LAB Online Exhibition

Operation Manual

knowledge

Action movie

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Contents

Read these operating instructions carefully before you use the **Büchi B-440** Wet Digester. Keep these instructions near to the equipment so that you can refer to them at all times.
The equipment should not be connected to the mains and switched on until the necessary preparations have been made. Chapter 2 contains important safety instructions. It is essential to be familiar with these if the equipment is to be used safely.

en, Version C (22 pages)	Order No.
B-440 Operating instructions	**96791**

1 Scope of Delivery

Fig. 1: Overview of B-440

Components:		**Order No.**
1	Basic system with B-440 and B-414 230 V 50 / 60 Hz *	**37531**
1	Vitrified ceramic plate	
	with 6 openings, 39 mm dia.	**26661**
	with 4 openings, 48 mm dia.	**36676**
	with 4 openings, 56 mm dia.	**26663**
	without openings	**26982**
	Ceramic crucibles	
	40 mm dia., H = 32 mm, V = 20 ml	**26936**
	50 mm dia., H = 40 mm, V = 49 ml	**36675**
	60 mm dia., H = 48 mm, V = 90 ml	**26938**

* Operating instructions, warning notices and spare parts for the B-414 scrubber can be found in the appropriate operating instructions for B-414.

Description:			**Order No.**
Scope of delivery:			
B-440	230 V	50 / 60 Hz	**26674**
B-414 Scrubber	230 V	50 Hz	**37879**
Connecting cable			**30983**

Table 1: Scope of delivery

Fig. 2: Standard accessories

	Description:	**Order No.**
	Standard accessories:	
1	Glass hood	**36081**
1	Separator, complete	**36119**
1	Viton tubes 12/8 1 m	**20136**
1	Corrugated hose	**26096**
1	Connection to glass hood	**36076**
3	Clip for receiving flask 35	**03275**
1	Clip set	**26968**
1	Glass wool	**33701**
1	Crucible tongs	**34184**
1	Glisseal laboratory grease	**17595**
1	Mains cable for the following types:	
	CH type	**10010**
	Earth contact type	**10016**
	GB type	**17835**
	AUS type	**17836**
	USA type	**33763**
1	Operating instructions in one of the following languages:	
	German	**96790**
	English	**96791**
	French	**96792**
	Italian	**96793**
	Spanish	**96794**

Table 2: Standard accessories

2 Safety

The equipment is built in accordance with state of the art technology and complies with recognized safety regulations. However, the equipment may still pose risks and hazards:

- If the equipment is operated by inadequately trained personnel.
- If the equipment is not used for the purpose for which it was intended.

2.1 Symbols

Stop
Information on hazards that may result in considerable damage to property and serious or life-threatening injuries.

Warning
Information on hazards that may result in damage to property or have a detrimental effect on health.

Note
Information that refers to technical requirements. If these instructions are not followed, it may result in faults, inefficiency and production losses.

2.2 Requirements to be met by the operator

The equipment may only be used by laboratory personnel and other persons who, because of their training or professional experience, have a clear picture of hazards which may occur when the equipment is operated.

2.3 Use for the purpose intended

The equipment is designed and built as laboratory equipment. It is designed to be used to heat up concentrated sulphuric acid for the oxidation of organic compounds in samples.

2.4 Uses for purposes other than those intended

Any use other than the one stated above and any application that does not comply with the technical data is considered to be incorrect use. The operator alone shall bear the risk for any damage that can result from use of this kind.

In particular, the following uses shall not be permitted:
- Use in areas that require explosion-protected equipment to be used.
- Samples that may explode or catch fire as a result of impact, friction, heat or the formation of sparks.

2.5 Basic hazards

Basic hazards are posed by:
- Hot sulphuric acid
- Combustible gases or vapours from solvents in close proximity to the equipment
- Damaged glassware
- Inadequate clearance between the equipment and the wall (See Chapter 4.1, Mounting position)
- Burns caused by touching hot glass components.

It is forbidden to remove covers using normal commercial tools, except by authorized maintenance personnel. The equipment must not be started up if the glassware is damaged.

You risk your life if you touch live components!

2.6 Safety measures

It is advisable only to use the equipment under an fume hood. Personal protective equipment such as **goggles**, **gloves** and a **laboratory coat** must be worn.
These instructions are an important part of the B-440 Wet Digester and must be made available at all times to the operating personnel at the place where the equipment is deployed. This also applies to the additional language versions of these instructions, which can be reordered separately.

