (cooling water in) at the rear side of the instrument and place it into a waste container. Loosen the water inlet of the left condenser to let the water flow out.

### Dismantling the condenser

Remove the sample holder from the condensation tube. Take out the condensation tube from the top of the condensers. The condensers can be screwed out of the holder rings.

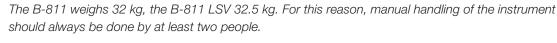
### 9.2 Storage and transport



Store the instrument at a dry place. Store and transport the instrument in its original packaging.



### **WARNING**



### 9.3 **Disposal**

To dispose of the instrument in an environmentally friendly manner, a list of materials is given in chapter 3. This helps to ensure that the components are separated and recycled correctly. Please follow valid regional and local laws concerning disposal.

# 10 Spare parts

This chapter lists spare parts, accessories, and options including their ordering information. Order the spare parts from Buchi. Always state the product designation and the part number when ordering spare parts.

Use only genuine Buchi consumables and genuine spare parts for maintenance and repair 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.

Table 10-1: Spare parts				
Product	Order number B-811	Order number B-811		
	Standard	LSV		
Set of glass sample tubes (4 pieces)	37281	37563		
Condensation tube	37482	37903		
Set of solvent beakers (4 pieces)	37276	38597		
Extraction chamber	36710	37902		
Operating panel	371	166		
Membrane with anchor for valve unit	375	534		
Front shield with grip	376	640		
Side shield	366	695		
Conduct in condenser	365	530		
Magnet valve, complete	366	687		
Cover for valve	365	537		
Condenser	367	711		
PTFE band	086	08607		
Set of seals for extraction chambers (4 pieces)	37388			
Set of fluororubber seals (4 pieces)	42654			
Holder ring (black ring)	36709			

### **Declarations and requirements** 11

### 11.1 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, 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.

### 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 Canadian Department of Communications. Ces limites sont destinées à fournir une protection adéquate contre les interférences néfastes lorsque l'appareil est utilisé dans un environnement

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 les zones résidentielles peut causer des interférences néfastes, auquel cas l'exploitant sera amené à prendre les dispositions utiles pour palier aux interférences à ses propres frais.





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