





Imprint

Product Identification: Operation Manual (Original), Melting Point M-565

093256G en

Publication date: 07.2018

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

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The English manual is the original language version and serves as basis for all translations into other languages. Other language versions can be downloaded at www.buchi.com .

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1 About this manual

This manual describes the Melting Point M-565 and provides all the information required for the safe operation and to maintain it in good working order.

It is addressed in particular to laboratory personnel and operators.

NOTE

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

1.1 Reference documents

For more information regarding melting point, refer to the corresponding literature:

- The Laboratory Assistant 94187
- Melting Point M-560, Operating Manual numbers 93251–93255
- Melting Point M-565, Operating Manual numbers 93256–93260

1.2 Abbreviations

Chemicals:

- PP Polypropylene
- PE Polyethylene
- EPDM Ethylene-propylene-diene rubber
- POM Polyoxymethylen
- PUR Polyurethane

Miscellaneous:

тр	Melting	point
----	---------	-------

- bp Boiling point
- pharm. Pharmacopoeia
- therm. Thermodynamic
- L Sample left
- C Sample center
- R Sample right

2 Safety

This section highlights the safety concept of the Melting Point M-565, Sample Loader M-569 and the MeltingPoint Monitor software and contains general rules of behavior and warnings about hazards concerning the use of the product.

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

2.1 User qualification

The instrument may be used only by laboratory personnel or other persons whose training or professional experience give them an overview of the dangers which can develop when operating the instrument.

Personnel without this training or persons who are currently being trained require careful supervision. This Operation Manual serves as a basis for training.

2.2 Proper use

The instrument has been designed and built for laboratory use only. It is intended to be used to determine melting and boiling points and melting ranges at ambient temperatures up to 400 °C.

2.3 Improper use

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

The following applications in particular are expressly forbidden:

- Use in rooms requiring explosion-proof equipment.
- Extraction of samples which may explode or ignite as the result of a blow, friction, heat, or spark (e.g. explosives, et cetera).

2.4 Warning notices used in this manual



WARNING

WARNING Hot surface.

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



WARNING Electrical hazard.

WARNING Biohazard.



ATTENTION

The "read this" symbol for ATTENTION indicates that equipment damage, malfunctions, or incorrect processes may result if the instructions are not followed.

NOTE

Useful tips to facilitate operation of the instrument.

2.5 Product safety

The Melting Point M-565, Sample Loader M-569, and the MeltingPoint Monitor software are designed and built in accordance with current state-of-the-art technology. However, risks to users, property, and the environment can arise when the instrument is used carelessly or improperly.

The manufacturer has determined residual dangers emanating from the instrument

- if the instrument is operated by insufficiently trained personnel.
- if the instrument is not operated properly.

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

2.5.1 Instrument-related hazards

Pay attention to the following safety notices:



WARNING

Potentially hot surfaces during operation, especially the heating oven (up to 400 °C).

• Always be aware of the danger of being burned.

2.5.2 Other hazards



WARNING

Certain solvents within or in the vicinity of the Melting Point M-565 can form peroxides and/or are highly inflammable.

- Always be aware of the explosion risk when working with hazardous substances or with substances of unknown composition.
- Always use the instrument in an adequately ventilated work area.

2.5.3 Safety measures



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

2.5.4 Safety elements

<u>Anti-seismic tie-down</u>

• The instrument is equipped with a fixture to tie it down in the event of an earthquake (see bottom side of the instrument).

2.6 General safety rules

Responsibility of the operator

The head of laboratory is responsible for training the lab personnel.

The operator shall inform the manufacturer without delay of any safety-related incidents that occur during the operation of the instrument. Legal regulations, such as local, state, and federal laws applying to the instrument, must be strictly followed.

Duty of maintenance and care

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

Spare parts to be used

Use only recommended consumables and spare parts for maintenance in order to ensure continued optimum system performance and reliability. Modifications to the spare parts used are allowed only with the prior written permission of the manufacturer.

Modifications

Modifications to the instrument are permitted only after prior consultation with and written approval from the manufacturer. Modifications and upgrades should be carried out only by an authorized BUCHI technical engineer. The manufacturer reserves the right to decline any claim resulting from unauthorized modifications.

3 Technical data

This section introduces the reader to the Melting Point M-565 and its main components. It contains technical data, requirements, and performance data.

3.1 Scope of delivery

Check the scope of delivery according to the order number.

NOTE

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

3.1.1 Standard accessories



Table 3-1: Standard accessories		
Product	Order number	
Melting Point M-565	11058004	





Calibration set M-560 / M-565	11055018
(4 substances; 4-nitrotoluene, diphenyl	
acetic acid, caffeine, potassium nitrate)	

Melting point capillaries, 100 units 017808





Table 3-1: Standard accessories (c	(cont.)	
Product	Order number	
Sample holder	11055014	

Cleaning tool	051978

A) Boiling point tubes, 10 units	019697
B) Boiling point capillaries, 10 units	051850

11066387
051935
036721
051983
11055310
93256
93257
93258
93259
93260

3.1.2 Optional accessories

Netherferenzen 10 FDR	Table 3-2: Optional accessories	
Andrew York Seek 8 To Hone 8	Product	Order number
a Status Etablic Etabl	MeltingPoint Monitor software with license	11055332
\sim	Sample Loader M-569	051997
	Verification set M-560 / M-565	11055019
	(3 substances; benzil, p-anisic acid,	
Consts e	Melting point capillaries, 1000 units	001759
	Mortar and pestle, agate	041867

	Table 3-2: Optional accessories (cont.)	
	Product	Order number
Little the state of the	Compact keyboard German (CH)	029509
	Compact keyboard English (USA)	029508
	Serial dot matrix printer (including ribbon, paper roll, and cables)	11055438
ME DE		
	Ribbon for printer	044306
C starr		
	Paper roll for printer	038684
I	A) Boiling point tubes, 100 units	019007
	B) Boiling point capillaries, 100 units	051890



Table 3-2: Optional accessories (cont.)		
Product	Order number	
IQ/OQ: After installation		
OQ: For repeat use		
For further information please contact		
your local affiliate or distributor.		
IQ/OQ M-565 English	11055 004	
OQ M-565 English	11055 009	

3.2 Technical data overview

Table 3-3: Technical data of the Melting Point M-565

	Melting Point M-565	
Manual melting point determination	\checkmark	
Manual boiling point determination	\checkmark	
Automatic melting point determination	\checkmark	
Automatic boiling point determination	\checkmark	
Homogeneous sample loading	_	
Positions for melting capillaries	3	
Positions for boiling capillaries	1	
Precision magnifying lens	\checkmark	
Magnification of lens	2.5×	
Digital camera	\checkmark	
Video function	\checkmark	
Magnification, display	6 x	
Display	Colour, TFT, 320×240, 3.5"	
Determination temperature range	Ambient + 10 °C to 400 °C	
Temperature resolution	0.1 °C	
Accuracy melting point at 0.5 °C/min	± 0.2 °C	
Repeatability melting point at 0.5 °C/min	± 0.1 °C	
Accuracy boiling point at 1.0 °C/min up to	± 0,5 °C	
400 °C		
Repeatability boiling point at 1.0 °C/min	\pm 0.3 °C	
Temperature gradients, °C/min	0.1, 0.2, 0.5, 1, 1.5, 2, 2.5, 3, 5, 10, 20	
Heat-up time (50 °C–350 °C) at 25 °C	~ 4 min	
Cool-down time (350 °C–50 °C) at 25 °C	~ 13 min	
Electrical supply	100–240 V (±10%), 50–60 Hz	
Power consumption	150 W	
Contact termination	L, N, PE	
Video run time	350 min at 1 °C/min, 700 min at 0.5 °C/min	
Approval	CE, CSA, UL	
Dimensions (W×H×D), mm	190×200×370	
Weight, kg	4.5	
Environmental conditions	For indoor use only	
Temperature	5–40 °C	
Altitude	up to 2000 m a.s.l.	
Humidity	Maximum relative humidity 80% for temperatures up to 31 $^{\circ}\mathrm{C},$	
	decreasing linearly to 50% relative humidity at 40 °C	
Over voltage category	ll	
Degree of protection	IP 20	
Pollution degree	2	
Storable methods for melting point	50	
Storable methods for boiling point	50	

Table 3-3: Technical data of the Melting Point M-565 (cont.)

