LAB Online Exhibition

Operation Manual

knowledge

Action movie

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Contents

Please read these instructions thoroughly before using the **BÜCHI** Melting Point **B-545** Unit. The instructions should be kept near the unit for general reference.

Chapter 2 contains important notes on safety which must be observed if the Melting Point Unit is to be operated safely.

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en, Version G (58 pages)	**Order No.**
B-545 Instructions	**96630**

1 Scope of delivery

Figure 1: Complete view of the unit

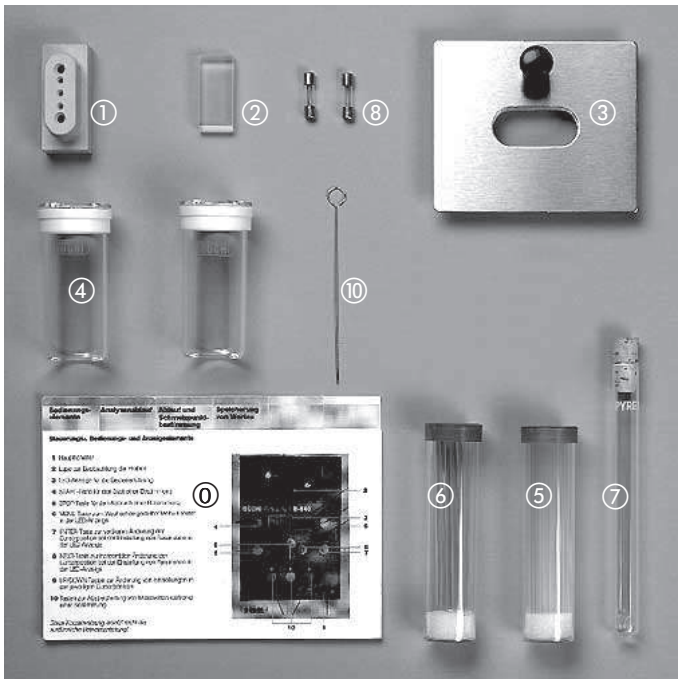


Figure 2: Accessories

Description

Appliance models:	**Order No.**
Melting Point Unit B-545, 220–230 V, 50 or 60 Hz	**36408**
Melting Point Unit B-545, 100–120 V, 50 or 60 Hz	**36582**

Table 1: Versions of the unit

Accessories:	**Order No.**
① 1 Oven, upper part of ceramic	**34564**
② 1 Glass panel	**34236**
③ 1 Metal cover for heating range	**36569**
④ 2 Sample holders of glass	**36110**
⑤ 1 Set of 100 melting point capillaries, each	**17808**
⑥ 1 Set of 10 boiling point tubes, each	**19697**
⑦ 1 Set of 10 boiling point capillaries, each	**19698**
⑧ 2 Spare fuses for 230 V	**01657**
2 Spare fuses for 120 V	**02998**
⑨ 1 Spare bulb	**29573**
1 Mains cable PNE, 2.5 m:	
Type CH	**10010**
Type earthed (shock-proof)	**10016**
Type GB	**17835**
Type USA	**10020**
Type AUS	**17836**
1 Capillary holder of plastic	**36629**
⓪ 1 Brief operating instructions:	
German	**96635**
Englisch	**96636**
French	**96661**
⑩ 2 Compaction wires	**36721**
1 Protective hood	**36625**
1 Operating instructions manual	
German	**96630**
English	**96631**
French	**96632**
Italian	**96633**
Spanish	**96634**
1 Packaging	

Table 2: Enclosed parts

	Optional accessories:	**Order No.**
1	Keyboard for simple parameter input:	
	Swiss keyboard arrangement	**29599**
	USA keyboard arrangement	**29548**
1	Printer for documentation	
	230 V, 50/60 Hz	**38706**
	115 V, 60 Hz	**38707**
1	Printer cable	**29547**
1	Melting point capillary (1000 off)	**01759**
1	Boiling Boiling point tube (100 off)	**19007**
1	Boiling point capillary (100 off)	**01762**
1	Büchi Certificate for Calibration at Works G/E/F	**97722**

Table 3: Optional accessories

2 Safety

This unit has been designed in accordance with the state of engineering and the recognised rules of safety. Nevertheless, it can still be a source of risks and hazards:

- If used by persons with insufficient training
- If used for purposes other than that for which it is intended.

2.1 Symbols

Stop
Information about hazards which can lead to serious material damage or cause serious or potentially fatal injury.

Warning
Information about hazards which can be harmful to your health or lead to material damage.

Please note
Information about technical requirements. Non-observance can lead to malfunctions, inefficiency and lost production.

2.2 Requirements to be met by the customer

The device must only be operated by persons who have the training or professional experience to comprehend the hazards that can arise when using the unit.
Personnel without such training or persons currently undergoing a course of training require thorough instruction. This instruction Manual should form the basis of these instructions.

2.3 Proper use

The unit is designed and built for use in laboratories. It is intended to be used to determine the melting and boiling points and melting ranges at ambient temperatures of up to 400°C.

2.4 Improper use

Any use other than those already listed, and any application that does not conform with the technical data is deemed to be a case of mis-use. The customer shall bear sole responsibility for any damage due to such mis-use.

The following applications in particular are inadmissible:
- Use in rooms requiring an explosion-proof equipment.
- Extraction of samples which may explode or ignite by ablow, friction, heat or spark (e.g. explosives, etc.).

2.5 General hazards

General hazards arise from:
- Mixtures of unknown composition or contaminations.
- Combustible gases or solvent vapours in the immediate vicinity of the unit.
- Damaged glass components.
- An insufficient distance from the back of the unit to the wall (see chapter 4, Commissioning the unit).
- Burning by touching hot parts of the heater.

Apart from authorised maintenance personnel, no one is allowed to remove any safety devices and covers with a standard tool. The unit must not be put into operation with damaged glass components.

Touching life parts may result in fatal injury!

2.6 Safety measures

Personal protective garments must be worn, such as goggles and a laboratory coat.
This Instruction Manual is a component of the melting point unit and as such it must remain available at any time to the operating staff where the unit is operated. This also applies to manuals in other languages which are available on request.

2.7 Modifications

Modifications on the unit or on the spare parts or accessories as well as the use of any other spare parts or accessories as those indicated in this Manual are only permitted after consulting with Büchi Labortechnik AG and obtaining its written consent.

2.8 Responsibility of the operator

The operator is responsible for instructing his personnel. For that purpose, this Manual can be supplied in other languages. The operator shall inform the manufacturer without delay on any events relevant to safety which may occur with operating the Melting Point Unit.

3 Function

The Melting Point Unit B-545 is a unit to determine melting and boiling points at ambient temperatures of up to 400° C. Three different samples can be determined at the same time. Values can be detected by viewing the samples.

3.1 Operating principle

Each of the three samples is located within a light barrier. If the sample is still crystalline, it will be opaque to light rays (= transmission 0%). The clear molten sample however is transparent and this condition is recognised by the unit as being the melting point. Apart from the specific temperature value, the gradient of the transmission curve is stored as well and is thus available for an interpretation of results.

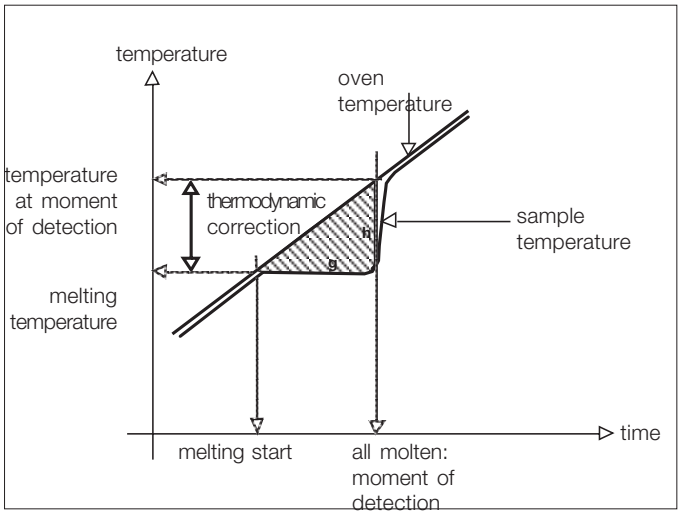


Figure 3a: Steep ramp

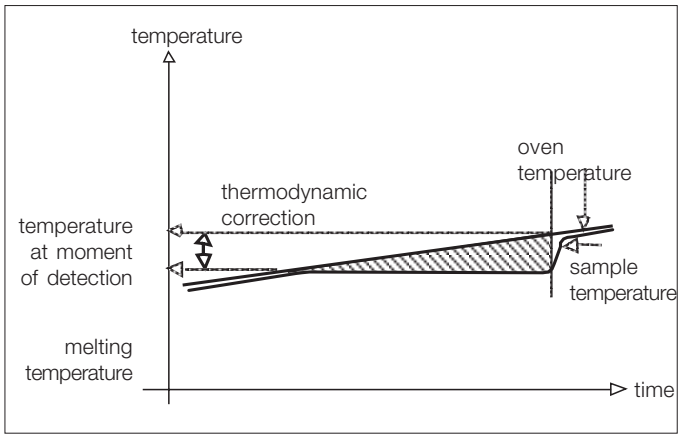


Figure 3b: Flat ramp

1. Thermodynamic correction of the automatically determinedmelting point

In its standard configuration, the unit supplies, together with the automatically determined melting points, the values which have been corrected thermodynamically. In order to obtain the correct melting temperature, it is therefore necessary to subtract the thermodynamic correction from the detected temperature.

Melting temperature = detected temperature -K • $\sqrt{r}$, r being the ramp ascent in centigrades/min.

2. Melting point values according to pharmacopoeia

The thermodynamic correction can be eliminated in the configuration menu (see Chapter 5.9, Adjustment of subparameters). With the correction eliminated, the values correspond to the current oven temperature and are as such dependent, to a certain extent, on the rising speed of the temperature ramp.

The type of determined values is printed out on the record (thermodynamical or pharmacopoeia).

4 Putting into operation

Check the unit for any damages immediately after opening the packaging. It is important that damage incurred during transit is determined and reported immediately. If this is the case, a full statement of facts must be made immediately (and reported to the post office, railway or transport firm).

It is advisable to keep the original packaging for future transport.

4.1 Location of unit

The unit must be erected on a stable, clean and even surface.

For safety reasons and to ensure sufficient cooling in the electronic compartment, the side walls of the unit must be placed at least 30 cm away from the walls or other objects. No containers, chemicals or other appliances should be placed behind a unit.

4.2 Mounting of accessories

The sample holders of glass are inserted into the opening of the housing cover.

Figure 4: Mounting the glass sample holder

The sample holders of glass protect the unit from dust and small particles as well as the user from touching electrical parts.

Therefore, never put the unit into operation without the sample holders inserted.

The glass panel is placed into the metal heating block, taking care to touch the panel only laterally.

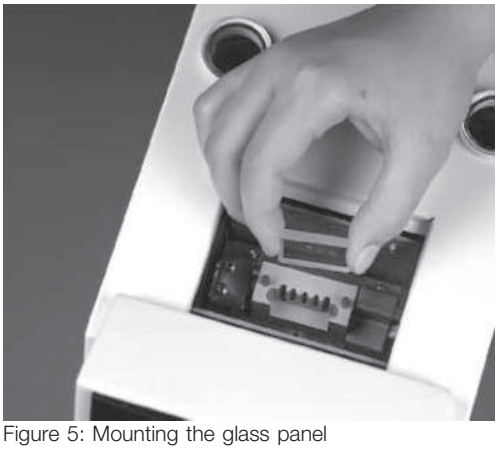


Figure 5: Mounting the glass panel

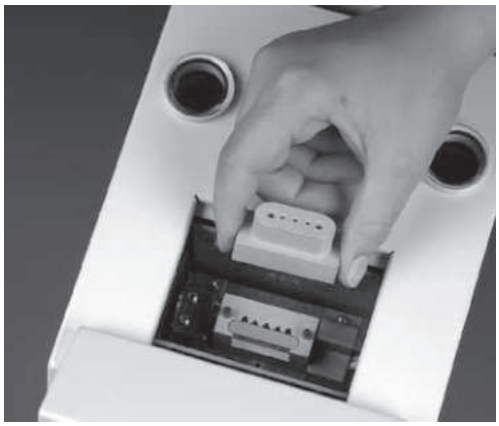


Figure 6: Inserting the upper ceramic part

The upper ceramic part of the oven is placed onto the metal block.

