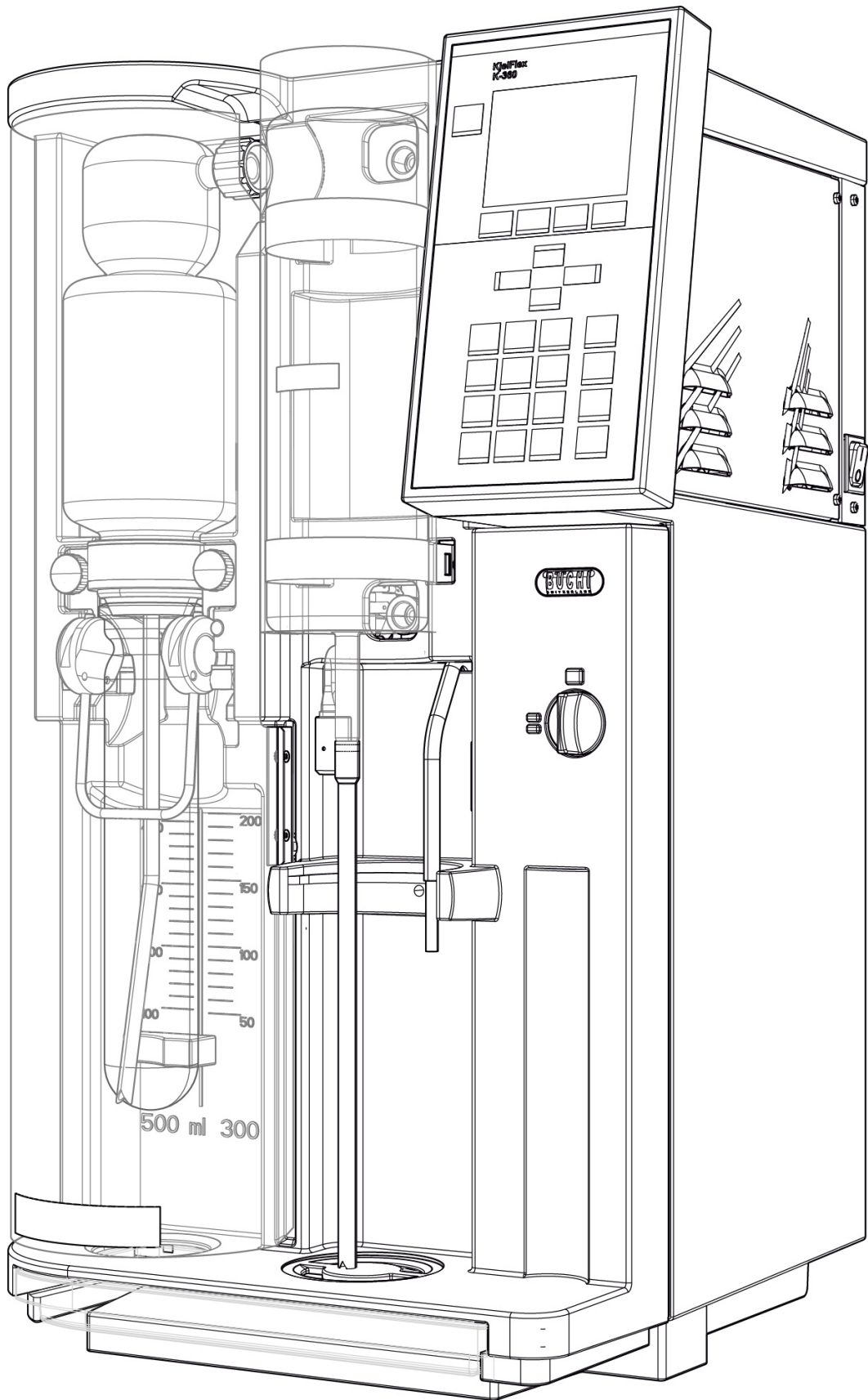




# KjelFlex K-360

## Operation Manual



**Imprint**

Product Identification:  
Operation Manual (Original), KjelFlex K-360

093176D en

Publication date: 06.2016

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Read this manual carefully before installing and running your system and note the safety precautions in chapter 2 in particular. Store the manual in the immediate vicinity of the instrument, so that it can be consulted at any time.

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# 1 About this manual

This manual describes the KjelFlex K-360 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.

**NOTE**

*The symbols pertaining to safety messages are explained in chapter 2.*

## 1.1 Abbreviations

*EPDM*: Ethylene Propylene Dimonomer

*FCC*: Federal Communications Commission

*FEP*: Fluorethylene Propylene

*NBR*: Nitrile Butadiene Rubber

*PE*: Polyethylene

*PMMA*: Polymethyl methacrylate

*PP*: Polypropylene

*PTFE*: Ethylenetetrafluoroethylene (Teflon)

*PUR*: Polyurethane

*RSD*: Relative Standard Deviation

## 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 Safety warnings and safety signals used in this manual

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

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








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

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

	 <b>SIGNAL WORD</b>
Space for supplementary safety information symbols.	Supplementary text, describing the kind and level of hazard / risk seriousness. <ul style="list-style-type: none"> <li>• List of measures to avoid the herein described, hazard or hazardous situation.</li> <li>• " "</li> <li>• " "</li> <li>• " "</li> </ul>

### Table of supplementary safety information symbols

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

Warning symbols	Meaning
	Electrical hazard
	Hot machine parts, hot surface
	Corrosive substances
	General warning sign
	General system damage
Mandatory action symbols	Meaning
	Wear safety gloves
	Wear protective goggles

### Additional user information

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

#### **NOTE**

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



## 2.2 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.

## 2.3 Proper use

The instrument has been designed and built for laboratories. It serves for the distillation of steam-volatile substances.

## 2.4 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.
- Use on samples, which can explode or inflame (e.g. explosives, etc.) due to shock, friction, heat or spark formation.

## 2.5 Product safety





The instrument 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.

Safety warnings in this manual (as described in section 2.1 ff) serve to make the user alert and to avoid hazardous situations emanating from residual dangers by giving appropriate counter measures.

### 2.5.1 General hazards and safety measures

	<p><b>! WARNING</b></p> <p>Death or serious injuries by explosive solvent atmosphere.</p> <ul style="list-style-type: none"> <li>• Operate the system in well ventilated environments only.</li> <li>• Do not use open flames in explosive environments.</li> <li>• Avoid electrostatic and electric spark formation in explosive environments.</li> </ul>
  	<p><b>! WARNING</b></p> <p>Death or serious injuries by strong corrosives.</p> <ul style="list-style-type: none"> <li>• Observe supplementary data sheets of all used chemicals.</li> <li>• Handle corrosives in well ventilated environments only.</li> <li>• Always wear protective goggles.</li> <li>• Always wear protective gloves.</li> </ul>

### 2.5.4 Safety elements

The instrument is provided with the following safety elements:

- Protective door: Safety appliance to protect users from burns at the splash protector (distillation area), which is hot during distillation.
- Protective door sensor: Prevents the start of a distillation with the protective door open and stops a running distillation and the dosing of reagents immediately when the protective door is opened during the process.
- Sample tube sensor: Prevents the start of a distillation without a sample tube inserted.
- Protective shield (cover) at condenser: Protects the glass parts.
- Service door sensor/switch: Electrical power is disconnected immediately when the service door is opened, thus preventing electrical shock during maintenance.
- Cooling water flow sensor: Stops a running distillation.
- Optional level sensors for reagent and waste tank: A running distillation is interrupted when the tanks are too full or too empty respectively.
- Drip tray: Collects overflowing liquids.
- Automatic chemical dosage: Ensures the use of homogeneous liquid amounts.

## 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](http://www.buchi.com) or contact your local dealer.

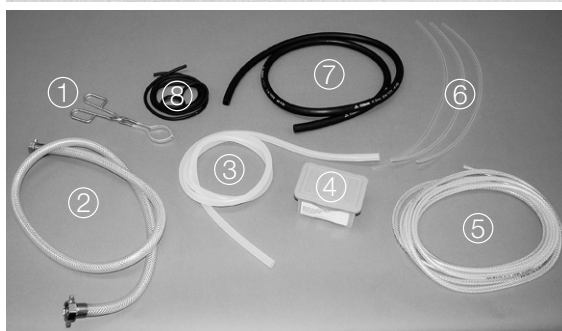
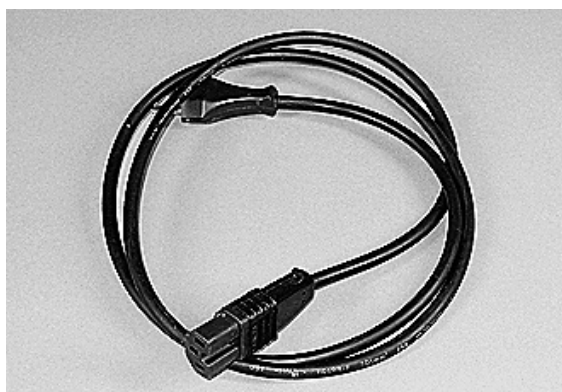
#### 3.1.1 Standard instrument



**Table 3-1: Standard instrument**

Product	Order number
KjelFlex K-360 Standard with glass splash protector, 230 V, 50/60 Hz	43600
KjelFlex K-360 Standard with splash protector made of fiber glass reinforced polypropylene, 230 V, 50/60 Hz	43601
KjelFlex K-360 with glass splash protector and acid resistant pump, 230 V, 50/60 Hz	43605
KjelFlex K-360 with splash protector made of fiber glass reinforced polypropylene and acid resistant pump, 230 V, 50/60 Hz	43604
KjelFlex K-360 Standard with glass splash protector and titration set, 230 V, 50/60 Hz	43607
KjelFlex K-360 with splash protector made of fiber glass reinforced polypropylene and titration set, 230 V, 50/60 Hz	43606
KjelFlex K-360 with glass splash protector, acid resistant pump and titration set, 230 V, 50/60 Hz	43609
KjelFlex K-360 with splash protector made of fiber glass reinforced polypropylene, acid resistant pump and titration set, 230 V, 50/60 Hz	43608

## 3.1.2 Standard accessories

**Table 3-2: Standard accessories**

Product	Order number
<b>Mains cable of the following types</b>	
Type CH	10010
Type Schuko	10016
Type GB	17835
Type AUS	17836
Type USA	33763
Type Japan	10016
① Pair of glass tongs	02004
② Cooling water hose complete: G 3/4", 1/2", L = 1.5 m	37780
③ Hose cooling water drain, silicone, L = 1.8 m, Ø 14/10 mm	43439
④ Weighing boats	40444
⑤ Hose chemical supply, Nylflex, L = 6 m, Ø 10/5 mm	43185
⑥ 3 suction hoses to tanks, FEP, L = 580 mm	43407
⑦ Hose waste drain, EPDM, L = 1.8 m, Ø 18/11 mm for acid resistant pump	43457
⑧ Hose Viton (acid resistant), L = 1.2 m, Ø 6/3 mm (only supplied for instruments with acid resistant pump)	43693
4 clamps Ø 11.9	43841
⑨ 3 tanks 10 L, without caps	43410
⑩ 1 cap for 10 L tanks, large	25869
⑪ 12 tank labels	43434
⑫ 1 cap for 10 L and 20 L tanks, small	43477
<b>Operation Manual:</b>	
English	93176
German	93177
French	93178
Italian	93179
Spanish	93180

## 3.1.3 Optional accessories K-360

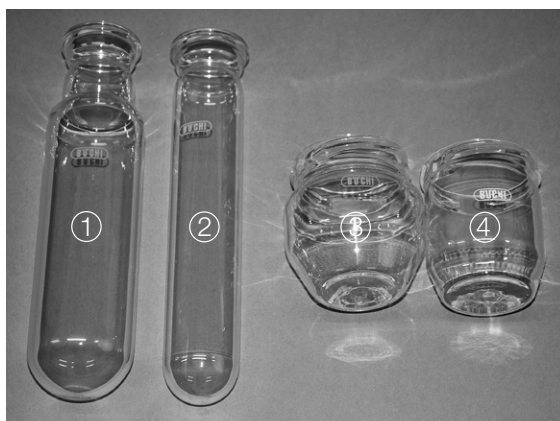
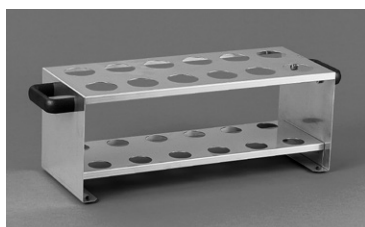
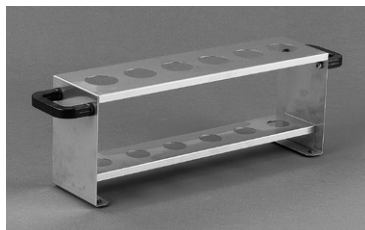
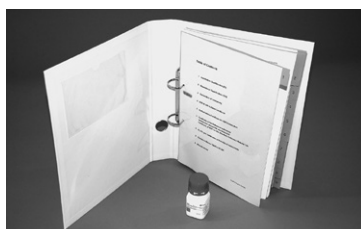


