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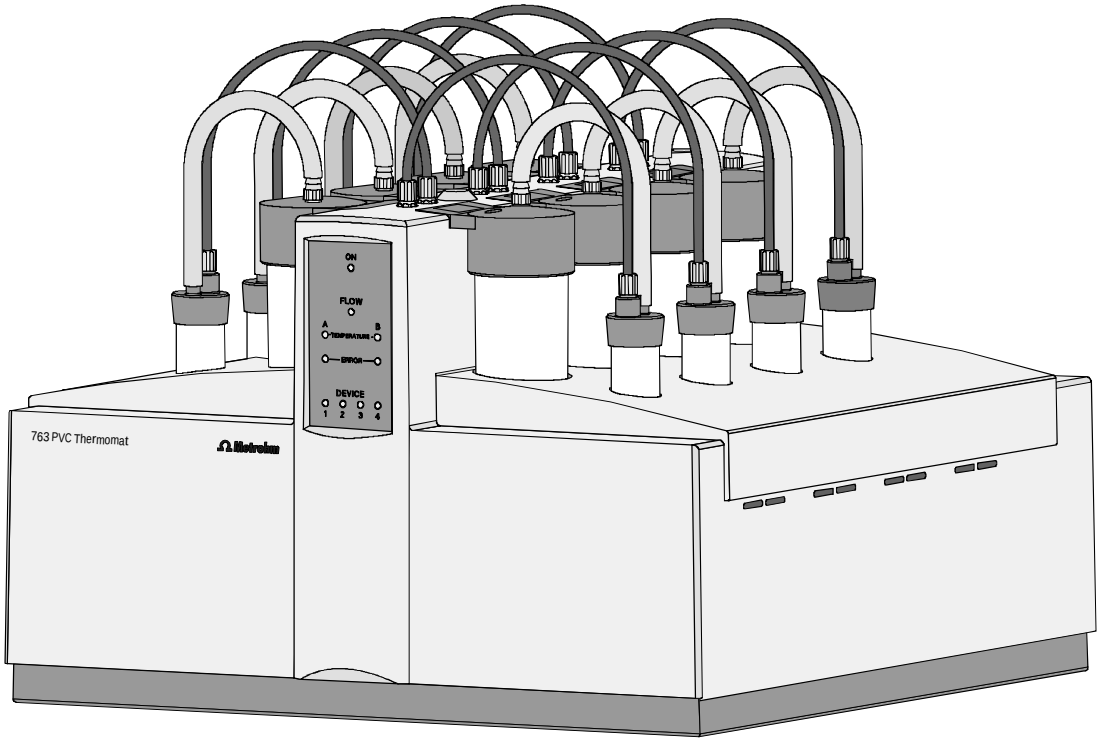
763 PVC Thermomat

with «763 PVC Thermomat 1.0» PC program



8.763.1003 Instructions for Use

26.10.1999 / dö

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1 Introduction

1.1 Determination of thermal stability

The determination of the thermal stability of polyvinyl chloride according to DIN 53 381, part 1, is based on the fact that PVC decomposes at higher temperatures with the release of HCl. The hydrochloric acid formed is taken up by a gas stream (normally N_2) and transferred into the measuring vessel where it is absorbed in the measuring solution (dist. water). The decomposition process is monitored by measuring the conductivity of the aqueous HCl solution. Both the stability time and the induction time are determined. The **stability time** is defined as the time required to achieve a conductivity difference of 50 µS/cm. The **induction time** is the time that elapses before the conductivity starts to rise sharply. The method is used for testing PVC in all stages of processing as well as for testing stabilizers.

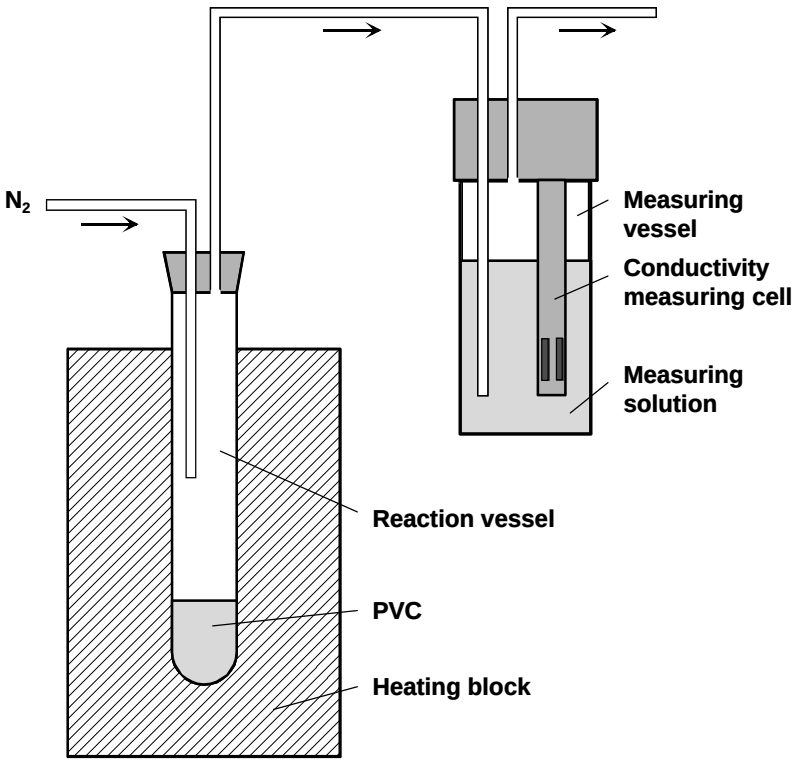


Fig. 1: Schematic measuring arrangement

1.2 Instrument description

The **763 PVC Thermomat** is a PC-controlled instrument for determining the thermal stability of PVC and similar halogenated plastics and is available in the following two versions:

- **2.763.0014 PVC Thermomat for 230 V, 50¼60 Hz**
- **2.763.0015 PVC Thermomat for 115 V, 50¼60 Hz**

The 763 PVC Thermomat is equipped with two **heating blocks** each with 4 measuring positions. Each block can be individually heated, i.e. 2 sets of 4 samples can be measured at two different temperatures or 8 samples can be measured at the same temperature. Measurements at the individual measuring positions can be started individually.

The complete **operation** of the 763 PVC Thermomat is carried out via a PC connected to the RS232 interface with the aid of the **«763 PVC Thermomat»** control and evaluation program. Up to 4 instruments can be connected to 1 PC allowing a maximum of 32 samples to be analyzed at the same time. The evaluation algorithm of the PC program determines the point of inflection of the Thermomat curve and therefore the **induction time** fully automatically. Apart from the induction time, the so-called **stability time**, i.e. the time taken until a certain alteration in the conductivity is reached, can also be determined. The results obtained can be subjected to further mathematical processes. In particular, induction times can be converted to the standard temperatures contained in the relevant standards.

Each Thermomat curve can also be evaluated **manually**. A PC-supported tangent method is available for this in which the tangents can be applied to the curve as required. This means that evaluations are possible even in extreme cases.

The results of the determinations are stored in a **database** together with all the data concerning the method and determination. In the results display part of the program determinations can be searched for, sorted, filtered, exported and printed out. Apart from the graphical display of single and multiple curves it is also possible to carry out recalculations with altered parameters and to extrapolate the results to a particular temperature.

GLP (Good Laboratory Practice) and instrument validation are continuously increasing in importance. In the 763 PVC Thermomat GLP tests are provided for the measurement of temperature, conductivity and gas flow. They determine whether and which tests are to be carried out. The time interval between the tests and the required accuracy can also be laid down as required. If the GLP function is selected then on each result report a remark will be made as to whether the GLP test requirements have been fulfilled. As an option Metrohm offers a GLP test set (6.5616.000), with which the most important tests can be carried out.

1.3 Operating elements

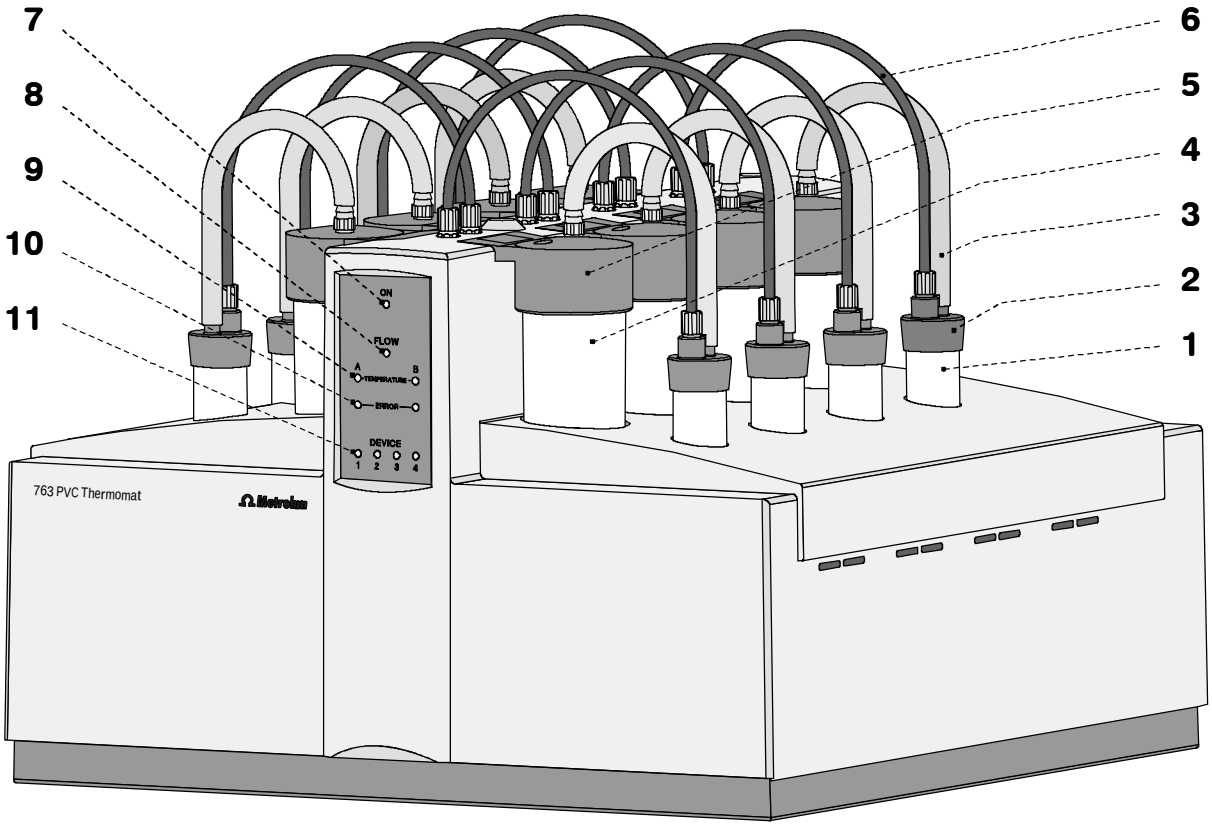


Fig. 2: Front of the 763 PVC Thermomat

1	**Reaction vessel (6.1429.040)**	**7**	**Mains pilot lamp** lit up when instrument switched on
2	**Reaction vessel cover (6.2753.100)**	**8**	**Gas flow display** lit up when gas flow switched on
3	**Connecting tubing (6.1816.010)** between reaction vessel and measuring vessel	**9**	**Temperature display** flashes when heating switched on lit up when temperature reached
4	**Measuring vessel (6.1428.100)**	**10**	**Error display (red)** lit up at error
5	**Measuring vessel cover (6.0913.130)** with built-in conductivity measuring cell	**11**	**Instrument display** indicates numbers (1…4) of units connected
6	**Connecting tubing (6.1805.080)** for gas supply into reaction vessel		

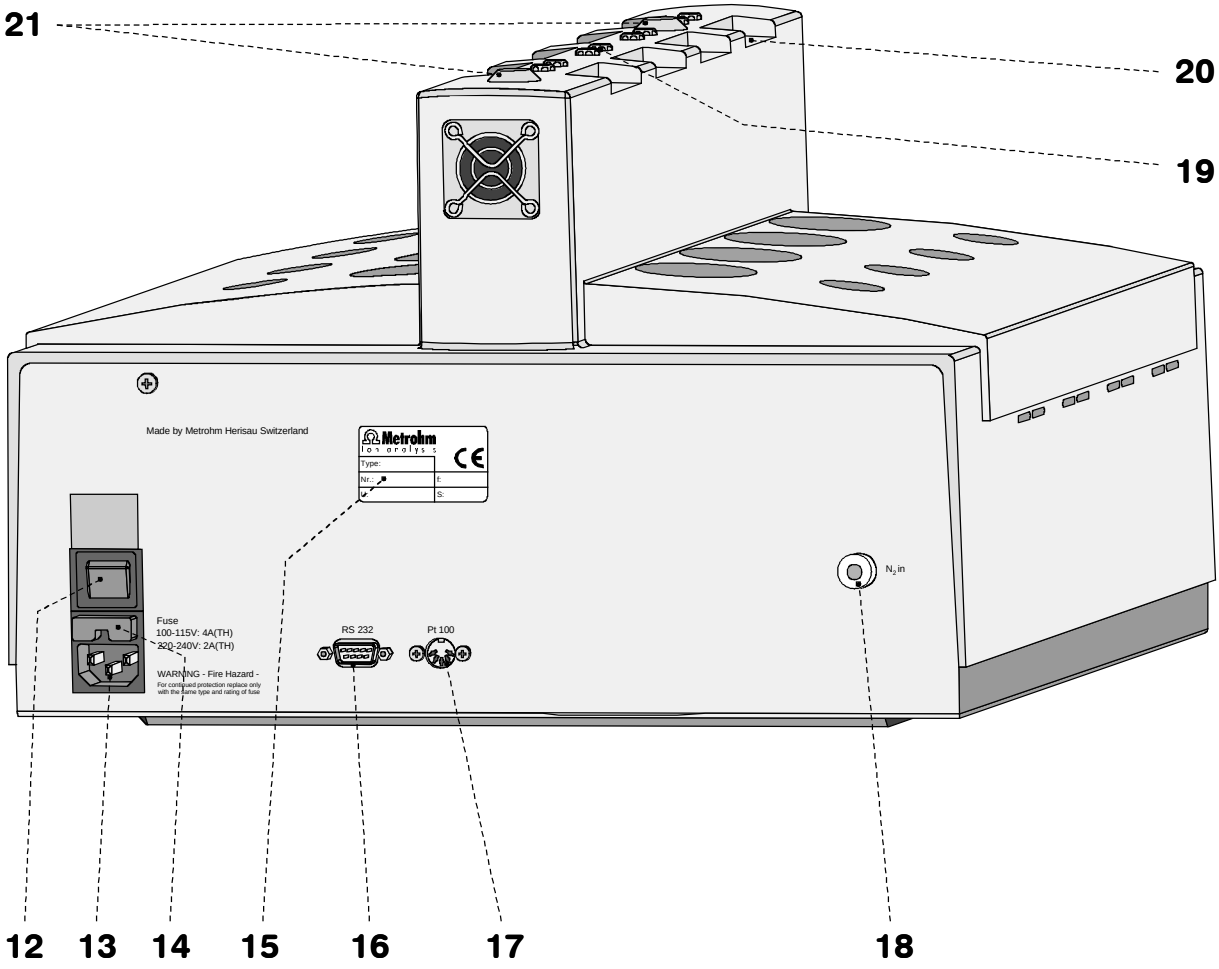


Fig. 3: Rear of the 763 PVC Thermomat

12 Mains switch
to switch instrument on and off:
I = ON 0 = OFF

13 Mains connection plug
mains connection, see *section 2.3*

14 Fuse holder
changing the fuses, see *section 2.3*

15 Model plate
indicating mains voltage and serial number

16 PC connection
RS232 interface

17 Pt100 connection
Connection for external temperature sensor

18 Connection "N_2 in"

19 Gas supply connection
for connection tubing **6**

20 Electrode connection
for measuring vessel cover **5**

21 Mounting
for 6.2757.000 Exhaust collection tube (option)

1.4 Information about these Instructions for Use

Please read through these Instructions for Use carefully before you put the 763 PVC Thermomat into operation. The Instructions for Use contain information and warnings to which the user must pay attention in order to assure safe operation of the instrument.