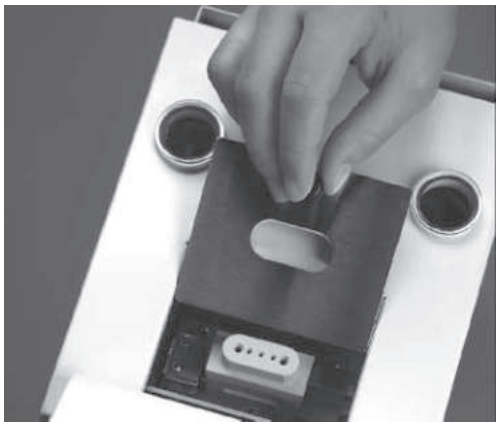


Figure 7: Fitting the metal cover

The metal cover is placed over the heating area.

4.3 Power connections

Check that the voltage of the socket is identical to the voltage specified on the unit plate. Only connect the Melting Point Unit to a plug which is grounded.

External couplings and extension cables must be grounded (3-contact couplings, cables and connectors). Any interruption of the earth wire is prohibited. Risks caused by possible internal defects are thus prevented.

4.4 Instruction: Calibration

The apparatus must be calibrated by the user before being used to determine melting points. The calibration substances set supplied should be used for this.

Chapter 5 of these operating instructions should be read before the calibration. Calibration is then carried out in accordance with section 6.3 of these operating instructions.

5 Operation

Please note that the unit has been commissioned correctly according to the instructions in chapter 4 Commissioning.

5.1 Control, operating and display elements

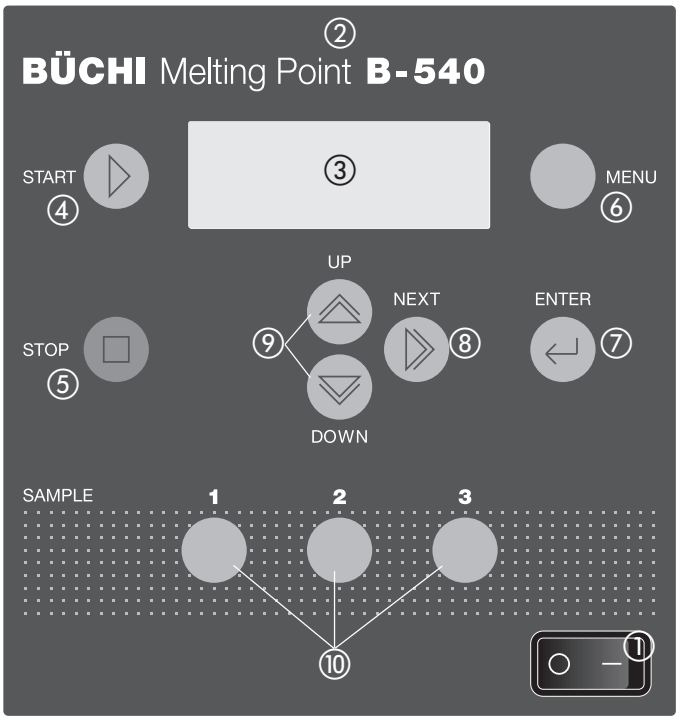


Figure 8: Front view, B-545

① Master switch
② Magnifying glass for viewing the samples
③ LED display for prompt facility
④ START key to start a determination
⑤ STOP key to interrupt a determination
⑥ MENU key to change displayed menu windows
⑦ ENTER key to change the cursor position vertically when adjusting parameters on the display
⑧ NEXT key to change the cursor position horizontally when adjusting parameters on the display
⑨ UP/DOWN keys to change adjustments of the relative cursor position
⑩ Keys to store measured values during a determination

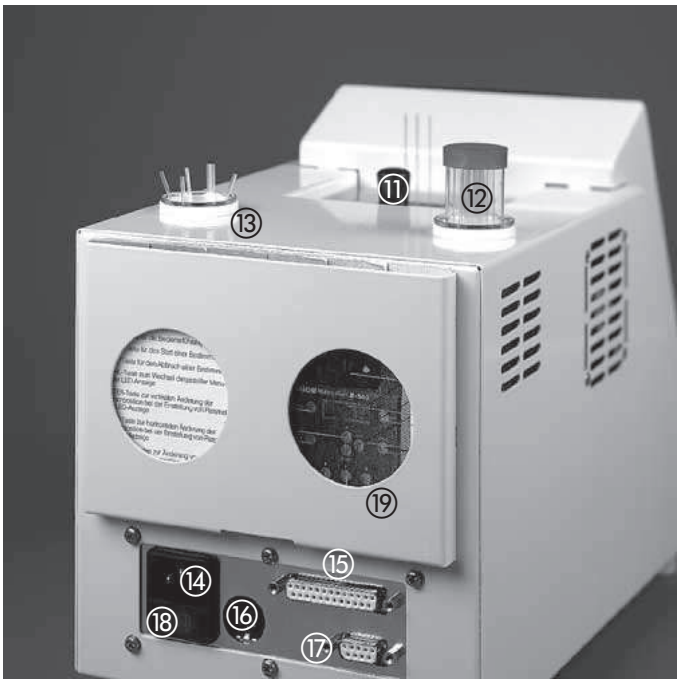


Figure 9: Rear view, B-545

⑪ Sample holder for melting point capillaries and boiling point tubes during determination
⑫ Receptacle for unused melting point capillaries and boiling point tubes
⑬ Receptacle for used melting point capillaries and boiling point tubes
⑭ Appliance plug
⑮ Plug for printer connection
⑯ Plug for keyboard connection
⑰ Plug for PC connection
⑱ Main fuse
⑲ Pocket for brief instruction manual

5.2 Operating setup of the menus

5.2.1 Main menus

The user may move through these menus for every new melting point determination.

Main menu 1

TEMPERATURE	:	25.0°C
SETPOINT	:	100°C
GRADIENT	:	2.0°C
MAXPOINT	:	120°C

Main menu 2

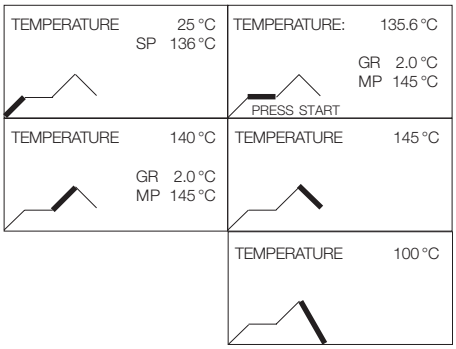
TEMPERATURE	:	25.0°C
NAME	:	
ID#	:	

Main menu 3

TEMPERATURE	:	25.0°C
RECALL		CLEAR
STORE		PRINT

5.2.2 Graphics

These graphics will accompany the user during a determination and explain which part of the heatingprofile is activated.



5.3 Definition of concepts on displays

5.3.1 Main menus

TEMPERATURE	:	25.0°C
SETPOINT	:	100°C
GRADIENT	:	2.0°C
MAXPOINT	:	120°C

Basic condition (main menu 1)

TEMPERATURE: Current temperature inside of the oven.

SETPOINT: Temperature
As from which the adjusted temperature program will run down.

GRADIENT: Temperature rise from 0.1/0.2/0.5/1.0/2.0/3.0/5.0/10.0/20° C/min, at which the unit will be heated up as from SETPOINT to MAXPOINT.

MAXPOINT: Maximum temperature to be reached for the current determination. This is set automatically at 15°C above the SETPOINT but may also be adapted.

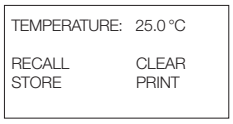
TEMPERATURE	:	25.0°C
NAME	:	
ID#	:	

Storage menus (main menu 2)

Temperature: Current temperature inside of the oven.

Name: Name of the method under which the parameters are to be stored for a determination.

ID#: Assignment of a sample name to a current determination which should appear equally on a subsequent printout.

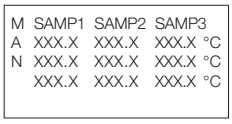


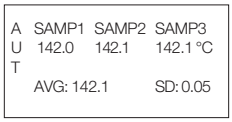
(Main menu 3)

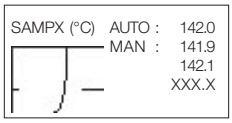
Temperature: Current temperature inside of the oven.
RECALL: Calling stored methods for determination
STORE: For storing new methods for determination
CLEAR: For cancelling stored methods of a determination.
PRINT: For switching on and off the automatic printout of the values obtained from a determination.

5.3.2 Display of values

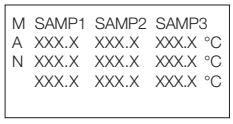
Melting point

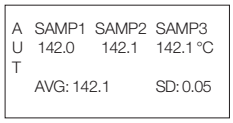


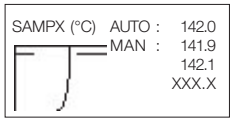




Melting range







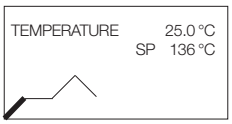
Manual display
This display permits to read off the melting points after a determination. A maximum of three values will be displayed per sample capillary which are arranged in vertical order.

Automatic display
The automatically detected values for melting point determination as well as the average value (AVG) and standard deviation (SD) are listed on this display.

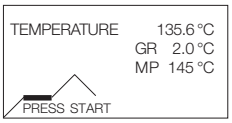
Display of the manually and automatically detected results
In this display the manually and automatically detected values are indicated. Furthermore the curve of the transmission is shown.

The automatic determination has been performed thermodynamically. If it had been performed due to Pharmacopoeia, there would have been indicated AUTP instead of AUT.

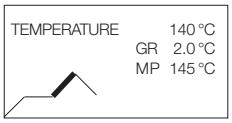
5.3.3 Graphics



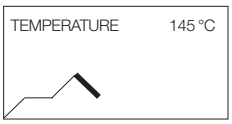
The unit heats up to the preselected SETPOINT.



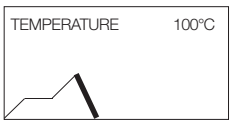
The unit has reached the preselected SETPOINT and waits for the temperature program to be started.



With the preselected GRADIENT, the unit heats up to the maximum point of the temperature program (MAXPOINT).



The unit has reached the highest point of the temperature program (MAXPOINT) and cools down to the previously selected SETPOINT.



The unit cools down to ambient temperature.

5.4 Preparation of samples

Granular crystalline and non-homogenous samples are crushed in the mortar and then filled into the melting point capillary to at least 4-6 mm. The use of Büchi capillaries is recommended (Item No. 01759). We very strongly advise against the use of capillaries made by other manufacturers because these capillaries usually have different diameters which can seriously affect the quality of the measurement values.

In order to obtain comparable results, it is essential that all three capillaries are filled to the same height and that the substance is well compacted in the capillaries.

Compaction in the capillary is obtained by:
- Knocking the capillary on a hard base
- Letting the capillary drop onto the table through a glass tube of approx. 1 m length (cleaning after the glass tube!)
- Compress sample with compaction wire

5.5 Determination of a melting point or a melting range

The following items must be known before a melting point can be determined:

- Is the approximate melting point known?
  if not → see 5.6

- Is a sample name to be entered?
  if yes → see 5.6.1

- Where is the SETPOINT of the determination located?
  SETPOINT input → see 5.5.1

- How fast should the temperature rise (GRADIENT) between SETPOINT and MAXPOINT?
  GRADIENT input → see 5.5.2

- Is the difference between SETPOINT and MAXPOINT to be more than 15° C?
  Change MAXPOINT → see 5.5.3

Substance with known melting point / range

Procedure for determining the melting point of a known substance.