Table 3-3: Optional accessories K-360

Product	Order number
① Sample tube (set of 4), 500 mL	43982
② Set of sample tubes (set of 4), 300 mL	37377
③ Receiving vessel 420 mL	43390
④ Receiving vessel 340 mL	43333
<b>Tanks without level sensors, including caps</b>	
⑤ 10 L chemicals	43468
⑤ 10 L waste	43470
⑧ 20 L chemicals	43469
⑧ 20 L waste	43471
<b>Tanks with level sensors, including caps</b>	
⑦ 10 L chemicals	43472
⑦ 10 L waste	43374
⑥ 20 L chemicals	43473
⑥ 20 L waste	43475
Indicator according to Sher, 100 mL	03512
<b>External PC keyboard (US)</b>	
External PC keyboard (US)	31456
<b>External PC keyboard (D)</b>	
External PC keyboard (D)	31457
<b>External dosage device for back titration, 115 V</b>	
External dosage device for back titration, 115 V	43367
<b>External dosage device for back titration, 230 V</b>	
External dosage device for back titration, 230 V	43596
<b>Cable for external dosage device for back titration (Schott Titronic)</b>	
Cable for external dosage device for back titration (Schott Titronic)	43621

**Table 3-3: Optional accessories K-360**

Product	Order number
Sample tube holder for 4 sample tubes, 500 mL each	16951
Holder for 6 sample tubes, 300 mL	43039
Holder for 12 sample tubes, 300 mL	43041
Titration set	43698
Splash protector for Devarda method	43335
Connection cable to:	
Mettler (DL15/22) titrator	43616
Metrohm (785/719/702) Mettler (Txx) titrator	43617
Metrohm (848) titrator	11055333
Schott titrator	43618
Radiometer titrator	43619
Adapter for Metrohm 719/702	47803

**Table 3-3: Optional accessories K-360 (cont.)**

Product	Order number
SO <sub>2</sub> accessory	48680

IQ/OQ set for K-360, English	93189
IQ/OQ set for K-360, German	45541
Repeating OQ, English	11055493
Repeating OQ, German	11055494

## 3.2 Technical data K-360

**Table 3-4: Technical data K-360**

	KjelFlex K-360
Power consumption	max. 2.2 kW
Connection voltage	220 - 240 VAC ± 10%
Frequency	50/60 Hz
Mains connection	3-pole (P, N, E) via power cord
Recovery rate	≥ 99.5%
Reproducibility (RSD)	≤ ± 1%
Detection limit	≥ 0.1 mg Nitrogen
Vapor output control	30 - 100%
Environmental conditions	for indoor use only
Temperature	5 – 35 °C
Altitude	up to 2000 m
Humidity	maximum relative humidity 80% for temperatures up to 31 °C decreasing linearly to 67% relative humidity at 35 °C
Installation category	II
Pollution degree	2
Dimensions (W x H x D)	405 x 660 x 400 mm
Weight	22 kg
Printer interface	USB 2.0, PCL 6



### 3.3 Titration solution

The amount of sample and the concentration of the titrant should be optimized, so that the titrant volume is between 2 and 18 ml (burette volume: 20 ml).

**Table 3-5: Titration solution 1**

N-amount	N-content	Sample size	Titration	Titration concentration	Titration volume
5 mg N	0.5 % N	1 g	H <sub>2</sub> SO <sub>4</sub>	0.01 mol/L	17.8 mL
10 mg N	1.0 % N	1 g	H <sub>2</sub> SO <sub>4</sub>	0.05 mol/L	7.1 mL
50 mg N	5 % N	0.1 g	H <sub>2</sub> SO <sub>4</sub>	0.01 mol/L	17.8 mL
100 mg N	10 % N	1 g	H <sub>2</sub> SO <sub>4</sub>	0.25 mol/L	14.3 mL
100 mg N	10 % N	1 g	H <sub>2</sub> SO <sub>4</sub>	0.5 mol/L	7.1 mL
200 mg N	20 % N	1 g	H <sub>2</sub> SO <sub>4</sub>	0.5 mol/L	14.3 mL
200 mg N	20 % N	1 g	H <sub>2</sub> SO <sub>4</sub>	1 mol/L	7.1 mL

**Table 3-6: Titration solution 2**

P-content	P-factor	N-content	Sample size	Titration	Titration concentration	Titration volume
1 % P	6.25	0.16 % N	2 g	H <sub>2</sub> SO <sub>4</sub>	0.01 mol/L	11.42 mL
2 % P	6.25	0.32 % N	1 g	H <sub>2</sub> SO <sub>4</sub>	0.01 mol/L	11.42 mL
5 % P	6.25	0.80 % N	2 g	H <sub>2</sub> SO <sub>4</sub>	0.1 mol/L	5.71 mL
10 % P	6.25	1.6 % N	2 g	H <sub>2</sub> SO <sub>4</sub>	0.1 mol/L	11.42 mL
10 % P	6.25	1.6 % N	2 g	H <sub>2</sub> SO <sub>4</sub>	0.25 mol/L	4.57 mL
20 % P	6.25	3.2 % N	2 g	H <sub>2</sub> SO <sub>4</sub>	0.25 mol/L	9.14 mL
50 % P	6.25	8.0 % N	2 g	H <sub>2</sub> SO <sub>4</sub>	0.5 mol/L	11.42 mL

#### General recommendation

Hydrochloric acid has the disadvantage of degassing. Therefore Buchi recommends to use sulfuric acid as titrant.

The correction factor for self prepared solutions is called a titer.

The use of standardized titration solutions make a titer determination unnecessary.

Exact titrant concentration = concentration x titer

The titer of the titrant must be known. In case, it is unknown, it must be determined.

**Example:** Exact titrant concentration = 0.100 mol/L x 0.998

### 3.4 Reference substances

**Table 3-7: Reference substances**

Name	Purity	% N theoretical (100 % purity)	Recommended sample size	Recommended titrant concentration	Digestion necessary
Ammonium dihydrogen phosphate	99.5	12.18	0.8 g	c(H <sub>2</sub> SO <sub>4</sub> ) = 0.25 mol/L	No
Glycine	99.7	18.66	0.5 g	c(H <sub>2</sub> SO <sub>4</sub> ) = 0.25 mol/L	Yes
Phenylalanine	99.0	8.47	0.9 g	c(H <sub>2</sub> SO <sub>4</sub> ) = 0.25 mol/L	Yes
Ammonium sulfate	99.5	21.21	0.4 g	c(H <sub>2</sub> SO <sub>4</sub> ) = 0.25 mol/L	No

### 3.5 Materials used

**Table 3-8: Materials used for the K-360**

Component	Material designation	Material code
Housing	Polyurethane	PUR / UL V0
Glass parts	Borosilicate glass 3.3	DIN/ISO 3585
Steam generator isolation	Ceramic fiber	Multitherm 550
Steam generator housing	Stainless steel	1.4301
Protective door	Polymethyl methacrylate	PMMA
Condenser cover	Polymethyl methacrylate	PMMA
Connecting stopper	Hypalon	CSM
Plastic splash protector	Glass fiber reinforced polypropylene	PP
Stop-cock	Polypropylene/polyethylene	PP/PE

## 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 overview



- ① Splash protector
- ② Sample tube
- ③ Protective door

- ④ Condenser
- ⑤ Operating panel
- ⑥ Service door

Fig. 4.1: Instrument overview

## 4.2 Function principle

The KjelFlex K-360 is suitable for determining nitrogen using the Kjeldahl (TKN; Total Kjeldahl Nitrogen) and Devarda methods as well as for other distillations of steam-volatile substances (e.g. of alcohol, SO<sub>2</sub>, volatile acids).

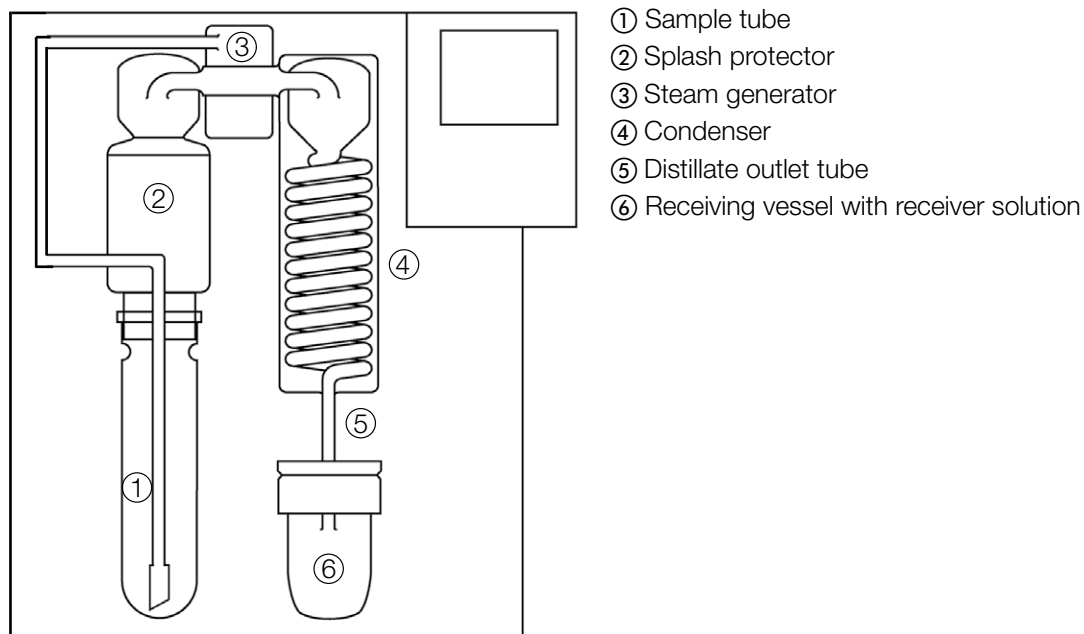


Fig. 4.2: Function principle

Steam is introduced into the sample solution (in sample tube ①) to drive out volatile components (such as ammonia, alcohol, etc.). After condensation (in condenser ④) the distillate is collected in a receiver solution (in receiving vessel ⑥).

## 4.3 Kjeldahl applications

Determination of nitrogen/protein.  
 For system configuration with reagent-pump:  
 Warnmeldung

## 4.4 Non-Kjeldahl applications

Typical examples for non-Kjeldahl methods.

Determination of:

- SO<sub>2</sub>
- Phenol
- Formaldehyde
- TVBN
- Alcohol
- Volatile acids

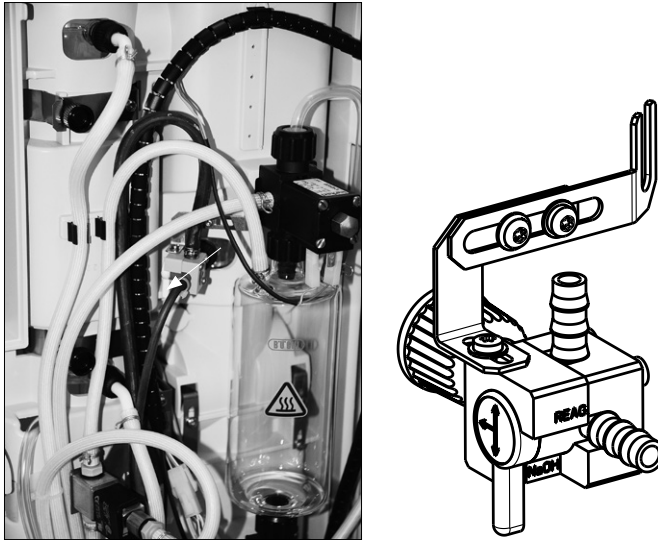




Fig. 4.3: Stop-cock

The stop-cock is located at the inside of the service door.

To carry out a Kjeldahl application the stop-cock must be in downwards position.

To carry out a non-Kjeldahl application the stop-cock must be in horizontal position.