1.4.1 Organization

These **8.763.1003 Instructions for Use** for the 763 PVC Thermomat provide a comprehensive overview of the installation, startup procedure, operation, fault rectification and technical specifications of this instrument. The Instructions for Use are organized as follows:

Section 1 Introduction
Measuring method, description of instrument, operating elements and safety notes

Section 2 Installation
Installation of accessories, mains connection, connection to PC

Section 3 Operating tutorial
Introduction to the operation using an example

Section 4 Operation
Detailed description of the operation

Section 5 Maintenance – Faults
Maintenance, fault display and fault rectification

Section 6 Appendix
Technical data, standard equipment, options, warranty, declarations of conformity, index

To find the required information on the instruments, you will find it an advantage to use either the **Table of contents** or the **Index** at the back.

You will find additional information on the PVC Thermomat method in the relevant "**Application Bulletins"**, which are available on request free of charge from your Metrohm agency, as well as in the "**Application Notes**" concerning the PVC Thermomat method, which can be downloaded from the Internet under «www.metrohm.ch».

1.4.2 Notation and pictograms

The following notations and pictograms (symbols) are used in these Instructions for Use:

Range	**Menu item, parameter or entry value**
763 PVC THERMOMAT RESULTS	**Program window**
<OK>	**Button**
[Ctrl]	**Key**
35	**Operating element of 763**
	**Hazard** This symbol draws attention to a possible danger to life or of injury if the associated directions are not followed correctly.
	**Warning** This symbol draws attention to possible damage to instruments or instrument parts if the associated directions are not followed correctly.
	**Caution** This symbol marks important information. First read the associated directions before you continue.
	**Comment** This symbol marks additional information and tips.

1.5 Safety notes

1.5.1 Electrical safety

While electrical safety in the handling of the 763 PVC Thermomat is assured in the context of the specifications IEC 1010-1 (protection class 1, degree of protection IP20), the following points should be noted:

- **Mains connection**

*Setting of the **mains voltage**, checking the **mains fuse** and the **mains connection** must be effected in accordance with the instructions in section 2.3.*

- **Opening the 763 PVC Thermomat**

*If the 763 PVC Thermomat is connected to the power supply, the instrument must not be opened nor must parts be removed from it, otherwise there is a danger of coming into contact with components which are live. Hence, always disconnect the instrument from all voltage sources before you open it and ensure that the **mains cable is disconnected from mains connection** **13** !*

- **Protection against static charges**

Electronic components are sensitive to static charging and can be destroyed by discharges. Before you touch any of the components inside the 763 PVC Thermomat, you should earth yourself and any tools you are using by touching an earthed object (e.g. housing of the instrument or a radiator) to eliminate any static charges which exist.

1.5.2 General precautionary rules

- **Take care with hot reaction vessels**

Avoid any contact with hot reaction vessels. Place them in the supplied vessel holder to cool them down.

2 Installation

2.1 Setting up the instrument

2.1.1 Packaging

The 763 PVC Thermomat is supplied together with the separately packed accessories in special packaging containing shock-absorbing foam linings designed to provide excellent protection. The instrument itself is packed in an evacuated polyethylene bag to prevent the ingress of dust. Please store all these special packaging as only they assure transport of the instrument free from damage.

2.1.2 Check

After receipt, immediately check whether the shipment is complete and has arrived without damage (compare with delivery note and list of accessories in *section 6.2*). In the case of transport damage, see instructions in *section 6.4.1* "Warranty".

2.1.3 Location

Position the instrument in the laboratory at a location convenient for operation, free from vibrations and protected against a corrosive atmosphere and contamination by chemicals. It is advisable to set up the instrument in a fume cupboard to guard against odor emission, but the PC can be positioned outside on the regular laboratory bench.

In order to improve accessibility to the measuring places the instrument can also be placed on the 6.2059.000 Rotation ring, which is available as an option.

2.2 Attaching the accessories

2.2.1 Accessories for external gas supply

Nitrogen (N_2) is required for external gas supply to the 763 PVC Thermomat. The following accessories must be mounted on the rear panel of the PVC Thermomat:

1 Fitting the connecting tubing

- Screw one end of connecting tubing 6.1805.030 (150 cm) onto connection **18** "**N_2 in**" on the rear panel of the PVC Thermomat (see *Fig. 3*).
- Screw the 6.1808.020 connection piece onto the other end of connecting tubing 6.1805.030.

2 Connect up gas supply

- Attach the gas supply from the cylinder containing nitrogen to the tubing nozzle of 6.1808.020 Connection piece.
- Set gas pressure at the nitrogen cylinder to ca. 1.5 bar using the reducing valve.

2.2.2 Reaction and measuring vessels

Fig. 4 shows the details of how the accessories for measuring the thermal stability are to be attached and connected to each other. Proceed as follows:

1 Fitting the measuring vessel cover

- Insert PTFE tube **27** from above into opening **24** "In" of measuring vessel cover **5**.
- Screw connection piece **22** into opening **24** of measuring vessel cover **5**.

2 Inserting the measuring vessel in the instrument

- Fill 60 mL dist. water into measuring vessel **4**.
- Place measuring vessel cover **5** on measuring vessel **4**.
- Insert measuring vessel **4** with attached measuring vessel cover **5** into the opening provided for it on the 763 PVC Thermomat and connect measuring vessel cover **5** with connection plug **26** to electrode connection **20** (see *Fig. 3*).

3 Fitting the reaction vessel

- Insert gas inlet tube **32** from below into connection **34** of reaction vessel cover **2**.
- Push sealing ring **33** over the upper end of gas inlet tube **32**.
- Screw connection nipple **30** loosely into connection **34** and press gas inlet tube **32** against connection nipple **30** from below; then fix gas inlet tube **32** to reaction vessel cover **2** by firmly tightening connection nipple **30**.

- Place reaction vessel cover **2** on reaction vessel **1**.
- Connect the white connecting tubing **3** to connection **31** of reaction vessel cover **2** (see *Fig. 3*).

4 Inserting the reaction vessel into the instrument

- Place the sample in the reaction vessel (see section *4.6.2*).
- When the required temperature has been reached insert reaction vessel **1** with attached reaction vessel cover **2** in the openings provided for it on the 763 PVC Thermomat.

5 Making the tubing connections

- Connect the white connecting tubing **3** attached to reaction vessel cover **2** to connection piece **22** of measuring vessel cover **5**.
- Screw one end of brown connecting tubing **6** to connection nipple **30** of reaction vessel cover **2**.
- Screw the other end of brown connecting tubing **6** to the corresponding connection **19** on the 763 PVC Thermomat (see *Fig. 3*).

The 6.1428.020 transparent glass measuring vessel available as an option can be used instead of the 6.1428.100 polycarbonate measuring vessel; in contrast to the polycarbonate vessel this can be cleaned with acetone.

2.2.3 Exhaust collection tube (option)

For selective exhaust lead off the **6.2757.000 Exhaust collection tube** available as an option can be fitted to the 763 PVC Thermomat. In addition to the exhaust collection tube 8 x **6.1816.010 Silicone tubing** (22 cm) must also be ordered. Attach the exhaust collection tube as described below:

1 Positioning the exhaust collection tube

- Place the exhaust collection tube with the two supports in the two mountings **21** (see *Fig. 3*) on the 763 PVC Thermomat so that the connection for leading off the exhaust is located to the rear.

2 Connecting the measuring vessel

- Screw a connection piece **22** into opening **23** of the measuring vessel cover **5**.

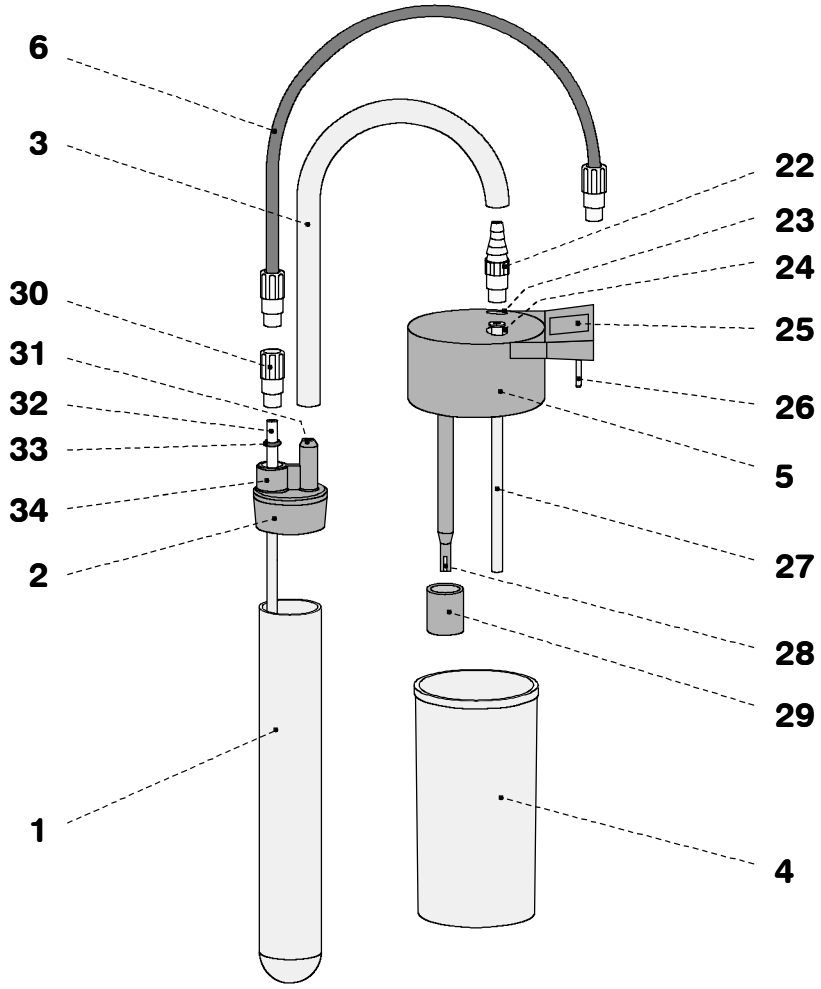


Fig. 4: Installation of reaction and measuring vessels

1	**Reaction vessel (6.1429.040)**	**26**	**Connection plug**
2	**Reaction vessel cover (6.2753.100)**	**27**	**PTFE tube (6.1819.080)** for air supply into measuring vessel
3	**Connecting tubing (6.1816.010)**	**28**	**Electrodes**
4	**Measuring vessel (6.1428.100)**	**29**	**Protective ring (4.422.0520)**
5	**Measuring vessel cover (6.0913.130)** with built-in conductivity measuring cell	**30**	**Connection nipple (6.1808.090)**
6	**Connecting tubing (6.1805.080)** for gas supply to reaction vessel	**31**	**Connection** for connecting tubing **3**
22	**Connection piece (6.1808.050)**	**32**	**Gas inlet tube (6.2418.110)**
23	**Opening "Out"** for measuring vessel exhaust	**33**	**Sealing ring (6.1454.040)**
24	**Opening "In"** for measuring vessel inlet	**34**	**Connection** for connection nipple **30**
25	**Marking field** for entering the cell constant		

- Connect one end of the 6.1816.010 Silicone tubing to connection piece **22**.
- Plug the other end of the 6.1816.010 Silicone tubing into the corresponding exhaust collection tube opening.
- Close the unused openings on the exhaust collection tube with the included E.400.0010 stoppers.

3 Connecting the exhaust collection tube

- Attach a piece of suitable tubing to the exhaust collection tube connection and connect it to an active aspirating device (e.g. water-jet pump).

2.3 Mains connection

Follow the instructions below for connecting to the power supply. If the instrument is operated with a mains voltage set wrongly and/or wrong mains fuse, there is a danger of fire!

2.3.1 Checking the mains voltage

Before switching on the 763 PVC Thermomat for the first time, check that the mains voltage indicated on the model plate **15** (see *Fig. 3*) matches the local mains voltage. If this is not the case, call the Metrohm service.

2.3.2 Exchange of fuses

Two fuses 4 A/slow-blow for 115 V or 2 A/slow-blow for 230 V are installed in fuse holder **14** of the 763 PVC Thermomat as standard.

Ensure that the instrument is never put into operation with fuses of another type, otherwise there is danger of fire!

For changing fuses, proceed as follows:

1 Disconnect mains cable

Disconnect mains cable from mains connection plug **13** of the 763 PVC Thermomat.

2 Remove fuse holder

Using a screwdriver, loosen fuse holder **14** above the mains connection plug **13** and take out completely.

3 Change fuses

Remove old fuses carefully from fuse holder **14** and replace them with two new fuses which are suitable for the set mains voltage, type TH (slow-blow, with high switching capacity):

115 V	**4 A (TH)**	Metrohm-No. U.600.0022
230 V	**2 A (TH)**	Metrohm-No. U.600.0107

4 Install fuse holder

Push fuse holder **14** back into the instrument until it clicks into position.

2.3.3 Mains cable and mains connection

Mains cable

The instrument is supplied with one of three mains cables

- 6.2122.020 with plug SEV 12 (Switzerland, …)
- 6.2122.040 with plug CEE(7), VII (Germany, …)
- 6.2133.070 with plug NEMA 5-15 (USA, …)

which are three-cored and fitted with a plug with an earthing pin. If a different plug has to be fitted, the yellow/green lead (IEC standard) must be connected to protective earth (protection class 1).

Any break in the earthing inside or outside the instrument can make it a hazard!

Mains connection

Plug the mains cable into mains connection plug **13** of the 763 PVC Thermomat (see *Fig. 3*).

2.3.4 Switching the instrument on/off

The 763 PVC Thermomat is switched on and off using mains switch **12**. When the instrument is switched on, the mains pilot lamp **7** "ON" (see *Fig. 2*) lights up.

2.4 Connection to the PC

2.4.1 Connection PVC Thermomat – PC

Always switch off 763 PVC Thermomat and PC before you connect the two instruments with the 6.2134.100 Cable.

The PC program «763 PVC Thermomat» allows a maximum of 4 PVC Thermomats to be controlled. The following possibilities exist for connecting these PVC Thermomats to serial PC interfaces:

- Connection to built-in COM interfaces on the PC
- Connection to an additional built-in interface extension board
- Connection to a softswitch (order number: 2.145.0014 for 230 V, 2.145.0015 for 115 V) which is connected to one of the COM interfaces.

Connect the PC connection **16** at the 763 PVC Thermomat to one of the serial COM ports at the PC or to a softswitch using the 6.2134.100 Cable (9 pin/9 pin). If only a 25-pin COM interface is available on the PC then the 6.2125.110 Adapter cable or a commercially available adapter must be used.

2.4.2 Software installation

The PC program **«763 PVC Thermomat 1.0»** is required for the operation of the 763 PVC Thermomat; this is contained on the 6.6037.000 CD included in the accessories. This program runs under Windows NT (recommended, especially if several instruments are connected), Windows 95 and Windows 98 operating systems and is installed as follows:

1 Install program

- For Windows NT: log in as operator with administrator rights.
- Insert 6.6037.000 Installation CD into CD drive.
- Select **<Start>** and **Run**. Browse for the **setup.exe** file on the installation CD and click on **<OK>**.
- Click on "**763**" and follow the instructions given in the setup program.