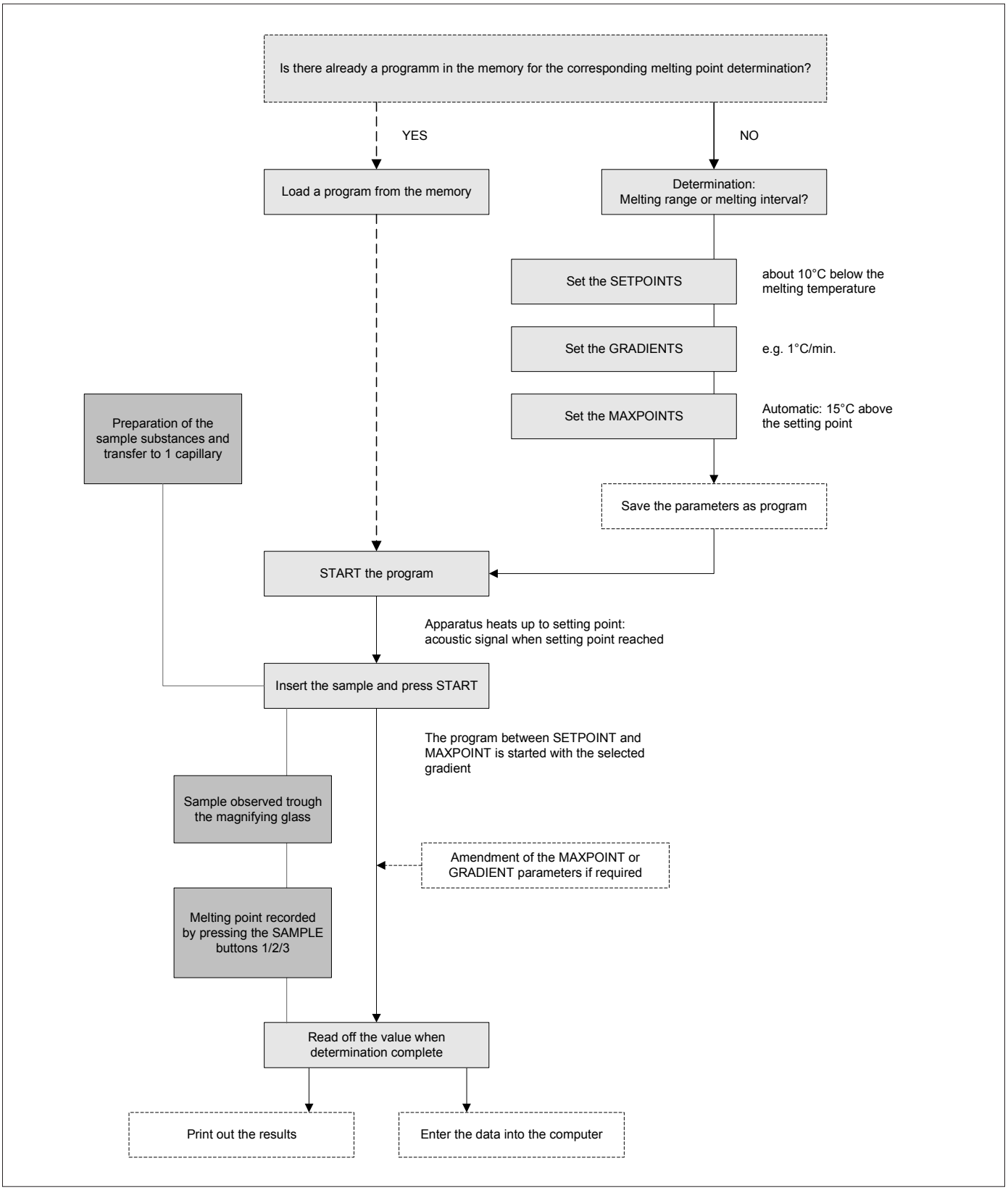


Figure 10: Flow chart of the melting point determination of a known substance

Substance with unknown melting point/range

If the approximate melting point of a substance is unknown, the rough position of the melting point is estimated before the actual melting point determination. The actual melting point can then be determined (see previous page).

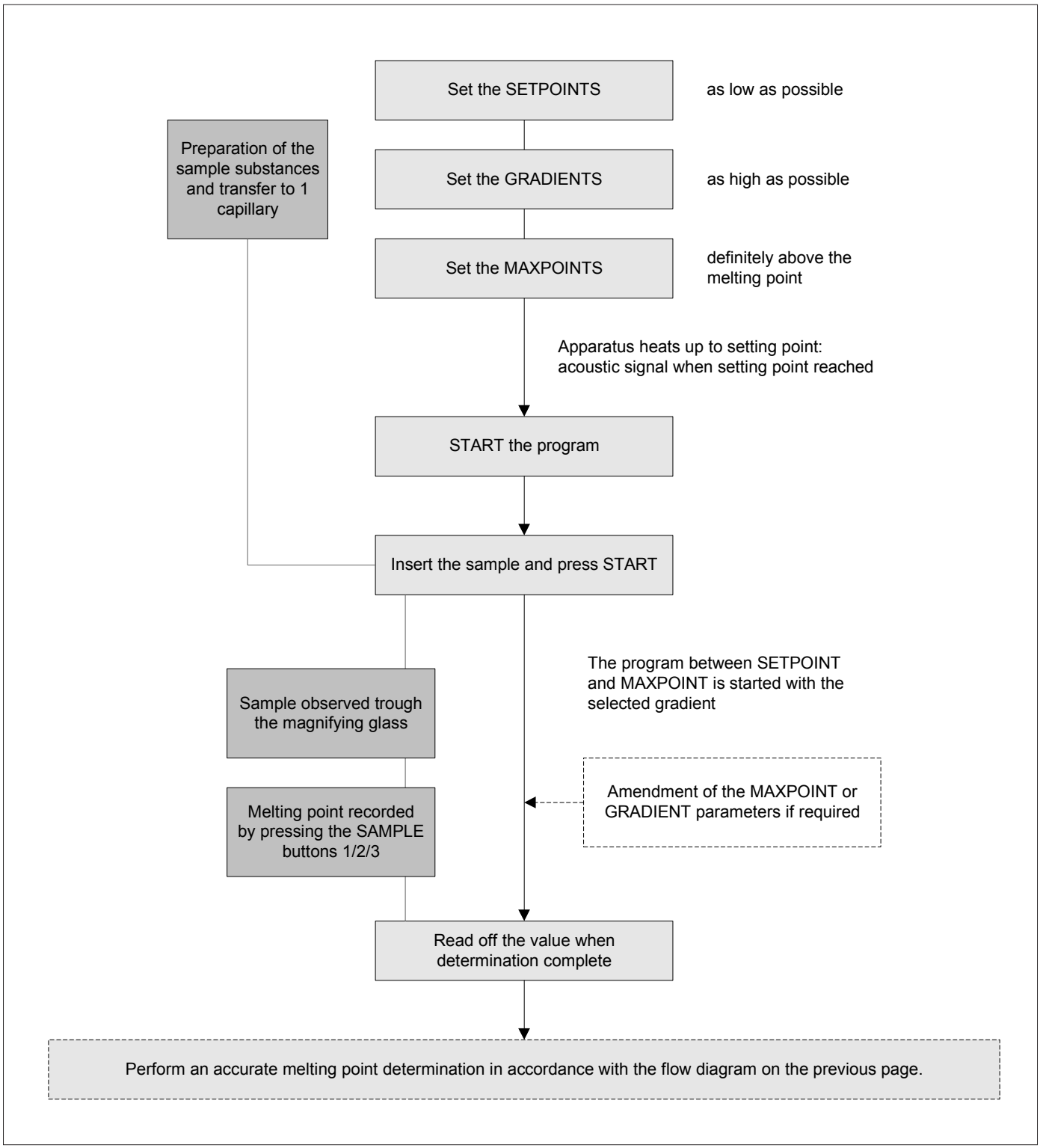


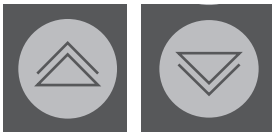
Figure 11: Flow diagram of melting point determination of a substance with unknown melting point /range

5.5.1 Determination of the SETPOINT

The SETPOINT can be adjusted in the main menu. This is done on every individual digit of the relative number.
The SETPOINT is adjusted with keys UP/DOWN and NEXT, the number is transmitted with the ENTER key. By way of example, a SETPOINT of 180° C is to be adjusted.

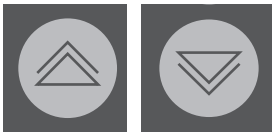
TEMPERATURE	:	25.0°C
SETPOINT	:	100°C
GRADIENT	:	2.0°C
MAXPOINT	:	120°C

Press ENTER until the cursor is on the SETPOINT field.



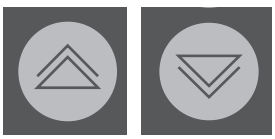
Increase or reduce the value until the number 1 is in the first digit.

NEXT until the cursor is on the second digit.



Increase or reduce the value until the number 8 is in the second digit.

NEXT until the cursor is on the third digit.



Increase or reduce the value until the number 0 is in the third digit.

ENTER to transmit the complete SETPOINT temperature.

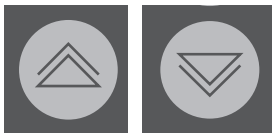
For the best results, we recommend a setting point of 5-10°C below the expected melting point.

5.5.2 Determination of the GRADIENT (temperature rise per minute)

The gradient is the heating rate at which heating is continued as from reaching the SETPOINT temperature to MAXPOINT. Adaptation of the gradient is made in 0.1/0.2/0.5/1/2/3/5/10/20 °C/min. in the main menu. By way of example, a gradient of 1° C is to be selected.

TEMPERATURE	:	25.0°C
SETPOINT	:	100°C
GRADIENT	:	2.0°C
MAXPOINT	:	120°C

Press the ENTER key until the cursor is on the GRADIENT field.



Increase or reduce the temperature rise until the value of 1° C/min is displayed.

Press ENTER to transmit the selected temperature rise.

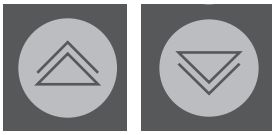
For the best results, we recommend a heating rate of 0.5°C/minute. A heating rate of 1°C/minute should be used for measurements in accordance with a Pharmacopoeia.

5.5.3 Determination of the MAXPOINT

Normally, the temperature of the MAXPOINT is adjusted automatically at 15° C above the SETPOINT. If this temperature differential exceeds 15° C in an individual case, the MAXPOINT may be adapted manually. The MAXPOINT is equally adapted in the main menu. By way of example, the MAXPOINT temperature of 195° C is to be increased to 230° C.

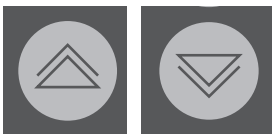
TEMPERATURE	:	25.0°C
SETPOINT	:	100°C
GRADIENT	:	2.0°C
MAXPOINT	:	120°C

Press ENTER until the cursor is on the MAXPOINT field.



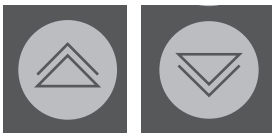
Increase or reduce the value until the number 2 is in the first digit.

NEXT until the cursor is on the second digit.



Increase or reduce the value until the number 3 is in the second digit.

NEXT until the cursor is on the third digit.



Increase or reduce the value until the number 0 is in the third digit.

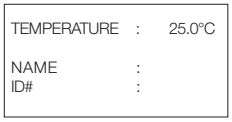
ENTER to transmit the MAXPOINT temperature.

5.6 Program administration

5.6.1 Assignment of a sample identification

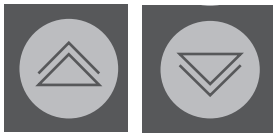
If similar parameters are used all over again, it is recommended to store them with sample names. By simply calling them up from the memory, their repeated entering becomes dispensable. As far as necessary, an identification (e.g. batch number or test number) may be assigned to this method. The name and the sample identification will then be printed out. A sample name and the sample identification are entered into the menu of the memory. The name used may consist of letters, figures or a combination of figures with letters.

For easy entering of sample names and sample identifications we recommend to use a keyboard (Büchi Art.No. 29599).



Press ENTER until the cursor is on the NAME field for entering the method name.

Press NEXT until the cursor is on the correct position of the horizontal which is to be modified.



An individual cursor position can be changed by actuating UP/DOWN.

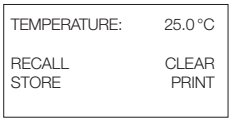
Press ENTER to transmit the method name. Enter the identification again with UP/DOWN and NEXT.

Press ENTER to transmit the sample identification.

The name and sample identification are now printed out. But the method has not yet been stored. If the method is to be stored in the memory, change over to main menu 3.

5.6.2 Storing programs

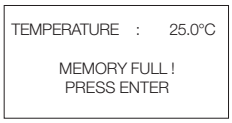
Press MENU to change to main menu 3.

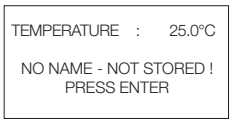


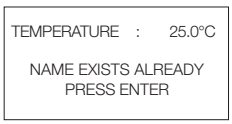
Press NEXT until the cursor is on the STORE field.

By pressing ENTER, the program is stored with the selected parameters under the entered name and the display will change to main menu 1. The sample identification is not stored.

Possible error messages.

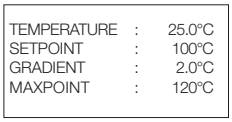






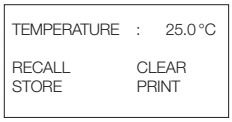
5.6.3 Recalling stored programs

If similar parameters are used all over again, it is recommended to store them with sample names. By simply recalling them from the memory, their repeated entering becomes dispensable.



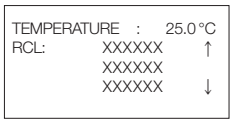
Stored programs can only be recalled from main menu 3.

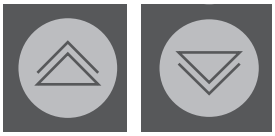
Press the MENU key twice to enter the main menu 3.



The NEXT key is pressed until the cursor is on the RECALL field.

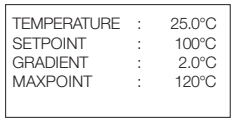
By pressing the ENTER key, the stored programs are listed.