Modifications

Modifications to the equipment, replacement parts and accessories and the use of spare parts or accessories other than those mentioned in these operating instructions shall only be permitted with the written permission of Büchi Labortechnik AG.

Responsibility of the operator

The operator is responsible for instructing his personnel. The operating instructions can be ordered in other languages for this purpose.

The operator shall inform the manufacturer immediately of any incidents relating to safety that occur during the use of this equipment.

If it is assumed that the equipment can no longer be operated without posing any risk, the equipment is to be taken out of operation and secured to prevent it being used inadvertently.

It can be assumed that the equipment can no longer be operated without posing any risk:

- if the equipment shows visible signs of damage,
- if the equipment no longer works,
- after it has been stored for a long time in unfavourable conditions,
- after it has been subjected to considerable stresses during transport.

The equipment may not be operated using combustible substances.

Should it be necessary to obtain help from Büchi's service department, hazardous substances (e.g. as defined by EC Directive L 360, 1976/79 or VBG 16) must be indicated and declared in order to comply with safety at work and environmental criteria.

3 Function

The set-up is suitable for all wet ashing operations using sulphuric acid. The ratio between the weight of the sample and sulphuric acid and the temperature program must be selected in such a way that practically only gaseous products are produced by the reaction.

3.1 Layout of operator's controls

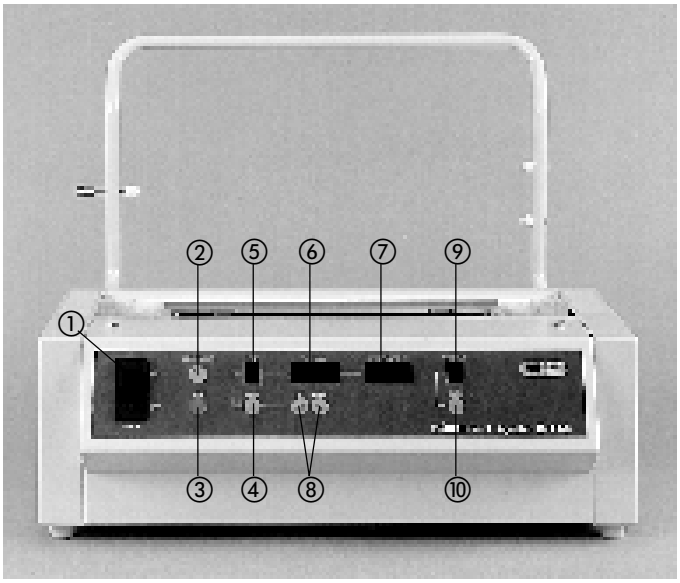


Fig. 3: Layout of operator's controls at front

① Main switch

② Start (continue after stop)

③ Stop

④ Select button to pre-select individual program/time/temperature steps

⑤ LED display of the relevant step

⑥ LED display for time in minutes

⑦ LED display of the heating temperature in °C

⑧ UP/DOWN buttons to change the values: Program step, time, temperature

⑨ LED display: Program number

⑩ Button to pre-select the program

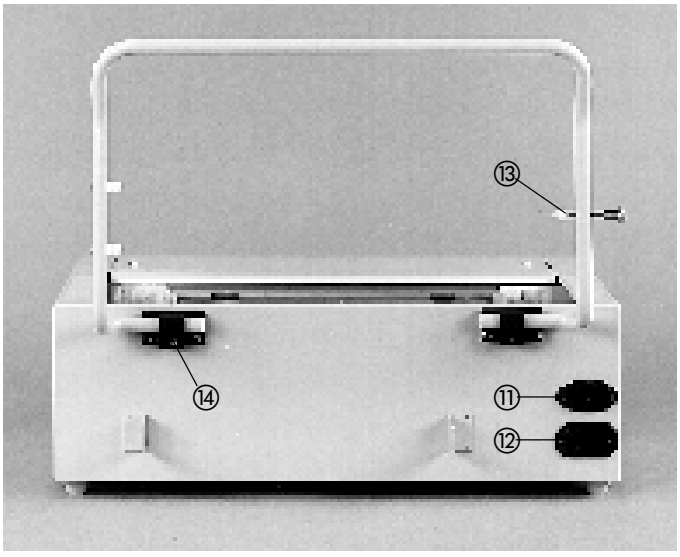


Fig. 4: Layout of operator's controls at back

⑪ Mains socket for the Scrubber

⑫ Mains connection

⑬ Counterpin

⑭ Clamping screw for setting hinge friction

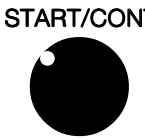
3.2 Operator's controls and their function