Melting Point M-565

Compliant with Pharmacopeia methods

PH. EUR., USP and JP

NOTE

Temperature measuring accuracy refers to pharmacopoeia melting point.

3.3 Materials used

Table 3-4: Materials used	
Component	Material designation
Print holder	РА
Heating block	Aluminium
Lenses	Glass
Axial fan	Aluminium
Housing	PU, stainless steel, glass
Cover	POM, ceramic, aluminium, stainless steel

4 Description of function

This section explains the basic principle of the Melting Point M-565 and provides a functional description of the assemblies.

4.1 Functional principle

The Melting Point M-565 is an instrument for automatic and visual (manual) determination of melting point, melting range, and boiling point at ambient temperatures +10 °C up to 400 °C. The melting point of three samples can be determined at the same time. The boiling point can be determined for one sample. Samples can be observed through the lens or on the color display.

Detection principle of the Melting Point M-565



Fig. 4.1: Reflection mode due to Ph. Eur. 6.1 2.2.60.

glass capillary tube
 sample
 camera
 temperature sensor
 heating block
 light source
 glass window

The melting point capillary sits in a hollow of a metal block, which is heated electrically and controlled by a temperature sensor. The heating block can be maintained accurately at a predefined temperature by the heating element and can be heated at a defined rate.

Detection is done as follows: The melting point capillary is illuminated from the front and the camera records the image. Melting and boiling points are detected by image processing.

4.1.1 Pharmacopoeia and thermodynamic melting points

The melting process of a substance does not take place instantaneously - it requires a finite amount of time. The melting process begins at the point where the first particles of the substance turn into the liquid state (thermodynamic melting point). The end of the melt is reached when the last solid particles have gone over into the liquid phase (pharmacopoeia melting point).

During the entire melting process of a pure compound, the temperature of the pure substance remains constant while heat is constantly transferred from the heating block to the sample.

For pure substances the thermodynamic melting point can be approached by multiplying the thermodynamic correction factor by the square root of the gradient and subtracting the result from the pharmacopoeia melting point.

$$mp_{[thermodyn.]} = mp_{[pharma.]} - (k \times gradient)$$



k = thermodynamic factor

Fig. 4.2: The amount of thermodynamic correction depends on the gradient selected: The smaller the gradient, the less the correction required.

4.1.2 Boiling points



The boiling point is determined by the "Siwoloboff" method.

The Melting Point M-565 can be used to determine the boiling point of a small amount of liquid. The heating block has one insert available for boiling point tubes (outside left). The moment of boiling is determined automatically or visually.

Detection is done as follows:

The beam of light illuminates the boiling point tube from the front, and the camera records the image.

Fig. 4.3: Boiling point

The process for boiling point determination is analogous to that for determining a melting point:

- The start temperature is set 5 to 10 °C lower than the expected boiling point.
- The sample is put into the heating block as soon as the start temperature is reached.
- A delay time allows the equilibrium between sample oven temperature and sample temperature. During this delay time some air bubbles turns out of the boiling point capillary.
- Starting from the start temperature, the sample is heated at a temperature gradient of 1 °C/min.
- As the temperature rises, bubbles of gas rise slowly and regularly from the immersed end of the boiling point capillary.
- The boiling point of the liquid has been reached when the flow of steam bubbles reaches a frequency of 0.6 Hz [Hertz].

The instrument records the curve and displays the result. At the beginning of the measurement, the current pressure has to be entered to obtain correct results. The system detects the boiling temperature. The boiling point is calculated and corrected for current pressure.

4.2 Controls and connections



- ① Power switch; turns the instrument on/off
- Start; starts process
- ③ Stop; stops process, starts/stops ventilation, returns to idle screen
- (4) 3 Select and Set buttons
- (5) Rotary knob for navigation in the menu and for select characters
- ⑥ Display

Fig. 4.4: Front view



Fig. 4.5: Rear view

- 1) Mains plug
- ② PS/2 connection for keyboard
- ③ RS 232 connection for serial printer
- ④ USB connection to the PC if software Melting Point Monitor is used, needed for service
- (5) Housing cooling fan
- (6) Heating block cooling fan

5 Putting into operation

This section describes the installation of the Melting Point M-565 and gives instructions for initial startup.

NOTE

Inspect the instrument for damage during unpacking. If necessary, prepare a status report immediately to inform the postal company, railway company, or transport company. Keep the original packaging for future transport.

5.1 Installation site

Place the instrument on a stable, horizontal surface adequate for the maximum product dimensions. It is advisable to place the instrument in a fume hood due to the fact that it will be used to measure chemical substances. For safety reasons and to ensure sufficient cooling in the electronic compartment, of the unit must be placed at least 30 cm away from rear walls or other objects. No containers, chemicals, or other appliances should be placed behind the unit.

NOTE

Do not expose the instrument to direct sunlight or very high illumination. This could influence the automatic detection process.

General hazards arise from:

- Mixtures of unknown composition or contaminations
- Combustible gases or solvent vapors in the immediate vicinity of the unit
- Damaged glass components
- Insufficient distance from back of the unit to the wall
- Burning by touching hot parts of the heater

5.2 Commissioning

5.2.1 Unpacking and installation



• Unpack the instrument and place it on a table. Remove the packing, and make sure that the following parts are installed:



• Glass window



- Install the glass window with glass holder on the front side of the heating block.
- Press the glass holder down.



• Glass window with glass holder



• Install the cover.





• Place the melting point capillaries in the hole of the housing. The second hole is a reservoir for used capillaries.

Accessories

5.2.2 Connecting to a PC with MeltingPoint Monitor software



The Melting Point M-565 can be operated in connection with the MeltingPoint Monitor software. In this case, connect the instrument with the PC or laptop using the USB cable. For detailed information check installation guide on software CD.

Melting Point M-565
 Lapton or PC with Melting

② Laptop or PC with MeltingPoint Monitor software

Fig. 5.1: Connection of Melting Point M-565 with the MeltingPoint Monitor software.

NOTE FOR USER MANAGEMENT

The software is not intended for use in the user level of the optional user management (11066387). The melting point instrument blocks the connection to the software. In the administrator mode the connection is possible.

5.2.3 Power connection

ATTENTION



Make sure that the voltage of the socket corresponds to the voltage given on the type plate of the instrument. Ensure that the instrument is grounded. External connections and extension cables must be provided with a grounded conductor lead (3-pole couplings, cable, or plug equipment) as the mains lead has a molded plug to avoid risks due to inadvertently defective wiring. Make sure that no electric sparks form in the instrument or its surroundings as they might damage the instrument. Make sure that the mains connector is freely accessable at any time.

5.2.4 Calibration

NOTE

Before use it is recommended to calibrate the apparatus. Use of supplied calibration substances is

recommended. Calibration is carried out in accordance with section 6.4 of these operating instructions.

6 Operation

This section explains the operating elements and possible operating modes. It gives instructions on how to operate the Melting Point M-565 properly and safely.

ATTENTION



Before use it is recommended to calibrate the apparatus. Use supplied calibration substances only. The calibration mode is described in section [6.4 Calibration].

6.1 Basic operating principles



WARNING

The heating oven can reach temperatures up to 400 °C.