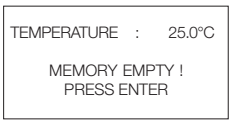




Choose the selected program with UP/DOWN keys (e.g.benzoic acid).

Recall the selected program by pressing the ENTER key. The unit is now ready for the next determination with the recalled method.
The display switches to main menu 1.

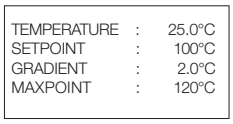




Possible error messages

5.6.4 Deleting stored programs

If stored programs are no more needed or if the capacity of storage locations is exhausted, programs may be deleted (erased).

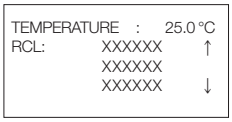


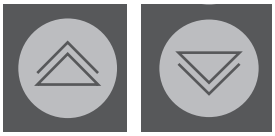
Stored programs can only be deleted from main menu 3.

Press MENU twice to go to main menu 3.

Press NEXT until the cursor is on the CLEAR field.

By pressing ENTER, the stored programs are listed.

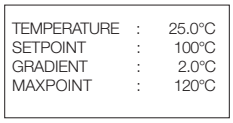


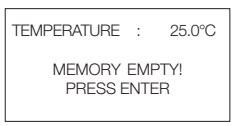


Select the program to be cancelled with UP/DOWN (e.g.benzoic acid).

Press ENTER to cancel the selected program.
If necessary, further programs may be cancelled now.

By pressing MENU, return to the main menu.





Possible error messages.

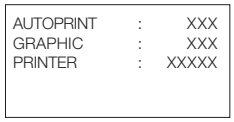
5.6.5 Printing results

1. Automatically printing out results after each determination

After completing a determination, results and selected parameters can be automatically printed out. If automatic printing is desired, you must switch to configuration menu 3.

The MENU key is pressed for 3 seconds in order to switch to the submenu.

Press the MENU key twice to switch to configuration menu 3.



The printer connected to the device is selected in the PRINTER field.

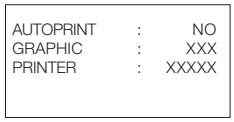
In the AUTOPRINT field, you specify if:

YES: a printout should be made automatically after every determination.

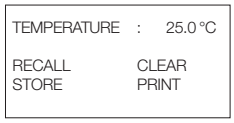
NO: a printout should not be made automatically after every determination.

2. Manually printing results after each determination

The user can specify the determination that should be printed.

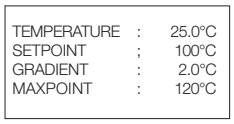


AUTOPRINT = NO must be selected in configuration menu 3.

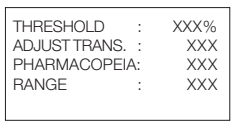


If a printout is desired after completion of a determination, then you must switch to the PRINT field using the MENU key and confirm by pressing ENTER.

5.7 Carrying out a determination



You must first decide if the melting point or the melting range is to be determined.

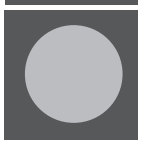


After pressing the MENU key for three seconds, you enter your selection in the first configuration menu.

The ENTER key is pressed three times. The cursor is now in the RANGE field.

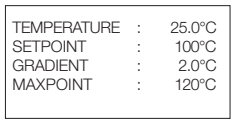
YES → Determination **Melting range**
NO → Determination **Melting point**

By pressing ENTER your selection is stored.



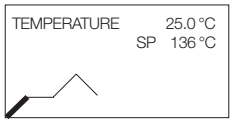
By pressing the MENU key for three seconds, you can return to the main menu.

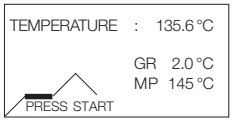
5.7.1 Melting point determination with a known melting point



The melting point determination may be started from any main menu. The SETPOINT, GRADIENT and MAXPOINT must first have been defined as described in 5.5.1-5.5.3.

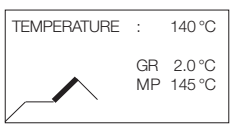
By pressing START, the unit will heat up to the previously selected SETPOINT. The display shows the temperature gradient graphically.

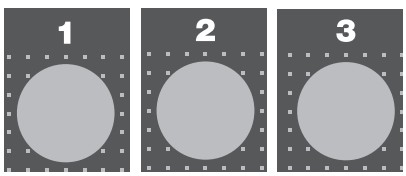




The user is informed acoustically when the SETPOINT has been reached (AUTO START: NO). The graphic will change at the same time. Samples are introduced into the sample holder.

By pressing START again, the temperature program is started between SETPOINT and MAXPOINT and the display changes to the detection phase.
The sample tubes must not be moved during this phase.

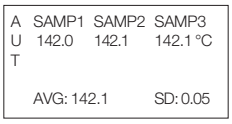




The samples are viewed through the magnifying glass as required. The melting point is retained by pressing SAMPLE keys 1/2/3. Every pressing of the SAMPLE keys is acknowledged with a beep and confirmed with an indication on the screen. A maximum of three modifications of condition per sample may be transmitted to the memory.

If the transmission does not change for 30 seconds during the melting process and automatic detection is on (DETECTION: YES), then the determination is automatically terminated and the device cools down.

When the preselected MAXPOINT has been reached, the unit will cool down automatically to the previously used SET-POINT. The temperature program may be stopped before reaching the MAXPOINT temperature by pressing STOP. Even in this case, the unit will cool down to the previously selected SETPOINT and display the automatically obtained values.



Samp 1:
Automatically determined value for substance in capillary 1

Samp 2:
Automatically determined value for substance in capillary 2

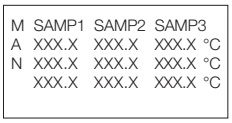
Samp 3:
Automatically determined value for substance in capillary

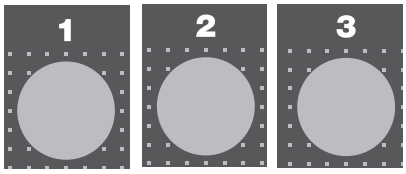
P:
Method Pharmacopeia

AVG:
Average value of the three determined values

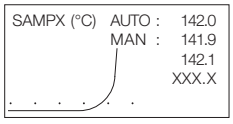
SD:
Standard deviation of the three determined values

By pressing the NEXT key the visually determined values can be displayed on the screen.





By pressing one of the three SAMPLE keys, the gradient of the transmission per capillary can be displayed graphically.



AUTP: Automatically determined value (Pharmakopoe)
MAN: Manually determined value

By pressing the MENU key, the determination can be repeated.

By pressing the STOP key, the unit will cool down to ambient temperature.

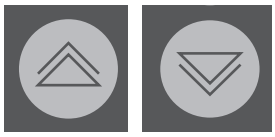
5.7.2 Melting point determination with unknown melting point / range

If the user has no information about the location of the melting point of his substance it can be found out by means of a previous fast determination. For that purpose, it is recommended to select the SETPOINT as low as possible and the temperature GRADIENT as high as possible. Here, the MAXPOINT should be selected to ensure that it is definitely above the melting point. The determination is started from the main menu.
A sample designation is dispensable. The SETPOINT, GRADIENT and MAXPOINT must be defined. By way of example, a melting point is to be determined which is between the ambient temperature and 300° C. For this, it is sufficient to determine the approximate melting point with one capillary only.

Establishing the SETPOINT
(for unknown melting points/ranges)

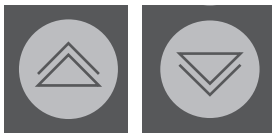
TEMPERATURE	:	25.0°C
SETPOINT	:	100°C
GRADIENT	:	2.0°C
MAXPOINT	:	120°C

Press ENTER until the cursor is on the SETPOINT field.



Increase or reduce the value until the number 0 is in the first digit.

Press NEXT until the cursor is on the second digit.



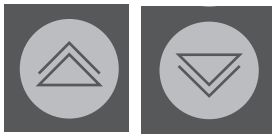
Increase or reduce the value until the number 3 is in the second digit.

Press NEXT until the cursor is on the second digit.

Increase or reduce the value until the number 0 is in the third digit.

ENTER to transmit the SETPOINT temperature.
The cursor will jump to the next line.

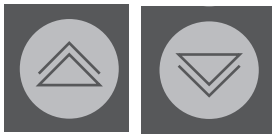
Adjusting the temperature program (GRADIENT)
(for unknown melting points/ranges)



Increase or reduce the temperature program until the value of 20° C/min is displayed.

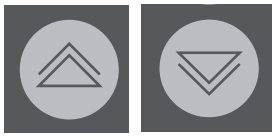
Press ENTER to transmit the selected temperature program. The cursor jumps to the next line.

Adjusting the MAXPOINT
(for unknown melting points/ranges)



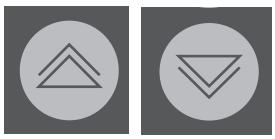
Increase or reduce the value until the number 3 is in the first digit.

Press NEXT until the cursor is on the second digit.



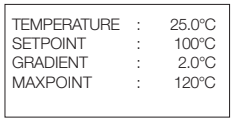
Increase or reduce the value until the number 0 is in the second digit.

Press NEXT until the cursor is on the third digit.

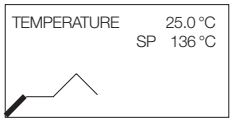


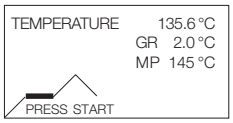
Increase or reduce the value until the number 0 is in the third digit.

Press ENTER to transmit the complete MAXPOINT temperature.



By pressing the START push-button, the unit will heat up to the previously selected SETPOINT. The display will show the temperature gradient graphically.

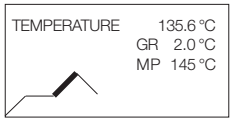


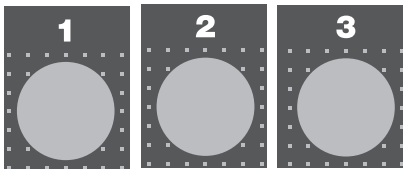


The user is informed acoustically that the SETPOINT has been reached. At the same time, the graphic will change. The samples are introduced into the sample holder.

The cappilaries have to be inserted before pressing the START button.

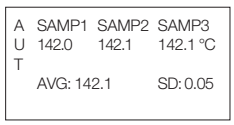
By pressing the START key again, the temperature program is started between SETPOINT and MAXPOINT and the display changes to the detection phase. (If Auto-Start had been switched off, refer to 5.9).





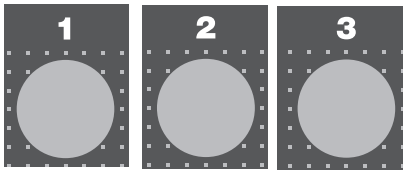
The samples are viewed through the magnifying glass as required. The melting point is retained by pressing SAMPLE keys 1/2/3. Every pressing of the SAMPLE keys is acknowledged with a beep and confirmed with an indication on the screen. A maximum of three modifications of condition per sample may be transmitted to the memory.

When the preselected MAXPOINT has been reached, the unit will cool down automatically to the previously used SETPOINT. The temperature program may be stopped before reaching the MAXPOINT temperature by pressing STOP. Even in this case, the unit will cool down to the previously selected SETPOINT and display the values obtained.

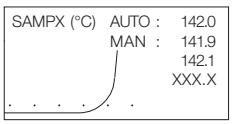


Samp 1:
Automatically determined value for substance in capillary 1

By pressing the NEXT key the visually determined values can be displayed on the screen.



By pressing SAMPLE key no. 1, the gradient of the transmission per capillary can be displayed graphically.



Aut.: Automatically determined value (thermodynamic determined)
Man.: Manually determined value

The melting point thus determined is approx. 25 °C too high, but it is still sufficient to permit a more accurate determination. (See chapter 5.5.5.)

By pressing the MENU key, the determination can be repeated.

By pressing the STOP key, the unit will cool down to ambient temperature.

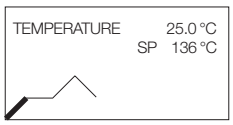
5.7.3 Modifications of parameters during a determination

Quite frequently, the user will find out during an actual determination that his selected parameters have to be adapted, e.g. because the SETPOINT or the MAXPOINT are too low or because the GRADIENT runs too slowly.
In order to adapt these parameters, it is not necessary to start the determination again from the beginning.
The following modifications are possible:

Modifications before reaching the SETPOINT
The SETPOINT can be adapted.

Modifications in the ready mode
The MAXPOINT can be adapted.
The GRADIENT can be adapted.

Modifications between SETPOINT and MAXPOINT
The MAXPOINT can be adapted.
The GRADIENT can be adapted.



Modifications before reaching the SETPOINT
Problem: The SETPOINT has been selected too low.