	<p><b>⚠ CAUTION</b></p> <p>Risk of burnings by hot machine parts and water steam.</p> <ul style="list-style-type: none"> <li>• Let the system cool down before performing any service.</li> <li>• Do not operate the instrument when enclosure is open or removed.</li> </ul>
	<p><b>Notice</b></p> <p>Wrong stop-cock setting will result in system overpressure. Tubing may come off.</p> <ul style="list-style-type: none"> <li>• Set stop-cock to correct working-position.</li> </ul>

**NOTE**

A warning message will be displayed, when the method is changed from a Kjeldahl to a non-Kjeldahl method or vice versa:

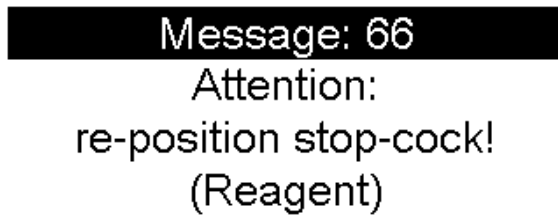


Fig. 4.4: Warning message on the display

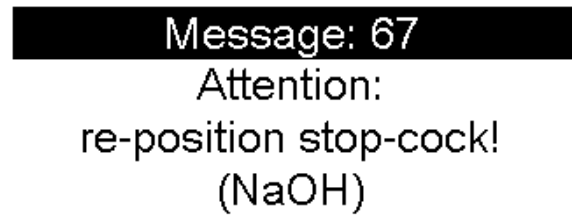


Fig. 4.5: Warning message on the display

## 4.5 Operational elements of the instrument

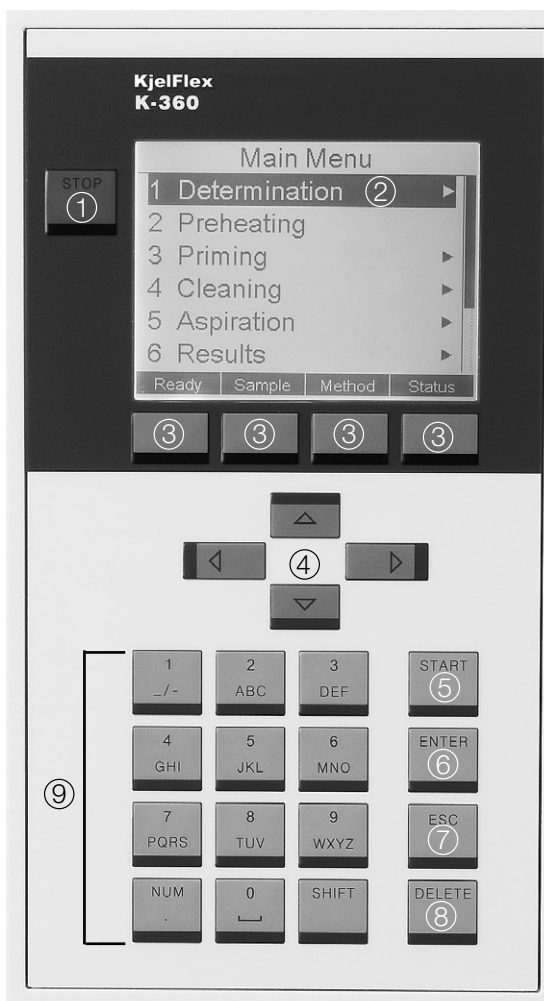


Fig. 4.6: Controls of the instrument

- ① Stop button to stop a process
- ② Display
- ③ Functional buttons to operate the software
- ④ Navigation buttons to navigate within the software
- ⑤ Start button to start a process like preheating, priming, cleaning, etc.
- ⑥ Enter button to move forward within the sub-menu structure
- ⑦ Escape button
- ⑧ Delete button to delete a digit entered via the keypad
- ⑨ Keypad

## 5 Putting into operation

This chapter describes how the instrument is installed and gives instructions on initial startup.


### NOTE

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

Keep the original packaging for future transportation.

### 5.1 Installation site

Install the instrument on a clean, flat and stable base and place the tanks for chemicals beside the instrument (not higher and not more than 1 m lower).

	<b>Notice</b>
	<p>Reduced instrument safety and lifetime due to improper handling and installation location.</p> <ul style="list-style-type: none"> <li>• Do not place any object on top of the instrument.</li> <li>• Keep a free safety space of 30 cm around the instrument.</li> <li>• Do not place items behind the instrument.</li> <li>• Do not operate the instrument within an fume cabinet.</li> </ul>

### 5.2 Electrical connections

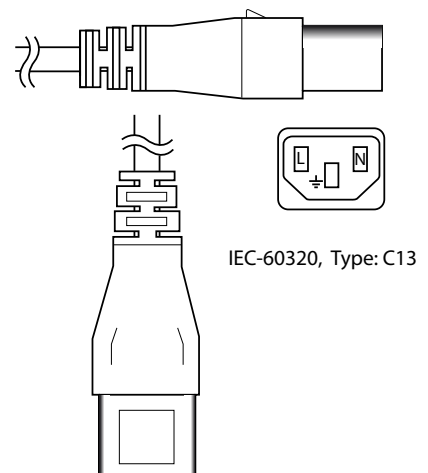



Fig. 5.1: Electrical connections

	<b>WARNING</b>
	<p>Death or serious burnings by electric shock.</p> <ul style="list-style-type: none"> <li>• Mains voltage must match the voltage reading on the type plate.</li> <li>• Instrument must be earthed by the mains socket.</li> <li>• Only use a molded 3-pole device plug (IEC-60320, type C13 or C15).</li> <li>• Do not use damaged cables.</li> </ul>

### 5.3 Reagent and water connections



- ①  $\text{H}_3\text{BO}_3$  inlet from tank
- ②  $\text{H}_2\text{O}$  inlet from tank
- ③  $\text{NaOH}$  inlet from tank
- ④ Reagent inlet for acid resistant pump
- ⑤ Cooling water inlet
- ⑥ Cooling water outlet
- ⑦ Drain to waste

Fig. 5.2: Reagent and water connections

All pumps are self-priming, no overpressure is necessary at the tanks.

#### 5.3.1 Cooling water connection

The water pressure should be at a maximum of 4 bar. The built in valve reduces the water flow to 1.2 liters per minute.

The flanged screw coupling for the water connection has a standard screw thread of G 3/4".

#### 5.3.2 Drainage of cooling water

Place the drain hose for the cooling water directly into the drain. For this purpose, shorten the silicone hose to the optimal length. The drain hose should not show any kinks, sharp bends and/or siphoning effect. Prevent flooding inside and outside the instrument by securing the drain hose.



### 5.3.3 Waste/aspiration hoses

The distillation and sample residue can be aspirated and collected separately from the cooling water. For this purpose a separate collection tank is necessary. The collection tank must be located lower than the instrument to guarantee proper drainage.

Connect the waste/aspiration hose to the waste outlet and place it directly into the drain (sink). For this purpose, shorten the EPDM hose to the optimal length. In order to prevent any back-flow, the hose may be placed maximum 10 cm into the tank.

### 5.3.4 Storage tank connection

To connect the storage tanks, proceed as follows:

- Cut the Nylflex hose into pieces of the appropriate length (use a Viton hose for the acid resistant pump).
- Insert a FEP suction hose into the Nylflex hose.
- Push an EPDM sealing ring over the Nylflex hose.
- Now fasten the hoses to the tank with the red screw cover.
- Connect the hoses at the appropriate connections at the instrument side and secure them with the clamps.

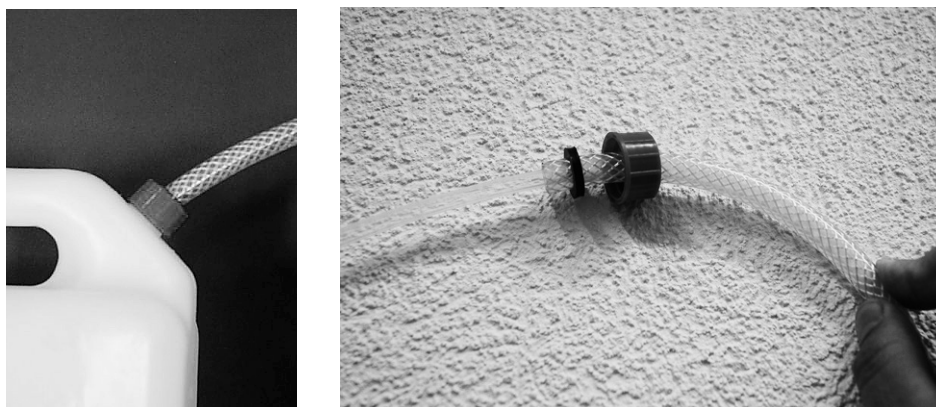



Fig. 5.3: Tank connection

<b>Notice</b>	
	<p>Possible instrument damage by calcification or corrosion.</p> <ul style="list-style-type: none"> <li>• Use only distilled water in the H<sub>2</sub>O storage tank to operate the system.</li> <li>• Do not mix up liquids at the instrument inlets, observe the inlet labelling.</li> </ul>

## 5.4 Level sensors (optional)

Level sensors are optionally available for all Buchi tanks. The individual sensors are connected to the corresponding socket.

H<sub>2</sub>O: Connection to the tank with distilled water for the steam generator and sample tube.

NaOH: Connection to the tank with sodium hydroxide.

H<sub>3</sub>BO<sub>3</sub>: Connection to the tank with boric acid.

Waste: Connection to the waste tank.

The length of the level sensor for chemicals can be adapted by means of the the rubber cap, see Fig. 5.4.

### NOTE

*The level sensor for the waste tank must be set active at: Configuration, Settings, Level Sensor Waste. This is not necessary for the other sensors.*

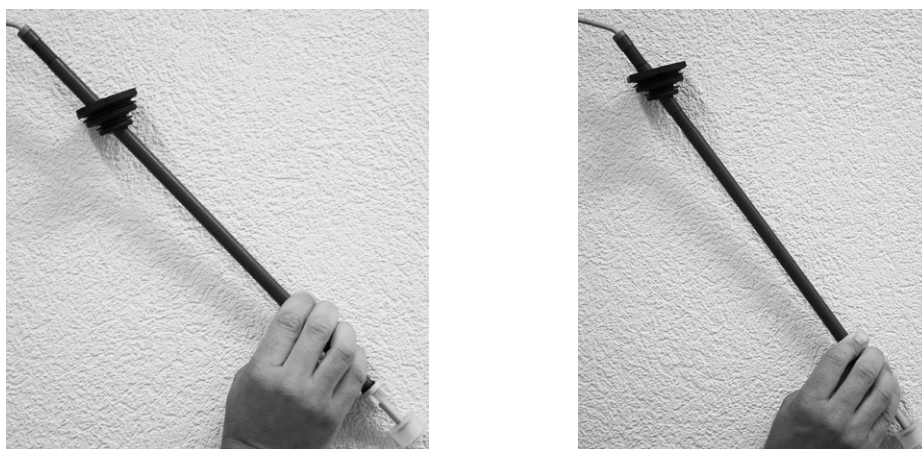


Fig. 5.4: Adapting the length of the level sensor

Adapt the length of the level sensor for chemical by moving up and down the rubber cap according to the size of the tank.

## 5.5 Connections to peripheral devices

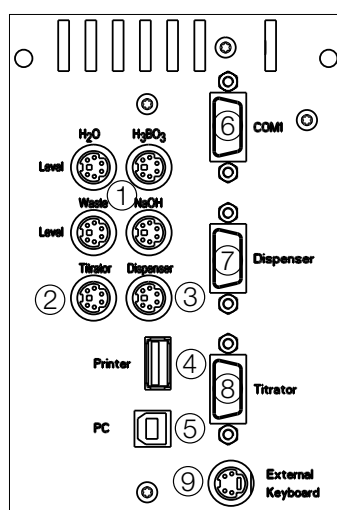


Fig. 5.5: Connections to peripheral devices

The following instruments and accessories can be connected to the K-360:

- ① Level sensors
- ② TTL interface to a titrator
- ③ TTL interface to a dispenser (external dosage device for back titration)
- ④ USB interface to a printer
- ⑤ USB interface (for service purposes)
- ⑥ COM1 (reserve)
- ⑦ RS interface to a dispenser
- ⑧ RS interface to a titrator
- ⑨ External keyboard

### 5.5.1 Connecting a printer

The K-360 supports printers with USB port and language PCL 3 or higher (e.g. PCL 5, PCL 5e, PCL 6, PCL 7 etc.) which are available e.g. from Hewlett Packard or Lexmark.

The printer is connected to the USB port.

To print out the results directly after each determination, go to Configuration > Titrator and set Print Data to Yes. You also have to configure the titrator for data printout. For this purpose, please consult the corresponding manual.

The following printers have been tested together with the K-360:

- HP Color LaserJet 3700DN
- HP Deskjet 1280c
- Brother mfc 8820d
- Lexmark E120
- Lexmark E240
- HP OfficeJet Pro K550
- Brother HL-5240
- OKI B4250

The following printer causes problems:

- HP LaserJet 1022

### 5.5.2 External keyboard

As an option, an external keyboard can be connected to the instrument to make data entry more comfortable. The internal keyboard is still active, even if an external keyboard is connected.

Special functions on the external keyboard are:

**Table 5-1: External keyboard**

K-360 keyboard	External keyboard
START	F3
STOP	F2
Function key below display	F5...F8

Other functions (e. g. up/down) are the same as on the internal keyboard.

## 5.6 Boric acid titration and back titration



Fig. 5.6: K-360 for boric acid titration (with titrator)

The K-360 offers the possibility to work with boric acid titration or back titration. It is recommended to choose a permanent configuration as different hardware is needed to convert between the two titration methods.

Buchi recommends to work with boric acid titration.

### Boric acid titration

Receiving vessel: Boric acid solution 2 % or 4 %, adjusted to pH 4.65.

Titration: Standardized sulfuric acid solution or hydrochloric acid solution

It is recommended to use boric acid solution 4 %, adjusted to pH 4.65 and standardized sulfuric acid solution.

The K-360 includes all necessary hardware to work with boric acid titration. Boric acid is dosed via the integrated pump.



Fig. 5.7: K-360 for back titration (with external dosage device)

### Back titration


Receiving vessel: Standardized sulfuric acid




Titration: Standardized sodium hydroxide solution

An external dosage device must be connected for the exact addition of the receiving solution. See chapter 6.4.6 for information on the installation and set-up of the external dosage device.