  The software package will be installed in the desired directory. In addition to the program files, the following folders are installed:

  Database Folder for database files (***.mrd**)

  Log Folder for event log files (***.mel**) and temperature recording files (***.txt**)

  Template Folder with empty database (**Default.mrd**) and copy of the result program (**Nachaus.prg**)

2 Registration

- Please send us your 8.763.8007 Registration card as soon as possible.

2.4.3 Basic settings

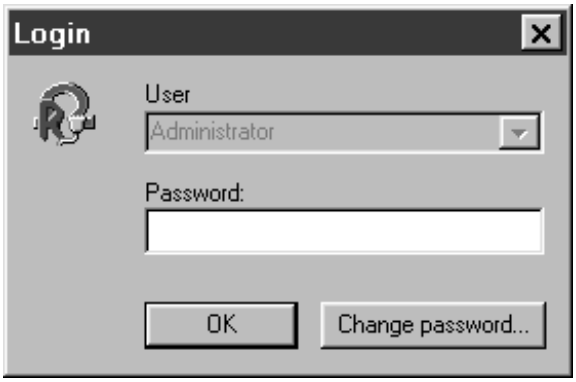
When the program is started for the first time several basic settings must be made for the 763 PVC Thermomat. Proceed as follows:

1 Switch on instruments

- Check if the PVC Thermomat has been connected correctly to the PC (see *section 2.4.1*).
- Switch on the PVC Thermomat using mains switch **12**.
- Switch on the PC.

2 Start program and login

- Double-click the software icon to start the program. The program window with the opening picture is opened and the **Login** window appears on the screen:

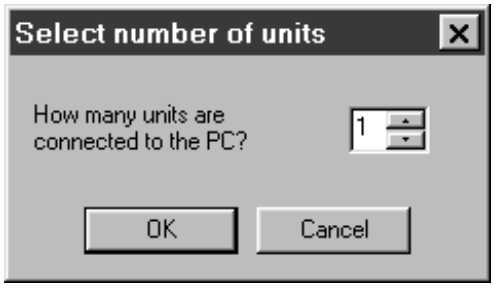


- Do not enter any password here, just click on **<Change password>**. The following window appears:

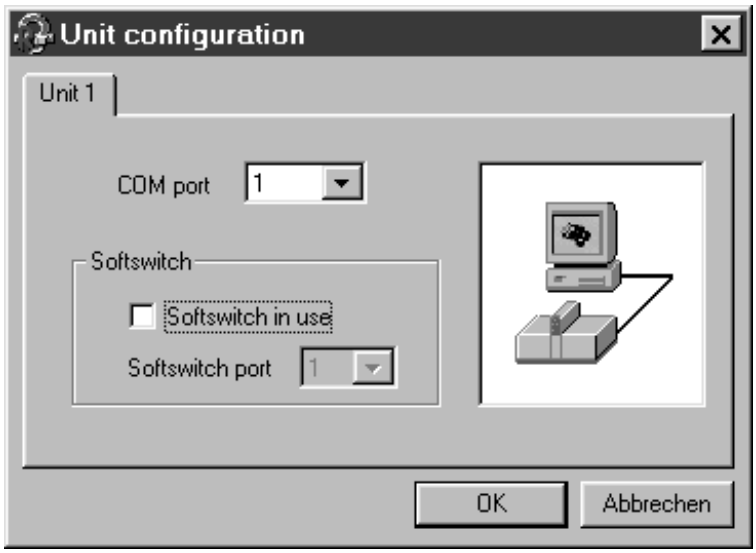
- Leave the **Old password** field empty. Under **New password** enter the required password which will in future allow you to log in as **Administrator**. Confirm the new password by renewed entry in the **Confirm new password** field and click on **<OK>**.
- Enter the new **Password** again in the **Login** window. You are now logged in as **Administrator**.
- Confirm the message **There are no units configured yet** with **<OK>**. The **763 PVC THERMOMAT CONTROL** window appears.

3 Communication settings

- Click on **763 PVC THERMOMAT CONTROL / Options / Communication** to open the **Select number of units** window:



- Enter the number of PVC Thermomats (**1**…**4**) connected to the PC and click on **<OK>**. The **Unit configuration** window appears:



- Under **COM port** select for each instrument the number of the serial interface (**1**…**8**) to which this instrument is connected. If a softswitch is used then the option **Softswitch in use** must be switched on. Under **COM port** the number of the serial interface to which the softswitch is connected is selected; under **Softswitch port** the number of the interface on the softswitch to which the instrument is connected is selected.
- Click on **<OK>** to close the **Unit configuration** window.
- Confirm the message which then appears with **<OK>**.

4 Close the program and start it up again

- Close the program with **763 PVC THERMOMAT CONTROL / File / Exit**.
- Restart the «PVC Thermomat 1.0» program by double-clicking the program icon.
- In the **Login** window enter the **Password** for the administrator. The control window appears. In the status line the message **Unit#: Ready** should appear for all connected instruments.

3 Operating tutorial

This section introduces you to the operation of the 763 PVC Thermomat by means of a brief operating tutorial which describes the basic operating steps needed for performing determinations and display of results.

For further explanations of the operation, please refer to section 4.

3.1 Determinations

The determination of induction and stability time of a PVC sample at 200 °C and a gas flow of 7 L/h is used an illustrative example. Please note that the steps and parameter settings described apply only to this example. If you use a different sample and different parameters, the procedures described in the tutorial must be modified appropriately.

3.1.1 Installation

Before you start this brief tutorial, the 763 PVC Thermomat must be correctly installed as described in s*ection 2.* In what follows, the most important points for the installation are listed once again (for details, see the sections mentioned).

⇒ Setting up instrument	*section 2.1*
⇒ Installing air supply accessories	*section 2.2.1*
⇒ Installing reaction and measuring vessels	*section 2.2.2*
⇒ Mains connection	*section 2.3*
⇒ Connection to PC	*section 2.4*

3.1.2 Preparing for determinations

Before starting determinations, the method must be selected, the heating must be started and the reaction and measuring vessels must be prepared. Proceed as follows:

1 Switch on 763 PVC Thermomat

⇒ Switch on 763 PVC Thermomat with mains switch **12** on the rear of the instrument. After the instrument has been switched on the mains pilot lamp **7** lights up.

2 Switch on PC

⇒ Switch on PC and start «763 PVC Thermomat» program.

3 Select method "Default"

⇒ In the opened control window, select the determination method **Default** for Block A (and – if more than 4 samples are to be determined – also for Block B) in the **Method** field.

⇒ Click on  , if you want to change the parameters of the default method (details see *section 4.5.2*).

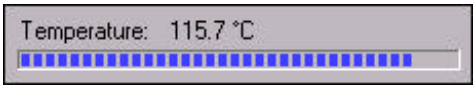
*The determinations described in this tutorial have been carried out using the default settings (***Temperature = 200 °C***,* **Delta T = 0.6 °C***,* **Gas flow = 7 L/h***, etc.).*

4 Start heating

⇒ To start the heating of Block A and – if desired – of Block B click on the following button:

As soon as the heating is switched on the color of the frame of this button switches to **red**. At the same time the temperature display **9** "**TEMPERATURE"** LED on the 763 PVC Thermomat starts to blink for the selected block.

The actual temperature is shown digitally beside the button. A bar below shows the absolute variation of the actual temperature from the target temperature; the bar length corresponds to 50 °C.



5 Prepare measuring vessels

⇒ Clean the used measuring vessels and their covers (details see *section 4.6.3*).

⇒ Fill each cleaned measuring vessel **4** with **60 mL distilled water**.

⇒ Place the measuring vessel cover **5** fitted with a PTFE tube **27** on the measuring vessel **4** (see *Fig. 4*).

⇒ Place the measuring vessel **4** with its cover **5** in one of the openings provided for it on the 763 PVC Thermomat and connect the electrode plug **26** on the cover to the corresponding socket **20** on the 763 PVC Thermomat.

6 Prepare reaction vessels

⇒ Weigh out **0.5 g PVC** into the reaction vessels **1**.

⇒ Take the upper rim of the reaction vessel **1** in your hand (e.g. in the space between thumb and index finger) and rotate the glass though 360°. This provides the degreased glass with a thin grease film, without which it is very difficult to remove the cover from the vessel after the determination.

⇒ Insert an gas inlet tube **32** into the reaction vessel cover **2** and fasten it by screwing down the connection nipple **30** (see *Fig. 4*).

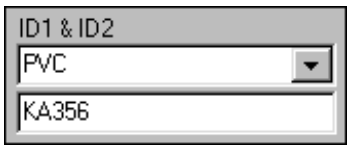
⇒ Place the reaction vessel cover **2** on the reaction vessel **1**. Rotate the cover so that the air inlet tube **32** is as close as possible to the vessel wall.

⇒ Fasten the white connecting tubing **3** on to the connection **31** of the reaction vessel cover **2**.

⇒ Place the prepared reaction vessel into the vessel holder.

7 Enter sample identifiers

⇒ Enter the sample identifiers **ID1** and **ID2** in the corresponding fields of the operating area in the control window.



3.1.3 Carrying out determinations

When the preparations are complete the determinations can be started. Proceed as follows:

1 Wait until the temperature has been reached

⇒ Wait until the heating block reaches the temperature defined in the method. This condition is indicated by temperature display **9** "**TEMPERATURE"** which stops blinking and remains lit up. At the same time or after a slight delay the frame color of the **<Heat to...>** button turns **green**.

2 Insert reaction vessels

⇒ Place the prepared reaction vessels **1** in the heating block openings on the 763 PVC Thermomat. Close any openings which are not used with stoppers or empty reactions vessels to protect against contamination.

⇒ Connect the white connecting tubing **3** fastened to the reaction vessel cover **2** to the corresponding connection **22** on the measuring vessel cover **5**.

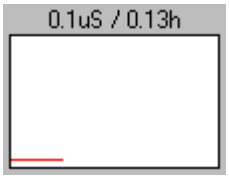
⇒ Screw the brown gas supply tubing **6** onto the connection nipple **30** of the reaction vessel cover **2** and the corresponding connection **19** on the 763 PVC Thermomat (see *Fig. 3*).

3 Start determination

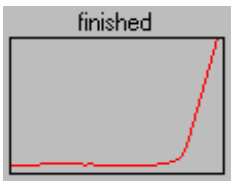
⇒ In the operating field of the control window for every determination you want to start you must click on the button

.

After the start of a determination the **live curve** of the started determination is shown in the operating window beside the channel number. The actual measured value appears above the curve.



As soon as a point of inflection is found during the measurement the determination is terminated. The window with the live curve becomes gray again and the message **finished** appears.



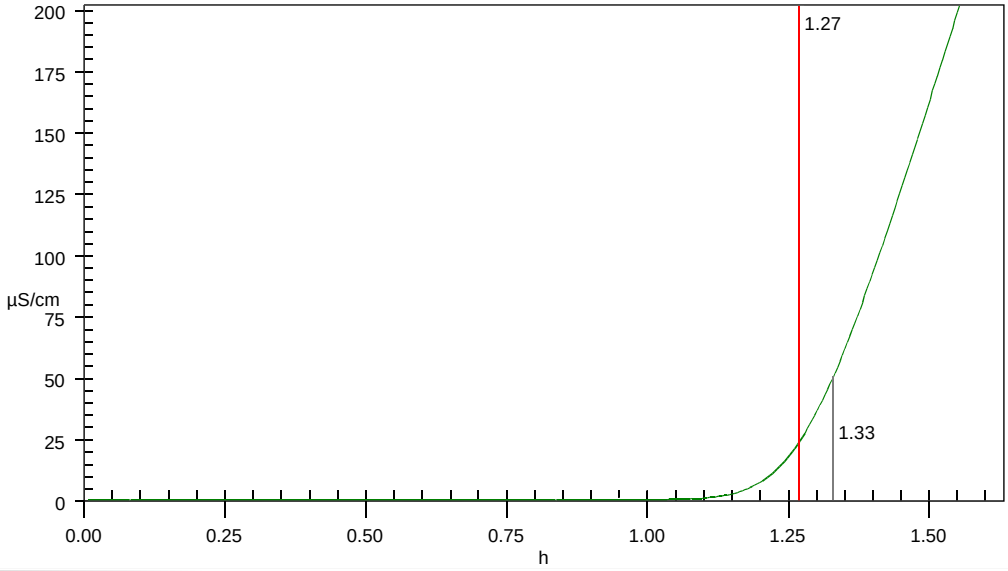
4 Automatic report output

At the end of the determination the report defined in the method is printed out automatically.

Unit	1	**Serial number**	01109	**Druckdatum**	09.09.1999 09:34:03
Block	A	**Cell constant**	1	**Bestimmungsdatum**	23.06.1999 13:25:52
Channel	1	**User**	Administrator		

ID 1	**ID 2**
PVC	KA356

Induction time	1.27 h		
Stability time	1.33 h	**Delta Kappa**	50 µS/cm
Standard time	6.45 h	(F: 2.254; T: 180°C)	



Method name	PVC 200°C		
Creator	Administrator	**Creation date**	23.06.1999 08:27:20

Temperature	200 °C	**Stop time**	0.00 h
Delta T	0.60 °C	**Stop at conductivity**	200 µS/cm
Gas flow	7 L/h	**Stopat endpoint**	☑
Start delay	0 min	**Delta Kappa**	50 µS/cm
Start mode	◉ per channel	**Evaluation delay**	0.00 h
	○ per 2 channels	**Evaluation suppression start**	0.00 h
	○ per block	**Evaluation suppression end**	0.00 h

Send to file

GLP test passed.

3.2 Results

The demo database **Demo.mrd** *is used as an example for the display and further processing of the results of recorded determinations. Please note that the steps and parameter settings described apply only to this database. If you use a different database, the procedures described in the tutorial must be modified appropriately.*

3.2.1 Determination overview

The determination overview displays an overview table with selected data fields of all the determinations contained in the opened database.

1 Open database "demo.mrd"

⇒ Click on  or **File / Results** in the control window in order to open the **763 PVC THERMOMAT RESULTS** window. This loads the **Repos.mrd** database and opens the determination overview as standard.

⇒ Click on **File / Open database** in the results window. Select the **demo.mrd** database in the **Open database** window and click on **<Open>**. A sub-window opens in the results window which shows the data from 5 determinations in an overview table.

Determination data

	ID 1	ID 2	Determination date	Temperature	Induction time automatic
	PVC	KA356	24.06.1999 13:32:43	200°C	1.27 h
▶	PVC	KA356	24.06.1999 13:32:41	190°C	2.90 h
	PVC	KA356	22.06.1999 13:47:30	170°C	14.14 h
	PVC	KA356	21.06.1999 17:53:40	160°C	33.43 h
	PVC	KA356	21.06.1999 17:53:38	180°C	6.33 h

2 Format determination overview

⇒ **Select fields**: Click on  or **Format / Select fields** in the results window. In the **Display fields** window move the fields until the fields required for the determination overview are visible in the **Display** field and then click on **<OK>**.

⇒ **Adjust field width**: Mark the column in the determination overview whose width is to be altered. Click on **Format / Adjust width** in the results window. Enter the column width in the **Column width** window or click on **<Best fit>** in order to automatically adapt the column width to the longest field contents. Alternatively the column width can also be set directly by using the **mouse**. This is done by moving the cursor between two columns in the title line of the table until it appears as ⇹. The column can now be pulled to the required width with the **left-hand mouse key** pressed down. A **double-click** on the

selected column automatically adapts the column width to the longest field contents (including title).