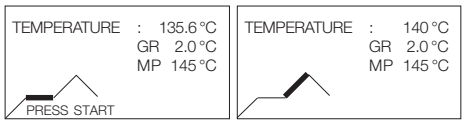
Press the MENU key:
The field for SETPOINT temperature begins to flash and may be increased by actuating UP/DOWN.

The new SETPOINT temperature is confirmed with ENTER and the unit returns to the determination mode.

Problem: The determination is to be aborted.

The determination is aborted. The unit cools down to ambient temperature.

Modifications in the ready mode

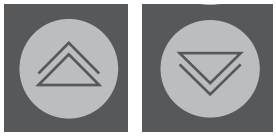


Modifications between SETPOINT and MAXPOINT

Problem: The temperature GRADIENT and/or MAXPOINT have been selected too low.

Press the MENU key:
The field for temperature GRADIENT begins to flash and can be increased via UP/DOWN.

The new temperature GRADIENT is acknowledged with ENTER and the MAXPOINT temperature flashes.



The MAXPOINT temperature can be increased via UP/DOWN.

The newly selected MAXPOINT is confirmed with ENTER and the unit returns to the determination mode.

Problem: The determination is to be aborted.

The determination is aborted and the unit cools down to ambient temperature.

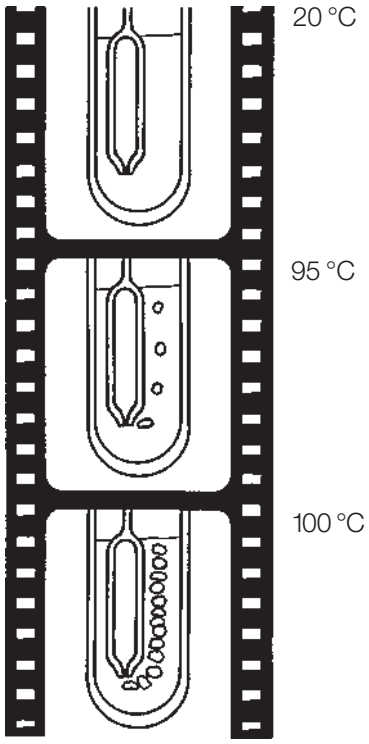


Figure 12: Boiling point determination

5.8 Boiling point determination

The sample liquid is filled into the capillary with an internal diameter of 2.25 mm to a height of 5-10 mm. Use a syringe for easy filling. The boiling capillaries are plunged into the sample with their thick end.
Switch the master switch on. Place the sample capillary into the sample holder. The position for boiling point determination is either fully to the left or to the right of the sample holder.

If the approximate boiling point is not known, a quick determination for orientation is carried out analogous to the melting point determination, section 5.6.
If the approximate boiling point is known, a determination is carried out analogous to the melting point determination with known melting point, section 5.5.5).

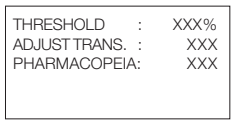
The boiling point has been reached when a quick continuous flow of bubbles (like a string of pearls) is formed at the plunged-in end of the capillary.

The boiling point can only be determined visually.
(not automatically)

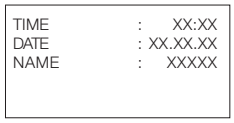
5.9 Device configuration

Subparameters are basic adjustments which are set by the user once when commissioning the unit. These are to be found in submenus which the user will enter only very seldom.
These submenus are entered by actuating the MENU key which should be pressed for three seconds for that purpose. Once the user has entered the first submenu, he may scroll through the individual menu windows by pressing the MENU key.

5.9.1 Configuration menus

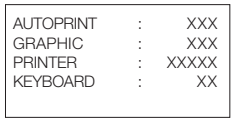


Büchi defines the melting point at a transparency of 40 %. This transparency can be adapted in this window of the menu.
The melting range is defined by Büchi to be at transparencies between 1% and 90%. These values can be changed, however.



Menu window for setting data which are to appear on the printout.

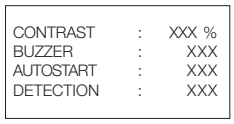
Time:	Time
Date:	Date
Name:	Company name



Menu window for adjusting the displays to be printed out.

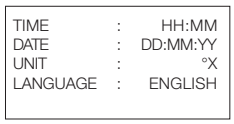
Autoprint:	Automatic printing after every determination yes/no
Graphic:	Representation of the curve on transparency yes/no
Printer:	Selection of the printer to be used
Keyboard:	Keyboard selection

Menu window for adjusting the serial interface to the PC connection.



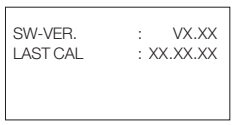
Menu window for adjustments of the unit

Contrast:	Adjustments of the LCD display
Buzzer:	Beep yes/no
Auto Start:	No / Yes
Detection:	No / Yes; Switch on/off the automatic

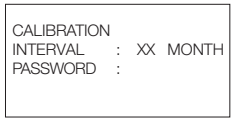


Menu window for basic adjustments

Time:	Time subdivided into 12 or 24 steps
Date:	American or European date
Unit:	Temperature in °C or °K
Language:	German / English / French



SW version:	Indication of the software installed
Last cal.:	Date of the last calibration



Interval:	Time interval before the unit informs the user that calibration should be repeated.
Password:	Entering the menu level for calibrating the unit by the user.

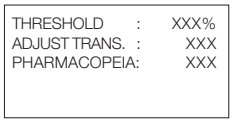
5.9.2 Modification of the transmission for automatic detection

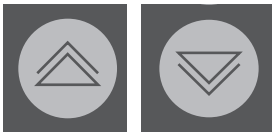
A light source located at the front of the measuring cell illuminates the samples. Behind the sample there are three photoelectric resistors to measure the remaining transmitted light. As long as the samples are not molten, the transparency is close to zero. When the melting point is about to be reached the light transmission increases considerably. This effect is used for automatic detection of the melting point.

The unit has been adjusted at works to a transparency of 40 % for an automatic detection of the melting point. This value of 40 % may be corrected by the user if he chooses to establish the melting point higher or lower after a visual check.

To determine the melting range, there is a an upper and a lower transmission threshold, both of which can be changed. The device is set at the factory with a lower transparency threshold of 1% and an upper transparency threshold of 90%.

The MENU key is pressed for 3 seconds to return to the range of submenus.





By pressing the UP/DOWN keys, the intensity of the transparency which is to apply from then on for all automatic detections of the melting point can be changed.

If the melting range is to be determined, the lower transmission threshold will be displayed for setting. By pressing the NEXT key, you can have the upper transmission threshold appear in the display for setting.

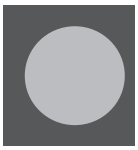
By pressing the ENTER key, the newly entered value will apply for all new determinations.

Press the MENU key again for 3 seconds to return to main menu 1

5.9.3 Modification of time, date and company name

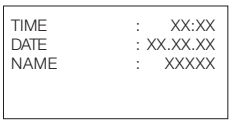
If you connect the unit to a printer, the melting points may be printed out with other parameters. In order to permit a better assignment of the printouts, each printout will bear the following:

- the name of the own company
- the time of melting point determination
- the date of melting point determination



Press the MENU key for three seconds to return to the range of submenus.

Press the MENU key a second time to return to the menu window for setting the data as they are to appear on the printout.



By pressing the UP/DOWN keys, the individual digits are changed; by pressing the NEXT key, the horizontal cursor position is changed.

By pressing the ENTER key, the date field will be displayed and this is then adapted in the same way as described above.

By pressing the ENTER key, the field for the company name is displayed and this is then adapted in the same way as described above.

By pressing the ENTER key, the newly entered company name will be used for all new determinations.

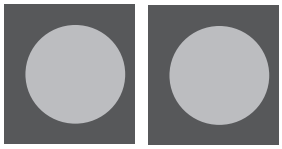
In order to return to main menu 1, press the MENU key again for three seconds.

5.9.4 Automatic printout after every determination, printing of the transmission curve, selection of the printer

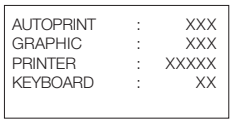
In this menu, the following adjustments can be carried out:

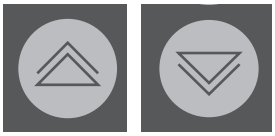
- Automatic printout after every determination
- Printing of the transmission curve on every printout as well
- Selection of the connected printer
- Selection of the keyboard used

Press the MENU key for three seconds to return to the range of submenus.



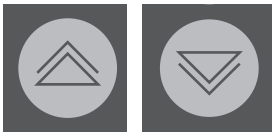
Press the MENU key twice to change to the menu window.





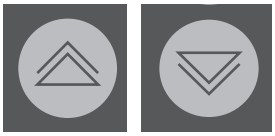
By pressing the UP/DOWN keys, the automatic printout will be activated after every determination, and made inoperative with NO.

Press the ENTER key to go to the graphic field.



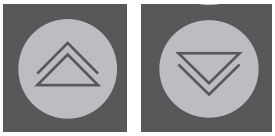
By pressing the UP/DOWN key, the automatic printout of the transmission curve will be activated and prevented with NO.

Press the ENTER key to go to the printer field.



Press the UP/DOWN key to choose the printer to be used.
- CITIZEN
- EPSON (Canon and Seiko also)
- Hewlett Packard
- STAR
- NONE

By pressing the ENTER key, the newly entered printer will now apply for all new determinations.



Press the UP/DOWN keys to choose the keyboard:
- German
- USA

By pressing the ENTER key the keyboard selection will be stored.

In order to return to the main menu, press the MENU key again for three seconds.

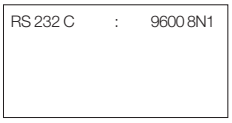
5.9.5 Definition of the RS-232 interface parameters

In this menu, you can change the following settings for the RS232 C interface:

- the baud rate
- length of a word of data
- parity
- number of stop bits

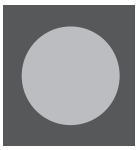
Press the MENU key for three seconds to return to the range of submenus.

Press the MENU key three times to change to the menu window.



Press the UP/DOWN key to select the RS-232 C parameters.

When pressing the ENTER key, the newly selected parameters are stored.

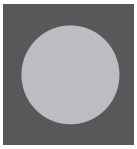


Press the MENU key again for three seconds to return to main menu 1.

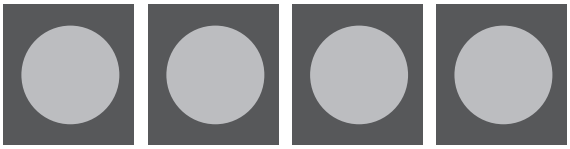
5.9.6 Display adjustments, switching on/off, beep

In this menu, the following adjustments can be carried out:

- Increase, reduce background illumination on the display
- Switch the beep on/off



Press the MENU key for three seconds to return to the range of submenus.



Press the MENU key four times to change to the menu window.

Menu window for settings of the unit:

CONTRAST	:	XXX %
BUZZER	:	XXX
AUTOSTART	:	XXX
DETECTION	:	XXX

- CONTRAST: Adjustments of the LCD display
- BUZZER: Beep yes/no
- AUTOSTART:

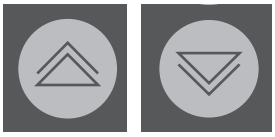
YES: Direct transition after reaching the SETPOINT in the detection phase without having to press START a second time.

NO: After reaching the SETPOINT, you must press START a second time to enter the detection phase.

- DETECTION:

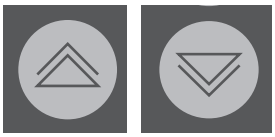
YES: The determination is done automatically. This means that the determination is automatically terminated after the melting process if the transmission does not change for 30 seconds. The values needed to create a diagram are also recorded.

NO: The automatic detection is switched off. The determination of the melting point/range is done manually and is only terminated after MAXPOINT is reached. No data is recorded during the determination and therefore no diagram can be created.



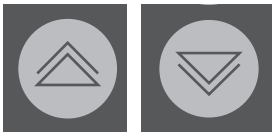
By pressing the UP/DOWN key, the contrast will be increased or reduced.

By the pressing the ENTER key, the user will return to the buzzer field.



By pressing the UP/DOWN key, the beep will remain with ON and be made inoperative with OFF.

By pressing the ENTER key, the user returns to the AUTO-START field.



By pressing the UP/DOWN keys, a direct switch to the detection phase will occur from now on, without having to press START a second time.

By pressing the UP/DOWN key, the following occurs for:
YES: an automatic detection
NO: a manual determination

Press the MENU key again for three seconds to return to the main menu.