6.1.1 Display during idle

After switching on, the display shows the menu for melting point.



- Function category with main setting parameters
- Hint
- ③ Parameters used after pressing START
- ④ Shows current heating block temperature and time
- (5) Functions selectable by the keys below

Different menus can be accessed by turning the rotary knob. Each menu has its own symbol. These idle screens are the starting points for all actions. The corresponding symbol is displayed during all processes.

25.0 °C 07	7:47	25.0 °C	11:5
10 11 + X		10 1ú 🔶 🔨	
Melting Point		Boiling Point	
Settings		Settings	
 Automatic detection: enabled Stop after detection: enabled 		 Automatic detection: e Stop after detection: e Boiling sample conditi 	enabled enabled oning: 30 s
Press START for measuring		Press START for meas	uring
76- 96 °C, 0.5 °C/	min	40- 60 °C,	0.5 °C/mir
Result Method Edit	t	Result Method	Edit
Melting Point		Boiling Point	
25.0 °C 18	8:23	25.0 °C	09:22
		10 10 + 1	
Calibration		Tools	
Press START to continue			
Buchi calibration set 6 valid results out of	12		
User calibration			
6 valid results out of	12		
Result Select Verifie	ed	Test SysInfo	Settings
Calibration		Tools	

6.1.2 Display during a process or in menus



- Shows where you are and gives hints and instructions.
- ② Working area shows:
 - items to select
 - samples during measurement
 - specific information in connection with the current menu

(3) Functions selectable by the keys below.



Screen during melting point determination



Result indication (numeric result)

6.1.3 Entering text





Settings menu



Result indication (replay)

Text can be entered by using the rotary knob or an external keyboard.

Rotary knob:

• Turn the rotary knob to select a character and press Enter. Press Save after entering all characters.

6.1.4 Using the external keyboard

NOTE

The instrument can be operated using an external keyboard.

For the softkeys, the following keys of the external keyboard are assigned:

- ENTER = right softkey
- Alt = center softkey
- Esc = left softkey
- In the method menu: To navigate quickly to a method name press the initial letter.

6.2 Melting point determination

NOTE

For exact melting point determination, use original capillaries from BUCHI only. If other capillaries are used, the results can be wrong. Use the following items:



Product	Order number
Melting point capillaries, 100 units	017808
Melting point capillaries, 1000 units	001759

Fig. 6.1: Melting point capillaries

6.2.1 Sample preparation

NOTE

Use only capillaries from BUCHI. They are precise and adequate for this kind of operation. Other capillaries have other dimensions and wall thicknesses. Using others may result in results that are incorrect.

Each sample has to be prepared.

The following methods to compact the samples are recommended:

- Using the Sample Loader M-569
- Knocking the capillaries on a hard surface.

NOTE

Sample preparation by letting the capillaries fall through a tube is not advised, because cross contamination may occur.

Preparation of the samples:

- The samples being investigated must be fully dry, homogenous, and in powdered form. Moist samples must be dried first (the pharmacopoeias prescribe that the substance needs to be dried in a vacuum for 24 hours over silica gel R). Coarse crystalline samples and non-homogeneous samples are finely ground in a mortar.
- To fill the capillary tubes with the sample, the open ends of the tubes are pressed into the substance. The substance is moved to the bottom of the tubes by repeatedly pounding the tubes against a hard base.
- Enough substance must be filled into the glass capillary to form a compact column 4 to 6 mm in height. (A height of 4–5 mm is recommended for precision measurements.) To ensure comparable results, it is important to fill all three capillary tubes to the same height and to compact the substance well in the tubes using the Sample Loader M-569.

6.2.2 Determination without pre-registered melting point method

- Choose the menu for melting point determination.
- Make sure that all samples are removed.
- Press START to start the determination process immediately with the last parameters used.

NOTE

If the current parameters do not meet your needs, press Edit and set start temperature, stop temperature, and temperature gradient.



- Start temperature
 Stop temperature
 Temperature gradient
 Edit
- To adjust the desired melting parameters, press **Edit**.



• Chose start temperature with the rotary knob, and press **Next**.



• Chose **stop temperature** with the rotary knob, and press **Next**.

25.0 °C		11:05
10 1 <u>8</u>	+	
Melting Point		
Settings		
 Automatic detection: enabled Stop after detection: enabled 		
Press START for measuring		
Temperature gradient		
79 - 99 °C, 0.5 °C/min		
Cancel		Save

• Chose temperature gradient with the rotary knob, and press **Save**.

NOTE

Insert samples only when the start temperature is reached (as soon as prompted).



• Press **START** to start the determination, and follow the instructions on the screen.



The instrument requests a sample ID during preheating (for details on entering text see 6.1.3). This request can be switched off by changing the setting "Sample ID request" (see 6.5).



Insert samples and press
 START to start the determination.



 To register additional temperature stamps, manually press the SET button for the related sample. Each button can be pressed three times. Registered temperature stamps are displayed in green.

NOTE

The result is indicated automatically after the melting point of each sample is detected.

I Result Press START to measure	e again
M-565 Melting Point	JCHI)
Parameters: Start temperature: Temp. gradient: 0.3	75 °C 5 °C/min
Melting result: acc. Pha Sample ID:	rmacop.
Auto (°C) L C 82.2 82.2	R 82.2
Average: Standard deviation:	82.2 °C 0.03 °C
Manual (°C) End Replay	Print

- To run through the results, turn the rotary knob up and down.
- To replay the recorded melting process, press **Replay**.
- Press **End** or **STOP** to return to the idle screen.
- Press **START** to measure again with the identical measuring parameters.
- The result is printed out automatically if a printer is connected.



- **Back**: Turns back to the result menu.
- **Sample**: Switches between sample left, middle, and right.
- **Play**: Replays recorded video automatically.
- Rotary knob: Changes temperature value and its corresponding picture of the sample.



- Changes replay speed of video using rotary knob.
- Stop: Stops replay mode.

NOTE

The last result is stored in the device until a new measurement is started or the instrument is switched off.



 After turning back to idle, the last result can be checked again by pressing **Result**.

6.2.3 Creating a method

Instead of entering and using the parameters directly from the idle screen, it is also possible to store parameter sets as methods.



• To enter the method menu, press **Method**.



 To create a new method, press Options → New then OK.

I Method Press START for measuring
Method Name:
ABCDEFGHIJKLM
NOPQRSTUVWXYZ
0123456789
+ * : % & / () = ? !
Cancel Enter Save

• Enter a method name. To finish press **Save**.



• Adjust a parameter and press **Next**. Then **Save**.

NOTE FOR USER MANAGEMENT

Creating a method is not possible when operating the instrument in the user level.

6.2.4 Using and handling methods

Nethod Press START				
PHENOLP	HTHA	LEIN		
:	230-2	250°C,	0.5°C/	min
VANILLI	N			
	75 -	95°C,	0.5°C/	min
Back	I Or	tions	Ed	lit
Datk	Lot	nions		ne

- Turn the rotary knob to select a method.
- Press **START** to start the determination.
- Use **Edit** to adjust method parameters.
- Press the **Options** key to get further functions:
- **New**: Creates a new method.
- **Delete**: Deletes the method.
- **Rename**: Changes the method name.

NOTE FOR USER MANAGEMENT

Methods can only be started in the user level. All other actions are not possible.

6.2.5 Adjusting parameters during a determination

NOTE

Measuring parameters can be modified during preheating or a determination. This function is possible only if enabled by the setting "Param. adj. during meas." (see 6.5).



- Turn the rotary knob to display the current parameter set.
- Adjust the parameters by pressing Edit. Adjusting the parameters does not affect the saved method.



If the temperature gradient is changed during a measurement, the result shows CHANGED (①) for the temperature gradient.