## 6 Operation

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

	<b>⚠ CAUTION</b>
	<p>Risk of cuts by defective glassparts.</p> <ul style="list-style-type: none"><li>• Handle glassparts with care.</li><li>• Exchange defective glassparts directly.</li><li>• Do not operate the instrument when glassparts are damaged.</li></ul>

  	<b>⚠ WARNING</b>
	<p>Serious chemical burnings by corrosives.</p> <ul style="list-style-type: none"><li>• Observe supplementary data sheets of all used chemicals.</li><li>• Wear protective goggles.</li><li>• Wear protective gloves.</li><li>• Wear protective clothing.</li></ul>

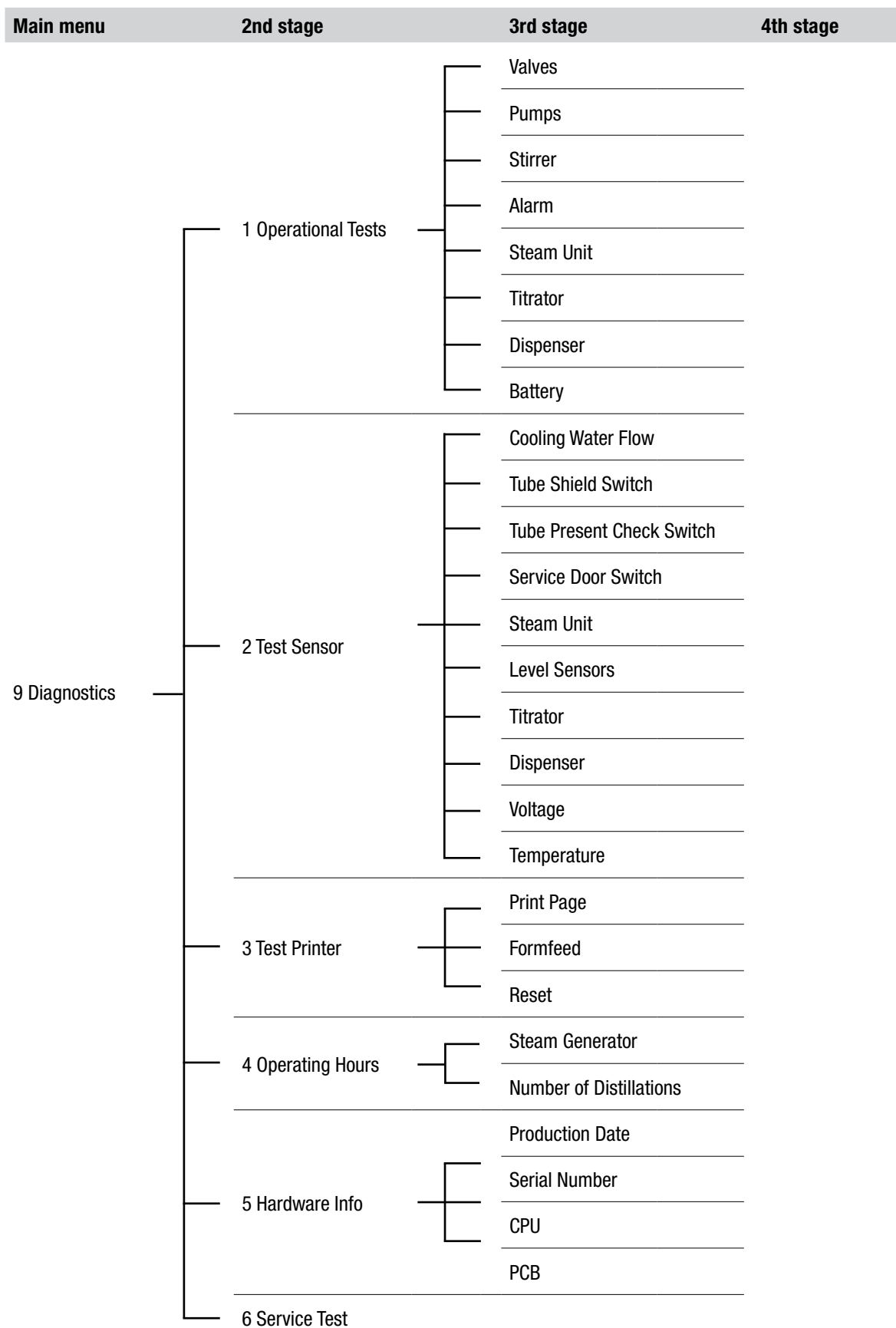
### **NOTE**

*Close unused connections with plug caps.*

## 6.1 Overview over the software structure

Main menu	2nd stage	3rd stage	4th stage
1 Determination	Sample List		
2 Preheating			
3 Priming	Priming Method	<ul style="list-style-type: none"> <li>H<sub>2</sub>O, NaOH, H<sub>3</sub>BO<sub>3</sub>, (Reagent) volumes</li> <li>Reaction Time</li> <li>Steam Power</li> <li>Distillation Time</li> <li>Titration Start</li> <li>Titr. Type</li> <li>Stirrer Speed</li> <li>Aspiration Sample/Receiver</li> </ul>	
4 Cleaning	Cleaning Method	<ul style="list-style-type: none"> <li>H<sub>2</sub>O</li> <li>Steam Power</li> <li>Distillation Time</li> </ul>	
5 Aspiration	Aspiration Method	<ul style="list-style-type: none"> <li>Aspirate Sample</li> <li>Aspirate Receiver</li> </ul>	
6 Results	Result List		
7 Methods	Method List		

Main menu	2nd stage	3rd stage	4th stage	
8 Configuration	1 Pump Calibration	Calibration of: H <sub>2</sub> O, NaOH, H <sub>3</sub> BO <sub>3</sub> , (Reagent) Pump		
	2 Set Date and Time	Date/Time Details		
	3 Settings	Language		
		Display Contrast		
		Demo Mode		
		Endless Mode		
		Level Sensor Waste		
		Rec. Vessel Present		
	4 Titrator	Reagent Pump		
		Print Data		
Titrator Present				
5 Dispenser	Titrator Signals			
	Dispenser Signals	Signal "ready"		
		Signal "active"		
6 User / Password	User / Password List			
7 Keypad	Keypad Time			
			Signal "end"	










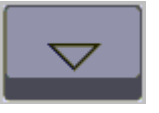




## 6.2 General information on buttons

The following control buttons are available in the software for navigation and input confirmation:

<b>Menu</b>	Go to the main menu
<b>OK</b>	Confirm a message and get back to the previous screen
<b>Save</b>	Save
<b>Close</b>	Close a screen (without saving) and get back to the previous one
<b>Delete</b>	Delete
<b>Edit</b>	Edit
<b>First</b>	Mark the currently highlighted result/method in the list as first result/method on a report
<b>Last</b>	Mark the currently highlighted result/method in the list as last result/method on a report
<b>H<sub>2</sub>O</b>	Add 10 ml of H <sub>2</sub> O
<b>H<sub>3</sub>BO<sub>3</sub></b>	Add 10 ml of H <sub>3</sub> BO <sub>3</sub>
<b>NaOH</b>	Add 10 ml of NaOH
<b>Reagent</b>	Add 10 ml of reagent
<b>Method</b>	Go to the method screen to view, define or edit a method
<b>List</b>	Go to the Method List screen
<b>Print</b>	Print
<b>Ready</b>	Switch from Standby to Ready mode
<b>Sample</b>	Go to the sample screen to define a sample
<b>Standby</b>	Switch from Ready to Standby mode
<b>Status</b>	Go to the status screen to view the status, the time and the user logged in
<b>Reset</b>	Reset the operating hours / numbers of distillation
<b>Actors</b>	Switch to the screen with Operational Tests
<b>Sensors</b>	Switch to the screen with Test Sensors
<b>Offline</b>	Switch to offline operation in the Operational Tests screen
<b>Online</b>	Switch to online operation in the Operational Tests screen
<b>Run</b>	Run the pump in the Pump Calibration screen
<b>Cal</b>	Calibrate the pump in the Pump Calibration screen

	Change password
	Define a new user
	Switch to previous screen e.g. in Service Test
	Switch to next screen e.g. in Service Test
	Confirm a message or affirm a question
	Negate a question
	Backward button to move to the previous digit
	Forward button to move to the next digit
	Move up within the entries of a screen
	Move down within the entries of a screen

## 6.3 Overview on how to prepare the instrument for routine operation

Configure the software according to the following steps to prepare it for routine operation:

1. Standard instrument configuration
2. Distillation methods
3. System preparation
4. Individual working methods

### 6.3.1 Standard instrument configuration

This configuration consists of typical settings, which have to be defined before the instrument is used for the first time. As long as the instrument is not updated or extended by any additional optional accessory these settings do not have to be changed again.

The settings are carried out in the menu Configuration.

### 6.3.2 Distillation methods

The distillation methods you like to use can be defined in the menu Method. For this purpose you use an existing Buchi standard method and modify it to your needs. Thus you can define up to 50 individual methods according to e.g. distillation time, aspiration yes/no, volume of boric acid/NaOH/water, etc.

### 6.3.3 System preparation

The system preparation is carried out every day before the instrument is taken into operation and an analysis is started. It consists of e.g. filling the hoses and priming the system.

### 6.3.4 Individual working method

The software offers two working methods for sample determination, the single sample analysis and the rack oriented analysis.

#### Single sample analysis

This working method is straight forward and is used especially when you are not interested in sample name input and storing the volume consumption of the titrant solution in the system memory, see also chapter 6.8.1.

#### Rack oriented analysis

Here all sample names and the corresponding weight is entered prior to determination. The volume consumption of the titrant solution is stored together with the sample name, see also chapter 6.8.2.

You can switch between these two working methods at any time.

## 6.4 Configuring the instrument

To configure the instrument in the Main Menu go to Configuration and press Enter. The following screen appears:

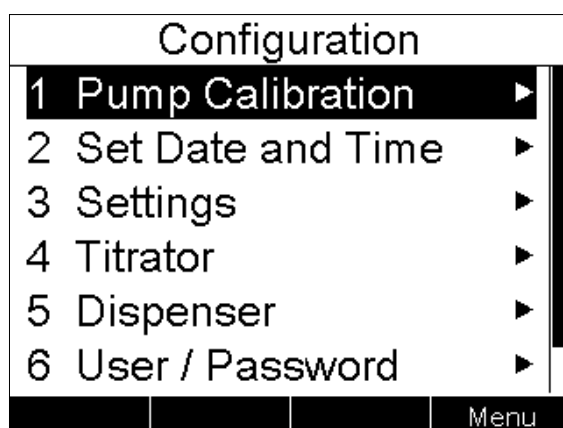


Fig. 6.2: Configuration

#### **NOTE**

*To access the individual menus you can either use the arrow keys to move up and down in the list and press Enter or the Forward button or you can type in the number of the corresponding menu directly (e.g. 3 for Settings).*

### 6.4.1 Pump calibration

Go to Pump Calibration and press Enter. The following screen appears:

Pump Calibration	
Pump:	H2O
Dose Volume:	50 ml
Measured Volume:	0 ml
Calibration Factor:	0.8300
<div style="display: flex; justify-content: space-between;"> <span>Run</span> <span>Close</span> </div>	

Fig. 6.3: Pump calibration

It is recommended to calibrate the pumps with the same volume as used for the methods. To perform the calibration a graduated cylinder is needed.

To carry out the pump calibration, proceed as follows:

- Choose the required pump by pressing left/right arrow repeatedly, when e.g. "H<sub>2</sub>O" is highlighted.
- Enter the quantity to be dosed (Dose Volume), e.g. 50 ml.
- Press Run to start dosing.
- Measure the dosed volume and enter the measured volume.
- Press Cal to calibrate the pump. When the calibration is finished a corresponding calibration factor appears.

#### **NOTE**

*Repeat this procedure until the entered and the dosed volume correspond. An acceptable difference at 50 ml is  $\pm 2$  ml. NaOH/H<sub>2</sub>O/Reagent can be dosed into the sample tube and then poured into a graduated cylinder. H<sub>3</sub>BO<sub>3</sub> is dosed directly into the receiving vessel and then poured into a graduated cylinder.*

*It is recommended to repeat the pump calibration regularly (once a month or every 100 distillations).*

### 6.4.2 Setting date and time

Go to Set Date and Time and press Enter. The following screen appears:

Date / Time	
Day:	21
Month:	6
Year:	7
Hour:	10
Minute:	35
U.S. Date Format:	No
<div style="display: flex; justify-content: space-between;"> <span>Save</span> <span>Close</span> </div>	

Fig. 6.4: Date/Time

Enter the corresponding settings via keypad and press Save to store them.

**NOTE**

The day, month and year settings can be entered with one or two digits.

**6.4.3 Defining general instrument settings**

Go to Settings and press Enter. The following screen appears:

Settings	
Language:	English
Display Contrast:	8
Demo Mode:	No
Endless Mode:	No
Level Sensor Waste:	No
Rec. Vessel Present:	Yes
Save	Close

Fig. 6.5: Settings

The following settings can be entered:

- Language
- Display Contrast
- Demo Mode
- Endless Mode
- Level Sensor Waste
- Receiving Vessel present
- Reagent pump present

Enter the desired settings via arrow buttons and press Save to store them. These settings have to be defined before the instrument is used for the first time.

**6.4.4 Defining the titrator settings**

Go to Titrator and press Enter. The following screen appears:

Titrator	
Print Data:	No
Titrator present:	1
Signal 'ready':	Low
Signal 'activ':	No
Signal 'end':	RS232
Baud:	9600
Edit	Close

Fig. 6.6: Titrator

Press Edit to edit the settings. By changing the number at "Titrator present" the settings for the titrators listed in the following table will be adjusted automatically.