5.10 Removal of errors

Problem	Reason	Solution
In the automatic melting point determination no value and no transmission curve are determined	• Sample is unstable, it decomposes, turns brown or melts non-uniformly • The temperature parameters have been entered incorrectly and the sample does not melt at all	• e.g. select higher maximum point
Sometimes no results or only 1 or 2 results instead of 3 results	• START button was pressed before insertion of all sample capillaries.	• Put all sample capillaries in the oven first, then press the START button. Do not move the sample capillaries again.
Melting point is measured but is incorrect	*Sample preparation* • poorly filled capillaries (can lead to the formation of bubbles so that the melting point is recorded too early) • Use of the wrong capillaries	• Ensure substance is compact in the capillaries (by knocking the capillary on a hard surface) • Use Büchi standard capillaries of the correct sizes
	Sample-specific preparations • Sample is not dry or contaminated by another substance • Sample decomposes during the melting process (formation of bubbles, sample turns brown etc.)	• Sample should be dried beforehand
	Apparatus Parameters • Apparatus is not or is poorly calibrated • Setting point is too close to the melting point • Incorrect adjustment of the transmission • Gradient is set too high • Method "according to Pharmaco-poeia" or "thermodynamic melting point determination" is incorrectly selected	• Regular calibration of apparatus with Büchi calibrating substances • Select setting point 5-10°C below melting point • Appropriate gradient selection (≤ 0.5°C/min. for accurate measurements) • Alter the corresponding parameters

Table 4: Removal of errors

6 Maintenance

All instructions intended to keep the Melting Point Unit in a fully operative condition should be duly noted. This also includes a periodic cleaning and checking for any damages. It is also recommended to calibrate the unit periodically with adequate calibration substances.

6.1 Cleaning

The housing of the Melting Point Unit B-545 is coated with paint. It must only be cleaned with a rag to be either dry or moistened with alcohol.
Do not use any chlorinated solvents, acetone or similar chemical to clean the magnifying glass. Use ethanol only.

6.2 Upkeep

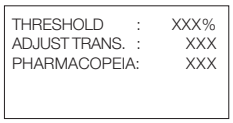
The upkeep of the unit is mainly limited to:
- a periodical calibration of the transmission
- a periodical calibration of the temperature

6.2.1 Periodical calibration of the transmission

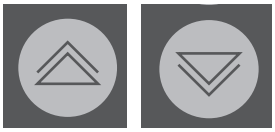
A light source located at the front of the measuring cell illuminates the samples. Behind the sample there are three photoelectric resistors to measure the remaining transmitted light. As long as the samples are not molten, the transparency is close to zero. When the melting point is about to be reached the light transmission increases considerably. This effect is used for automatic detection of the melting point. The transmission must be recalibrated from time to time with empty capillaries.

Press the MENU key for approx. 3 seconds.

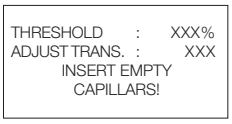
No capillaries should be in the oven!



Press the ENTER key to change to the field “ADJ. TRANSM.” Insert three empty, clean capillaries into the oven.

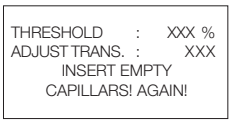


Set the display to YES by pressing the UP/DOWN key.

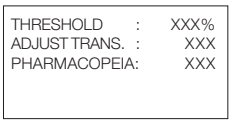


3 empty, cleaned capillaries habe to be inserted.

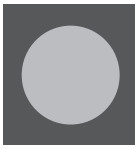
By pressing the ENTER key, the transmission will be calibrated.



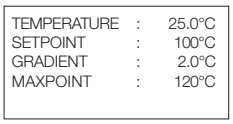
If no capillaries were inserted into the oven, it will not be possible to calibrate the transmission. The unit will recognise this error automatically and prompt the user to correct the error and repeat the procedure.



If the transmission has been correctly adjusted, the display will return to the preceding menu (Adjust. Transmission: NO).



Press the MENU key again for 3 seconds to return to main menu 1.



6.3 Calibration of the apparatus with reference substances

The apparatus should be periodically checked with reference substances. The melting points measured are compared with the theoretical melting points. If the two values deviate, the temperature measurement in the apparatus heating block can be adjusted with correction values.
The correction factors are stored in the permanent memory and can be corrected again by the user at any time. This adjustment does not alter any of the basic settings of the correction factors pre-determined by the user that are used for the temperature measurement in the heating block.

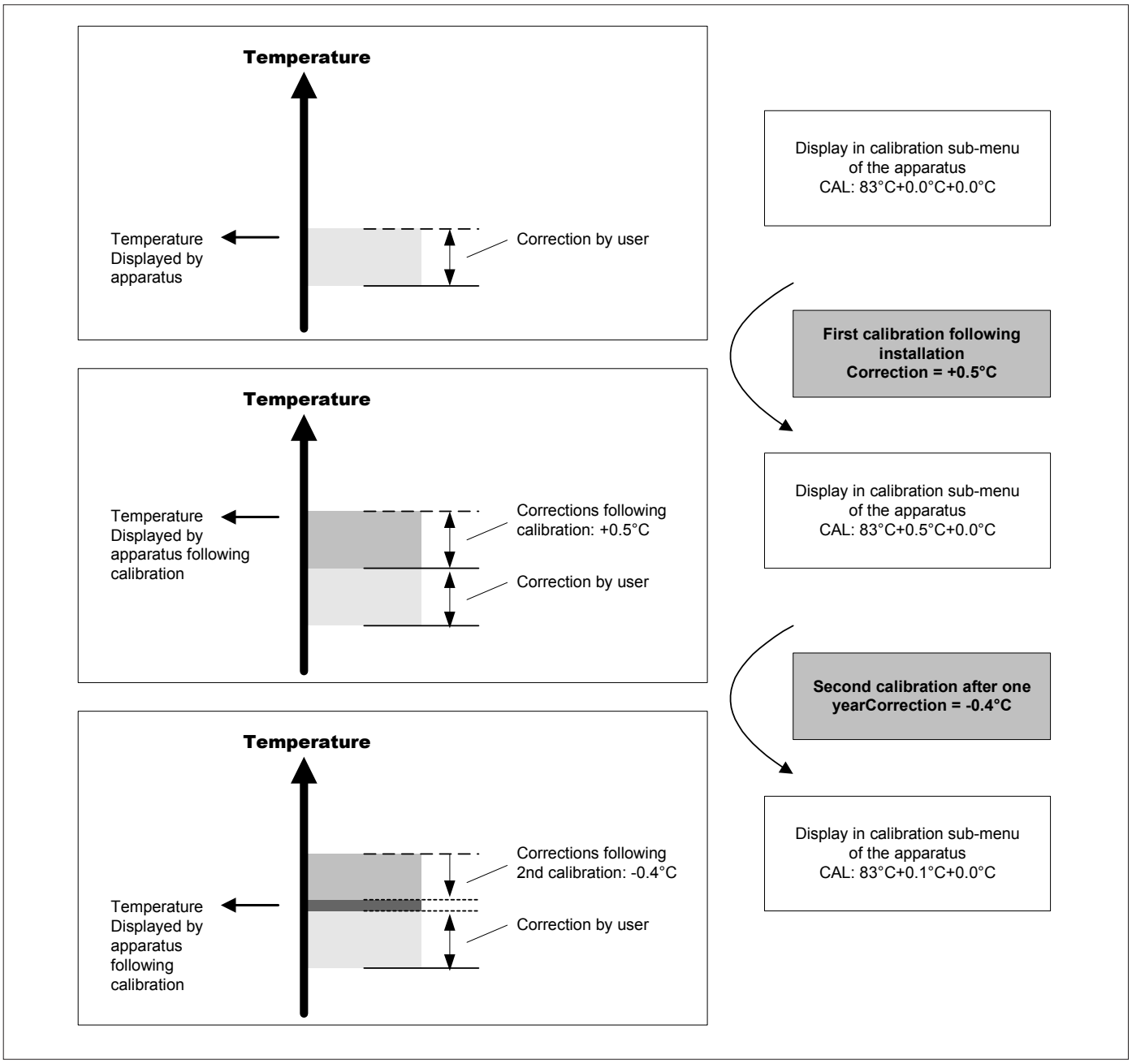


Figure 13: Diagram of correction sectors

The following reference substances are provided for the calibration of the melting point apparatus B-545:

Vanillin	Theoretical melting point	approx. 83°C
Phenacetin	Theoretical melting point	approx. 136°C
Caffeine	Theoretical melting point	approx. 237°C

We recommend the use of the reference substances from the calibration set, Büchi Item No. 037454.

6.3.1 Melting point determination for the calibration

- The instrument should be calibrated at least once a year.
- The reference substances should be stored protected from light in a desiccator over desiccant at room temperature (15-25°C).
- The expiry date must be checked and the reference substances replaced if necessary.
- The theoretical melting points of the reference substances are known. An analysis certificate is provided and describes how the theoretical melting points are determined.
- The transmission is calibrated before determination of the melting point (see section 6.2.1)
- Only Büchi melting point capillaries, item no. 017808 or 001759, should be used
- Three measurements are performed simultaneously for each reference substance.

Recommendation:
The substances should be ground in a mortar, filled to a height of 4-5mm as compactly as possible by knocking the filled capillaries on a hard surface.

Gradient:	0.5°C/min.
Setting point:	5°C below the expected melting point
Pharmacopoeia method	
Automatic detection at 40%	

6.3.2 Evaluation of the melting point determination

All measurements should be evaluated in accordance with strict criteria and checked for their usability. The measurement must be repeated in the case of doubt. New substances should be used if necessary. The transmission procedure complies with the following requirements:

Example: These measurements should be repeated.

Example: These measurements are acceptable.

The mean value is calculated and the standard deviation from all 3 measurements shall not exceed <0.3°C.

6.3.3 Imput of corrections

If it is ascertained during a routine check of the temperature display, that the actual and rated values of the calibration substances differ, the unit may be recalibrated by the user as follows:

Press the MENU key for three seconds.

Press the MENU key again 7 times to change to the calibration window.

```
CALIBRATION
INTERVAL    :  XX  MONTH
PASSWORD    :
```

Press the ENTER key to change to the PASSWORD field. The password for the calibration may then be entered: **CAL**

Press the ENTER key to get to calibration.

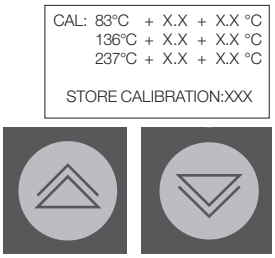
Calculation of the correction

As rated melting point use the temperature indicated on the reference substance. If there is indicated an interval, use its mean as rated melting point.

Correction =
Rated melting point - actual melting point

Examples:

- The rated melting point of the standard vanilline is 83.0°C. With the Melting Point B-545 was found an actual melting point of 82.3°C. Correction: 83.0°C - 82.3°C = +0.7°C
- The rated melting point of the standard vanilline is 83.0°C. With the Melting Point B-545 was found an actual melting point of 83.3°C. Correction: 83.0°C - 83.3°C = -0.3°C



Now the pertinent corrections can be entered for the threetypes of calibration substances. The middle column indicates the last correction, the right column is used to enter the new corrective value.

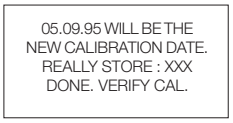
By pressing the UP/DOWN keys, the necessary corrective value at 83° C is entered.

By pressing the ENTER key, change to the 2nd temperature value and adapt this analogously.

Then press the ENTER key to change to the 3rd temperature value and adapt this analogously.

Press the ENTER key to change to the STORE CALIBRATION field and press the UP/DOWN key to obtain YES.

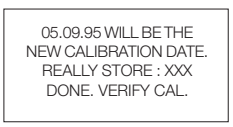
By pressing the ENTER key, the new calibration is confirmed.



For reasons of safety, the user is inquired again whether the corrective factors should be stored. If this is the case, set the display to YES and confirm with the ENTER key.

Press the MENU key to leave the calibration menu.

After the calibration, verifiation of the calibration has to be performed (chapter 6.3.4).



6.3.4 Control measurement

The calibration is checked with verification substances. For this we recommend the Set of verification substances, Büchi Item No. 037667. It allows checks at the following temperatures using four different substances:

Substance	Temperature
1,8-Octandiol	approx. 60°C
Metatoluic acid	approx. 110°C
Sulphanilamide	approx. 165°C
Phenolphthalein	approx. 260°C

The measurements are performed with the measurement parameters recommended in section 6.3.1. The melting point of each reference substance must be checked in at least three measurements. The standard deviations should be less than 0.3°C. The maximum permitted deviations of the mean value from the theoretical melting point are as follows:

Substance	Tolerated deviation
1,8-Octandiol	0.4°C
Metatoluic acid	0.4°C
Sulphanilamide	0.5°C
Phenolphthalein	0.6°C

All measurement results of the calibration, the correction factors entered and control measurement values should be recorded. This allows the calibration to be checked and repeated at a later date.