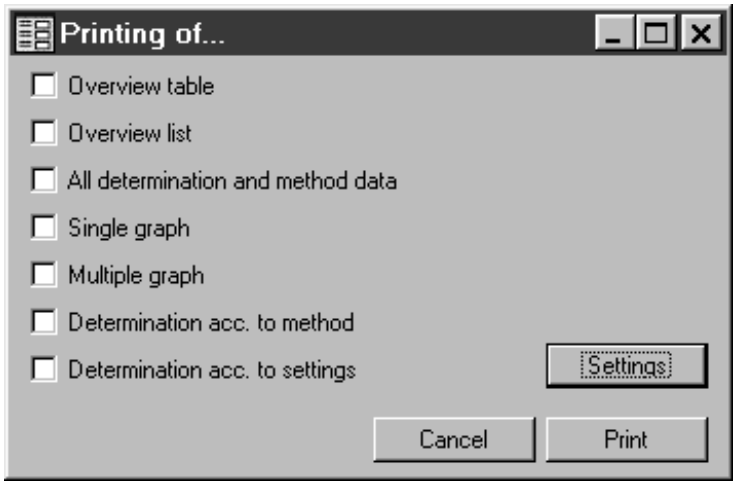
⇒ **Format fields**: Click on **Format / Fields** in the results window. Set down the format for the fields in the determination overview in the **Cells effects** window and click on **<OK>**.

⇒ **Select fonts**: Click on **Format / Fonts** in the results window. Select the font to be used in the determination overview in the **Font** window and click on **<OK>**.

⇒ **Save settings**: Click on **Options / Save settings now** to save the new format of the determination overview permanently.

3 Print determination overview

⇒ Click on  or **File / Print** in the results window to open the **Printing of** window.



⇒ Select **Overview table** if the determination overview is to be printed out in tabular form.

⇒ Select **Overview list** if the determination overview is to be printed out as a list with all the fields selected in the determination overview.

⇒ Click on **<Print>**.

⇒ In the **Print** window select the printer, printing area and number of copies and click on **<Print>**.

4 Export determination overview

⇒ **Copy to clipboard**: Mark the lines or columns in the determination overview which are to be copied. Click on  or **Edit / Copy**. The marked texts are loaded into the clipboard from where they can be further processed with other programs.

⇒ **Export to other database**: Mark the determinations (lines) which are to be exported in the determination overview. Click on **Determination / Export / Export to database**. In the **Export data to...** window select the database to which the determinations are to be exported and click on **<Save>**.

⇒ **Export to Word**: Mark the lines or columns in the determination overview which are to be exported. Click on  or **Determination / Export / Export to MS Word**. The marked texts are loaded into an RTF file which is automatically opened with Microsoft Word.

⇒ **Export** to **Excel**: Mark the lines or columns in the determination overview which are to be exported. Click on  or **Determination / Export / Export to MS Excel**. The marked texts are loaded into an XLS file which is automatically opened with Microsoft Excel.

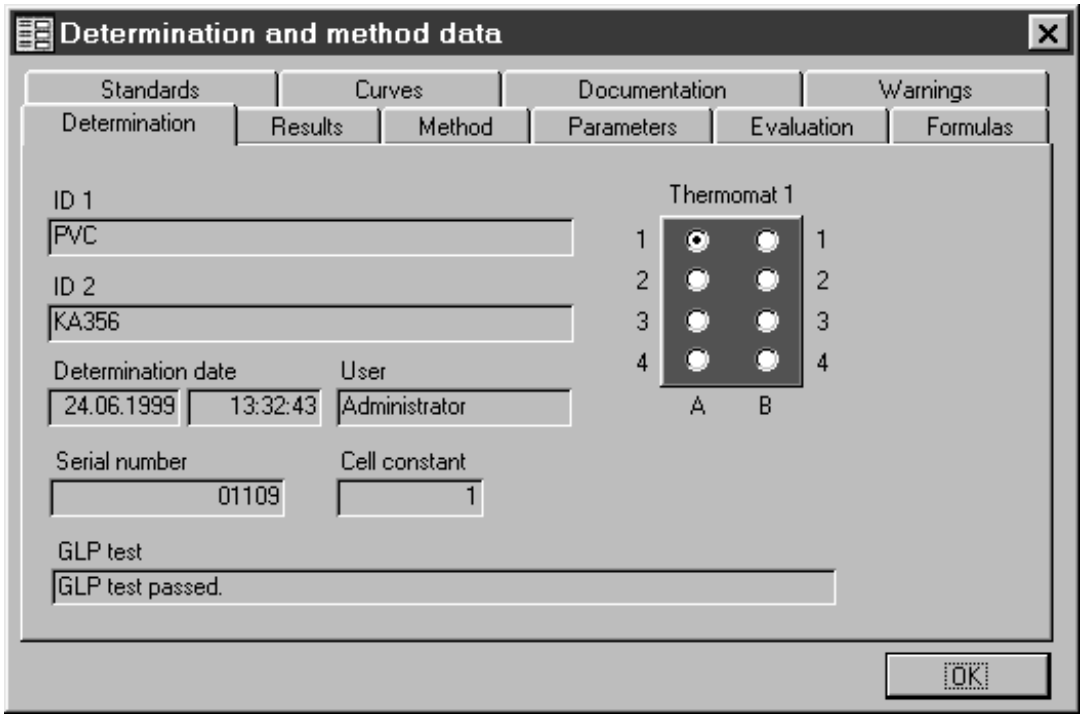
3.2.2 Determination and method data

All the determination and method data of each determination in a determination overview can be shown, printed out and exported.

1 Display determination and method data

⇒ In the opened determination overview mark the determination for which all parameters and results are to be displayed.

⇒ Click on  or **View / Determination and method data** in the results window or double-click on the required line in the determination overview. The **Determination and method data** window opens in which the data are shown on several register cards.



2 Print determination and method data

⇒ Click on or **File / Print** in the results window to open the **Printing of** window (see *section 3.2.1*).

⇒ Select the **All determination and method data** option.

⇒ Click on **<Print>**.

⇒ In the **Print** window select the printer, printing area and number of copies and click on **<Print>**.

3 Export determination and method data

⇒ Mark the determinations (lines) in the determination overview from which all data is to be exported.

⇒ Click on or **Determination / Export / Determination and method data.**

⇒ In the **Save to export file** window select the required folder and the file name of the ***.txt** export file under which the data are to be stored and click on **<Save>**.

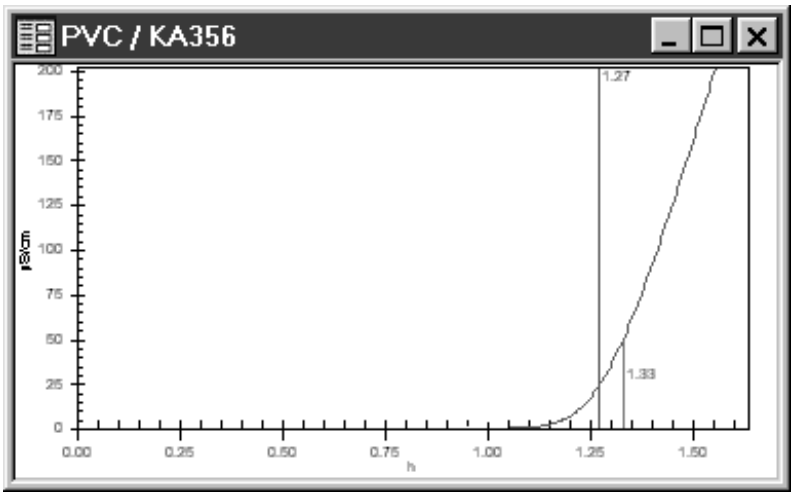
3.2.3 Graphics

Curves for each determination in a determination overview can be shown, printed out and copied.

1 Open single graph

⇒ Mark the determination in the determination overview whose single curve is to be displayed.

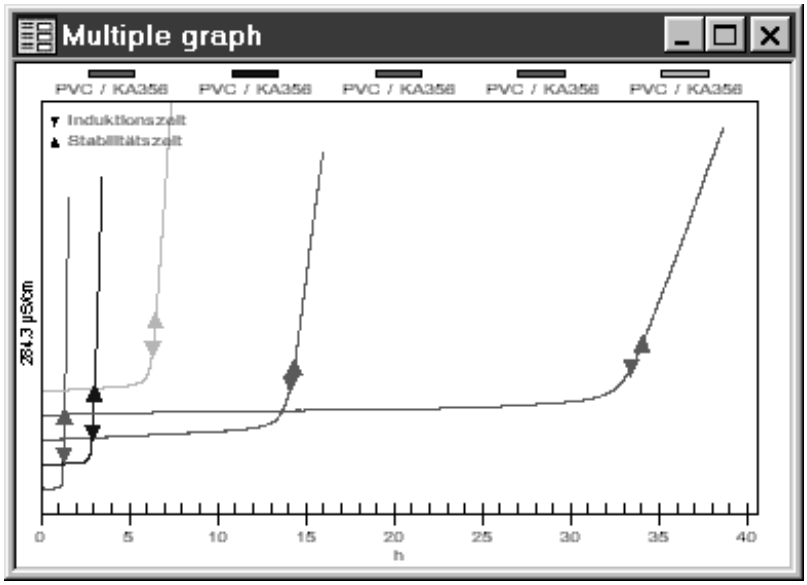
⇒ Click on  or **Determination / Graphics / Single graph** in the results window. A new window is opened in which the **single graph** for the determination selected in the determination overview is shown. The sample identifiers **ID 1** and **ID 2** are shown in the window title. If present then the determined **Induction time** (red vertical line and numbers) and **Stability time** (black vertical line and numbers) will be shown in the graphics window as well as the **measuring curve** (green).



2 Open multiple graph

⇒ Mark the determinations in the determination overview whose curves are to be displayed in a multiple graph.

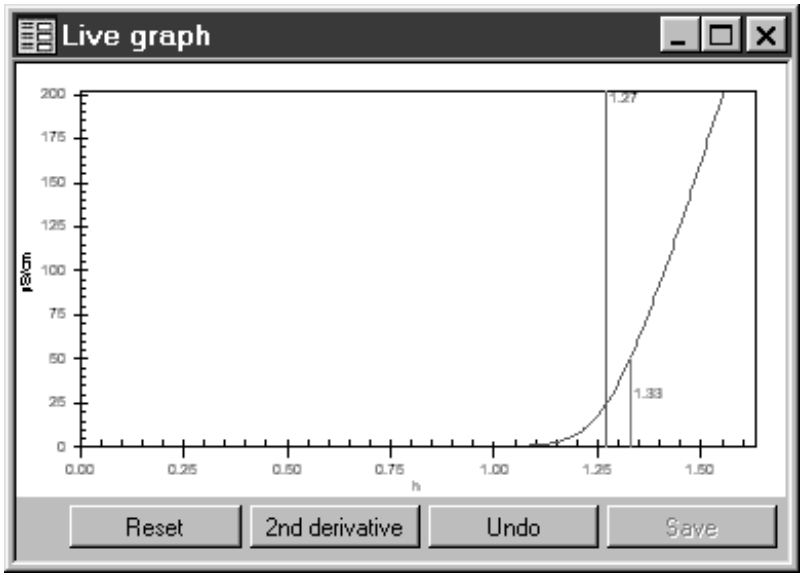
⇒ Click on  or **Determination / Graphics / Multiple graph** in the results window. A new window is opened in which **all curves** for the determinations marked in the determination overview are shown. The individual measuring curves are shown in color, the legends referring to the colors (**ID 1** and **ID 2**) are located in the graph header. If present the determined **Induction time** (▼) and **Stability time** (▲) will also be shown.



3 Open live graph

⇒ Mark the determination in the determination overview whose curve is to be displayed.

⇒ Click on  or **Determination / Graphics / Live graph with reevaluation** in the results window. A new window is opened in which the **measuring curve** of the marked determination is shown. If a different determination is marked in the determination overview then the curve is automatically updated. If present then the determined **Induction time** (red vertical line and numbers) and **Stability time** (black vertical line and numbers) will be shown in the graphics window as well as the **measuring curve** (green). With **<2nd derivative>** the second derivative of the measuring curve can be shown as a blue line. The live graph offers the possibility of the manual **reevaluation** of the measuring curves (see *section 4.7.3*).



4 Print graphics

⇒ Open the required graph in the results window which is to be printed.

⇒ Click on  or **File / Print** in the results window. The curve shown in the active graph window will be printed out.

⇒ If you want to print several single or multiple curves at the same time first mark the required determinations in the determination overview. Click on  or **File / Print** (see *section 3.2.1*). In the **Printing of...** window select the option **Single graph** or **Multiple graph** and click on **<Print>**.

5 Copy graphics

⇒ Open the required graph in the results window which is to be copied.

⇒ Use the right-hand mouse key to click on the graph window and select the option **Copy** in the context-sensitive menu. The selected curve is copied into the clipboard where it can be further processed with other programs.

4 Operation

This section describes the most important points concerning the operation of the 763 PVC Thermomat. For further details please refer to the on-line help in the PC program which can provide you with the required information rapidly and conveniently from any place in the program.

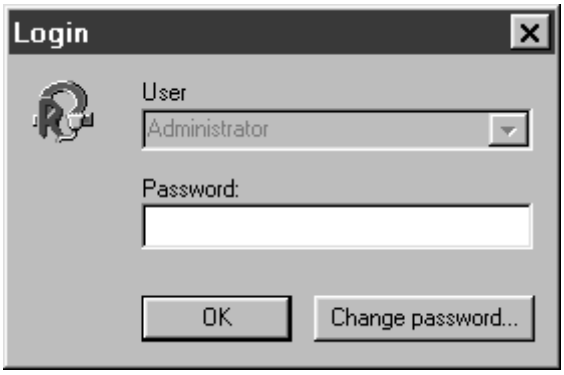
4.1 Fundamentals of the operation

4.1.1 Starting/closing the program

Start the «763 PVC Thermomat» program


PvcTm.exe

Start the program
Double-click this icon or the **PvcTm.exe** file to start the «763 PVC Thermomat 1.0» program. The **Login** window appears:



Select the desired **User** name and enter the **Password**. Then click on **<OK>**.

After software installation, the program can be started without entering a **Password***. For the definition of users, see section 4.2.2.*

Close the «763 PVC Thermomat» program

763 PVC THERMOMAT CONTROL / File / Exit
Exit the «763 PVC Thermomat» program.

The program is also quitted by clicking on  in the upper right part of the **763 PVC THERMOMAT CONTROL** window.

4.1.2 Glossary

Control window

The main window **763 PVC THERMOMAT CONTROL** is known as the control window. It contains all the functions required for controlling the 763 PVC Thermomats connected to the PC.

Method

A method includes all parameters for carrying out and evaluating a determination.

Determination

A determination is understood to be the automatic determination of the induction time and/or stability time of a sample. In order to carry out a determination a suitable method must be selected for the samples.

Results

The results of a determination are automatically stored in the **Repos.mrd** database and can be observed in the results window.

Reevaluation

Reevaluation is the subsequent reprocessing of determinations, in particular the manual determination of the induction time with the aid of tangents.

Recalculation

Recalculation means that results, equations and standards can be calculated again at a later date.

Extrapolation

Extrapolation can be used to convert the results obtained at various temperatures to a required target temperature. In addition, this method can be used for converting the induction time to the standard time.