The individual parameters (step, time, temperature) can be pre-selected in stages. The display flashes for 10 seconds and then stops if no change is made. The figures entered are stored at the same time. The individual parameters are only displayed during the process cycle: they cannot be changed.

The flashing value can be increased in stages. If the button is held down, the speed will increase.

The flashing value can be reduced in stages. If the button is held down, the speed will increase.

Pre-select program 1 - 9.



Start of the process cycle. The LED in the start button lights up. The heating is adjusted to the selected set point. Here the temperature display changes to the actual value and the time display shows the total remaining running time.

If the START/CONT button is pressed when the LED is flashing, it means that the process is started up again at the point where it was interrupted.

The process cycle stops, the LED in the start button flashes and the heating switches off. The temperature display is set to the actual value and the total remaining running time display does not move.

If the button is pressed again, the LED extinguishes and the process is finally aborted. The temperature display changes to the set point for step 1.

Display during the process cycle:
During the cycle the actual temperature and the total remaining running time for each step is displayed. The total remaining running time is counted down minute-by-minute. The dot in the time display flashes during this operation. The pre-selected program data can be displayed via SELECT.

Value ranges of the parameters:

Step:	1-5
Temp. set point T1-T5: (600°C corresponds to approx. 550°C in melting crucible)	0 to 600°C
Time value t1 - t5	0 to 240 mins.
Program:	1-9
Temp. actual value T1-T5:	Room temp. ...999°C

4 Putting Into Operation

4.1 Mounting position

The equipment must be mounted on a stable, clean and level surface.
For safety reasons, the clearance between the back of the equipment and the wall or between the back and other objects must be a minimum of 30 cm. It is not permissible for any containers, chemicals or other equipments to be placed behind the equipment.
The B-440 must be operated with the B-414 scrubber.
The equipment must be operated in conjunction with the laboratory fume hood.

4.2 Unpacking

The original packaging is to be stored in a safe place for use if the equipment needs to be transported at a later date.

If the packaging is damaged, the equipment is to be inspected carefully to see if the glass is broken or the housing damaged.

Check that the voltage supply at the socket agrees with the voltage shown on the equipment's rating place. Always plug the equipment into an earthed socket. This will avoid any risk that may result from an internal defect.

4.3 Assembling the vitrified ceramic plate

Every time this is opened, a clip must be fitted to prevent the crucibles from jamming. Place the vitrified ceramic plate on the heating dish.

4.4 Assembling the glass hood

Place the glass hood into the two retaining cams ② and fix it in place by means of the counterpin ①.