**Table 6-1: Titrator settings**

Titrator present	Configuration	Signal ready	Signal active	Signal end	Baud	Data bit	Parity	Stop bit
1	e.g. Mettler Toledo DL 15/22	Low	No	RS232	9600	8	no	2
2	e.g. Mettler Toledo T50/Txx	No	No	RS232	4800	8	even	1
3	e.g. Metrohm Food Titrino DMP785/719/702	No	No	RS232	9600	8	no	1
4	e.g. Schott TitroLine Easy	No	RS232	RS232	4800	7	no	1
5	e.g. Radiometer TitraLab 840	Low	High	Low	9600	8	no	1
6	e.g. Metrohm 848 Plus	Low	High	RS232	19200	8	no	1
Custom	other titrators							

When you have selected “Custom” you can enter the corresponding settings via the arrow buttons and press Save to store them.

At the moment there are the following titrator types that enable a signal transfer and thus an automatic result display in the K-360 software:

- Mettler Toledo DL 15/22/Txx
- Schott TitroLine Easy
- Metrohm Titrino DMP 785/719/702/848 Plus

To assure a correct signal transfer, the respective titrator has to be configured according to the Büchi specifications.

#### 6.4.5 Boric acid / end point titration

Receiving vessel: see chapter 5.6

Titrant: see chapter 5.6

It is recommended to use boric acid solution 4%, adjusted to pH 4.65 and standardized sulfuric acid solution.

Titrate the distillate with sulfuric acid standard solution to the end point of pH 4.65.

##### Defining settings to work with a titrator

Configuration > Settings > Rec Vessel present: Yes

Configuration > Titrator: Titrator present: (1, 2, 3, 4, 5, 6, Custom)

Methods > Select the corresponding method from the method list and press Enter

> Aspiration Sample: Yes

> Aspiration Rec. Sol'n: Yes

##### Defining settings without a titrator

Configuration > Settings > Rec Vessel Present: No

Configuration > Titrator: Titrator present: No

Methods > Select the corresponding method from the method list and press Enter

> Aspiration Sample: Yes

### 6.4.6 Back titration with dispenser

#### External dosage device for back titration

The external dosage device is connected to the port for the manual key button. The volume of the receiving solution must be entered at the external dosage device. For this purpose, refer to the Operation Manual of the dosage device.

The K-360 sends an impulse to the connected device to start the dosing procedure.

Receiving vessel: Standardized sulfuric acid

Titrant: Standardized sodium hydroxide solution

#### Defining the K-360 settings for back titration

The following setting at the K-360 is a prerequisite for working with a dispenser:

Method > Titr. Type > Back Titration

Go to Configuration > Dispenser and press Enter. The following screen appears:

Dispenser	
Signal 'ready':	No
Signal 'activ':	No
Signal 'end':	60 s
Save	Close

Fig. 6.7: Dispenser

Enter the corresponding settings via the arrow buttons and press Save to store them.

**Table 6-2: Dispenser settings**

Configuration	Signal ready	Signal active	Signal end
Schott Titronic universal	No	No	60 s

### 6.4.7 Defining user and password settings

Go to User/Password and press Enter. The following screen appears:

User / Password	
buchi	
New	Delete
Pwd	Close

Fig. 6.8: User/Password

To define a new user, press New. The following screen opens:

User		ABC
Name:		
Password:		
New:		*
Confirm:		*
Save		Close

Fig. 6.9: User

Enter a new name and a password. To change your password, enter a new one and confirm it. Press Save to store your settings.

**NOTE**

*A password is not mandatory.*

- *When a password is used the corresponding user is indicated on the result printout list.*
- *When a password is forgotten, the determination can be started with the Buchi default login:  
User name: buchi, password: buchi01.*

*When the Buchi default login is used, all other passwords will be deleted.*

#### 6.4.8 Defining the keypad settings

Go to Configuration > Keypad and press Enter. The following screen appears:

Keypad	
Edit Time:	2.0 s
Save	Close

Fig. 6.10: Keypad

Set the desired Edit Time, i.e. the waiting time until a new character can be typed in, and press Save to store your setting.



## 6.5 Defining a distillation method

The Standard method that is stored in the instrument cannot be deleted. It can be used as basis to define further methods. Up to 50 different methods can be saved.

To define a method in the main menu go to Methods and press Enter. The Standard Method is displayed in the Method List by default. Press Enter again, the following screen appears:

Method	
Name:	Standard Method
User:	Büchi
H <sub>2</sub> O:	50 ml
NaOH:	90 ml
H <sub>3</sub> BO <sub>3</sub> :	60 ml
Reagent:	0 ml
Edit	Sample
List	Menu

Fig. 6.11: Method

A method consists of the following settings:

- Name
- User
- Amount of chemicals to be dosed (H<sub>2</sub>O/NaOH/H<sub>3</sub>BO<sub>3</sub> / Reagent)
- Reaction time
- Titration type
- Stirrer speed (Distillation/Titration)
- aspiration (Sample/Receiving vessel)

To define a new method, press Edit and enter the corresponding settings. Then press Save to store them.

### NOTE

*For Kjeldahl applications define a volume of H<sub>2</sub>O and NaOH, for non-Kjeldahl applications, define the volume of your used reagent (acid).*

A list of all defined methods is displayed on pressing the List button:

Method List	
Standard Method	
Beer Method	
Meat Method	
Milk Method	
Delete	Print
Menu	

Fig. 6.12: Method list

To print out a method, highlight the method-name in the list and press Print.

## 6.6 Instrument modes

The instrument has two different modes, Standby and Ready. In Ready mode the steam generator heats continuously, so that a distillation can be started immediately. When the instrument is not operated for thirty minutes, it switches to Standby mode and stops heating to save electric power. When it is switched back to Ready mode, it has to heat up first. You can see the icon “Process in progress” (see position 3 in Fig. 6.8.3) flashing during heating.

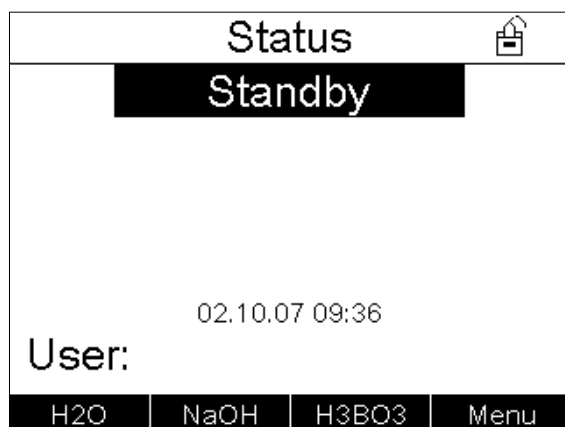


Fig. 6.13: Standby mode

To switch from Standby to Ready mode, press Menu and subsequently Ready.

## 6.7 System preparation

### 6.7.1 Preheating

The glass parts of the distillation system have to be preheated prior to analysis. Use a clean and empty sample tube for this processing. It is recommended to perform a preheating, when the glass (splash protector) has cooled down. The preheating time is predefined.

### 6.7.2 Priming

This preparation procedure includes distillation and titration with a clean and empty sample tube. It is recommended to perform a priming at least once a day, before starting analysis. The priming method can be modified.

### 6.7.3 Cleaning

At the end of a day, the system should be rinsed thoroughly by carrying out a cleaning. The splash protector and the condenser are rinsed with water to remove sodium hydroxide residues. With regular cleaning, the lifetime of the glass parts can be extended. The cleaning method is predefined, but can be modified and adapted to the size of the sample tube.

### 6.7.4 Aspiration

With this procedure sample residue in the sample tube and titration residue in the receiving vessel can be aspirated.

## 6.8 Carrying out a distillation

To carry out a distillation, proceed as follows:

- Switch on the instrument and wait until the steam generator is ready.
- Insert the sample tube containing the sample.
- If you do not work with the titration set, prepare a conical flask or a similar receptacle with a volume of about 250 ml as receiving vessel.

For sample analyses there are two different approaches, depending on how you prefer to work:

- Single sample analyses
- Rack oriented analyses

### 6.8.1 Single sample analysis

For single sample analyses, no predefined sample list is needed for identification. To start an analysis, provide a sample and press the Sample button in the main menu. The following screen opens:

Sample		ABC
Name:	Default	
Weight:	0.0000	
Unit:	mg	
Type:	Sample	
Method:	Standard Method	
		Method Menu

Fig. 6.14: Sample

Enter a sample name and sample weight via keypad. Choose the desired method and press Start to start the distillation process.

#### **NOTE**

- When you re-enter the screen, sample name, weight, unit, type and method are set to the latest entered settings.
- You can start a determination at any time without a sample name or weight input.

### 6.8.2 Rack oriented analysis

For the rack oriented analyses all sample data (name, weight, etc.) have to be entered.

The Sample List screen opens:

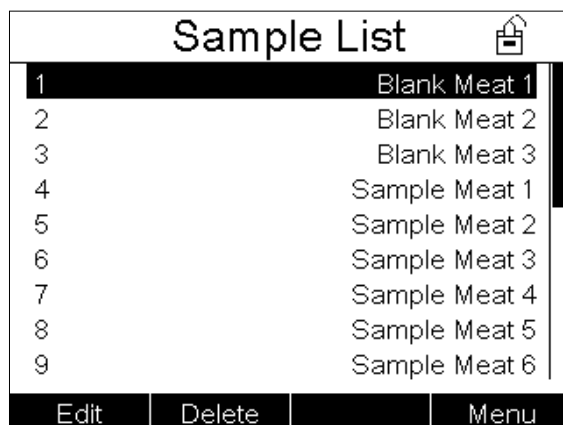
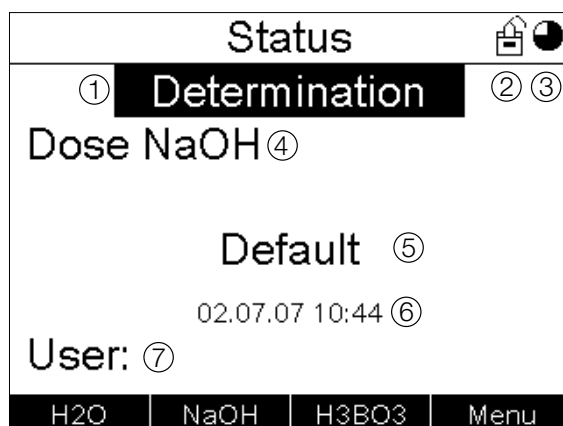


Fig. 6.15: Sample List

This screen allows to define a Sample List entry for every sample provided. To define a new entry, press Edit. The Sample screen opens. Enter sample name and weight and press Save. The newly created entry now appears in the list. Repeat this step for all provided samples.

To start distillation of a sample, enter Determination in the main menu, select an entry and press the Start button. The distillation starts.

### 6.8.3 Analysis screen



- ① Status designation
- ② Titrator icon (titrator connected)
- ③ Icon (turning when process is in progress, flashing when instrument is heating, standing still when instrument is ready)
- ④ Current process step
- ⑤ Name of currently processed sample
- ⑥ Current date and time
- ⑦ User currently logged in

Fig. 6.16: Analysis screen

#### 6.8.4 Result screen

If a titrator has been connected to the K-360, which does not provide direct data transfer, the result screen opens after a distillation. The corresponding ml-value indicated on the connected titrator can be manually added under Result via keypad. Press OK to save the entry.

Result	
Name:	Default
Weight:	50.0000 mg
Result:	0.0000 ml
Type:	Sample
Method:	Standard Method
Date:	02.07.07 10:53
OK	

Fig. 6.17: Result screen

In case the connected titrator provides direct data transfer, the result is stored automatically. If no titrator is connected, no Result screen is displayed.

#### 6.8.5 Result list

To view the list of results measured, go to Results in the main menu and press Enter or just press 6.

The Result List opens:

Result List		
Sample Milk 7	6.2230ml	04.07.07
Sample Milk 6	6.2310ml	04.07.07
Sample Milk 5	6.2510ml	04.07.07
Sample Milk 4	6.2450ml	04.07.07
Sample Milk 3	6.2800ml	04.07.07
Sample Milk 2	6.2100ml	04.07.07
Sample Milk 1	6.2010ml	04.07.07
Blank Milk 3	0.1080ml	04.07.07
Blank Milk 2	0.1020ml	04.07.07
First	Delete	Print
Menu		

Fig. 6.18: Result list

The result list can hold a maximum of 500 results. If this number is being exceeded, the “oldest” results in the list will be overwritten successively by new results.

To print out a selection of results, highlight the first result and press the First button. Then highlight the last result that should appear in the list and press the Last button to define your selection. Now press Print.

For a detailed printout of a result, highlight a single result in the list and press Print.

### 6.8.6 After distillation

If you do not work with the titration set, empty the receiving vessel. The content of the receiving vessel of the titration set is automatically aspirated at the end of the distillation, if this is configured within the method.

It is recommended to clean the system using the cleaning method at the end of the day.