4.1.3 Control window

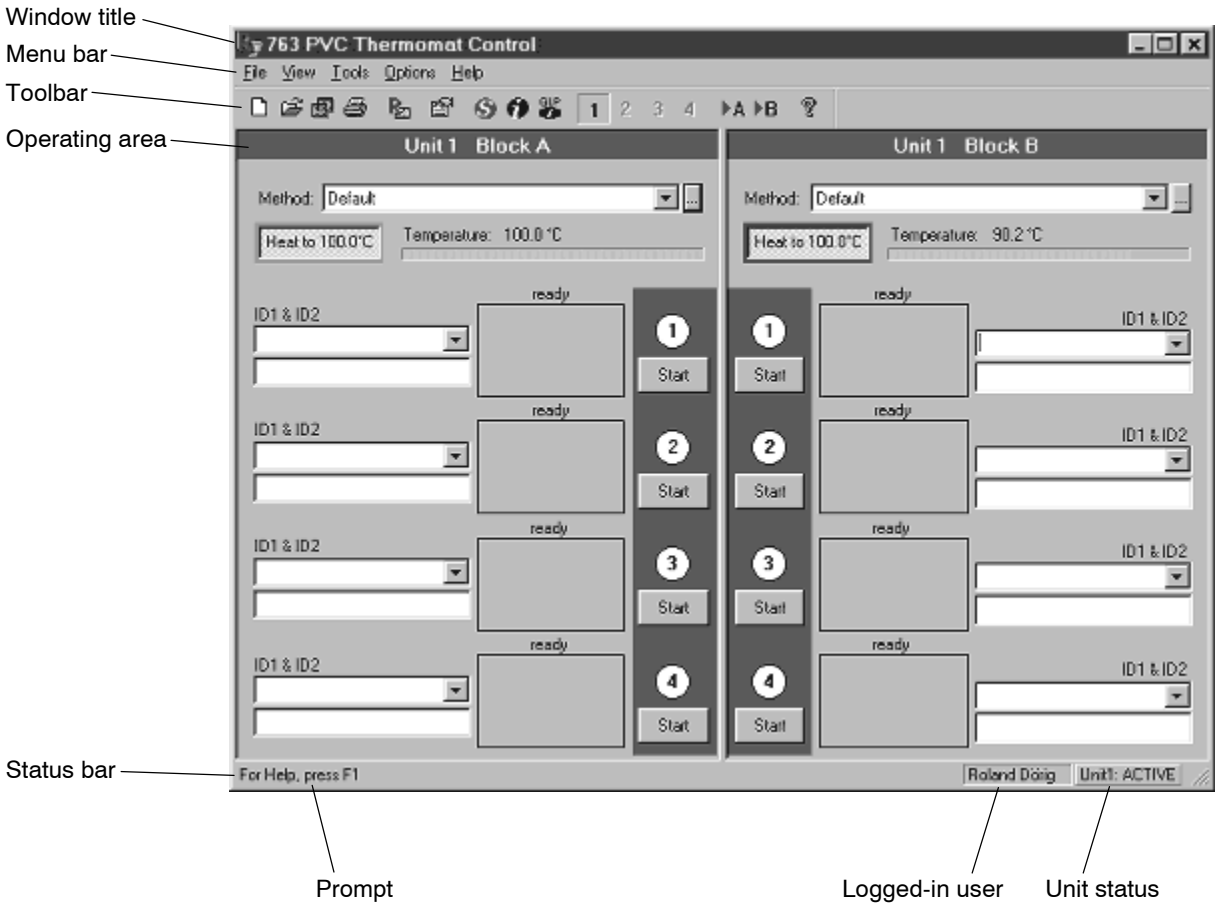
The **763 PVC THERMOMAT CONTROL** window is used for controlling the PVC Thermomats, managing methods, displaying the live curves and accessing various program functions.

Open

The **763 PVC THERMOMAT CONTROL** window is opened automatically when the program is started (see *section 4.1.1*).

Arrangement

The elements of the control window are the menu bar, the toolbar and the status bar, indicating prompts, the logged-in user and the instrument status. The operating area contains a diagram of the measuring unit of the 763 PVC Thermomat which can be used to start, display and stop determinations.



Menus

The control window contains the following main menus:

File Management of method files, opening the results window, printing, new log in.

View View of toolbar and status bar, unit information, live parameters, status overview, event log overview, unit selection.

Tools	Cell constants, GLP test, timer, gas flow control, start all channels, determination of Delta T, temperature recording, service diagnosis.
Options	General settings, instrument configuration, user permissions.
Help	Program-specific on-line help.

Icons

The following icons are displayed in the control window:


Create new method (see *section 4.5.1*).


Open existing method (see *section 4.5.1*).


Method manager (see *section 4.5.1*).


Print results (see *section 4.7.6*).


Open results window (see *section 4.1.4*).


Live parameters, which can be altered during a run (see *section 4.6.4*).


Status overview of the connected instruments (see *section 4.3.2*).


Unit information (see *section 4.3.1*).

Display GLP status (see *section 4.8.3*).

Select unit 1...4.

Start all channels of Block A (see *section 4.6.4*).

Start all channels of Block B (see *section 4.6.4*).


Call up help.

Close

The control window is closed by clicking on **763 PVC THERMOMAT CONTROL / File / Exit** or  in the upper right part of the window.

4.1.4 Results window

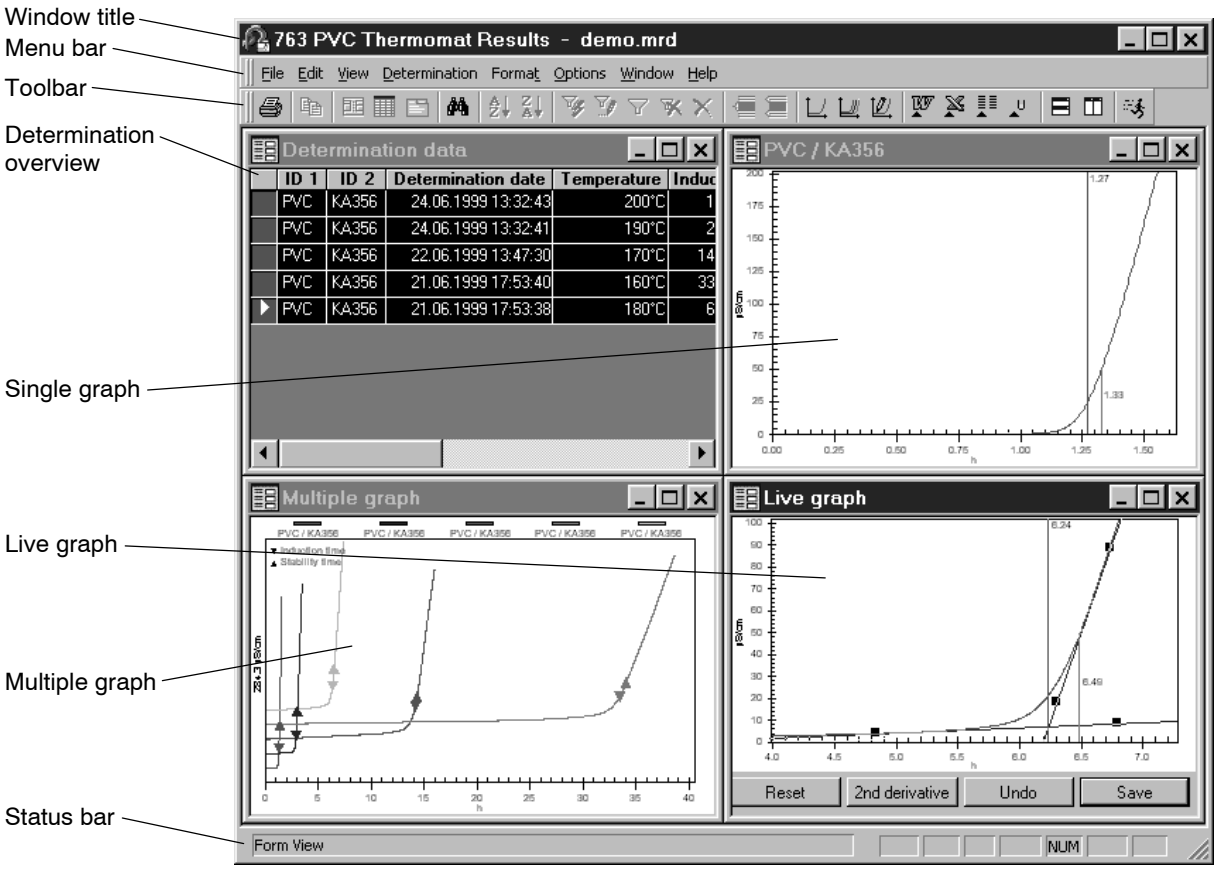
The **763 PVC THERMOMAT RESULTS** window is used to display, output and recalculate the results of determinations recorded by the 763 PVC Thermomat. The determination data are stored in the ***.mrd** database files and can be shown in this window as an overview table and as curves. The results window can run even when the control window is closed.

Open

The **763 PVC THERMOMAT RESULTS** window is opened by clicking on  or **763 PVC THERMOMAT CONTROL / File / Results** in the control window.

Arrangement

The elements of the results window are the menu bar, the toolbar and the status bar. Within the results window sub-windows can be opened showing determination overviews, single, multiple and live graphs.



Menus

The results window contains the following main menus:

File	Open database, print, close window.
Edit	Copy, mark, delete filter.
View	Select display: determination overview, method and determination data, GLP.

Determination	Seek, sort, filter, graphics, extrapolation, recalculation, export, delete.
Format	Format determination overview.
Options	General program settings.
Window	Display of windows.
Help	Program-specific on-line help.

Icons

The following icons are shown in the results window:


Print results, curves and overview lists (see *section 4.7.6*).


Copy to clipboard (see *section 4.7.6*).


Select fields for determination overview (see *section 4.7.1*).


Show determination overview (see *section 4.7.1*).


Show all method and determination data (see *section 4.7.2*).


Find (see *section 4.7.1*).


Sort ascending (see *section 4.7.1*).


Sort descending (see *section 4.7.1*).


Selection based filter (see *section 4.7.1*).


Special filter/sorting (see *section 4.7.1*).


Apply filter/sorting (see *section 4.7.1*).


Remove filter/sorting (see *section 4.7.1*).


Delete filter (see *section 4.7.1*).


Filter selection (see *section 4.7.1*).


Selection not in filter (see *section 4.7.1*).


Single graph (see *section 4.7.3*).


Multiple graph (see *section 4.7.3*).


Single graph with possibility of reevaluation (see *section 4.7.3*).


Export to MS Word (see *section 4.7.6*).


Export to MS Excel (see *section 4.7.6*).


Export measured values (see *section 4.7.6*).


Export determination and method data (see *section 4.7.6*).


Tile windows vertically (see *section 4.7.8*).


Tile windows horizontally (see *section 4.7.8*).


Close results window.

Close

The results window is closed by clicking on  or **763 PVC THERMOMAT RESULTS / File / Return**.

4.1.5 Overview of file types

The following file types are produced by the «763 PVC Thermomat» program:

***.mrd** **Database file**
Contains measuring data and results. ***.mrd** files are automatically stored in the **Database** folder.

***.mel** **Event log file**
This file contains a protocol of all events which have occurred with the connected PVC Thermomats. ***.mel** files are automatically stored in the **Log** folder.

***.txt** **Measurement table file**
Contains measurement tables in ASCII format. ***.txt** files are automatically stored in the **Log** folder.

4.1.6 Context-sensitive menus

A lot of menu functions of the program window can also be selected by clicking the required window or element with the **right-hand mouse key**. The menu options which are then opened depend on the selected active window or element

4.1.7 Mouse functions

The **mouse** can be used to carry out the normal program operating functions such as the selection of menu items and fields. It can additionally be used for magnifying a section of a curve (**zooming**). To zoom a portion of the curve place the mouse cursor to the upper left corner of the square to zoom, press the left mouse button and drag the cursor to the lower right corner of the rectangle. After releasing of the left mouse button the selected region will be zoomed full-screen.

4.1.8 Help

By clicking on  or **Help / Help** or by pressing the [F1] key you can get on-line help on the current topic anywhere in the program.

Green texts	can be clicked to jump to a different Help topic.
Violet texts	identify dialog items, parameters or buttons in the corresponding window.
Blue texts	Identify titles and important information.

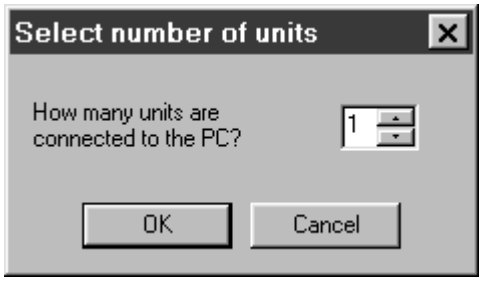
4.2 Instrument and software settings

4.2.1 Communication

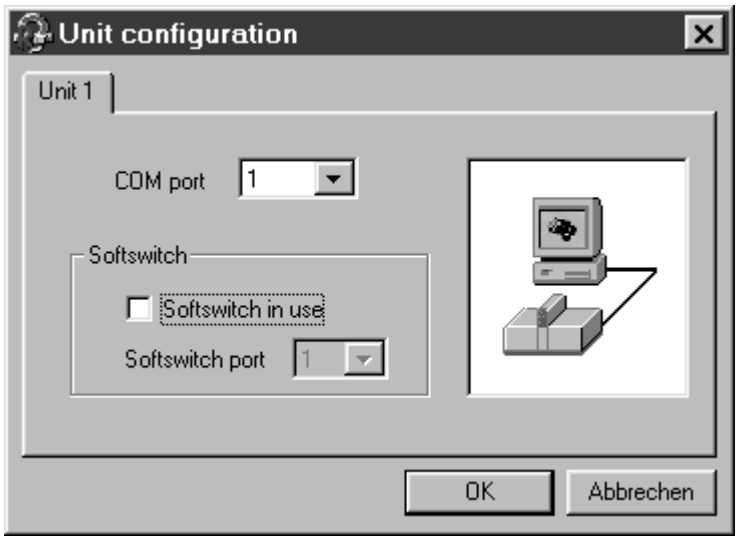
763 PVC THERMOMAT CONTROL / Options / Communication

This menu item is used for the configuration of the communication between the PC and up to 4 connected 763 PVC Thermomats.

The **Select number of units** window appears first; the number of 763 PVC Thermomats connected to the PC must be entered here.



Then the **Unit configuration** window opens; the following parameters can be selected here for each instrument which is connected:



COM port	Selects the COM port (**1...8**) to which the instrument or the softswitch is connected.
Softswitch	
Softswitch in use	Use a softswitch between PC and instrument.
Softswitch port	Selects the port (**1...4**) on the softswitch to which the instrument is connected.

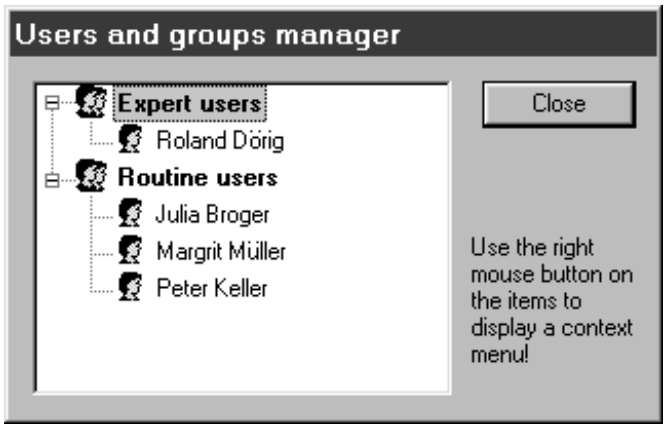
Two softswitches are available as options from Metrohm: 2.145.0014 (for 230 V) or 2.145.0015 (for 115 V). 4 PVC Thermomats can be connected to a softswitch.