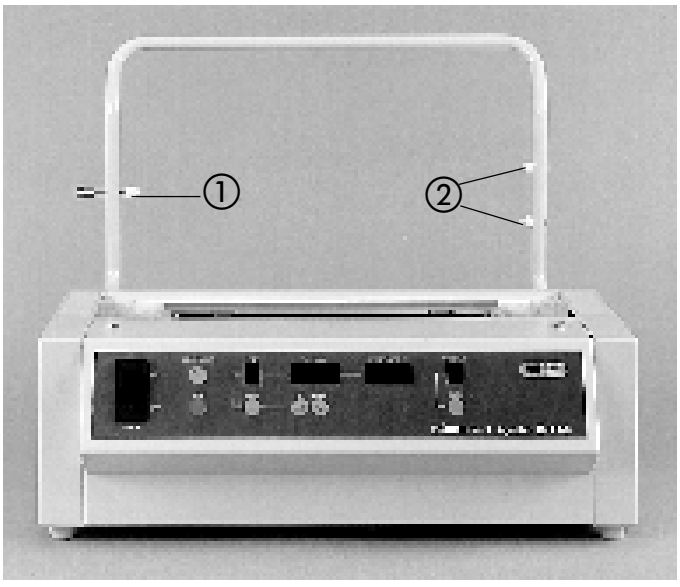


Fig. 5: Assembling the glass hood

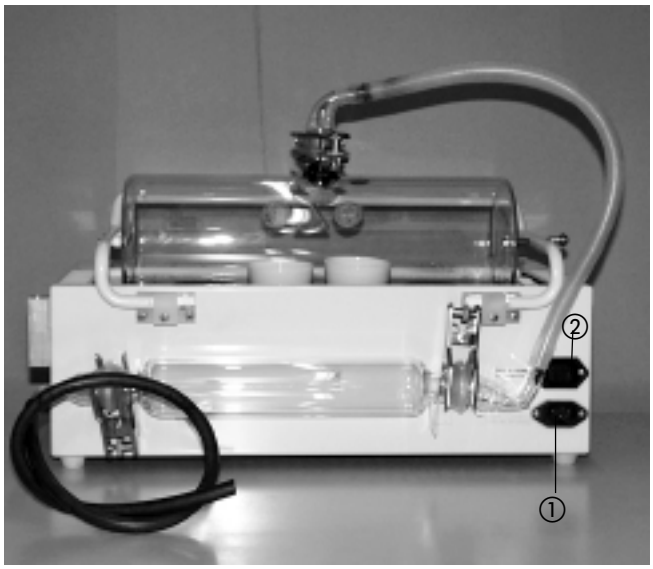


Fig. 6: Power connection

4.5 Power connection

Before switching on the B-440 for the first time, make sure that the voltage rating plate agrees with the available line (mains) voltage. Connect the power cable to the power connector ① and connect the system to the mains. Then connect the jumper cord to the power socket ② and the scrubber.

The system is designed so that the on-going process cycle is saved in the event of a power failure lasting up to 3 minutes, without a restart being required, i.e. ashing is continued to the end according to the program selected.
A power failure lasting longer than 3 minutes will result in the process cycle being discontinued and a defined RESET.
In no case will the saved program data be lost.

5 Operation

5.1 Set-up and working methods

Use

The set-up is suitable for all wet ashing operations using sulphuric acid. The ratio between the weight of the sample and sulphuric acid and the temperature program must be selected in such a way that practically only gaseous products are produced by the reaction.

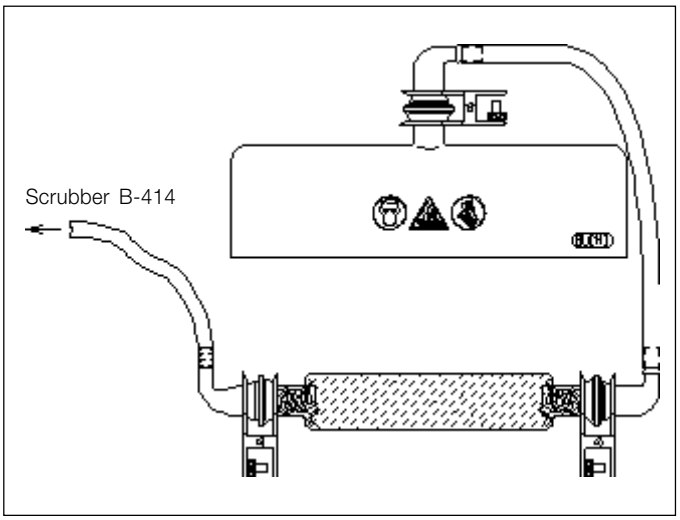


Fig. 7: Set-up

Set-up

The set-up consists of a glass hood with a ball joint and the condensate trap. This can be filled with activated carbon granules to reduce the load on the B-414 scrubber.

The condensate trap can be filled with activated carbon granules and the ends are plugged with glass wool. To prevent the glass wool causing a blockage, the glass wool may only be inserted very loosely. The active carbon can be obtained from companies such as Merck (granule size approx. 2.5 mm, Order No. 2518.1000 for 1 kg).

The waste gas hose must be connected to the scrubber inlet.

Description	Order No.
Glass hood	**36081**
Connector for Top 15	**36076**
Connector for Top 10	**36077**
PFA corrugated hose	**26096**
Clip for joint	**03275**
Condensate trap	**36080**

The connecting hose to the B-414 is contained in the scope of delivery for the scrubber.