6.4 Customer service

Work on or in the unit may only be carried out by authorised service personnel, i.e. skilled technicians who are familiar with the operating instructions and current regulations with regard to work safety and accident prevention. Büchi customer service depots have device-specific service manuals which are only available to authorised personnel. You will find a list of addresses of official Büchi customer service depots on the last page of this instruction manual. Please contact one of these depots if the unit develops any faults, or you have any technical enquiries or application problems.

The Büchi customer service will be pleased to provide you with the following services:

- Replacement parts service (when placing an order, please use the article numbers printed in this manual)
- Repair service
- Maintenance service
- Technical hotline

7 Taking out of operation

The unit must be cleaned thoroughly.

7.1 Storage/Transport

The unit should be stored and transported in its original packaging and in clean condition.

7.2 Disposal

To ensure that disposal of the Melting Point Unit is as ecologically friendly as possible, you will find a list of the materials used, some of which are marked with material codes, in Chapter 9, Annex. This ensures that components can be separated and, where appropriate, recycled.

Care must be taken that regulations are complied with, when disposing of electrical components. Furthermore, all methods of disposal should comply with regional and local legislations.

8 Replacement parts

Only original Büchi accessories and replacement parts guarantee the operational safety and reliability of this unit. Use of any replacement parts or accessories other than Büchi is only permitted with the express permission of the manufacturer. For assembly and dismantling purposes, replacement parts should only be used as described in chapter 6 of this instruction manual. The reproduction or manufacture of any parts found in this manual is equally prohibited.

Description	**Order No.**
Protective cover	**36625**
Brief instruction manual in German	**96635**
Brief instruction manual in English	**96636**
Brief instruction manual in French	**96661**
2 Compaction wires	**36721**
Capillary holder (plastic)	**36629**
Spare fuse 110 –120V (10 pcs.)	**20083**
Spare fuse 220 –240V (10 pcs.)	**19660**
Upper ceramic part of oven	**34564**
Glass panel	**34236**
Metal cover for heating area	**36569**
Glass beaker	**36110**
Paper holder for printer Citizen IDP-562	**33751**
Roll of paper for printer Citizen IDP-562	**28261**
Color ribbon for printer Citizen IDP-562	**37303**
Paper holder for printer Citizen IDP-460	**38684**
Color ribbon for printer Citizen IDP-460	**38683**

Table 5: Replacement parts

9 Appendix

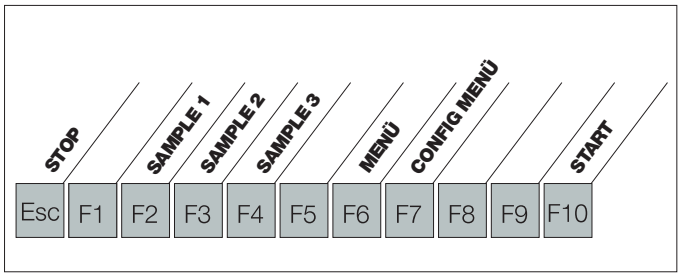


Figure 14: Function key layout

9.1 Connecting a keyboard

We recommend that you use a keyboard (Büchi Order No. 29548) to simplify the entering of sample names and sample identification information.

The row of function keys is laid out as shown in Figure 15.

9.2 Connection of a printer

We recommend use of a Citizen printer model IDP 460, Büchi item no. 038697 or 038698. The Citizen IDP 460 printer is connected using the communication cable supplied with the printer.

All printers are connected through the serial RS232 interface which is connected via the communication cable to the printer (see section 5.1).

Appropriate printer settings are selected in the configuration menu of the melting point apparatus. The interface is automatically correctly configured by the melting point apparatus.

The following printers may be used to print out measurement data:

- Citizen
- HP (Laserjet 3 or compatible)
- EPSON (LX 850 or compatible)
- Seiko (thermal printer DPU 411; in the menu under EPSON)
- Canon (BJC-600; in the menu under EPSON)
- Star Micronics SP2000

9.3 Interface configuration

Serial interface:	RS 232
Baud rate:	9600
Data bits:	8
Stop bits:	1
Parity:	none

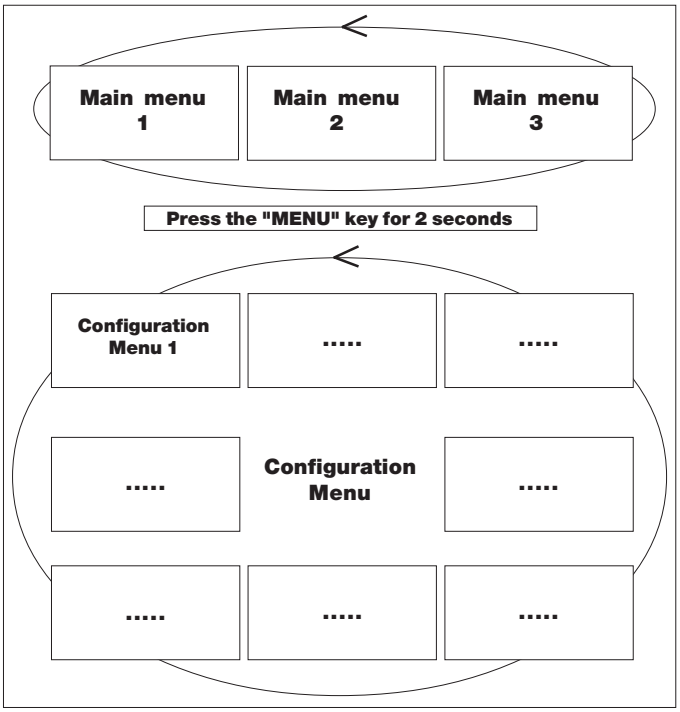


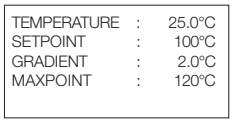
Figure 15: Menu tree

9.4 Menu tree

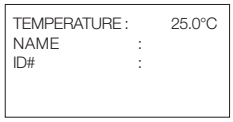
The menu tree of the B-545 Melting Point can be split into 2 major levels:

- the 3 main menus (Chapter 9.5.1)
- the 8 configuration submenus (Chapter 9.5.4)

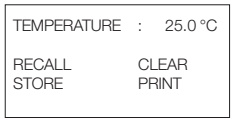
9.4.1 Main menus 1-3



Current temperature
Range of settings: 25 - 400°C
Parameters: 0.1 / 0.2 / 0.5 / 1.0 / 2.0 / 5.0 / 10.0 / 20.0
Automatic: SETPOINT + 15°C or variable up to 410°C

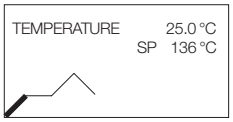


Current temperature

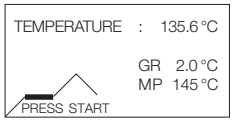


Program name (max. 15 characters)
Sample identification number (max. 15 characters)
Current temperature
RECALL/CLEAR: call/delete the stored program
STORE: store the program
PRINT: print the measurement results on the printer

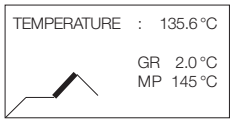
9.4.2 Heating/cooling phase



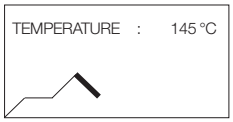
Quick heating (approx. 70°C/min.) up to the SETPOINT.
SP = SETPOINT



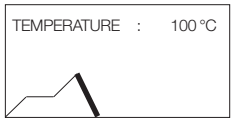
SETPOINT has been reached. Continue by pressing the START key.
GR = GRADIENT
MP = MAXPOINT



Slow heating using the gradient specified up to MAXPOINT; or stop beforehand by pressing the STOP key.
(manual) / Autodetection
GR = GRADIENT
MP = MAXPOINT

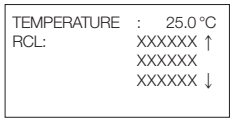


Quick cooling to the SETPOINT.

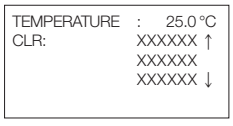


Quick cooling to room temperature.

9.4.3 Program storage



RC = Recall = Calls the stored program.



CLR = Clear = Deletes the stored program

9.4.4 Configuration menus

German	French	English
SCHWELLE : XXX% EINST. TRANSM : XXX PHARMAKOPOE : XXX BEREICH : XXX	SEUIL : XXX% REGLAGE TRANS.: XXX PHARMACOPEE : XXX PLAGE : XXX	THRESHOLD : XXX% ADJUST TRANS. : XXX PHARMACOPEIA: XXX RANGE : XXX

Detection threshold: (10-90%: default setting: 40%)
Transmission settings: (YES/NO): comparison with empty capillaries
YES = without thermodynamic compensation (Chapter 3)
NO = with thermodynamic compensation (Chapter 3)

Detection threshold:
melting point (10-90%: default setting: 40%)
melting range (lower threshold: default setting)
(upper threshold: default setting)

Transmission settings: (YES/NO): comparison with empty capillaries

Pharmacopoeia:
YES: without thermodynamic compensation (Chapter 3)
NO: with thermodynamic compensation (Chapter 3)

Range:
YES: determination of the melting range
NO: determination of the melting point

SCHWELLE : XXX% EINST. TRANSM. : XXX LEERE ROEHRCHEN EINSETZEN!	SEUIL : XXX% REGLAGE TRANS.: XXX INTRODUIRE CAPILL. VIDE!	THRESHOLD : XXX% ADJUST TRANS. : XXX INSERT EMPTY CAPILLARS!
SCHWELLE : XXX % EINST. TRANSM. : XXX LEERE ROEHRCHEN EINSETZEN! NOCHMALS!	SEUIL : XXX % REGLAGE TRANS.: XXX INTRODUIRE CAPILL. VIDE! ENCORE!	THRESHOLD : XXX % ADJUST TRANS. : XXX INSERT EMPTY CAPILLARS! AGAIN!

Transmission adjustment: (**Yes/No**)

Always remove the capillaries before turning the device on.

ZEIT : XX:XX DATUM : XX.XX.XX NAME : XXXXX	HEURE : XX:XX DATE : XX.XX.XX NOM : XXXXX	TIME : XX:XX DATE : XX.XX.XX NAME : XXXXX

Time: 12 (AM/PM) and 24 hour clock. HH = hours, MM = minutes
Date: DD = day, MM = month, YY = year
User name (max. 8 characters)

AUTO PRINT : XXX GRAFIK : XXX DRUCKER : XXXXX TASTATUR : XX	IMPR. AUTO : XXX GRAPHIQUE : XXX IMPRIMANTE : XXXXX CLAVIER : XX	AUTOPRINT : XXX GRAPHIC : XXX PRINTER : XXXXX KEYBOARD : XX

Automatic printing: (**YES**/**NO**)
Diagram: (**YES**/**NO**) Printout of results with/without diagram
Printer: - **CITIZEN** - **EPSON** (Canon and Seiko also)
- **HP** - **STAR MICRONICS**
- **NONE**
Keyboard: - **GERMAN** - **USA**

RS232 C : XXXX XXX	RS232 C : XXXX XXX	RS232 C : XXXX XXX

Serial interface for connecting to a PC
Baud rates: **1200 / 2400 / 4800 / 9600 Baud**
Parity
Start / Stop Bit

KONTRAST : XXX % PIEPSER : XXX AUTO START : XXX DETEKTION : XXX	CONTRASTE : XXX % BUZZER : XXX START AUTO : XXX DETECTION : XXX	CONTRAST : XXX % BUZZER : XXX AUTOSTART : XXX DETECTION : XXX

Contrast: **10-90%** (default setting: 50%)
Buzzer: **ON / OFF**
(at the start and end of a determination procedure)
Autostart: **YES / NO** (the "PRESS START" phase will be skipped)
Detection: **YES / NO** (Turns the automatic detection on/off)

ZEIT : HH:MM DATUM : DD:MM:YY EINHEIT : °X SPRACHE : DEUTSCH	HEURE : HH:MM DATE : DD:MM:YY UNITE : °X LANGAGE : FRANCAIS	TIME : HH:MM DATE : DD:MM:YY UNIT : °X LANGUAGE : ENGLISH

Time: 12 (AM/PM) and 24 hour clock. HH = hours, MM = minutes
Date: DD = day, MM = month, YY = year
Temperature unit: °**C** or **F** can be selected
Language: **German** (default setting), **English**, **French**

SW-VER. : VX.XX LETZ. KAL. : XX.XX.XX	SOFT. VER. : VX.XX DERNIER CAL : X.XX.XX	SW-VER. : VX.XX LAST CAL : XX.XX.XX

Software version
Last calibration

KALIBRATION INTERVALL : XX MONATE PASSWORT :	CALIBRATION INTERVAL : XX MOIS PASSWORD :	CALIBRATION INTERVAL : XX MONTH PASSWORD :

Calibration window:
Calibration interval: **1** to **36** months
Password
(for the calibration with calibration substances; see Chapter 6.3)

CAL: 83°C + X.X + X.X °C 136°C + X.X + X.X °C 237°C + X.X + X.X °C SPEICHERN KAL :XXX	CAL: 83°C + X.X + X.X °C 136°C + X.X + X.X °C 237°C + X.X + X.X °C MEMORISER CAL: XXX	CAL: 83°C + X.X + X.X °C 136°C + X.X + X.X °C 237°C + X.X + X.X °C STORE CALIBRATION:XXX

3 calibration substances are recommended.
(Calibration substances: vanillin, phenacitine, caffeine)
Middle column: shows the current correction factor.
Right column: enter new correction factor (see Chapter 6.3).