6.2.6 Printout

NOTE

The result is printed out automatically if a printer is connected and automatic printing is configured in the settings menu.

6.2.7 Melting result

NOTE

Changing the determination mode does not affect the principle of measurement, but it does influence the interpretation and the appearance of the results. The mode can be selected in the settings menu.

The default and most frequently used mode is the pharmacopoeia melting point. Melting range is convenient for substances with a wide melting range. The thermodynamic melting point should be used for special application only. For detailed information see Section 4.1.1.



 In the settings, change the melting result to Melting point pharmacopoeia and press Save.

Melting point pharmacopoeia



 In the settings, change the melting result to Melting range and press Save.

Settings		
Sample IE) request	
		enabled
Barometri	c pressure	request
		never
Boiling sa	Boiling sample conditioning	
		30 s
Molting re	eult	
acc. pharmacopoeia		
Tthermodynamic		
		range
Cancel		Save

 In the settings, change the melting result to Melting point thermodynamic and press Save.

Settings Sample ID request enabled Barometric pressure request never Boiling sample conditioning 30 s Melting recult acc. pharmacopoeia T thermodynamic range Cancel Save

In the settings, change the melting result to Melting point pharmacopoeia and press Save.



Printout

- 1) Instrument type
- ② Determination parameters
- ③ Automatically detected results
- ④ Manual results
- (5) Curve sample left
- 6 Curve sample center
- ⑦ Curve sample right
- (8) General information
- Signature of person making the determination

NOTE

The Average is calculated on the basis of the temperature values with two positions after the decimal point. Thus, the value indicated for the Average might deviate from the value you calculate on the basis of the temperature values on the printout, as there is just one position after the decimal point.

6.2.7.2 Melting range



• In the settings, change the melting result to **Melting** range and press **Save**.



An additional parameter is shown. The default value is set to 15%. This value can be used for most of substances.

M-565 Melting P	oint	BUI	<u>) (HI</u>
Method:			
Start tem Temp. gr Melting s	peraturi adient: tart thre	VAN e: 0.5 ' shold:	ILLIN 75 °C °C/min 15 %
Melting res	sult:		Range
Sample ID:	14.56		
Auto (°C) From: To:	L 79.5 82.2	C 79.5 82.2	R 79.5 82.2
Manual (°0 1: 2: 3:	>) 		
Sample left	79.5 -	82.2 °C	;

Threshold: This value is needed only for melting range determination. The default value is set to 15%. This value can be used for 80% of substances.

• If the beginning of the melting range differs from that observed, increase or decrease this value.



The result mode shows a range for each sample: XX.X–XX.X °C



In the replay mode, the melting range is shown in gray backlit.

0	M-565 Melting Point	(BUCHI)
2	Method: Start temperature: Temp. gradient: Melting start thres	VANILLIN 75 °C 0.5 °C/min hold: 15 %
	Melting result: Sample ID: 14.56	Range
3	Auto (°C) L From: 79.5 To: 82.2	C R 79.5 79.5 82.2 82.2
4	Manual (°C) 1: —— 2: —— 3: ——	
5	Sample left: 79.5 - 100%	82.2 °C
6	Sample center: 79.5	5 - 82.2 °C
	Sample right: 79.5 -	- 82.2 °C
8	*C 75 79 Date: Time: Last calibration: Last verification: Date, Signature:	09.10.2008 11:23 08.10.2008 08.10.2008 08.10.2008
- L	SN:	V 00.22

Printout

- ① Instrument type
- ② Determination parameters
- ③ Automatically detected results
- ④ Manual results
- (5) Curve sample left
- (6) Curve sample center
- ⑦ Curve sample right
- ⑧ General information
- Signature of person making the determination

6.2.7.3 Melting point thermodynamic



 In the settings, change the melting result to Melting point thermodynamic and press Save.



Experimental measurements have shown that in most cases a good approximation to the factor for thermodynamic correction of the BUCHI Melting Point M-565 is a value of ~1.5. Empirical calculation for thermodynamic correction is recommended when more accurate results are required for specific substances.

In order to obtain a thermodynamic melting point result within the accuracy of the unit, the following steps are advised:

- Perform complete automatic pharmacopoeia melting point determinations on your sample at three different temperature ramping rates: 0.2, 0.5, 1.0 °C/min.
- Plot the resulting melting points versus the square root of their corresponding temperature ramping rate (mp pharmacopoeia vs. $\sqrt{\text{gradient}}$)-a linear dependence should be observed.
- The slope is the thermodynamic correction factor. Enter this value in the setting 6.6 "Thermodynamic correction."

An example of this calculation procedure is shown below:

Gradient, r [°C/min]	Melting Point [°C]
0.2	236.1
0.5	236.5
1.0	236.9



"Melting point temperature vs. $\sqrt{\text{gradient}}$ " for a caffeine sample melted at 0.2, 0.5, and 1.0 °C/min. The slope of the straight line, factor for thermodynamic correction = 1.3, is programmed into the setting of instrument for this compound.

Fig. 6.2: "Melting point temperature vs. Vgradient"



Printout

- 1) Instrument type
- ② Determination parameters
- ③ Correction factor for thermodynamic determination
- (4) Automatically detected results
- (5) Manual results
- 6 Curve sample left
- ⑦ Curve sample center
- (8) Curve sample right
- (9) General information
- (1) Signature of person making the determination

NOTE

The Average is calculated on the basis of the temperature values with two positions after the decimal point. Thus, the value indicated for the Average might deviate from the value you calculate on the basis of the temperature values on the printout, as there is just one position after the decimal point.

6.3 Boiling point

NOTE

For exact boiling point determination, use boiling point tubes and capillaries from BUCHI only. If other glass parts are used, incorrect or no results can be obtained. Use the following items:



Product	Order number
A) Boiling point tubes, 10 units	019697
A) Boiling point tubes, 100 units	019007
B) Boiling point capillaries, 10 units	051850
B) Boiling point capillaries, 100 units	051890

Fig. 6.3: Boiling point tube and capillary

6.3.1 Sample preparation

In preparation, the boiling point tubes are filled with 5 to 10 mm of liquid sample. We recommend using a syringe for simple filling. Insert a boiling point capillary into the boiling point tube with its open/ thick end down. Put the sample immediately into the heating block.

6.3.2 Determination without pre-registered boiling point method

- Choose the menu for boiling point determination.
- Make sure that all samples are removed.
- Press START to begin the determination process immediately with the last parameters used. They are always saved in the instrument and are indicated in the green field in the lower part of the screen.

If the current parameters do not meet your needs, press Edit and set the start temperature, stop temperature, and temperature gradient.



- Start temperature
 Stop temperature
 Temperature gradient
 Edit
- To adjust the boiling parameters, press Edit.



• Use the rotary knob to choose the **start tempera-ture** and press **Next**.



• Use the rotary knob to choose the **stop tempera-ture** and press **Next**.



 Use the rotary knob to chose the temperature gradient and press Save.

NOTE

Insert sample only when the instrument has reached the start temperature.

25.0 °C 11:	53
10 1ú 💠 🔧	
Boiling Point	
Settings	
 Automatic detection: enabled Stop after detection: enabled Boiling sample conditioning: 30 s 	5
Press START for measuring	
40- 60 °C, 0.5 °C/m	In
Result Method Edit	

• Press START to start the determination, and follow the instructions on the screen.

ല്ല് 42.0 °C Insert sample, press START			
.0 Hz			
Sample ID:			
354-45			
ABCDEFGHIJKLM			
NOPQRSTUVWXYZ			
0123456789			
+ * : % &/() = ? !			
Cancel Enter Save			

The instrument requests a sample ID during preheating (for details on entering text see 6.1.3). This request can be switched off by changing the setting "Sample ID request" (see 6.5).