**NOTE**

*The water volume can be adapted in the menu Cleaning under H<sub>2</sub>O.*

## 6.9 Switching ON/OFF checklist

To switch on and preheat the instrument, proceed as follows:


- Check the filling level of the supply tanks.
- Open the main water tap.
- Switch on the instrument and wait until the steam generator is ready.
- Insert an empty sample tube and an empty receiving vessel.
- Close the protective door.
- Start the preheating/priming by pressing the Start button in the corresponding menu.
- Start the distillation by pressing the Start button.


To switch off the instrument, proceed as follows:


- Start the cleaning process (see chapter 7.12).
- Rinse the acid resistant pump with distilled water (in case it was used for the dosage of strong acids, e.g. HCl).
- Switch off the instrument.
- Close the main water tap.




## 7 Maintenance

This chapter gives instructions on all maintenance work to be performed in order to keep the instrument in good working condition. All working steps described herein shall only be carried out by trained personnel. After the maintenance has been finished, the person in charge with this maintenance must bring the system into standard operating condition (e.g. close the service door) and check the proper function of the instrument.

	<b>⚠ WARNING</b>
	<p>Death or serious injuries by electric shock.</p> <ul style="list-style-type: none"> <li>• Switch off the instrument and remove the power cord.</li> <li>• Do not bring any electrical component into contact with fluids.</li> <li>• Do not operate the instrument when enclosure is open or removed.</li> </ul>

	<b>⚠ CAUTION</b>
	<p>Risk of burnings by hot machine parts and water steam.</p> <ul style="list-style-type: none"> <li>• Let the system cool down for 30 minutes before performing any service.</li> <li>• Do not operate the instrument when enclosure is open or removed.</li> </ul>

	<b>⚠ WARNING</b>
	<p>Death or serious burnings by flammable vapors.</p> <ul style="list-style-type: none"> <li>• Remove all sources of flammable vapor.</li> <li>• Avoid spark formation.</li> <li>• Do not operate with open flames.</li> </ul>

  	<b>⚠ WARNING</b>
	<p>Serious chemical burnings by corrosives.</p> <ul style="list-style-type: none"> <li>• Observe supplementary data sheets of all used chemicals.</li> <li>• Wear protective goggles.</li> <li>• Wear protective gloves.</li> <li>• Wear protective clothing.</li> </ul>

### Note


*The service door may only be opened for maintenance or service purposes and to switch the stop-cock*

## 7.1 Daily maintenance

Regular cleaning is vital to ensure proper instrument functioning. Thus the deposits of caustic substances within the instrument (e.g. causing degradation of glassware) can be avoided.

### 7.1.1 Cleaning the housing

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

	<b>NOTICE</b>
	<p>Risk of housing damage by solvents and acids.</p> <ul style="list-style-type: none"><li>• Do not clean the housing with solvents.</li><li>• Wipe off any acid drops immediately.</li></ul>

Open the service door and perform a visual check of all parts inside. Make sure that there is no leaking at hoses and valves.

### 7.1.2 Cleaning the glass parts

To extend the lifetime of the glass parts, we recommend to perform a cleaning at the end of the day. For this purpose in the main menu select “Cleaning” and press the Start button. The system will start a distillation with 300 mL water for a period of 5 minutes.



### 7.1.3 Cleaning the rubber bung seal

Clean the seal with a damp cloth.

## 7.2 Monthly maintenance

### 7.2.1 Calibrating the pumps

We recommend to calibrate the pumps once a month or every 100 distillations. Before calibrating the pumps, make sure that the hoses are not bent inside the instrument. For a description on how to carry out the calibration, see chapter 6.4.1.

### 7.2.2 Checking the distillate amount

To check the distillate amount, wait until the steam generator is ready, i.e. the preheating has been performed. Then run a single sample with an empty tube and an empty receiving vessel with the following parameters:

**Table 7-1: Checking the distillate amount**

Parameter	Setting
Check distillate amount:	Distillation
Sample tube:	empty
Water:	0 mL
NaOH:	0 mL
Distillation time:	5:00

Measure the distillate amount by means of a measuring flask. With the above parameters it should be minimum 130 mL.

## 7.3 Maintenance as required

We recommend to replace the parts listed in this chapter depending on the instrument use or at least once a year.

### 7.3.1 Cleaning the splash protector


#### Glass splash protector:

It is recommended to unmount the splash protector if residues are visible in its upper part. It can be cleaned with commercially cleaning agent or in an ultrasonic bath.

#### Plastic splash protector:

It is recommended to unmount the splash protector if you see the blank values continually increasing. It can be cleaned with commercially cleaning agent or in an ultrasonic bath.

When unmounting the splash protector for cleaning, clean the seal as well to prolong its lifetime. Rinse it with water, dry it with a soft cloth, remount it and put the splash protector back in place.

	<b>NOTICE</b>
	<p>Risk of sealing damage by sharp edges or lubricants.</p> <ul style="list-style-type: none"> <li>• When installing, move sealing in perpendicular axis to the glass parts.</li> <li>• Do not touch sealing with potentially sharp objects.</li> <li>• Do not apply grease or any other lubricant to the sealing.</li> </ul>

For further information on how to unmount and remount the splash protector, see chapter 7.3.3.

### 7.3.2 Replacing the splash protector

Replace the Splash protector after approximately 5000 determinations.

### 7.3.3 Replacing the rubber bung seals and the splash protector

We recommend to replace the parts listed below depending on the instrument use, at least once a year.

The seals are subject to wear and tear, thus you should check them regularly. We recommend to replace them once a year. The following parts need to be replaced (see also the spare parts list):



- ① Accessories splash protector
- ② Gasket for splash protector, complete
- ③ SVL22 sealing



Make sure that the instrument is cooled down.  
Open the service door and loosen the two tubings shown on the picture.



Open the screw-type cap of the hose.

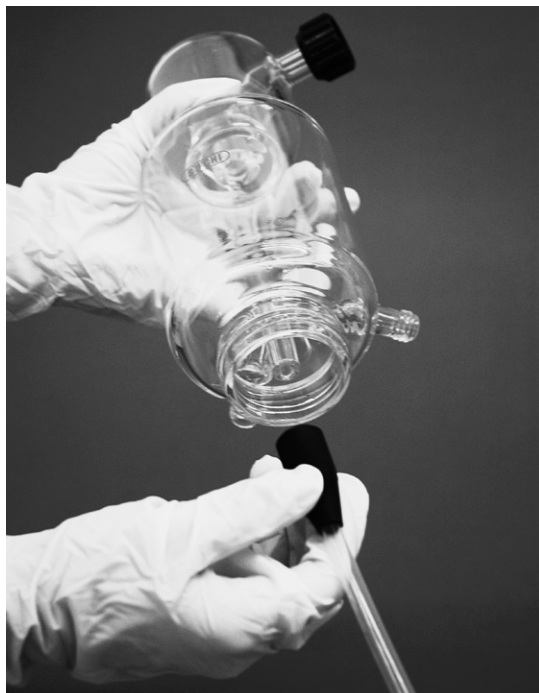


Now loosen the two screws with black head to remove the metal holder at the front side. Carefully remove the bracket.



Now unscrew the black plastic screw with a polygrip. Make sure to do this gently as you might break the glass parts. This part of the procedure is the most critical one. Carefully remove the splash protector.

Unscrew the gasket.



Pull out the steam tubing.



To replace the rubber parts pull off the connecting screw. The smaller sealing is located inside.

Fig. 7.1: Installing the glass assembly

Exchange all the rubber and plastic parts shown on the first picture of chapter 7.3.3. Put back the replaced parts in the reverse order and make sure to tighten the screw on top of the splash protector very carefully with the Polygrip.

#### 7.3.4 Glass parts

Replace the sample tubes and the condenser if broken.

#### 7.3.5 Replacing the hoses

Depending on the applications and the used chemicals, it might be necessary to replace some of the hoses inside of the instrument to avoid leakages or aspiration problems.

## 7.4 Yearly maintenance

### 7.4.1 Customer service

In order to avoid down times of a KjellFlex, we recommend to have the following parts replaced at least once a year by an authorized service person:

- Membranes of the aspiration module valves
- NaOH pump, the other pumps if required

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](http://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

### 7.4.2 Decalcification of the steam generator

To decalcify the steam generator proceed as follows:

1. Make sure that the steam generator is cooled down (switch off the unit and let it cool down for at least 30 minutes)
2. Remove the water of the steam generator (see 9.1 Emptying the steam generator)
3. Mix about 0.8 L of solution for decalcification (use e.g. approx. 160 g citric acid or approx. 80 g amidosulfonic acid dissolved in 0.8 L water)
4. Remove the hose from the H<sub>2</sub>O-inlet on the back of the instrument.
5. Connect a new hose to the H<sub>2</sub>O-inlet and put its free end into decalcification solution
6. Switch on the K-360
7. After initialization the pump starts running
8. Switch off the unit after the steam generator is filled up with the solution (pump stops running)
9. Let the solution dissolve the lime for 0.5 – 1 hour
10. Remove the solution of the steam generator (see step 1 and 2)
11. Perform a second decalcification (see step 6 – 10)
12. Connect the hose from the water tank with the inlet of H<sub>2</sub>O
13. Flush the steam generator 2 – 3 times with distilled water (see step 6 – 8 and 10)
14. Perform 2 – 3 times a CLEANING (cleaning method) of the instrument

## 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 malfunctions and their remedy**

Error number / Error indication	Possible cause	Corrective measure
0	The current process was stopped.	Press OK, aspirate manually and start again
1	Steam Valve (Y1 on) malfunction.	Contact the BUCHI customer service
2	Aspiration Out Valve (Y2 on) malfunction.	Contact the BUCHI customer service
3	Aspiration In Valve (Y3 on) malfunction.	Contact the BUCHI customer service
4	Receiver Valve (Y4 on) malfunction.	Contact the BUCHI customer service
5	Cooling Water Valve (Y5 on) malfunction.	Contact the BUCHI customer service
8	Injection Valve (Y8 on) malfunction.	Contact the BUCHI customer service
9	Water Valve (Y9 on) malfunction.	Contact the BUCHI customer service
11	Steam Valve (Y1 off) malfunction.	Contact the BUCHI customer service
12	Aspiration Out Valve (Y2 off) malfunction.	Contact the BUCHI customer service
13	Aspiration In Valve (Y3 off) malfunction.	Contact the BUCHI customer service
14	Receiver Valve (Y4 off) malfunction.	Contact the BUCHI customer service
15	Cooling Water Valve (Y5 off) malfunction.	Contact the BUCHI customer service
18	Injection Valve (Y8 off) malfunction.	Contact the BUCHI customer service
19	Water Valve (Y9 off) malfunction.	Contact the BUCHI customer service
20	H <sub>2</sub> O Pump (M1 on) malfunction.	Contact the BUCHI customer service
21	NaOH Pump (M2 on) malfunction.	Contact the BUCHI customer service
22	Reagent Pump (M3 on) malfunction.	Contact the BUCHI customer service
23	H <sub>3</sub> BO <sub>3</sub> Pump (M4 on) malfunction.	Contact the BUCHI customer service
25	H <sub>2</sub> O Pump (M1 off) malfunction.	Contact the BUCHI customer service
26	NaOH Pump (M2 off) malfunction.	Contact the BUCHI customer service
27	Reagent Pump (M3 off) malfunction.	Contact the BUCHI customer service
28	H <sub>3</sub> BO <sub>3</sub> Pump (M4 off) malfunction.	Contact the BUCHI customer service
30	Calibration out of range, check pump.	Check connection K-360/tank, check pump
33	The service door is open. If a process was in progress, it may have been stopped.	Close the service door, start again
34	The splash protector door is open. If a process was in progress, it may have been stopped.	Close the splash protector door, start again

**Table 8-1: General malfunctions and their remedy (cont.)**

Error number / Error indication	Possible cause	Corrective measure
35	There is no sample tube.	Sample tube present? Sample tube defective? Remove the sample tube and put it back again. Restart the process
37	Boric acid level is low. Do you want to continue determination?	Refill the boric acid tank
38	Water level for steam generator is low. Do you want to continue determination?	Refill the water tank for the system generator
39	Sodium hydroxide level is low. Do you want to continue determination?	Refill the sodium hydroxide tank
40	Waste tank is full. Do you want to continue determination?	Empty the waste tank
41	Titrator not ready. Retry?	Wait until the titrator is ready, try again
42	Not enough cooling water!	Cooling water connected? Cooling water flowing? Flow sensor may be defective Contact the Buchi customer service
43	Steam generator not ready.	Set status of steam generator to "Ready" mode until the steam generator is ready (up to 2 minutes)
44	Steam generator over temperature.	Check water connection
45	Cannot reach water level for steam generator.	Fill water tank, check H <sub>2</sub> O pump
46	Titrator not ready or not configured.	Configure titrator in Configuration menu
47	Dispenser not ready or not configured.	Configure dispenser in Configuration menu
48	The printer is not ready for printing. The current job will not be printed.	Switch on printer, check connection K-360/printer
49	Printer: No paper.	Add paper and start printing again
50	The printer is busy. The current job will not be printed.	Wait until the printer is ready and start printing again
51	The system is offline. To set the system online, go to Diagnostics.	Go to the Diagnostics menu and set the instrument online
52	Unit runs in demo mode.	Switch off Demo mode in the menu Configuration
53	Standby mode, switch to ready mode to heat steam generator.	Switch to Ready mode
54	Determination still in progress.	Wait until the active determination is finished and start again
55	Device cooled down! Priming or Preheating recommended. Do you want to continue determination?	Perform priming/preheating or continue determination
56	Battery voltage low.	Call the Buchi service to replace the battery
57	Unit supply voltage was too low! Please switch off unit and start again.	Check the power supply source in your lab
58	An internal error occurred! (Watchdog PCB) Please switch off unit and start again.	Contact the Buchi customer service

**Table 8-1: General malfunctions and their remedy (cont.)**

Error number / Error indication	Possible cause	Corrective measure
59	EEPROM, CPU	Contact the Buchi customer service
60	You cannot change the Standard Method!	Store the new parameter under a new method name
61	You cannot delete the Standard Method!	Do not try to delete the standard method
62	You have changed an existing method. Overwrite method?	Overwrite the existing method or store the new parameter under a new method name
63	The method list is full! You have to delete some methods.	Delete some methods and add new methods
64	Restart from Watchdog!	No corrective measure needed!
65	You changed between the demo and the normal mode.	Restart the system to activate the selected mode
66	Attention: Reposition stop-cock! (Reagent)	Check the correct position of the stop-cock
67	Attention: Reposition stop-cock! (NaOH)	Check the correct position of the stop-cock

## 8.2 Diagnostics

The K-360 software enables to carry out some operational, sensor, service and printer test and to view some hardware information and the operating hours.