4.2.2 User permissions

The «763 PVC Thermomat» program is equipped with a comprehensive **password protection** system which allows each menu item to be individually provided with **access rights**. The access rights are issued for various **groups**. The individual **user** can then be assigned to a group

763 PVC THERMOMAT CONTROL / Options / User permissions

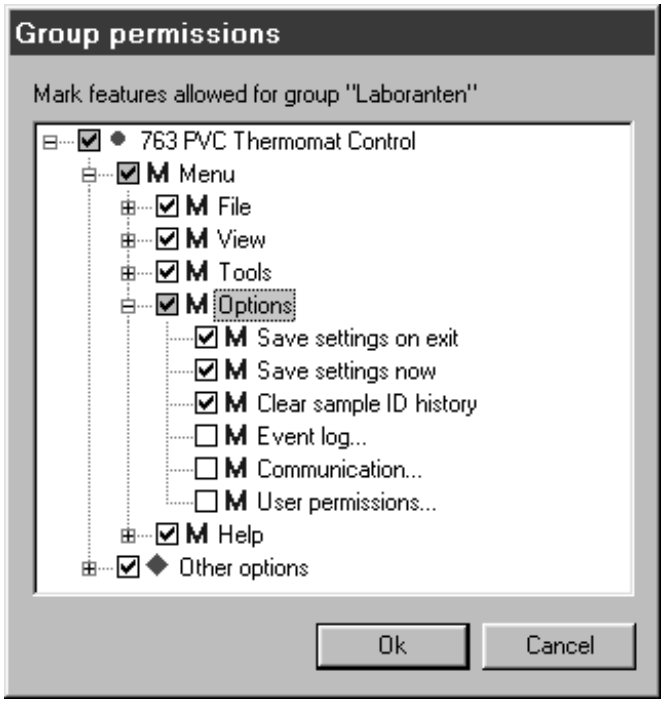
This menu item is used to issue access rights and is reserved for the **Administrator** only. The **Users and groups manager** window opens in which the already defined groups with their users are listed.



The following menu items are available by clicking on the entries with the right-hand mouse key.

Functions for groups

Add user	Adds a new user to the selected group. The name of the new user must be entered in the **Enter new user name** window.
Add group	Adds a new group to the previous groups. The name of the new group must be entered in the **Enter new group name** window.
Delete group	Deletes the selected group.
Properties	The **Group permissions** window opens in which access to all program functions can be switched on or off individually.



Functions for users

Delete user Deletes the selected user.

It is recommended to make user lists and enter passwords as a first action after software installation.

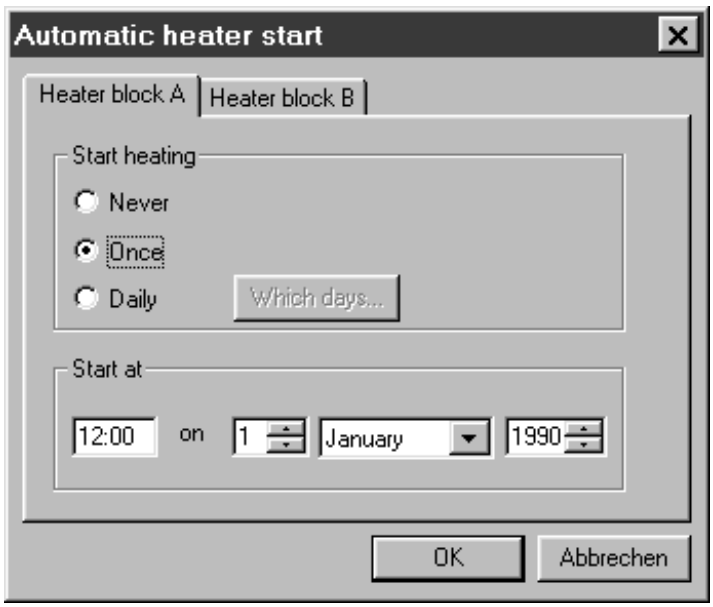
The **Administrator** *is not included in the list of operators but possesses all rights of access and cannot be deleted.*

After configuration of user permissions the program prompts for the selection of the user name and entry of the password in the **Login** window every time the program starts. The user name stamps all methods, determinations and reports. It is possible at any time to change the user with the menu item **763 PVC THERMOMAT CONTROL / File / New login**.

4.2.3 Timer

763 PVC THERMOMAT CONTROL / Tools / Timer function...

The **Automatic heater start** window opens in which the automatic start of heating at a predetermined time can be entered independently for block A or block B.



Start heater

Never	No automatic start of heating.
Once	Automatic start of heating at the time defined in the **Start at...** field.
Daily	Automatic start of heating on the day selected in the **<Which days>** field at the time defined in the **Start at...** field.
<Which days>	This button opens the **Select days** window in which the required day can be selected.
Start at...	If **Once** has been selected then the time and date for the automatic start of heating can be entered, if **Daily** has been selected the time.

4.2.4 Gas flow control

The flow of gas through the reaction vessels to the measuring vessels can be switched on and off manually and shown in a separate window.

763 PVC THERMOMAT CONTROL / Tools / Gas flow control / Gas flow on

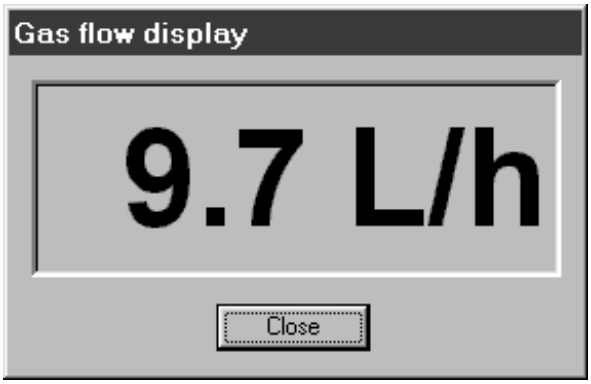
Switches on gas flow.

763 PVC THERMOMAT CONTROL / Tools / Gas flow control / Gas flow off

Switches off gas flow.

763 PVC THERMOMAT CONTROL / Tools / Gas flow control / Display gas flow...

The **Gas flow display** window opens in which the actual gas flow is shown. This function is only available if no determination is running.



4.2.5 Recording temperature

763 PVC THERMOMAT CONTROL / Tools / Temp. logging / Block A on

Switches on temperature recording for block A and external temperature sensor.

763 PVC THERMOMAT CONTROL / Tools / Temp. logging / Block A off

Switches off temperature recording for block A and external temperature sensor.

763 PVC THERMOMAT CONTROL / Tools / Temp. logging / Block B on

Switches on temperature recording for block B and external temperature sensor.

763 PVC THERMOMAT CONTROL / Tools / Temp. logging / Block B off

Switches off temperature recording for block B and external temperature sensor.

The temperature can be individually recorded at any time for each heating block. The measured values are written in a text file which contains the following values:

t(s)	Time in s
Block [°C]	Measured temperature in heating block
Ext. Sensor [°C]	Measured temperature with the external temperature sensor

The text file is stored under the name **U#X-YYMMDD-HHMMSS.txt** in the folder **Log**; **#** stands for the instrument number (1...4), **X** for the block (A or B) and **YYMMDD-HHMMSS** for the date and time of the start of data recording.

4.2.6 Options

Save settings

The program and window settings can be saved with the two following menu items:

763 PVC THERMOMAT CONTROL / Options / Save settings on exit

If this option is switched on then the current program and window settings will be automatically saved each time the program is shut down.

763 PVC THERMOMAT CONTROL / Options / Save settings now

The current program and window settings will be saved immediately.

Clear sample ID1 history

763 PVC THERMOMAT CONTROL / Options / Clear sample ID history

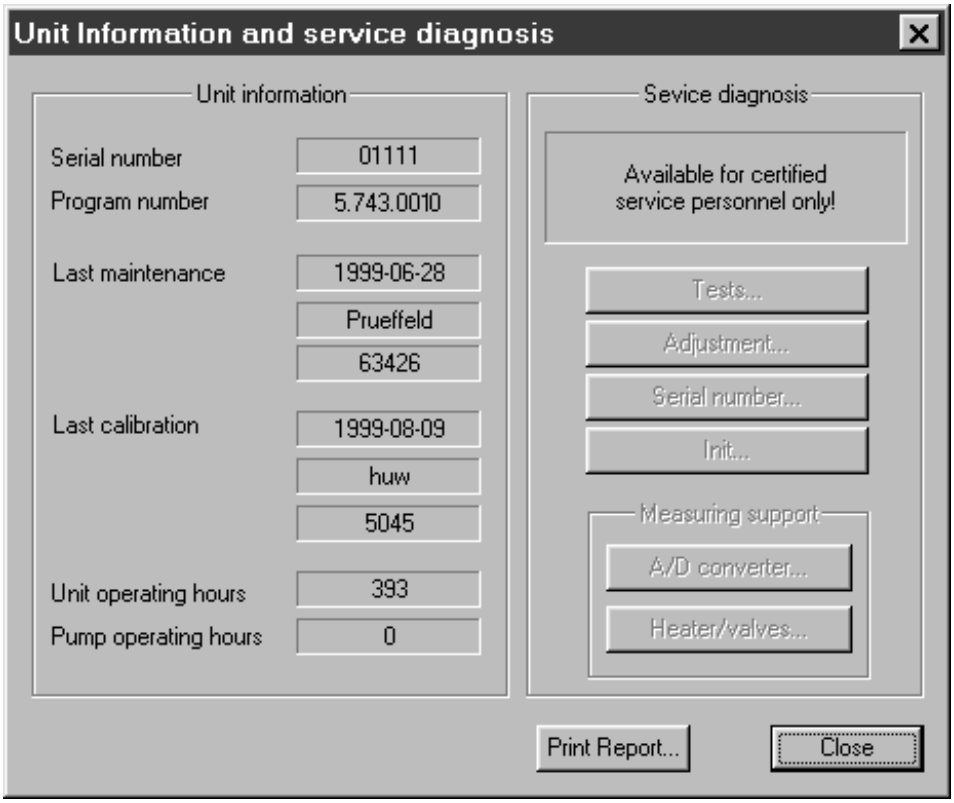
This menu item is used to delete the list automatically formed from the entered sample identifiers **ID1**.

4.3 Program information

4.3.1 Unit information

763 PVC THERMOMAT CONTROL / View / Unit information

The **Unit information** and **Service diagnosis** window opens.



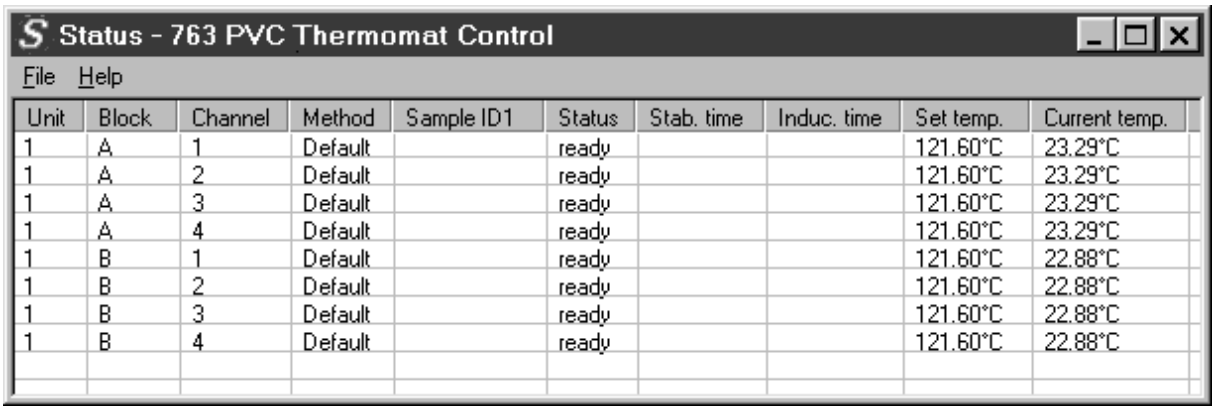
Serial number	Serial number of the selected 763 PVC Thermomat.
Program number	Number of the EEPROM program of the selected 763 PVC Thermomat.
Last maintenance	Date of the last maintenance with the signature of the service technician who carried out the maintenance work.
Last calibration	Date of the last adjustment with signature and code of the office which carried it out.
Unit operating hours	Number of hours that the instrument has been switched on.
Pump operating hours	Number of hours that the gas flow has been switched on.
<Print report>	Print out unit information report.

The **Service Diagnosis** *block is password-protected and is only accessible to trained service personnel.*

4.3.2 Status overview

763 PVC THERMOMAT CONTROL / View / Status overview

The **STATUS - 763 PVC THERMOMAT CONTROL** window opens and displays an overview of all available channels of the instruments connected to the PC.



This window contains the following columns:

Unit	Instrument number (1...4).
Block	Instrument block (A, B).
Channel	Channel (1...4).
Method	Method name.
Sample ID1	Sample identifier 1.
Status	Status of channel:
ready	No active measurement. The channel is ready for the start of a determination.
running	Running measurement.
finished	Determination finished. The channel is ready for the start of a new determination.
error	Communication error between instrument and PC.
Stab. time	Determined stability time.
Induc. time	Determined induction time.
Set temp.	Set temperature defined in method.
Current temp.	Actual block temperature.

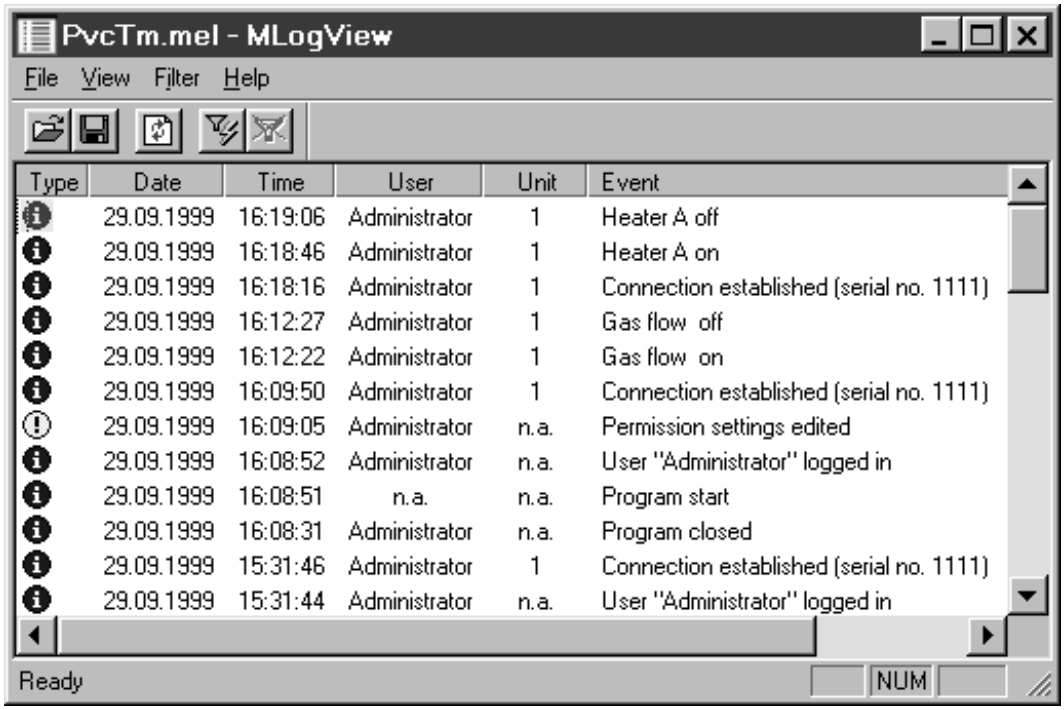
The width of the columns in the **STATUS - 763 PVC THERMOMAT CONTROL** window can be altered by pulling the right-hand field margin of the column title with the mouse. These settings together with the window size and window position can be permanently stored with **763 PVC THERMOMAT CONTROL / Options / Save settings now**.

4.3.3 Events overview

Event window

763 PVC THERMOMAT CONTROL / View / Event log overview

The **PVCTM.MEL - MLOGVIEW** window opens in which an overview of all the events concerning the 763 PVC Thermomats connected to the PC is displayed.