ाsert Sam	0 °C oles. Press S	TART
1.0 Hz		
0.6 Hz		
<u>0.0 Hz</u> *C	50 60	-
	Bar P.	Samp. ID

- **Bar P**: Enter the current barometric pressure.
- This request can be switched off by changing the setting "Barometric pressure request" (see 6.5).
- Insert sample and press **START** to start the determination.

ില് 42.0 °C Delay 29 s	
1.0 Hz	
0.6 Hz	
0 <u>.0 Hz</u>	-
Set	

Delay: to ensure equilibrium between oven temperature and sample temperature. During this delay time some air bubbles turns out of the boiling point capillary. This request can be changed in the setting "Boiling sample conditioning" (see 6.5).

Press START 1	lt to measu	re again
Sample ID: 354-45		
Automatic Boiling Point: Boiling Temper	<u>73.2</u> rature:	<u>76.4</u> °C —— °C
Manual (°C) 1: 2: 3:	Temp. <u>73.4</u>	Point <u>76.6</u>
1.0 Hz		I
Back		Print

• To run through the results, turn the rotary knob up and down.

Video replay is not available for boiling point determination.

• Press **End** or **STOP** to return to the idle screen.

• Press **START** to measure again with the identical measuring parameters.

6.3.3 Creating a method

NOTE

The process of creating a boiling point method is identical that used to create a melting point method (see 6.2.3).

6.3.4 Adjusting parameters during a determination

During preheating or a determination, measuring parameters can be modified. This function is possible only if enabled by the setting "Param. adj. during meas." (see 6.5).



- Turning the rotary knob displays the current parameter set.
- Adjust the parameters by pressing Edit. Adjusting the parameters does not affect the saved method.



If the temperature gradient is changed during a measurement, the result shows CHANGED (①) for the temperature gradient.

When a measurement is done, the last method parameters are always retained in the instrument. To start the same method, just press **START**.

NOTE

Results are stored in memory until a new measurement is started or the instrument is switched off.

6.3.5 Printout



Printout

Instrument type

Determination parameters

③ Automatically detected results

(4) Manual results

(5) General information

Signature of person making the determination

Boiling temp. = Temperature sample boiled.

Boiling point = Boiling temp. corrected according entered barometric pressure. If a barometric pressure of 1013 mbar is entered boiling temperature and boiling point results are equal.

6.4 Calibration

NOTE

BUCHI recommends calibrating each new instrument after installation. Furthermore it is recommended that the instrument be recalibrated every year.

Two calibration modes are available: with BUCHI substances and user defined substances

6.4.1 Calibration principle

NOTE

The instrument is calibrated using melting point standards. The calibration is valid for boiling points

as well. The calibration procedure is recommended to be performed with the BUCHI calibration

substances, in the BUCHI calibration set mode.

Use the calibration set (11055018). This calibration set contains the melting point standards listed below. Each standard is shipped with a certificate of analysis and an MSDS (material safety data sheet).

The calibration set contains the following substances:

•	4-Nitrotoluene:	approx.	52	°C
•	Diphenyl acetic acid:	approx.	148	°C
•	Caffeine:	approx.	237	°C
•	Potassium nitrate:	approx.	335	°C

From each substance, a minimum of 6 have to fulfill a standard deviation of less than +/- 0.2 °C. Otherwise the instrument will not move on to the next substance. The maximum number of samples for each substance to reach the deviation of +/- 0.2 °C is limited to 12 samples. The instrument automatically chooses the best 6 results from the determinations performed.

After a successful calibration is done, it may be checked using the verification set (11055019). Each standard of the set contains a certificate of analysis and the MSDS.

The verification set contains the following substances:

•	Benzil:	approx.	94	°C
•	p-Anisic acid:	approx.	182	°C
•	Phenolphthalein:	approx.	261	°C

NOTE FOR USER MANAGEMENT

The calibration procedure can only be performed in the administrator level.

6.4.2 Calibration procedure

To start calibration, turn the rotary knob to Calibration. With the select button the calibration principle can be chosen. Press **START** and follow the instruction on display. BUCHI calibration set:

25.0 °C 18:31 Melting Point Settings - Automatic detection: enabled - Stop after detection: enabled Press START for measuring 40 - 80 °C, 0.5 °C/min Result Method Edit

The instrument automatically heats to the start temperature of the first substance. In the meantime, prepare at least 6 samples of the given substance. For this process, follow section 6.2.1.



After 6 results are obtained within a standard deviation of +/- 0.2 °C, the process moves on to the next substance. The process is identical for the other substances.

25.0 °C		18:40
10 16	+ \	
Calibra Last calibra 08.09.2017 Last verific	tion ation: 7 Buchi calib ation:	ration
Press STA	RT to continu	ie
Būchi cali	ibration set	
	6 valid resu	Its out of 12
User calib	oration	
	6 valid resu	lts out of 12
Result	Select	Verified

The calibration date is now saved and indicated on the idle screen.



• Choose the correct pharmacopoeia melting point value with the rotary knob. The pharmacopoeia melting point is written on the certificate of analysis. Press **Save**.

Result Calibration complete		
Calibration Result Date Time Temp. Gradient Standard dev. limit No. of req. samples	11.07.2008 11:08 0.5 °C/min 0.2 °C 6	
4-Nitrotoluol	C R	l
New calibration c	late saved	

As soon as a complete calibration is obtained, the new calibration date is saved automatically.

 Press OK. All results obtained are displayed.



Follow the instructions on the display. After each run, the current result is indicated.

• Press **START** to perform the next measurement.

🔶 R Calibrati	esult	ete	
Calibra Date Time Temp. G Standard No. of re CALIBR	tion Res radient d dev. lim q. sample ATION (ult 11.0 0.5 it es COMPLI	17.2008 11:08 °C/min 0.2 °C 6 ETE
4-Nitrot	oluol		
	L	С	R
°C 1:	48.4	48.4	48.4
2:	48.5	48.5	48.5
3:			
Back			Print

- The rotary knob can be used to display all the results of the calibration.
- The **Print** key is used to print out the data.
- Back exits this menu.

User calibration:



By choosing the user calibration, 4 freely selectable substances can be chosen for calibration.

Result Press START to continue No. on reg. samples o CALIBRATION INCOMPLETE			
4-Nitrot	oluol L 48.4	C 48.4	R 48.4
2: 3: 4:	48.5	48.5	48.5
Average: Standarc Referenc	l Deviatio :e temp.:	in: 0 5	48.4 °C 0.05 °C 52.0 °C
Back	Cle	ar	Print

Set the melting point value for the substance using the rotary knob.



Choose the gradient for the calibration with the rotary knob.

Result Calibration complete	
Calibration Result Date 11.07.2008 Time 11.08 Temp. Gradient 0.5 °C/min Standard dev. limit 0.2 °C No. of req. samples 6 CALIBRATION COMPLETE	
4-Nitrotoluol	L
L C R	
°C 1: 48.4 48.4 48.4	ч,
New calibration date saved	I
ок	

Enter the Lot number for the substance used.



Insert the name of the substance with starting the lowest melting point used for the calibration.

Calibration complete			
Calibration Result Date 11.07.2008 Time 11:08 Temp. Gradient 0.5 °C/min Standard dev. limit 0.2 °C No. of req. samples 6 CALIBRATION COMPLETE 6			
4-Nitro	toluol		
	L	С	R
°C 1:	48.4	48.4	48.4
2:	48.5	48.5	48.5
3:			
Back			Print

Enter the correct reference temperature for the substance according to the data sheet. This value is used for the actual calibration. Measurement as described for BUCHI calibration.



The calibration date is now saved and indicated on the idle screen..