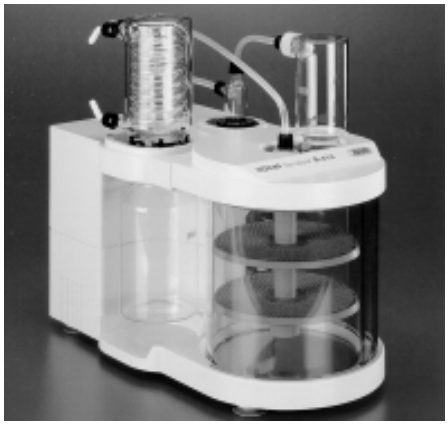


Fig. 8: B-414 scrubber with cold trap and collecting vessel, 1.2 litre

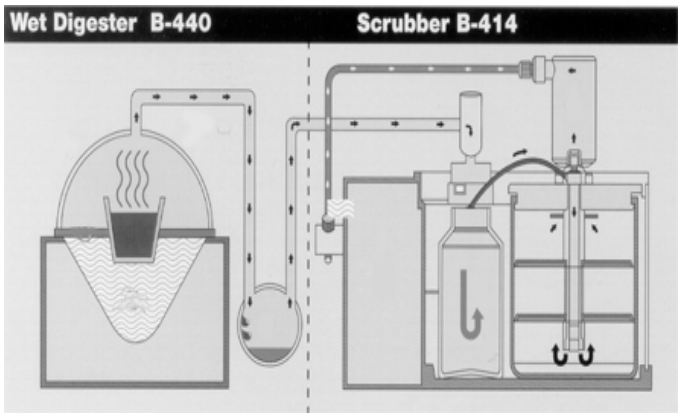


Fig. 9: Diagram of B-440 Wet Digester with B-414 scrubber

Configuration of the B-414 scrubber

The following B-414 scrubber configuration is recommended for pre-ashing of sulphated ash determination:

- Basic model with cold trap and collecting vessel, 1.2 litres.

Working methods

The working solution of the scrubber is used to neutralize the acidic gases of the ashing unit. One of the following solutions can be used to achieve this:

- Sodium hydroxide with a concentration of max. 20%
- A solution of 600 g Na_2CO_3 crystals, diluted with 3 litres of warm water
- A solution of 1.7 kg hydrated sodium carbonate (Na_2CO_3 x 10 H_2O) diluted with 3 litres of warm water

We recommend the use of colour indicators such as bromo-thymol blue. These indicators change colour at a pH-value of approx. 7.0 and thus show when the working solution has to be changed.

We recommend that the cooling trap be filled with iced water or an ice/salt mixture. This should be able to condense all volatile gases and improve the efficiency of the scrubber.

Optimization principle for a wet ashing program
The various molecule structures make it impossible to develop a general-purpose wet ashing procedure for all substances. Each substance has its own ashing kinetics and therefore its own profile of the decomposition temperature.

You should observe the following rules to optimize an ashing program:

- If the sample contains water, you should evaporate it during the first stage by heating the sample at approx. 120°C for as long as is necessary.
- The stages contained in the following example should be programmed to increase the temperature of the sample to approx. 550°C in the crucible (600°C for the heating element). Each temperature stage should last for as long as a reaction takes place.
- This temperature profile should be optimized for each sample type.

Please note that, if the sample is heated to too high temperature too quickly, it may ignite or be propelled out of the crucible.

- Chemicals such as H_2O_2 or H_2SO_4 can be added to the sample to facilitate wet ashing.

Example
Temperature program for the wet ashing of starch:

- Weigh the crucible (A).
- Weigh in 1 g sample (B).
- Add 1 ml w (sulphuric acid) = 0.98.
- Place the crucible in the equipment.
- Program the heating as follows and start the procedure:
  - Step 1: 175°C for 15 mins.
  - Step 2: 200°C for 25 mins.
- Add a further 1 ml w (sulphuric acid) = 0.98.
- Program the heating as follows and start the procedure:
  - Step 1: 200°C for 25 mins.
  - Step 2: 250°C for 15 mins.
  - Step 3: 350°C for 10 mins.
  - Step 4: 500°C for 10 mins.
- Complete the wet ashing in the muffle furnace at 600°C.
- Allow the crucible to cool off.
- Determine the crucible and ash weight (C).

$$T = \frac{C - A}{B}$$

5.2 Faults

Fault	Possible cause	Remedy
LED display does not illuminate	Fuse F1 faulty	Replace fuse
IR lamp does not warm up	Bulb faulty	Replace bulb
E2	1. Faulty EEPROM	Fit a new EEPROM and recalibrate
	2. EEPROM malfunctioning (e.g. EMC fault or lightning)	Calibrate EEPROM/thermoelement and save program information again
E4, E5, E7, E8, E9	Sensing electronics faulty	Replace controller PCB GS-2 and calibrate
E6	Thermo element faulty	1. Fit a new thermo element or break in the wiring → Contact Büchi's Service Dept. 2. Calibrate

Table 3: Faults

If faults E2 to E9 occur, also contact Büchi service department.