This window contains the following columns:

Type	Type of event:
	Information about an event which has taken place properly.
	Warning about a special event.
	Error message about the faulty event.
Date	Date of the event. The format depends on the settings defined under **Regional Settings / Date** in Windows.
Time	Time of the event. The format depends on the settings defined under **Regional Settings / Time** in Windows.
User	Name of the operator logged in when the event occurred.
Unit	Instrument number(1...4).
Event	Description of the event.

Menus

The events window contains the following main menus:

File Open and save event log files.

View View toolbar and status bar, update.

Filter Filter the events.

Help Program-specific on-line help.

Icons

The following icons are shown in the events window:

 Open existing event log file (***.mel**).

 Save event log file as text file (***.mel**).

 Update event overview.

 Selection-based filter.

 Remove filter.

Filter

 **PVCTM.MEL - MLOGVIEW / Filter / Selection based filter**

The selection-based filter is a method for filtering records rapidly and conveniently. This is done by first marking an entry in a field in the determination overview. After activating the selection-based filter the table is filtered so that only records which contain the marked entry are shown.

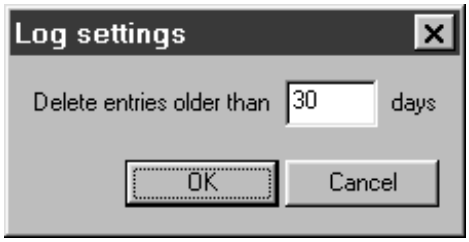
 **PVCTM.MEL - MLOGVIEW / Filter / Remove filter**

The filter used on the event overview is removed and all events are shown again.

Parameter for event recording

763 PVC THERMOMAT CONTROL / Options / Event log

The **Log settings** window opens:



Delete entries older than...

Number of days before events are automatically deleted.
Range: **1 ... 365**

4.4 Calibration functions

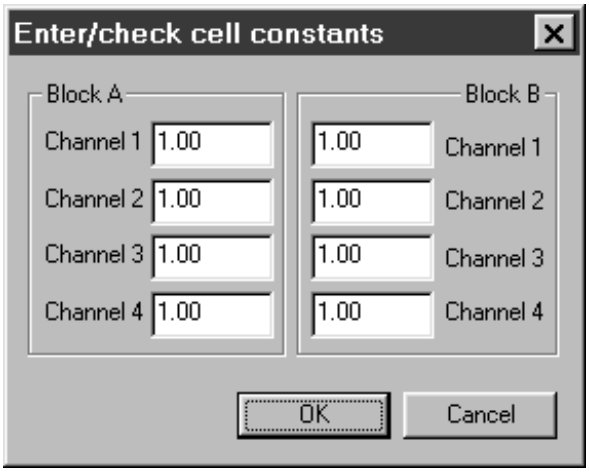
4.4.1 Determination of cell constants

As in PVC Thermomat measurements only the alterations in the conductivity are measured and evaluated, the cell constants of the conductivity cells used in the PVC Thermomat, which are normally in the range **1.00...1.20**, only need to be entered exactly if the absolute value for the conductivity is to be shown correctly. The exact entry is also required before carrying out the GLP test for conductivity. The cell constants can either be entered manually or determined automatically with the aid of a defined standard solution.

Manual entry of cell constants

763 PVC THERMOMAT CONTROL / Tools / Cell constants / Manual...

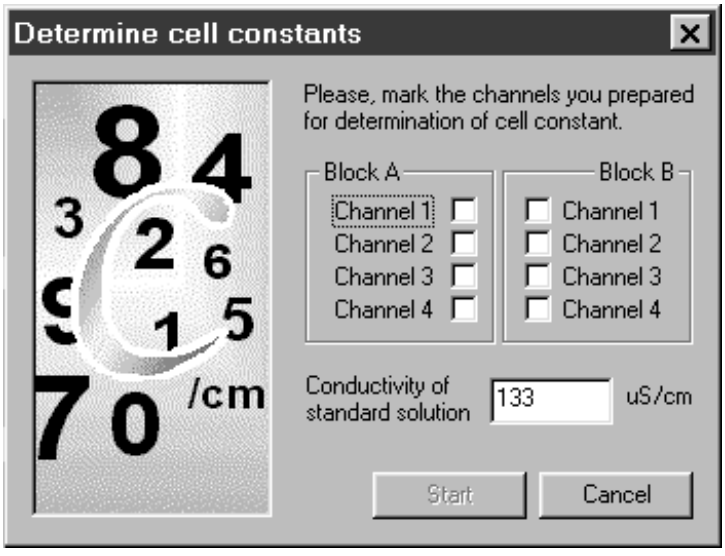
The **Enter/check cell constants** window appears in which the cell constant for the conductivity cell can be entered for each **Channel**.



Automatic determination of cell constants

763 PVC THERMOMAT CONTROL / Tools / Cell constants / Automatic...

The **Determine cell constants** window appears:



Block A

Channel 1...4 Each channel of Block A for which the cell constant of the conductivity cell is to be determined can be marked.

Block B

Channel 1...4 Each channel of Block B for which the cell constant of the conductivity cell is to be determined can be marked.

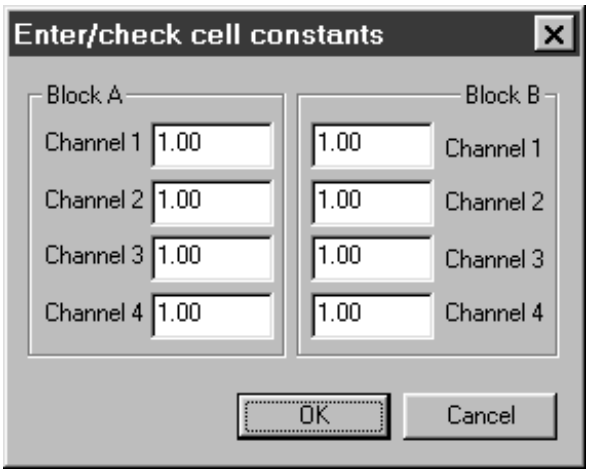
Conductivity of standard solution

Conductivity of the standard solution in µS/cm which is filled into all measuring vessels for which the cell constant of the conductivity cell is to be determined.

A solution of c(KCL) = 1 mmol/L, prepared from the 6.2301.060 Conductivity standard (KCl 0.1 mol/L) available as an option, can be used as standard solution. The conductivity of this standard solution is:

Temperature	Conductivity
18 °C	127 µS/cm
19 °C	130 µS/cm
20 °C	133 µS/cm
21 °C	136 µS/cm
22 °C	138 µS/cm
23 °C	141 µS/cm
24 °C	144 µS/cm
25 °C	147 µS/cm

<Start> Starts the automatic determination of the cell constants. When the determination has ended the **Enter/check cell constants** window appears in which the automatically determined cell constants have been entered:



Click on **<OK>** to accept these values or on **<Cancel>** to retain the previous values.

4.4.2 Determination of Delta T

The **temperature correction Delta T** describes the variation of the actual sample temperature from the temperature in the heating block and is a parameter of the method (see *section 4.5.2*). It can be automatically determined with the 6.1111.010 Calibrated external temperature sensor (part of the 6.5616.000 GLP test set available from Metrohm as an option). Since the direct temperature measurement in PVC samples is impossible, silicone oil must be used as sample instead of PVC.

Preparations

Fig. 5 shows in detail how to install the accessories for Delta T determination. Proceed as follows:

1 Prepare reaction vessel cover

- Install gas inlet tube **35** (part of the 6.5616.000 GLP test set, length = 148 mm) on the reaction vessel cover **2** (see *section 2.2.2*).
- Fasten distance piece **37** to the gas inlet tube **35** so that it is located at a distance of approx. 2 cm from the upper end of the reaction vessel.
- Fasten distance piece **37** to the gas inlet tube **35** so that it is located at a distance of approx. 5 cm from the lower end of the reaction vessel.
- Insert temperature sensor **36** from above in opening **31** of the reaction vessel cover **2** and fasten it in the corresponding openings of the distance pieces **37**.

2 Prepare reaction vessel

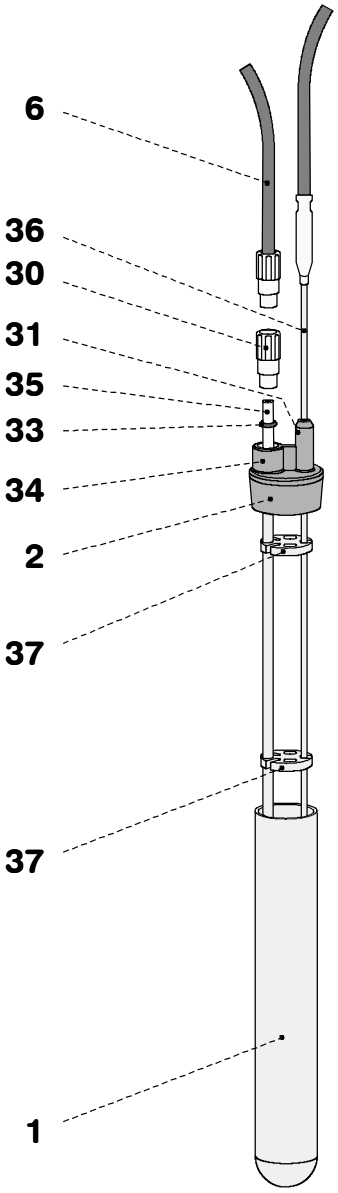
- Fill reaction vessel **1** with **5 g silicone oil** (e.g. FLUKA 85409).
- Place the reaction vessel cover **2** with temperature sensor **36** on the reaction vessel **1**.
- Slide the temperature sensor **36** right down (the sensor must touch the base of the vessel).

3 Insert and connect reaction vessel

- Place the prepared reaction vessel **1** in channel 2 or 3 of the heating block.
- Attach one end of the brown connecting tubing **6** at connection nipple **30** on the reaction vessel cover **2**.
- Attach the other end of the brown connecting tubing **6** at the connection **19** on the 763 PVC Thermomat (see *Fig. 3*).

4 Modify method parameters

- In the control window select the required **Method** for the heating block for which the temperature correction **Delta T** is to be determined.



1	**Reaction vessel (6.1429.040)**
2	**Reaction vessel cover (6.2753.100)**
6	**Connecting tubing (6.1805.080)** for gas supply to reaction vessel
30	**Connection nipple (6.1808.090)**
31	**Connection** Feedthrough for temperature sensor **36**
33	**Sealing ring (6.1454.040)**
34	**Connection** for connection nipple **30**
35	**Gas inlet tube (6.2418.000)** length = 148 mm
36	**Temperature sensor Pt100 (6.1111.010)**
37	**Distance piece (6.2042.040)**

Fig. 5: Accessories for Delta T determination

- Click on … to open the **Method parameters** window. Enter the required set temperature for the sample under **Temperature** and set **Delta T** to **0°C**. Set **Gas flow** to the desired value and click on **<Save>**.

5 Start gas flow and heating

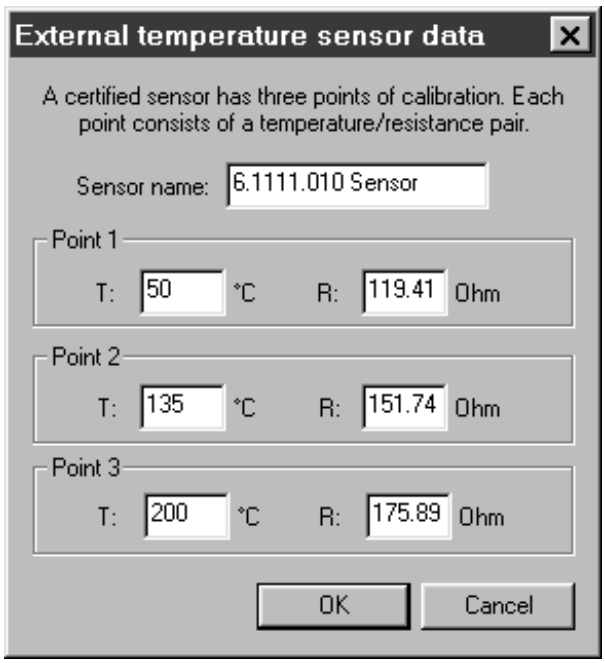
- Switch on gas flow manually.
- Start heating for the selected block manually.

Carrying out

763 PVC THERMOMAT CONTROL / Tools / Determine Delta T / Block A

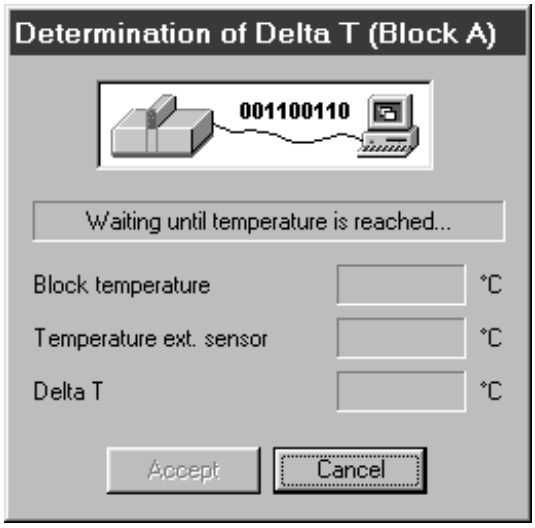
763 PVC THERMOMAT CONTROL / Tools / Determine Delta T / Block B

These menu items are used to start the automatic determination of **Delta T** for Block A or Block B. The **External temperature sensor** window first appears in which the calibration data of the temperature sensor used can be entered (once data have been entered they are retained):



Sensor name	Name of temperature sensor.
Point 1...3	Calibration points 1...3 of the temperature sensor.
T	Temperature at the calibration point in °C.
R	Resistance at the calibration point in ohms.

When the external temperature sensor data have been entered the **Determination of Delta T** window appears.



If the temperature defined in the method has not been reached then the heating is started automatically. The message **Waiting until temperature is reached...** appears in the window.

When the temperature defined in the method has been reached the message **Measuring temperature. Please wait...** appears in the window. The three following values are shown below this message:

Block temperature	The temperature in the heating block measured by the internal temperature sensor.
Temperature ext. sensor	The temperature in the reaction vessel measured by the external temperature sensor.
Delta T	The temperature correction determined from the difference between **Block temperature – Temperature ext. sensor**.

As soon as both the measured temperatures are stable the message **Measured temperature is now stable!** appears. The determined temperature correction **Delta T** can now be included in the method with **<Accept>**. The message **Delta T is now set in the corresponding method** then appears.

4.5 Methods

4.5.1 Method handling

Create method

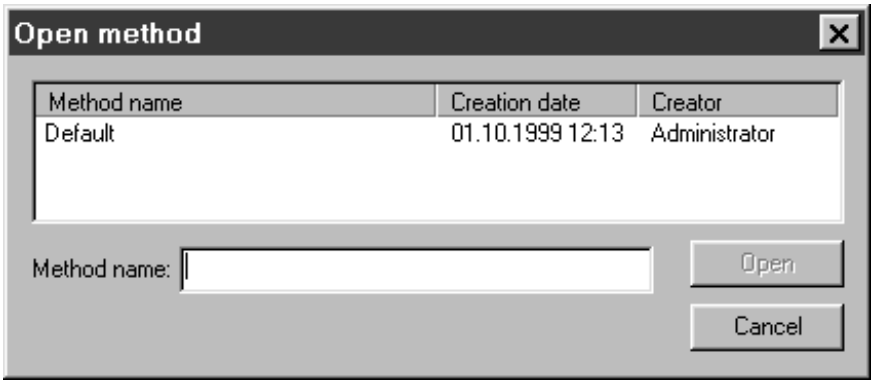
763 PVC THERMOMAT CONTROL / File / New method

Creates a new method. A window opens for the input of the method parameters (see *section 4.5.2*). When all parameters have been entered the method must be stored under a new name with **<Save as...>**.