6.4.3 Printout

(2) Temp. gradient 0.5 °C/min Standard deviation limit 0.2 °C No. of required samples 6 CALIBRATION COMPLETE 4-Nitrotoluene Auto (°C) L 2 52.0 3 4: Average: 52.1 52.0 52.0 3: 4: Average: 52.1 °C Standard deviation: 0.05 °C Reference temperature: 52.0 °C Diphenylacetic Acid Auto (°C) C R 1: 4: Average: 147.5 147.5 2: 147.7 3: 4: Average: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C <t< th=""><th>1</th><th>M-565 Calibration</th><th>CHI)</th></t<>	1	M-565 Calibration	CHI)
Image: Standard deviation: 0.05 vm cterrer Image: Standard deviation: 0.08 vm cterrer Image: Standa	2	Temp. gradient 0.5 Standard deviation limit No. of required samples	i°C/min 0.2°C 6 FTF
③ 4-Nitrotoluene Auto (°C) L C R 1: 52.0 52.1 52.0 3: 4: 2: 52.1 52.0 3: 4: 4: 4: 4: Average: 52.1 °C Standard deviation: 0.05 °C Reference temperature: 52.0 °C Ø Diphenylacetic Acid Auto (°C) L C R 1: 147.5 147.5 147.5 4: Auto (°C) L C R 1: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 147.6 °C Standard deviation: 0.08 °C Go Zaffeine Auto (°C) L C R 1: 236.8 236.7 2: 236.8 236.7 2: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard d			
Average: 52.1 °C Standard deviation: 0.05 °C Reference temperature: 52.0 °C Diphenylacetic Acid Auto (°C) L C R Auto (°C) L C R 1: 147.5 147.5 147.5 2: 147.7 147.6 147.5 2: 147.7 147.6 147.5 3: 4: Average: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 334.6 334.6 334.6 2: 334.6 334.4 334.7 3: 3: 4: Average: 334.6 °C Standard deviation: 0.10 °C Reference temperature	3	4-Nitrotoluene Auto (°C) L C 1: 52.0 52.1 2: 52.1 52.0 3: 4: 4:	R 52.1 52.0
(a) Diphenylacetic Acid Auto (°C) L C R 1: 147.5 147.5 147.5 2: 147.7 147.6 147.5 3: 4: (a) 1: 147.5 147.5 147.5 2: 147.7 147.6 147.5 3: 4: (a) Average: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 147.6 °C (b) Average: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 147.6 °C (c) Caffeine Auto (°C) L C R 1: 236.8 236.9 236.7 2: 236.8 236.8 236.7 3: 4: (c) Average: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C 		Average: Standard deviation: Reference temperature:	52.1 °C 0.05 °C 52.0 °C
Auto (°C) L C R 1: 147.5 147.5 147.5 2: 147.7 147.6 147.5 3: 4: 4: 4: Average: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 147.6 °C Caffeine Auto (°C) L C R 1: 236.8 236.9 236.7 2: 236.8 236.9 236.7 2: 236.8 236.9 236.7 2: 236.8 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Standard deviation: 0.08 °C 3: 4: Average: 334.6 °C Standard deviation: 0.10 °C 6 I 334.5 °34.6 °C Standard deviation: 0.10 °C Reference temperature: 334.5 °C 7 I I Average: 09.10.2008 <		Diphenylacetic Acid	
Average: 147.6 °C Standard deviation: 0.08 °C Reference temperature: 147.6 °C Reference temperature: 147.6 °C Reference temperature: 147.6 °C Reference temperature: 147.6 °C Caffeine Auto (°C) L C Auto (°C) L C R 1: 236.8 236.9 236.7 2: 236.8 236.8 236.7 3: 4: Average: 236.8 °C Aterage: 236.8 236.8 °C Standard deviation: 0.08 °C Reference temperature: Average: 236.8 °C C Atto (°C) L C R 1: 334.5 334.6 334.6 2: 334.6 334.4 334.7 3: 4: 4: 4: Average: 334.6 °C Standard deviation: 0.10 °C Reference temperature: 334.5 °C C Standard deviation: 0.10 °C Reference temperature: 334.5 °C Standard deviation:	4	Auto (°C) L C 1: 147.5 147.5 2: 147.7 147.6 3: 4:	R 147.5 147.5
(5) Caffeine Auto (°C) L C R 1: 236.8 236.9 236.7 2: 236.8 236.8 236.7 3: 4: 236.8 236.8 236.7 3: 4: 236.8 236.8 236.7 3: 4: 236.8 236.8 236.7 3: 4: 236.8 236.8 236.7 6 Potassium Nitrate Auto (°C) L C R 1: 334.5 334.6 334.6 2: 334.6 34.6 2: 334.6 334.4 334.7 3: 4:		Average: 1 Standard deviation: Reference temperature: 1	47.6 °C 0.08 °C 47.6 °C
Average: 236.8 °C Standard deviation: 0.08 °C Reference temperature: 236.8 °C Reference temperature: 236.8 °C Reference temperature: 236.8 °C Potassium Nitrate Auto (°C) L Auto (°C) L C R 1: 334.5 334.6 334.6 2: 334.6 334.4 334.7 3: 4: Average: 334.6 °C Standard deviation: 0.10 °C Reference temperature: 334.5 °C ⑦ Date: 09.10.2008 11:16 ⑧ Date, Signature: SN: V 00.22	5	Caffeine Auto (°C) L C 1: 236.8 236.9 2: 236.8 236.8 3: 4:	R 236.7 236.7
Image: Standard deviation: 0.10 °C Image: Standard deviation: 0.10 °C <td< th=""><th></th><th>Average: 2 Standard deviation: Reference temperature: 2</th><th>36.8 °C 0.08 °C 36.8 °C</th></td<>		Average: 2 Standard deviation: Reference temperature: 2	36.8 °C 0.08 °C 36.8 °C
Average: 334.6 °C Standard deviation: 0.10 °C Reference temperature: 334.5 °C Date: 09.10.2008 Time: 11:16 Date, Signature: SN:	6	Potassium Nitrate Auto (°C) L C 1: 334.5 334.6 2: 334.6 334.4 3: 4:	R 334.6 334.7
⑦ Date: 09.10.2008 Time: 11:16 Date, Signature: SN:		Average: 3 Standard deviation: Reference temperature: 3	34.6 °C 0.10 °C 34.5 °C
SN: V 00.22	⑦ ⑧	Date: 09.1 Time: Date, Signature:	10.2008 11:16
	_	SN:	V 00.22

Printout

- 1) Instrument type
- ② General information regarding calibration
- ③ Results 4-nitrotoluene
- ④ Results diphenylacetic acid
- (5) Results caffeine
- 6 Results potassium nitrate
- ⑦ General information
- (8) Signature of person making the calibration

NOTE

The Average is calculated on the basis of the temperature values with two positions after the decimal point. Thus, the value indicated for the Average might deviate from the value you calculate on the basis of the temperature values on the printout, as there is just one position after the decimal point.

6.4.4 Verification

To verify the calibration, proceed as follows:

• Measure all standards (BUCHI recommends using the verification set (11055019) but it is also possible to use your internal standards.

NOTE

The verification is not a guided process.

• When all measuring results were within the required tolerances, click on **Verified**. The following screen appears:

25.0 °C	14:20
10 10 🕈 🔧	
Colibration	
Last calibration:	
Password:	
VER	
ABCDEFGHIJKLM	(
NOPQRSTUVWXYZ	:
0123456789	
+ * : % & / () = ?	'!
Cancel Enter O	К

• Enter the following password: VER. The current date is now indicated under "Last verification".

NOTE FOR USER MANAGEMENT

Can only be accessed in the administrator level.

6.5 Settings, SysInfo, Test



NOTE FOR USER MANAGEMENT Can only be accessed in the administrator level. **Test:** Functional test of the instrument. To perform the test of several functions follow carefully the instuction on the screen. If a function test shows not **ok**, please contact the customer service. The Test protocol is described in section 6.5.1.