6 Maintenance

6.1 General and preparations

Wipe off any splashes of acid or lye immediately using a damp cloth in order to avoid any damage being caused to the plastic coating on the housing.

After each use:

- Check the glass components for damage.
- Clean the glass hood and vitrified ceramic plate with water.

Separate standard joints from each other.

Empty condensate bottle regularly.

Do not grease ground-glass joints under any circumstance.

6.2 Cleaning the system (B-440/B-414)

Condensate trap
The activated charcoal does not have to be cleaned or changed until the glass wool at the inlet has clearly changed colour or has become sticky. However, it is generally sufficient just to change the dirty glass wool.

Extraction hose
The extraction hose (PFA corrugated hose) just has to be rinsed through with water or ethanol. Cleaning using an ultrasound bath is also suitable.

Glass hood
In the case of the glass hood it must be noted that heating must be continued for a further 30 minutes at 600°C after pre-ashing. The hood can then be cleaned using a cleaning agent and a household sponge.

In the case of stubborn dirt, four crucibles can be heated using approx. 2 ml of 98 % sulphuric acid.

The following temperature program is used for this:

20 mins.	300 °C
30 mins.	600 °C

6.3 Duty to undertake inspection and maintenance work

All regulations designed to ensure that the equipment remains in good working order must be followed. This also includes occasional cleaning and inspection of any damage that may have occurred. Each time a repair is undertaken, the operator must first check to satisfy himself/herself that the equipment is operating correctly.

6.4 Changing components

Components may only be changed when the equipment has cooled down completely.
The equipment must be disconnected from all power sources before any components are changed. Electrical and electronic components may only be changed by appropriately qualified persons.

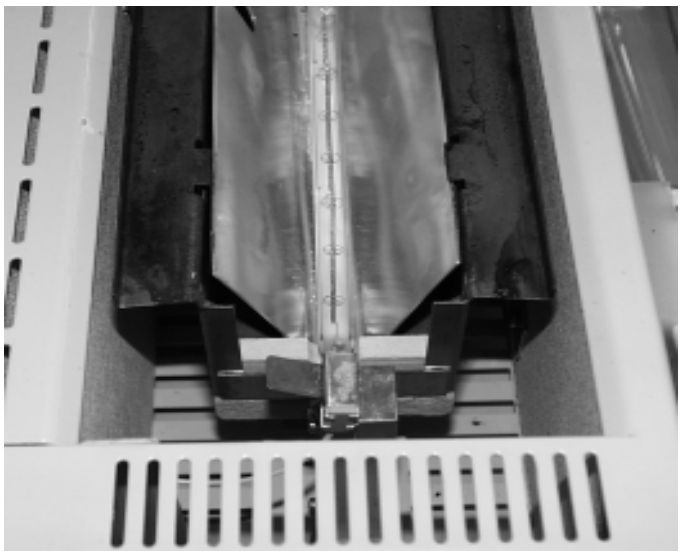


Fig. 10: Replacing the IR heating

Changing the side insulation
These two insulating panels are freely accessible and can be changed without having to use any tools.

Replacing the IR heater

- The equipment must be cold.
- Disconnect the mains plug.
- Remove the vitrified glass plate.
- Remove the insulation.
- Press down the clip and release the heater.
- Fit the new heater.

Do not touch the heater glass!

- Connect the equipment to the mains again.

Replacing the reflector

The reflector must be replaced for ashing if a temperature of 530°C can no longer be achieved in the crucible. You can check this with a Tempilstick® , which melts at 530°C.

- Disconnect the mains plug.
- Remove the vitrified ceramic plate.
- Remove the insulation.
- Remove the infrared heater.

Do not touch the heater glass!