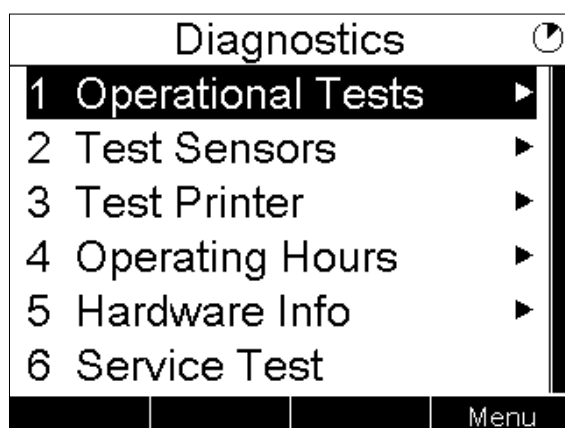


Fig. 8.1: Diagnostics

### 8.2.1 Operational Tests

This submenu enables to test the following components:

- Valves
- Pumps
- Stirrer
- Alarm
- Steam Unit Heating
- Titrator Out (Titrator suitable)
- Dispenser Out (Dispenser suitable)
- Battery Monitor



To carry out an Operational Test proceed as follows:

- Set the system offline by pressing the corresponding button.
- Go to the part you want to test, e.g. Steam valve and switch it on by pressing the forward or the backward button.
- If the valve works properly, a press sound is audible.
- Switch the valve off.
- Set the system online again.

**NOTE**

*The button „Sensors“ enables to switch to the submenu Test Sensors.*

### 8.2.2 Test sensors

This submenu enables to test the sensor functionality.

To test e.g. the tube shield sensor, proceed as follows:

- Open the tube shield. When the sensor is working properly “Open” will be indicated on the display.
- Close the tube shield. Now “Close” has to be indicated on the display.

**NOTE**

*The button „Actors“ enables to switch to the submenu Operational Tests.*

### 8.2.3 Test Printer

This submenu enables to test the functionality of the printer by e.g. printing a test page.

### 8.2.4 Operating Hours

This submenu indicates the operating hours of the steam generator, the number of distillations and the date and time of the last reset.

### 8.2.5 Hardware Info

This submenu indicates some hardware information, e.g. part and serial numbers and the production date of the instrument.

### 8.2.6 Service Test







This submenu enables the instrument manufacturers or service technicians to carry out a functionality test of the instrument.

## 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 transport

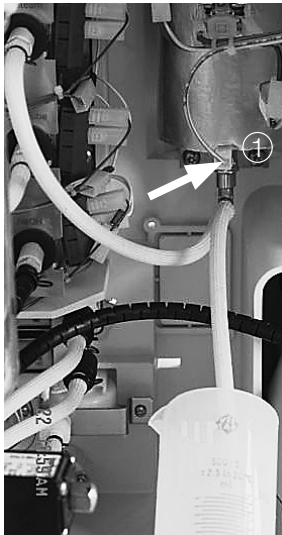
Take out the power cord and remove all water/reagent hoses. Rinse the sodium hydroxide pump/reagent pump/boric acid pump hose with distilled water. Clean the instrument thoroughly, so that all chemical residues are removed completely.

	<p><b>! WARNING</b></p> <p>Death or serious injuries by electric shock.</p> <ul style="list-style-type: none"> <li>• Switch off the instrument and remove the power cord.</li> <li>• Do not bring any electrical component into contact with fluids.</li> <li>• Do not operate the instrument when enclosure is open or removed.</li> </ul>
	<p><b>! CAUTION</b></p> <p>Risk of burnings by hot machine parts and water steam.</p> <ul style="list-style-type: none"> <li>• Let the system and the steam generator cool down for 30 minutes before performing any service.</li> <li>• Do not operate the instrument when enclosure is open or removed.</li> </ul>
	<p><b>! WARNING</b></p> <p>Death or serious burnings by flammable vapors.</p> <ul style="list-style-type: none"> <li>• Remove all sources of flammable vapor.</li> <li>• Avoid spark formation.</li> <li>• Do not operate with open flames.</li> </ul>
  	<p><b>! WARNING</b></p> <p>Serious chemical burnings by corrosives.</p> <ul style="list-style-type: none"> <li>• Observe supplementary data sheets of all used chemicals.</li> <li>• Wear protective goggles.</li> <li>• Wear protective gloves.</li> <li>• Wear protective clothing.</li> </ul>

To empty the steam generator, proceed as follows:

- Turn off the instrument.
- Let the steam generator cool down for 30 minutes.

- Open the service door.
- Attach an appropriate silicon hose to the drain cock at the steam generator.
- Insert the silicone hose into a vessel with at least 500 mL volume.
- Slowly open the screw (marked with an arrow in the figure below) with a screw driver and completely empty the steam generator.
- Close the stop-cock with a screw driver.



① Drain with screw

Fig. 9.1: Emptying the steam generator

## 9.2 Storage and transport

Store and transport the instrument in its original packaging.

## 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.

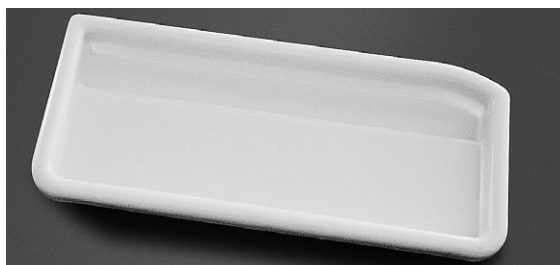
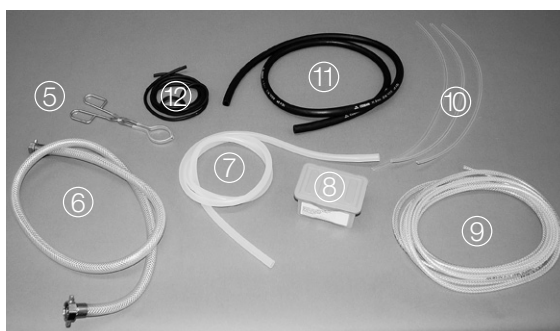
Use only genuine BUCHI consumables and genuine spare parts for maintenance and repair to assure good system performance and reliability. Any modifications to the spare parts used are only allowed with the prior written permission of the manufacturer.

### 10.1 Spare parts K-360



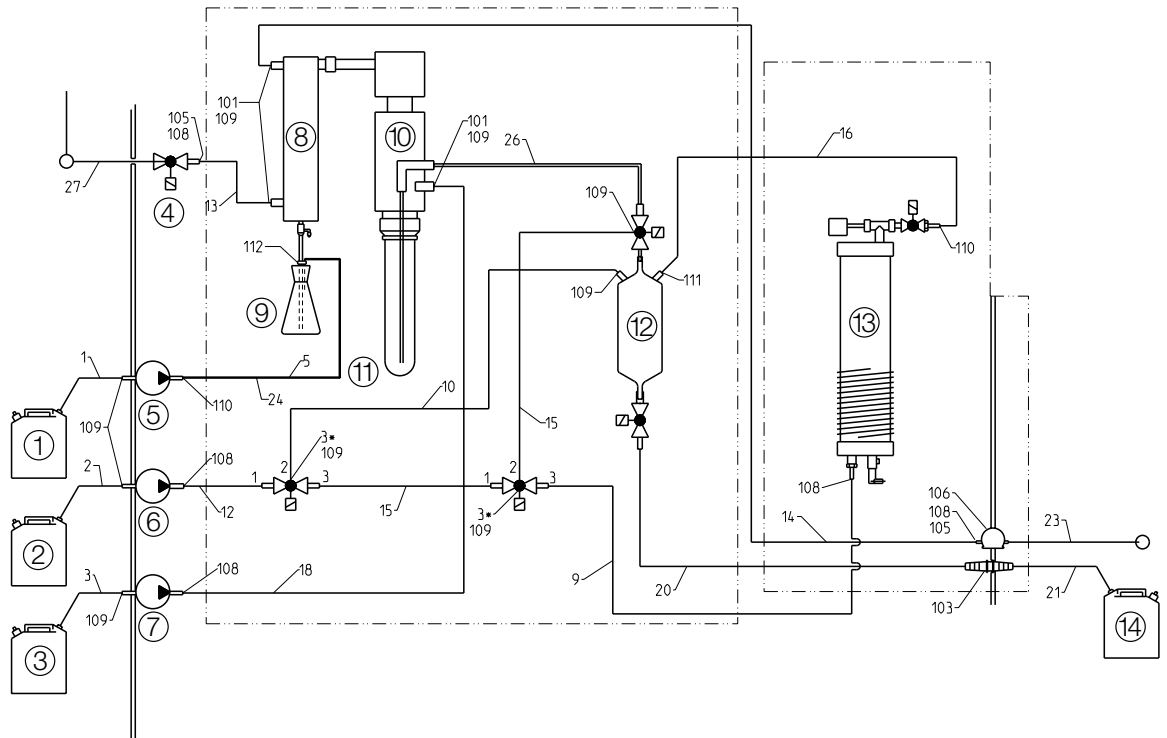
**Table 10-1: Spare parts K-360**

Product	Order number
① Splash protector for Devarda method	43335
② Splash protector glass	43332
③ Splash protector, polypropylene	43590
④ Accessories splash protector	43187
⑤ Gasket (splash protector) complete, 2 pieces	43065
⑥ SVL22 sealing	02073
⑦ Condenser K-360	43320
⑧ Check valve, complete	43356
⑨ Condenser cover, complete	43484
⑩ Stirrer shank	43316
⑪ Stirrer rotor blade	43466
⑫ Stirrer, complete	43080

**Table 10-1: Spare parts K-360 cont.**

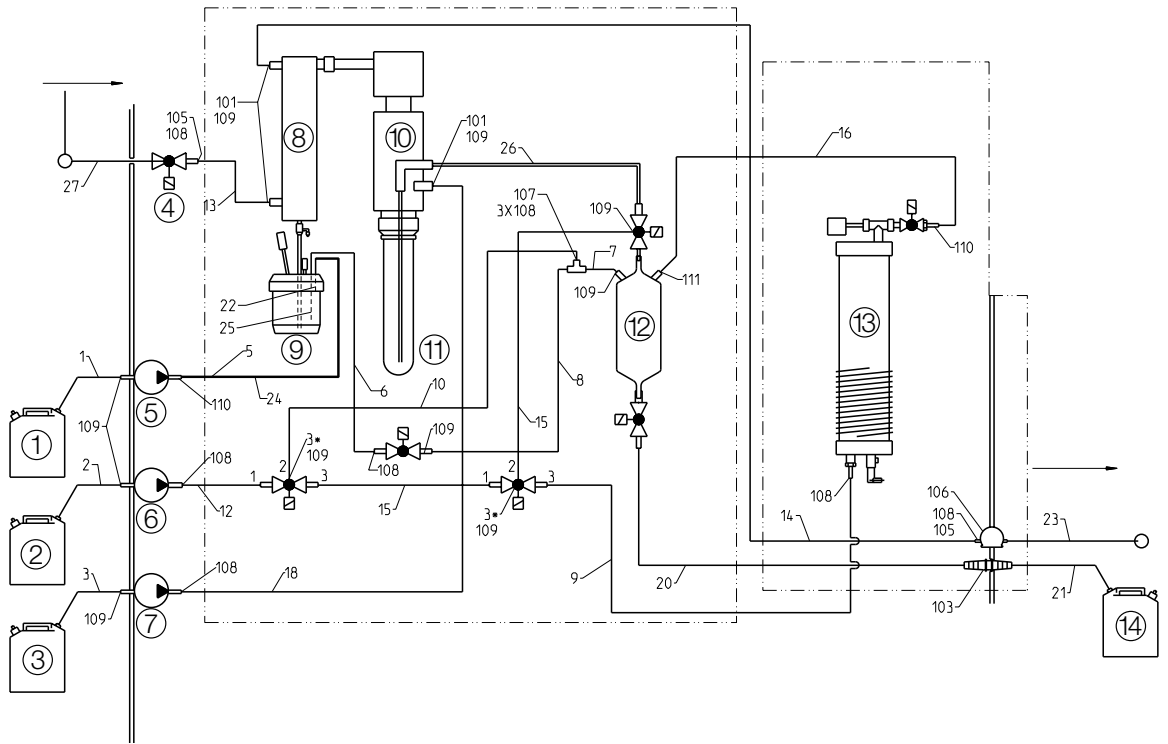
Product	Order number
① Set of sample tubes (set of 4), 500 mL	43982
② Set of sample tubes (set of 4), 300 mL	37377
③ Receiving vessel 420 mL	43390
④ Receiving vessel 340 mL	43333
⑤ Pair of glass tongs	02004
⑥ Cooling water hose complete: G 3/4", 1/2", L = 1.5 m	37780
⑦ Hose cooling water drain, silicone, L = 1.8 m, Ø 14/10 mm	43439
⑧ Weighing boat	40444
⑨ Hose chemical supply, Solaflex, L = 6 m, Ø 10/5 mm	43185
⑩ 3 suction hoses to tanks, FEP, L = 580 mm	43407
⑪ Hose waste drain, EPDM, L = 1.8 m, Ø 18/11 mm	43457
⑫ Hose Viton (acid resistant), L = 1.2 m, Ø 6/3 mm for acid resistant pump	43693
6 clamps Ø 11.9	43841
Tank 10 L	43408
Tank 20 L	43410
Cap for 10 L tanks, large	25869
12 tank labels	43434
Cap for 10 L and 20 L tanks, small	43477
Drip tray	43302
Set of sealings EPDM / NBR for cooling water hose	40043
Sealing special complete (to universal adapter)	43116
Aspiration hose special, PTFE (universal adapter)	43119