SysInfo: Instrument gives information regarding settings and connected devices.

Settings: This menu can be used to change parameters. The SysInfo protocol is described in section 6.5.2.



• Press the **Test** button to enter the functional test, and follow the instructions.

SysInfo	
M-565 SysInfo	(BUCHI)
Language	English
Display brightness	100 %
Temperature unit	°C
Pressure unit	mbar
Buzzer volume	inijai
Date format	4 %
	DD.MM.YYYY
Back	Print

• Press the **SysInfo** button to open the system information menu.

Printout is possible if a printer

is connected.

Settings

• Press the **Settings** button to enter the settings menu.

Table 6-1: Setting parameters	
Language	English, German, French, Italian, Spanish, Japanese, Chinese
Stop after detection	disabled, enabled: If the instrument finds a melting point or boiling point for every detected sample, measurement stops automatically.
Param. adj. during meas.	disabled, enabled: Whether or not it is possible to change parameters (start temperature, stop temperature, and temperature gradient) during a determination.
Result printout automatically	disabled, enabled: After a measurement is finished, results will be printed automatically.
Result graph indication	disabled, enabled: Each result contained is shown with a graphic.
Calibration interval	0–36 months: default 12 months. BUCHI recommends calibration or verification after 12 months using the calibration set and the verification set.
Sample ID request	enabled: After the start of determination, the sample ID window is displayed.
	disabled: The sample ID window is not automatically displayed. In combination with the MeltingPoint Monitor software, it is advisable to turn this feature off.
Barometric pressure request	Has impact only on boiling point determination.
	never: No pop-up screen is shown.
	daily: Enter current barometric pressure once a day.
	always: Enter current barometric pressure every time the instrument heats up to the start temperature for boiling point determination.
Boiling sample conditioning	0–600 s: The default value is set to 60 seconds. This is needed to ensure a temperature equilibrium between heating block and liquid in the boiling point tube. If this value is set too low, the result can be incorrect or no automatic boiling point detection.
Melting result	acc. pharmacopoeia
	thermodynamic
	range

Table 6-1: Setting parameters (cont.)	
Thermodynamic correction	0.0-3.0; default value: 1.1
Company name	The name of a company can be entered.
Date (DD.MM.YYYY)	XX.XX.XXXX
Time (24 h)	XX:XX
Date format	DD.MM.YYY, MM/DD/YYYY, YYYY-MM-DD
Time format	24 h, AM/PM
Temperature unit	°C, °F, K
Buzzer volume	0, 1, 2, 3, 4
External Keyboard	CH, USA
Display brightness	0–100%
Automatic detection	Enable or disable the automatic detection. If disabled, stop tempera-
	ture of method is the end of the determination.
Statistic	Enable or disable the statistic. If disabled, mean value and standard
	deviation are not shown in the results.
Camera position	Use the rotary knob to position the dashed rectangles at the bottom
	part of the substance according to the example.

Change Direction to move vertically or horizontally.



User management

Activation key will enable the user management setting. After an administrator password can be defined. The instrument can only be started in the administrator mode if the correct password is entered.

6.5.1 Test protocol

[M-565	
	Test Result	
	Keypad	OK
	Rotary knob	OK
	24V input voltage	OK
	24V after fuse	OK
	5V	OK
	Heating present	OK
	Cooling fan present	OK
	Housing fan present	OK
	Temp. sensor present	OK
	Temp. sensor function	OK
	External keyboard	OK
	Board temp. sensor	OK
	Heating function	OK
	Cooling fan	OK
	Housing fan	OK
	Sample illumination	ОК
	Camera position	ОК
	Camera brightness	ОК
	Internal clock	ОК
	Display	OK
_	Com volue w/e comp l	14
	Cam, value w/o samp, C	14
	Cam. value w/o samp. C	10
2	Cam. value w/o samp. R	100
	Cam. value w/ samp. L	100
	Cam. value w/ samp. C	114
	Cam. value w/ samp. R	112
3	Date, Signature:	
	SN:	V 01.05

- ① Power supply voltage
- (2) Values for sample brightness
- (3) Signature of person carrying out the test

6.5.2 SysInfo protocol

	M-565 SysInfo BUCHI) (1) Settin
	Settings	-
	Language Englisł	n
	Stop after detection	4
	Param. adjust. during meas.	4
	Result printout automatically	4
	Result graph indication	4
	Calibration interval	
	Sample ID request	
	Barometric pressure request	
	Boiling sample conditioning	5
	Melting result	5
\odot	acc. pharmacopoen Thermodynamic factor	a
	Company name	
	Date (DD.MM.YYYY)	
	Time (24 h)	7
	Date format	/
	Time format	
	Temperature unit	、 、
	Pressure unit	r
	Buzzer volume	
	External keyboard	4
	Display brightness	
	Automatic detection	4
	Statistic	4
L		<u> </u>

Setting parameters

ſ	Service Serial number
	1000005820
	01.05.11
	00.15
	576392.8 h
	No. of melting point meas. 97
	No. of boiling point meas.
	Number of calibrations 14
	Calibration date 20.03.2009
	Verification date 16.02.2009
	Factory adj. value at 20°C -0.26 °C
	Calibration value at 52°C -0.04 °C
2	Calibration value at 148°C −0.54 °C
	Calibration value at 237°C 1.46 °C
	Calibration value at 335 °C 2.62 °C
	Factory adj. value at 400°C 3 27 °C
	Sample illumin. brightness
	Camera position horizontally
	Camera position vertically -5
	Color gain green for meltingp.
	14U Color gain blue for meltingp.
	205 Color gain red for meltingp.
4	110 Color gain green for boilingp.
	75 Color gain blue for boilingp.
	110 Color gain red for boilingp.
	60 Board test date
(5)	—.—. End test date
	—.—. Board temperature
	42 °C Heating present
	OK Cooling fan present
	OK Housing fan present
	OK Temp. sensor present
	OK Temp, sensor function
	OK 24V before fuse present
	24V after fuse present
	OK OK
	OK Date, Signature:
"L	SN: V 01.05
L	

② Correction values for calibration

③ Camera position values

- (4) Color values (Factory default settings)
- (5) Date of the factory tests

(6) Signature of the person who performed the test

6.6 User management

An optional user management (11066387) is available to provide regulatory compliance. A serial number dependent activation code has to be entered in the settings menu. After entering the activation code, a password to enter the administrator level can be defined.

With the user management a password identification is requested when switching on the instrument.

- If the password is successfully entered, the user gets access to the administrator level.
- Without password identification or the wrong password the user has limited access to the instrument functionalities.

Administrator level

- 1. Full access to the instrument functionalities
- 2. Access to the Service menu
- 3. Possibility to change the password
- 4. Connection to MeltingPoint Monitor Software possible

User level

- 1. Possibility to choose between melting point and boiling point
- 2. Access to the following parameters: start temperature, heating rate and end temperature
- 3. Selection of methods, but no editing and deleting
- 4. No access to: changing of date and time, calibration data and calibration menu.
- 5. No connection to: MeltingPoint Monitor Software

The user management setting for the password protection can be undone by deleting the password in the administrator mode and leaving the field blank. The user management setting can be reactivated with the activation key.

The activation key is only valid in the year of purchase. For later activation please contact: registration@buchi.com

6.7 XML to PC data export

If no printer is connected to the MeltingPoint device, data can be sent to a PC by pushing the "XML2PC" button. The raw data is transmitted through the serial interface and can be received by the PC with the following settings:

Table 6-7: PC settings		
Baud rate:	19200	
Parity:	No	
Data bit:	8	
Stop bit:	1	

No PC software is supplied to receive the XML data. Picking up the data is the customers responsibility.