- Press the reflector together upwards until it can be removed from the hold-down cams.
- Carefully introduce the new reflector to locate over the ceramic tubes and press downwards until it clicks into place under the hold-down cams.
- Remove the protective plastic film.

6.5 Aftersales service

Work on or in the equipment may only be carried out by authorized service personnel. These are professionals who have received a thorough technical training and are aware of the risks that arise if the safety precautions are not taken. Büchi aftersales service offices have a service manual for the specific piece of equipment. This can only be obtained by authorized persons.

The addresses of official Büchi aftersales service offices can be found on the last fold-out page of these operating instructions. Please contact these offices if any faults occur or if you have any technical queries or application problems.

Büchi's after sales service organization can provide the following services:

- Spare parts
- Repairs
- Maintenance
- Technical advice

7 Taking the System Out of Operation

Hazardous substances must be removed and the equipment must be thoroughly cleaned. This will avoid any risk that persons may be injured as a result of coming into contact with hazardous substances.

7.1 Storage/Transport

After it has been cleaned, the equipment is to be stored and transported in its original packaging.

7.2 Disposal

So that the equipment can be disposed of in the most environmentally sensitive manner, a list of the materials used to manufacture the most important components can be found in the Appendix to Chapter 9, Table 6. This will allow the parts to be separated and sent for recycling. We would refer you to the relevant directives for information on the disposal of electronic components. Additionally, regional and local laws concerning disposal are to be observed.

8 Spare Parts

Only genuine Büchi accessories and spare parts will provide the product features required to ensure safe and correct operation of the equipment. Spare parts and accessories from sources other than Büchi are only permitted if prior written permission is obtained from the manufacturer. For the purposes of assembly and removal, spare parts may only be used in conjunction with Chapter 6 of these operating instructions. It is not permitted to allow third parties to inspect, take away for examination and manufacture copies using these instructions.

8.1 Spare parts

Fig. 11: Spare parts

Description:		Order No.
1	Vitrified ceramic plate with no bore holes	**26982**
1	Vitrified ceramic plate, 39 mm dia.	**26661**
1	Vitrified ceramic plate, 48 mm dia.	**36676**
1	Vitrified ceramic plate, 56 mm dia.	**26663**
1	Crucible 20 ml	**26936**
1	Crucible 49 ml	**36675**
1	Crucible 90 ml	**26938**
1	Separator (glass only)	**36080**
1	Glass hood	**36081**
1	Hinge	**26925**
1	Bracket, complete	**26996**
1	Infrared heater, complete	**26686**
1	Heater module, complete	**26997**
1	Thermo element	**26885**
1	Ceramic tube	**26881**
1	Reflector	**26924**
1	Viton tube 12/8	**20136**
1	Corrugated hose	**26096**
1	Set of clips	**26968**
1	Fuses 10*100mAT	**22555**

Table 4: Spare parts

9 Appendix

9.1 Technical data

Dimensions of equipment (Width x Height x Depth)	470 x 290 x 370 mm
Voltage rating	220 – 240 V, 50 – 60 Hz
Wattage	approx. 1100 Watts
Current consumption	approx. 5 amps
Weight (net)	approx. 12 kg
Number of ashing locations	4/6

Table 5: Technical data

9.2 Materials used

Part	**Material**	**Material code**
Housing	Steel, powder-coated with polyester-epoxide	1.0330
Glass parts	Borosilicate glass 3.3	DIN/ISO 3585

Table 6: Materials used

9.3 Declaration of Conformity

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

declare as the party solely responsible that the product:
BÜCHI B-440 Wet Digester

to which this declaration refers, complies with the following Standards:

EN 292-1:1991
Safety of machinery; basic terminology and methodology

EN 292-2:1991
Safety of machines, technical principles and specifications

EN 60335-1:1988 (~IEC 335-1 VDE 0700-1 SEV 1054-1)
Safety regulations for household appliances

EN 55011:1991/B (~VDE 0875/B VDE 0871/B)
Limits and methods of measurement of electromagnetic disturbance characteristics of industrial, scientific and medical radio-frequency equipment

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

EN 61000-3-3: 1995
Limitation of voltage fluctuations and flicker

EN 50081-1:1992
Electromagnetic compatibility – General emissions standard – Residential, commercial and light industry areas

In accordance with the provisions in the EU Directive:
89/392/EEC Directive on machinery

Flawil, 26.01.01

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