05.09.95 WIRD ALS KAL. DATUM GESETZT. WIRKLICH : XXX GEMACHT. KAL. PRUEFEN	05.09.95 DATE DE CAL. CONFIRMER LA CALIBRATION: XXX VERIFIER LA CAL.	05.09.95 WILL BE THE NEW CALIBRATION DATE. REALLY STORE : XXX DONE. VERIFY CAL.

Storing the correction factor: (**YES**/**NO**)

Check the calibration with the recommended parameters (see Chapter 6.3).

9.4.5 Error messages (see also Chapter 9.6)

TEMPERATURE : XXX.X°C MEMORY EMPTY ! PRESS ENTER

No existing entry, storage space empty.

TEMPERATURE : XXX.X°C NAME EXIST ALREADY PRESS ENTER

Program name already exists. Select a new name.

TEMPERATURE : XXX.X°C NO NAME - NOT STORED! PRESS ENTER

Program was not stored because no name was entered.

TEMPERATURE : XXX.X° MEMORY FULL ! PRESS ENTER

Memory is full. Delete a program to obtain storage space.

TEMPERATURE : XXX.X°C NO PRINTER SELECTED PRESS ENTER

No printer selected (configuration menu).

STORED DATA LOST PLEASE CHECK BATTERY AND SET TIME AND DATE

Check the battery (lithium cells in the device) and reset the time and date.

CALIBRATION INTERVAL EXCEEDED! LAST CAL.: XX.XX.XX

Calibration interval was exceeded.
(Press the menu key to continue working)
Last calibration.

CALL SERVICE ERROR: 001/099

Turn the device completely off using the main switch.
Press and hold the start key and turn the device back on.

Contact customer service (see Chapter 6.4).

9.5 Printout

9.5.1 Printout of a melting point determination

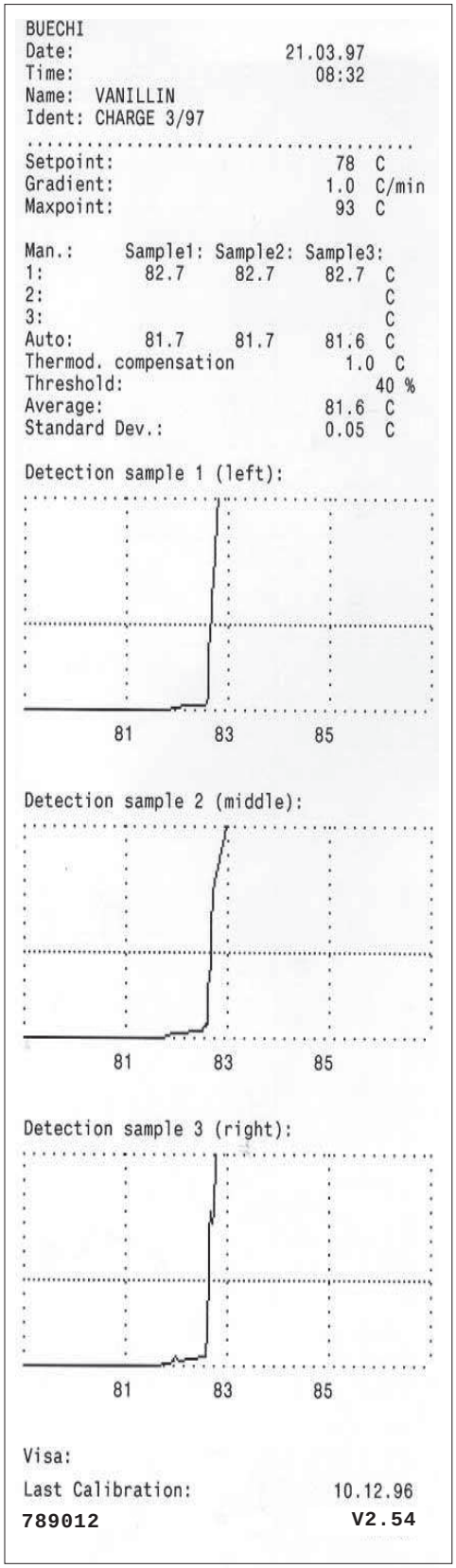


Figure 16: Printout of a melting point determination

What	Entry/change
Name of company	Configuration menu 2
Date of the determination	Configuration menu 2
Name of method	Main menu 2
Identification of the determination	Main menu 2
Start temperature of the determination	Main menu 1
Heating rate for the determination	Main menu 1
Final temperature of the determination	Main menu 1
Manually (visually) determined melting points per capillary	
Automatically determined melting points per capillary	
Factory setting of thermodynamic compensation	
Transmission threshold for the automatic melting point detection	Configuration menu 1
Average value of the three melting point determinations	
Standard deviation of the three melting point determinations	
Diagram of the transmission in the first capillary	Configuration menu 3
Diagram of the transmission in the second capillary	Configuration menu 3
Diagram of the transmission in the third capillary	Configuration menu 3
Signature of the person executing the calibration	
Date of last calibration	
Manufacturer's number and software version	

9.5.2 Printout of a melting range determination

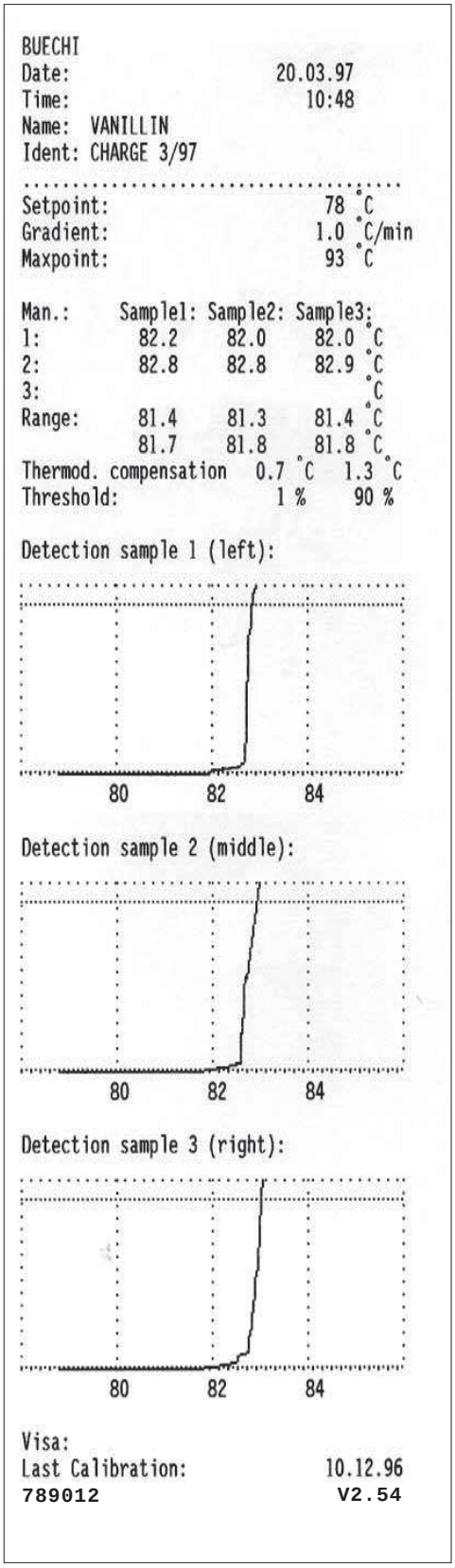


Figure 17: Printout of a melting range determination

What	Entry/Change
Name of company	Configuration menu 2
Date of the determination	Configuration menu 2
Name of method	Main menu 2
Identification of the determination	Main menu 2
Start temperature of the determination	Main menu 1
Heating rate for the determination	Main menu 1
Final temperature of the determination	Main menu 1
Manually (visually) determined melting ranges per capillary	
Automatically determined melting ranges per capillary	
Pharmacopoeia method (determination also possible thermodynamically)	
Upper and lower transmission thresholds for the automatic melting range determination	Configuration menu 1
Diagram of the transmission in the first capillary	Configuration menu 3
Diagram of the transmission in the second capillary	Configuration menu 3
Diagram of the transmission in the third capillary	Configuration menu 3
Signature of the person executing the calibration	
Date of last calibration	
Manufacturer's number and software version	

9.6 Error messages

Error	Cause	Remedy
STORED DATA LOST PLEASE CHECK BATTERY AND SET TIME AND DATE	The battery is exhausted	Press the Menu key for 5 seconds. Order new battery and have it installed by techn. Service. Date and time to be reprogrammed.
KALIBRATION INTERVAL EXCEEDED! LAST CAL: XX:XX	The selected calibration time has expired.	Press the Menu key for 5 seconds. Calibrate the unit. (See chapter 6, Maintenance)
The Citizen printer does not print	Configuration	The SEL key on the printer must be actuated whenever the printer is switched on.
CALL SERVICE ERROR 001/099	Device defective	Turn the device completely off using the main switch. Press the start key. Turn the device on and press the start key.
Wrong melting points.	Wrong transmission Calibration	Always remove capillaries when switching the unit on. Calibrate the unit.

Table 6: Error messages

9.7 Technical data

Dimensions of the unit (width x height x depth)	170 x 210 x 340 mm
Dimensions of packaging (width x height x depth)	340 x 390 x 580 mm
Weight	5.4 kg
Temperature range	Ambient temperature up to 400 °C
Cooling-down rate 350 °C – 50 °C	max. 10 min.
Heating-up spee 50 °C – 350 °C	max. 5 min.
Measuring accuracy up to 100 °C	+/– 0.3 °C
Measuring accuracy up to 250 °C	+/– 0.5 °C
Measuring accuracy up to 400 °C	+/– 0.8 °C
Temperature ramps	0.1/0.2/0.5/1/2/3/5/10/20 °C/min
Mains voltage	100 - 120V/50 or 60Hz
	220 - 230V/50 or 60Hz
Power consumption	Approx. 200 W
Current consumption	2 A (100 bis 120V) / 1 A (220 bis 230V)
Data saving with current failure	The status of the unit is stored
	Restarting is required
Ambient temperature	5 – 40 °C

Table 7: Technical Data

9.8 Materials used

Designation		Material	Material code
Lens holder		Grilamid	PA 12
Diaphragm		Grilamid	PA 12
Spacer ring		Polyamid	PA 6
Collar		Arosten	PBT
Print holder		Arosten	PBT
Upper part of heater		Ceramic	
Heater plate		Aluminium	ALMGSI 1
Spacer		PEEK	
Housing		Steel sheet	ST 12
Axia fan	Housing:	Aluminium	
	Propeller:	Ultem 254	
Lens		Glass	
Conical handle		Duroplast	
Foot of the appliance		Natural rubber	NR
Insulation cover		Saproplan	

Table 8: Materials

9.9 FCC requirements (for USA and Canada)

English:

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

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

Français:

Cet appareil a été testé et s'est avéré conforme aux limites prévues pour les appareils numériques de classe A et à la partie 15 des règlementation FCC à la règlementation des radio-interférences du Canadian Department of communications. Ces limites sont destinées à fournir une protection odéquate contre les interférences nétastes lorsque l'appareil est utilisé dans un environnement commercial.

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

9.10 Declaration of Conformity

We **BÜCHI** Labortechnik AG
Postfach, CH-9230 Flawil
Switzerland

declare under our sole responsibility that the product:
BÜCHI Melting Point **B-540/B-545**

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

EN 61010-1:1993 (~ IEC 1010-1, VDE 0411-1)
Safety requirements for electrical equipment for measurement, control an laboratory use: General requirements

EN 50014:1993
Limits and methods of measurement of radio disturbance characteristics of electrical motoroperated and thermal appliances for household and similar purposes, electric tools and similar electrical apparatus

EN 50082-1:1992
Electromagnetic compatibility - Generic immunity standard: Residential, commercial, light industry.

EN 60555-2:1987 (~ IEC 555-2)
Disturbances in supply systems caused by household appliances and similar electrical equipment: Harmonics

EN 60555-3:1987 (~ IEC 555-3)
Disturbances in supply systems caused by household appliances and similar electrical equipment: Voltage fluctuations

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

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

following the provisions of EU-Directive:
73/23/EEC (Low voltage)
89/336/EEC (Electromagnetic compatibility)

Flawil, 10.12.2001

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