7 Maintenance

This section provides instructions on all maintenance work to be performed to keep the instrument in good working condition.



WARNING

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



WARNING

Electrical hazard:

• Prior to any maintenance work on the instrument, switch off the power supply.



ATTENTION

Use only genuine BUCHI consumables and spare parts for any maintenance and repair work in order to assure continued system performance and reliability. Any modifications to the spare parts used should be carried out only with the prior written permission of the manufacturer.

7.1 Housing

Check the housing of your melting point instrument for defects (controls, plugs). The housing is coated with paint and should be cleaned only with a rag moistened in a soapy solution. Remove the glass window from the heating block periodically and wipe it clean with alcohol or acetone. Replace the window with a new one if it will not come completely clean.



ATTENTION

Never use any halogenated solvents, acetone, or similar chemicals, because such cleaning agents may damage the instrument.

7.2 Glass window

Remove the glass window from the heating block periodically and wipe it clean with alcohol or acetone. Replace the window with a new one if it will not come completely clean.

7.3 Upkeep

The upkeep of the unit is mainly limited to:

• Periodic calibration of the temperature.

7.4 Cleaning the heating block

Remove broken capillaries from the heating block.

ATTENTION



- Wait for the heating block to cool down before touching it!
- Remove the cover.

Follow the instructions mentioned below on the use of the cleaning tool 051978.





- If a melting point capillary is broken and is stuck in the heating block, use the **cleaning tool** (Order number: 051978).
- Remove the cover and glass holder with glass window.

• If glass parts of a melting point capillary are stuck in a hole of the metal heating block, use the cleaning tool as shown in the picture.



8 Troubleshooting

The following section describes how to resume operation of the instrument in the event of any minor problem. It lists some possible occurrences, their probable cause, and suggests how to remedy the problem. The troubleshooting table below lists possible instrument malfunctions and errors and describes courses of action that can be used by operators to correct some of those problems. The appropriate course of action is listed in the column "Remedy".

More complicated malfunctions or errors are usually handled by a BUCHI technical engineer who has access to the official service manuals. In this case, please contact your local BUCHI customer service agent.

Table 8-1: General malfunctions ar	nd their remedy		
Malfunction	Possible cause	Remedy	
Instrument does not work	Main switch off	Switch on mains switch	
	Instrument is not connected to mains	Check mains connection	
	supply		
No or unreadable printout	Not activated in the settings	Activate automatic printout in the settings	
	Bad cable connection	Check cable connection	
	Printer switched OFF	Switch ON printer	
	No paper	Replace paper roll	
	Baud rate settings of printer faulty	See section 8.1.1	
No melting curve is shown	Not activated in the settings	Activate graphic in the settings	
	No automatic detection of samples	Manually register the melting point/ boiling point	
	Measuring finished before detecting a result for all samples	Turn off in the settings "Stop after detection" and check stop tempera- ture.	
In the automatic melting point deter- mination, no value and no melting curve are determined	Sample is unstable, it decomposes, turns brown, or melts non-uniformly	e.g. select higher maximum point	
	The temperature parameters have been entered incorrectly and the sample does not melt at all		
	Automatic detection is disabled	Enable the Automatic detection in the Settings	
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.	
Unexpected results	Sample-specific preparations:		
	Sample is not dry or is contaminated by another substance	Sample should be dried before use	

8.1 Malfunctions and their remedy

Table 8-1: General malfunctions and their remedy					
Malfunction		Possible cause		Remedy	
		Sample decomposes durin	g the		
		melting process (formation	n of		
		bubbles, sample turns brow	wn, etc.)		
		Apparatus parameters:			
		Apparatus is not or is poor	ly cali-	Regular calibration of apparatus with	
		brated		BUCHI calibrating substances	
		Method "According to phar	rmaco-	Alter the corresponding parameters	
		poeia" or "thermodynamic	melting		
		point determination" is inc	orrectly		
		selected			
No results		Setting point is too close to	o the	Select setting point 5–10 °C below	
		melting point		melting point	
No statistic		Setting parameter Statistic	is	Enable the Statistic in the Settings	
		disabled			
Instrument doe	s not heat	Heating defective		Contact BUCHI customer service.	
Table 8-2: Ma	functions with Meltin	ng Point Monitor software			
Malfunction		Possible cause		Remedy	
No USB connec	tion	USB cable is not connected	d or defec-	Check USB connection, replace if	
		tive.		necessary.	
Driver not found	d	Device connected for the f	irst time.	Install recommended driver.	
Table 8-3: Wa	rning messages				
Warning number	Possible cause		Remedy		
Warning 01	Calibration outdated.		Calibrate o	or verify device.	
Warning 02	Housing temperature	too high.	Cool down	device and check clearance of vent	

Warning 03 Device not calibrated.

Table 8–4: Error messages				
Error number	Possible cause	Remedy		
Error 01	Memory data loss, all data are reset.	Calibrate device. In case of recurrence contact customer service.		
Error 02	Automatic restart, firmware problem possible.	In case of recurrence contact customer service.		
Error 03	Board temperature sensor defective.	Contact customer service.		
Error 04	Temperature sensor defective.	Contact customer service.		
Error 05	Heating defective.	Contact customer service.		
Error 06	Cooling fan defective.	Contact customer service.		
Error 07	Housing fan defective.	Contact customer service.		
Error 08	Camera defective, communication loss.	Contact customer service.		
Error 09	24 V fuse defective.	Contact customer service.		
Error 10	24 V input voltage missing.	Contact customer service.		

holes and fans.

Calibrate device.

Check ambient temperature.

Table 8–4: Error messages			
Error number	Possible cause	Remedy	
Error 11	Internal clock, power loss.	Contact customer service.	
Error 13	5V input voltage missing	Contact customer service.	

NOTE

If several errors are pending, the one with the highest priority will be displayed.

8.1.1 Setting the printer baud rate



• Open compartment on the bottom of the printer.



The following setting should be selected.
 1,2,3,4,5,7,8,9,10 = ON
 6 = OFF

8.2 Customer service

Only authorized service personnel are allowed to perform repair work on the instrument. They have comprehensive technical training and knowledge of possible dangers that might arise from the instrument.

Contacts for official BUCHI customer service offices are available on the BUCHI website at: www.buchi.com. If malfunctions occur on your instrument or you have technical questions or application problems, please contact one of these offices.

Customer Service offers the following:

- Spare part delivery
- Repairs
- Technical advice

9 Shutdown, storage, transport, and disposal

This section provides instructions on how to shut down the instrument, how to pack it for storage or transport, and specifies the storage and shipping conditions.

9.1 Storage and transport



WARNING

Biohazard:

- Remove all dangerous substances from the instrument, and clean it thoroughly.
- Store and transport the instrument in its original packaging.



WARNING

Electrical hazard:

• Always remove the power cord from the socket first to avoid having live cables in the laboratory.

9.2 Disposal

To dispose of the instrument in an environmentally friendly manner, a list of materials is given in section 3.3, please ensure that the components are separated and recycled correctly. Please follow current regional and local laws concerning disposal.

10 Spare parts

This section lists spare parts, accessories, and optional extras, including all of the relevant order information for ordering from BUCHI. Always state the product designation and part number when ordering any spare parts.

To ensure optimum system performance and reliability, use only genuine BUCHI consumables and spare parts for maintenance and repair. Prior written permission of the manufacturer should be obtained before any modifications are made to the spare parts used.



Fig. 10.1: Spare parts

Table 10-1: Spare parts						
Product	Order number	Product	Order number			
Cover	051900	Ring	051848			
Glass holder	051847	Rotary knob	11055017			
Glass window	051849					

11 Declarations and requirements

11.1 FCC requirements (for USA and Canada)

English:

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

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

<u>Français:</u>

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

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

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