## 10.2 Hosing connection schemes



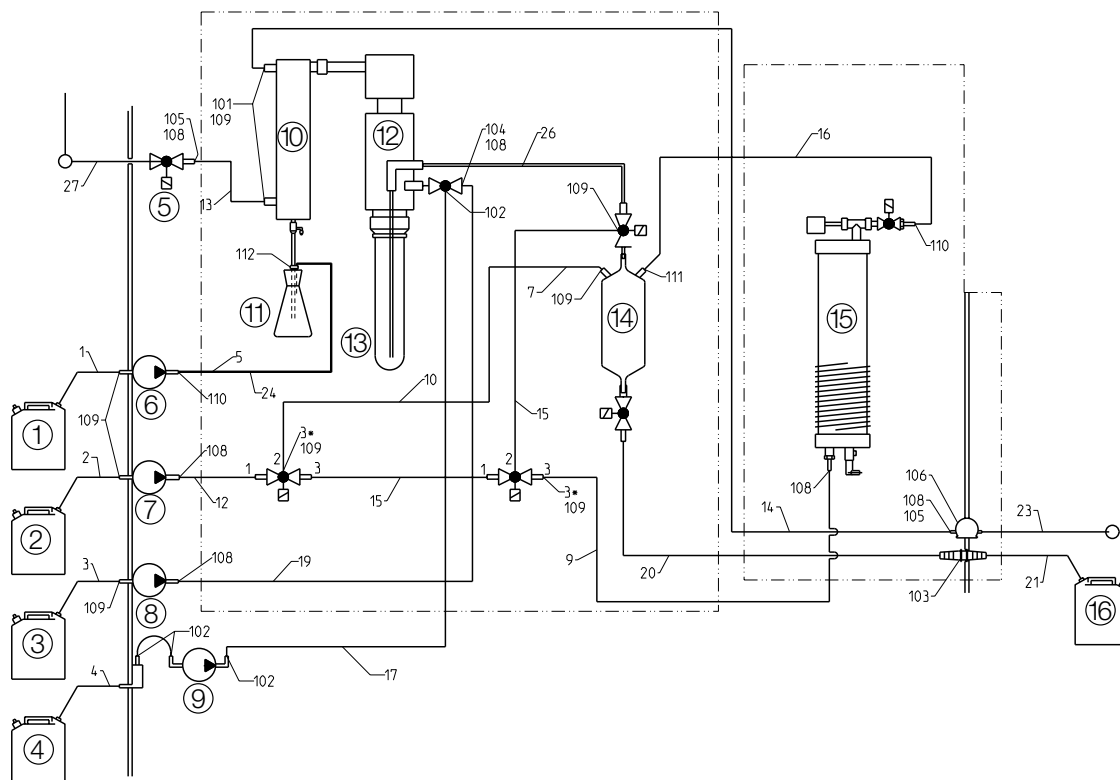
- |   |                    |
|---|--------------------|
| ① Boric acid tank                       | ⑧ Condenser        |
| ② H <sub>2</sub> O tank                 | ⑨ Receiving vessel |
| ③ NaOH tank                             | ⑩ Splash protector |
| ④ Cooling water valve in                | ⑪ Sample tube      |
| ⑤ Boric acid pump                       | ⑫ Expansion vessel |
| ⑥ H <sub>2</sub> O steam generator pump | ⑬ Steam generator  |
| ⑦ NaOH pump                             | ⑭ Waste tank       |

Fig. 10.1: Hosing connection scheme Standard



- |   |                                    |
|---|------------------------------------|
| ① Boric acid tank                       | ⑧ Condenser                        |
| ② H <sub>2</sub> O tank                 | ⑨ Receiving vessel (Titration set) |
| ③ NaOH tank                             | ⑩ Splash protector                 |
| ④ Cooling water valve in                | ⑪ Sample tube                      |
| ⑤ Boric acid pump                       | ⑫ Expansion vessel                 |
| ⑥ H <sub>2</sub> O steam generator pump | ⑬ Steam generator                  |
| ⑦ NaOH pump                             | ⑭ Waste tank                       |

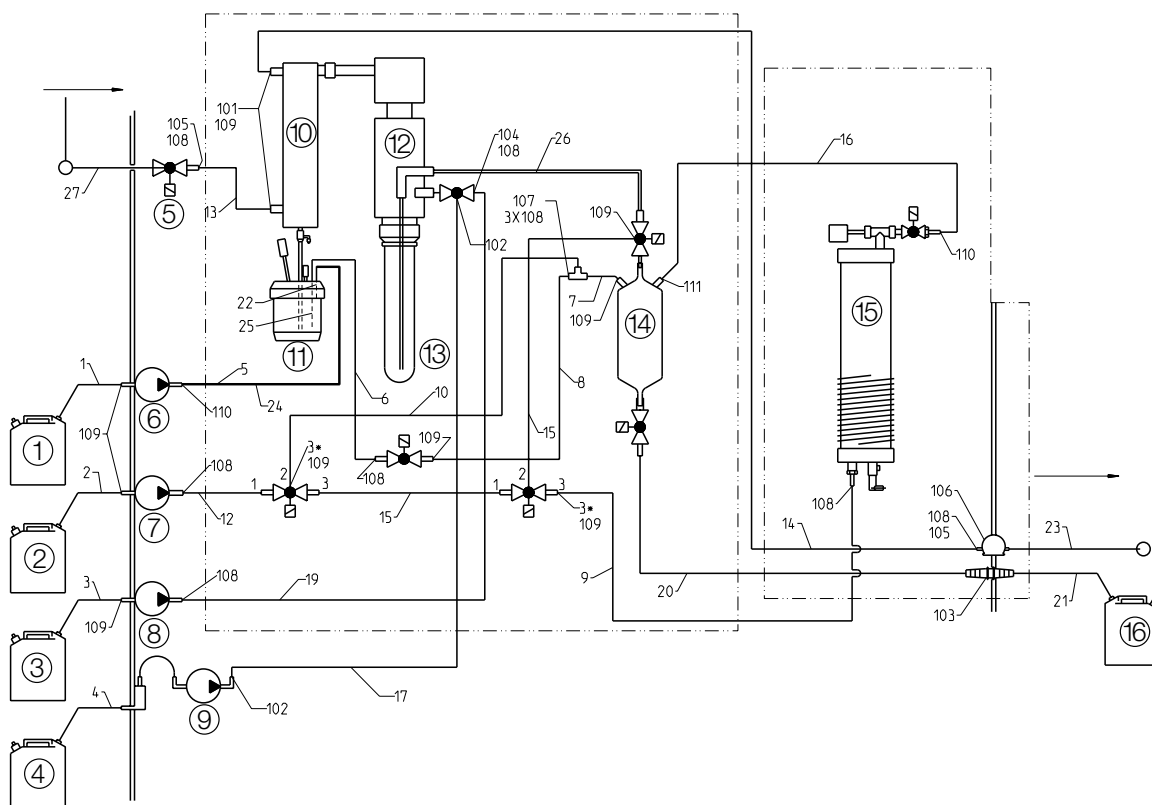
Fig. 10.2: Hosing connection scheme Titration



- |   |                    |
|---|--------------------|
| ① Boric acid tank                       | ⑨ Reagent pump     |
| ② H <sub>2</sub> O tank                 | ⑩ Condenser        |
| ③ NaOH tank                             | ⑪ Receiving vessel |
| ④ Storage bottle for reagent            | ⑫ Splash protector |
| ⑤ Cooling water valve in                | ⑬ Sample tube      |
| ⑥ Boric acid pump                       | ⑭ Expansion vessel |
| ⑦ H <sub>2</sub> O steam generator pump | ⑮ Steam generator  |
| ⑧ NaOH pump                             | ⑯ Waste tank       |

Fig. 10.3: Hosing connection scheme Standard with acid-resistant pump





- |   |                                    |
|---|------------------------------------|
| ① Boric acid tank                       | ⑨ Reagent pump                     |
| ② H <sub>2</sub> O tank                 | ⑩ Condenser                        |
| ③ NaOH tank                             | ⑪ Receiving vessel (Titration set) |
| ④ Storage bottle for reagent            | ⑫ Splash protector                 |
| ⑤ Cooling water valve in                | ⑬ Sample tube                      |
| ⑥ Boric acid pump                       | ⑭ Expansion vessel                 |
| ⑦ H <sub>2</sub> O steam generator pump | ⑮ Steam generator                  |
| ⑧ NaOH pump                             | ⑯ Waste tank                       |

Fig. 10.4: Hosing connection scheme with acid-resistant pump and titration

**Table 10-2: Hoses**

Position	Order number	Description	10.1	10.2	10.3	10.4
1						
2	43185	Solaflex hose Ø 10/5, L = 6000	1	1	1	1
3						
4	43693	Viton Ø 6/3, L = 1200			1	1
5	43157	Tygon Ø 8/4.8, L = 1030	1	1	1	1
6	43156	Tygon Ø 8/4.8, L = 350		1		1
7	43149	Unisil Ø 10/6, L = 85		1		1
8	43150	Unisil Ø 10/6, L = 250		1		1
9	43155	Unisil Ø 10/6, L = 350	1	1	1	1
10	43151	Unisil Ø 10/6, L = 370	1	1	1	1
12	43152	Unisil Ø 10/6, L = 560	1	1	1	1
13	43153	Unisil Ø 10/6, L = 640	1	1	1	1
14	43154	Unisil Ø 10/6, L = 920	1	1	1	1
15	43689	Unisil Ø 10/6, L = 970	2	2	2	2
16	43690	Tersil Ø 12/6, L = 300	1	1	1	1
17	43691	Viton Ø 6/3, L = 730			1	1
18	43029	EPDM Ø 6/2.8, L = 530	1	1		
19	43692	EPDM Ø 6/2.8, L = 820			1	1
20	43438	EPDM Ø 18.2/11 L = 340	1	1	1	1
21	43457	EPDM Ø 18.2/11 L = 1800	1	1	1	1
22	43366	FEP Ø 6/4, L = 40		1		1
23	43439	Silicone Ø 14/10, L = 1800	1	1	1	1
24	43184	Spiral band, L = 760	1	1	1	1
25	43321	Suction hose		1		1
26	43344	Suction hose, splash protector	1	1	1	1
27	37780	Cooling water hose, complete	1	1	1	1

**Table 10-3: Small parts**

Position	Order number	Description	Var 1	Var 2	Var 3	Var 4
101	43139	Hose clip, bent, GL14, EPDM	3	3	2	2
102	03562	Cable binder 2.5 x 98 mm			4	4
103	43205	Hose coupling	1	1	1	1
104	43660	Stop-cock			1	1
105	43207	Hose connector	2	2	2	2
106	29646	Impeller meter	1	1	1	1
107	43582	T-piece, PVDF		1		1
108	27738	Hose clamp Ø 9.6	6		11	1
109	43202	Hose clamp Ø 10.7	12	12	12	12
110	43841	Hose clamp Ø 11.9	1	1	1	1
111	43297	Hose clamp Ø 7.6	1	1	1	1
112	14295	Cable tie 4.8 x 178 mm	1		1	

# 11 Declarations and requirements

## 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 commercial.

Cet appareil génère, utilise et peut irradier une énergie à fréquence radioélectrique, il est en outre susceptible d'engendrer des interférences avec les communications radio, s'il n'est pas installé et utilisé conformément aux instructions du mode d'emploi. L'utilisation de cet appareil dans 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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