Open method

763 PVC THERMOMAT CONTROL / File / Open method

Opens an existing method. The **Open method** window opens for method selection.

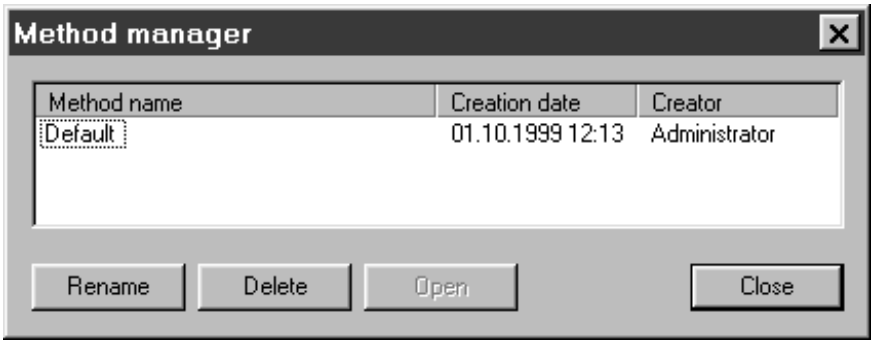


Method name	Name of the method.
Creation date	Date and time at method creation.
Creator	Name of the logged-in user at method creation.
<Open>	Open the method parameters window (see *section 4.5.2*).

Method manager

763 PVC THERMOMAT CONTROL / File / Method manager

The **Method manager** window opens for renaming, deleting and opening methods.



Method name	Name of the method.
Creation date	Date and time at method creation.
Creator	Name of the logged-in user at method creation.
<Rename>	Rename the selected method.
<Delete>	Delete the selected method.
<Open>	Open the method parameters window (see *section 4.5.2*).

Save method

In the **Method parameters** window (see *section 4.5.2*) the two following buttons are available for storing an opened method:

<Save>	Saves the method under the same name.
<Save as...>	Saves the method under a new name. The old method is retained. Methods can also be copied in this way.

Print method report

The following button is available in the **Method parameters** window (see *section 4.5.2*) for printing out all the method parameters of the opened method:

<Print>	Prints out method report.

4.5.2 Method parameters

Overview

763 PVC THERMOMAT CONTROL / File / Open method

By clicking on this menu item or on  beside the method name in the operating field of the **763 PVC THERMOMAT CONTROL** window the window with the method parameters opens; it contains the following tabs:

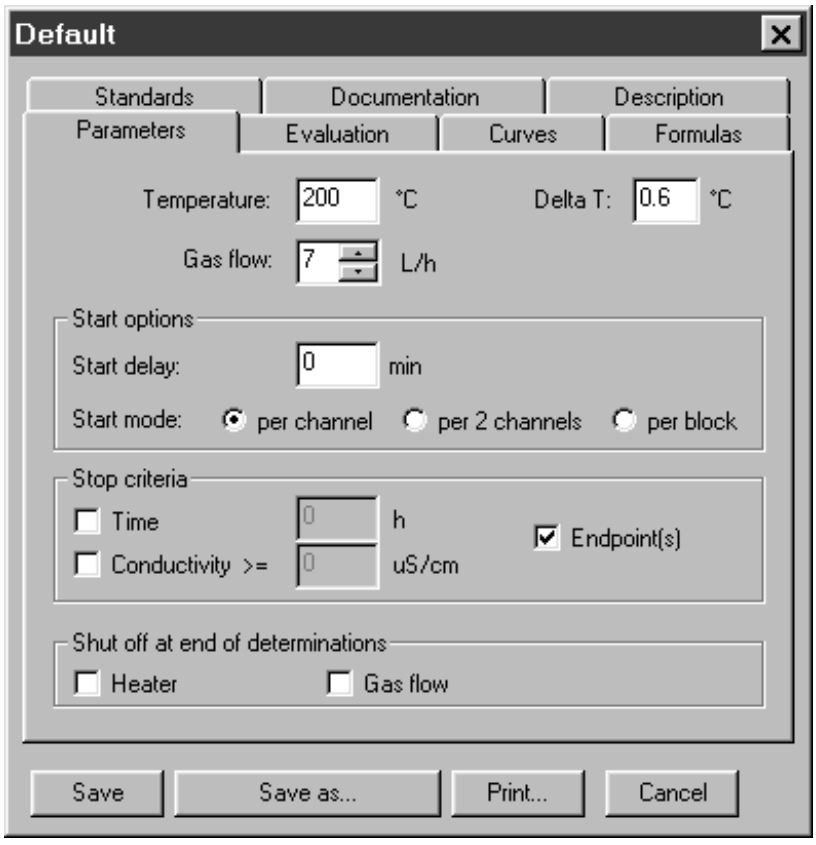
Parameters	Measuring parameters.
Evaluation	Parameters for curve evaluation.
Curves	Parameters for scaling the axes.
Formulas	Equation input for further calculation of the induction and stability times.
Standards	Parameters for calculating the standard time.
Documentation	Parameters for report output.
Description	Freely-definable comments about method.

The **Method parameters** window contains the following buttons:

<Save>	Saves method under the same name.
<Save as>	Saves method under a new name; the old method is retained.
<Print>	Prints out method report.
<Cancel>	Closes method parameter window without saving the modified parameters.

Parameters

Tab **Parameters** of the **Method parameters** window with measuring parameters.



Temperature	**Set temperature** of the sample. Range: **50...220 °C**
Delta T	**Temperature correction**: Correction value for increasing the block temperature in order to reach the set temperature in the sample. This value is determined by measuring the variation of the actual sample temperature from the block temperature using a calibrated external temperature sensor (see *section 4.4.2*). Range: **-9.9...9.9 °C**

If no temperature sensor is available for the **Delta T** determination, a value of **+0.6 °C** can be entered for temperatures of **160 °C to 200 °C** (this value was determined using 5 g silicone oil and a gas flow of 7 L/h nitrogen).

Gas flow

Gas flow through sample. It cannot be set individually for each block; the flow rate for both blocks depends always on the value entered in the method loaded for Block A.
Range: **4...15 L/h**

Start options

Start delay

Delay period until start of measurement.
Range: **0...1000 min**

Start mode

Mode for start and stop of the channels:

per channel

All 8 channels can be started and stopped individually.

per 2 channels

Each 2 channels can be started and stopped together.

per block

The 4 channels in one block can be started and stopped together.

Stop criteria

Time

Time until automatic termination of the determination.
Range: **0.1...100 h**

Conductivity

Limit value for conductivity. If this value is reached then the determination is terminated automatically.
Range: **1...400 mS/cm**

Endpoint(s)

Automatic termination of the determination when each active channel has reached all endpoints.

Shut off at end of determinations

Heater

Automatically switches off the heating at the end of the determination.

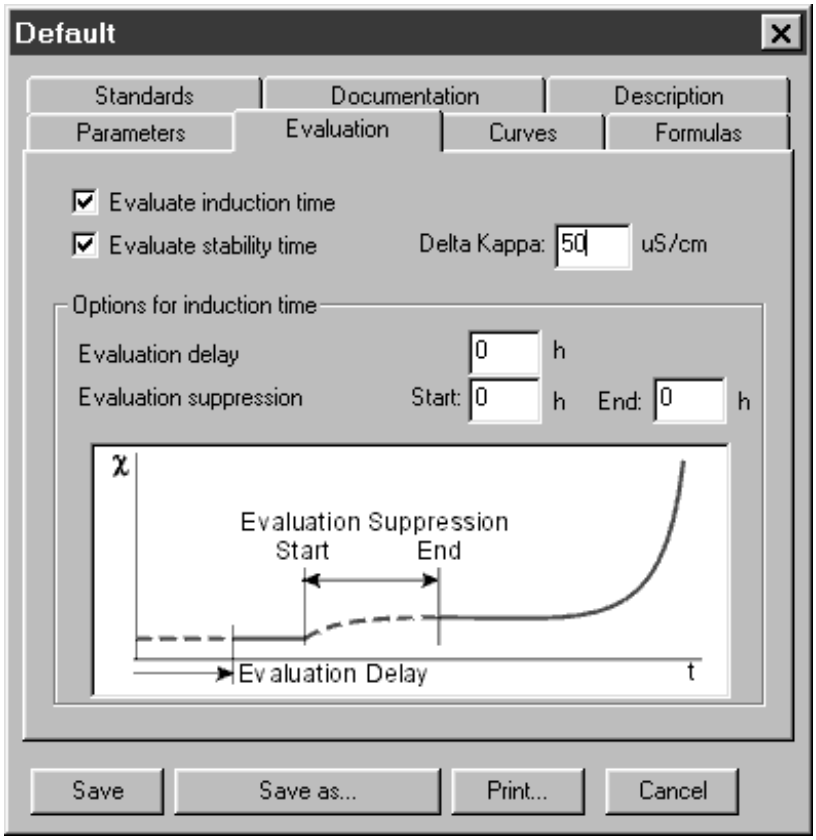
Gas flow

Automatically switches off the gas flow at the end of the determination.

*If the gas flow is automatically switched off, the **reaction vessels must be removed immediately** from the PVC Thermomat after the end of the determination. If the reaction vessels are not removed, the instrument can be damaged.*

Evaluation

Tab **Evaluation** of the **Method parameters** window with parameters for curve evaluation.

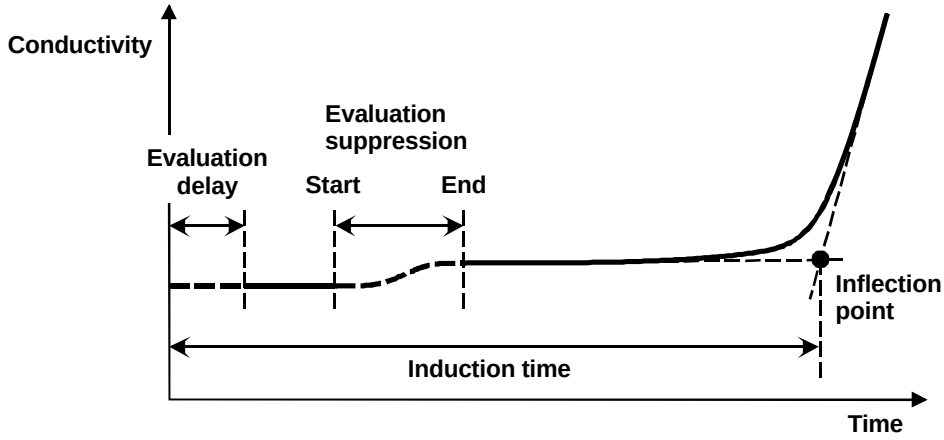


Evaluate induction time	Determines the **induction time** (see below).
Evaluate stability time	Determines the **stability time** (see below).
Delta Kappa	Conductivity alteration for the determination of the **stability time**. Range: **1...400 mS/cm**
Evaluation delay (option for the induction time)	Delay period until start of the curve evaluation. Range: **0...100 h**
Evaluation suppression (option for the induction time)	
Start	Time from start of the determination up to start of the evaluation suppression. Range: **0...100 h**
End	Time from start of the determination up to end of the evaluation suppression. Range: **0...100 h**

Induction time

The **induction time** is the time up to the inflection point of the *conductivity* vs. *time* curve recorded by the PVC Thermomat. The induction time is a characteristic value for the thermal stability of the sample under investigation.

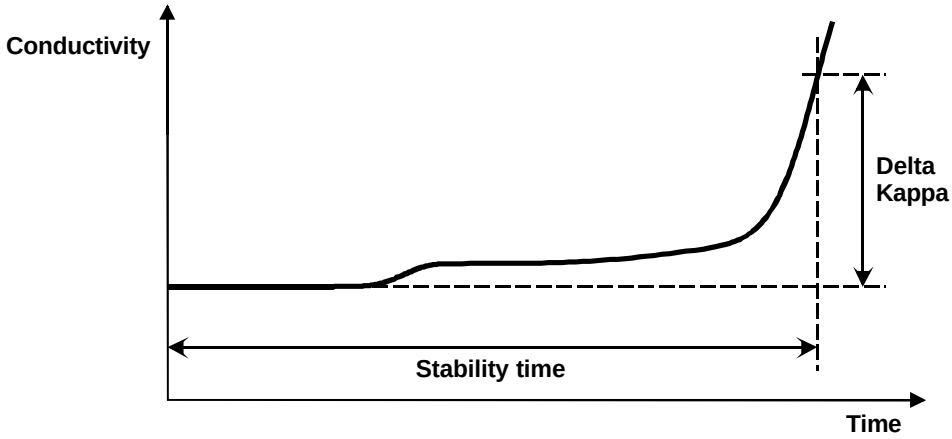
For automatic evaluation of the induction time the second derivative of the measured curve is used. This derivative has a maximum at the inflection point and can be displayed in the **Live graph** window. In order to recognize an inflection point certain criteria concerning the height and width of the peak in the second derivative must be fulfilled. The inflection point can also be evaluated manually as point of intersection of the two tangents of the extended straight arms of the curve.



With certain samples it is possible for the conductivity to increase in small steps a long time before the induction time itself. In order that this increase is not evaluated as the endpoint the operator can delay the evaluation or suppress it in a particular range.

Stability time

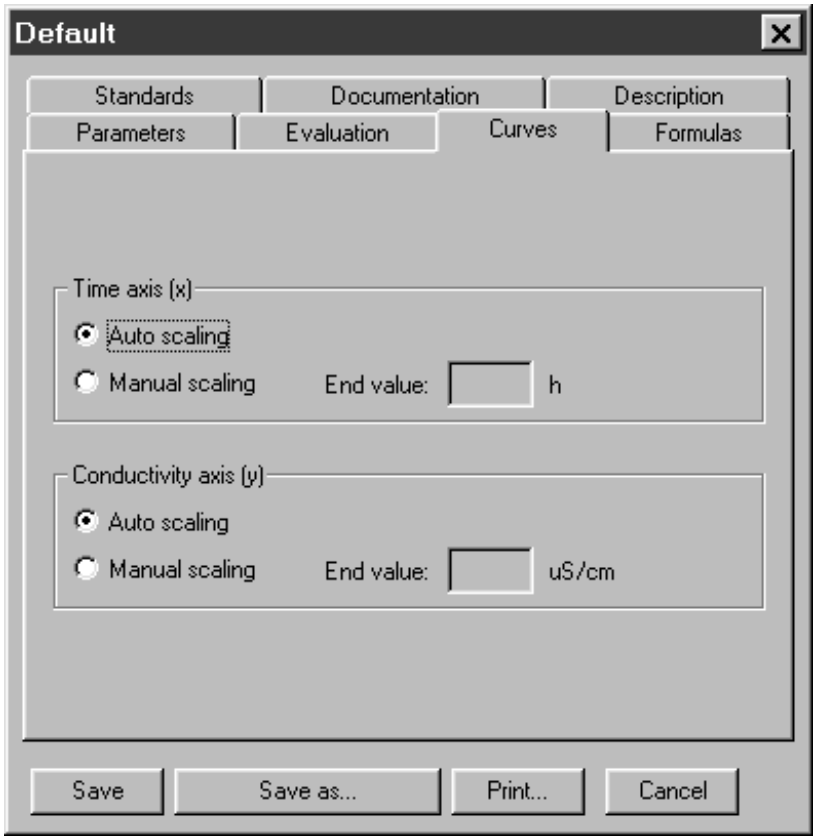
The **stability time** is the time required to achieve a given conductivity alteration of **Delta Kappa**.



The input of an evaluation delay or evaluation suppression has no influence on this evaluation mode; the calculation is always carried out from the time that the measurement was started.

Curves

Tab **Curves** of the **Method parameters** window with parameters for scaling the curve axes.



Time axis(x)

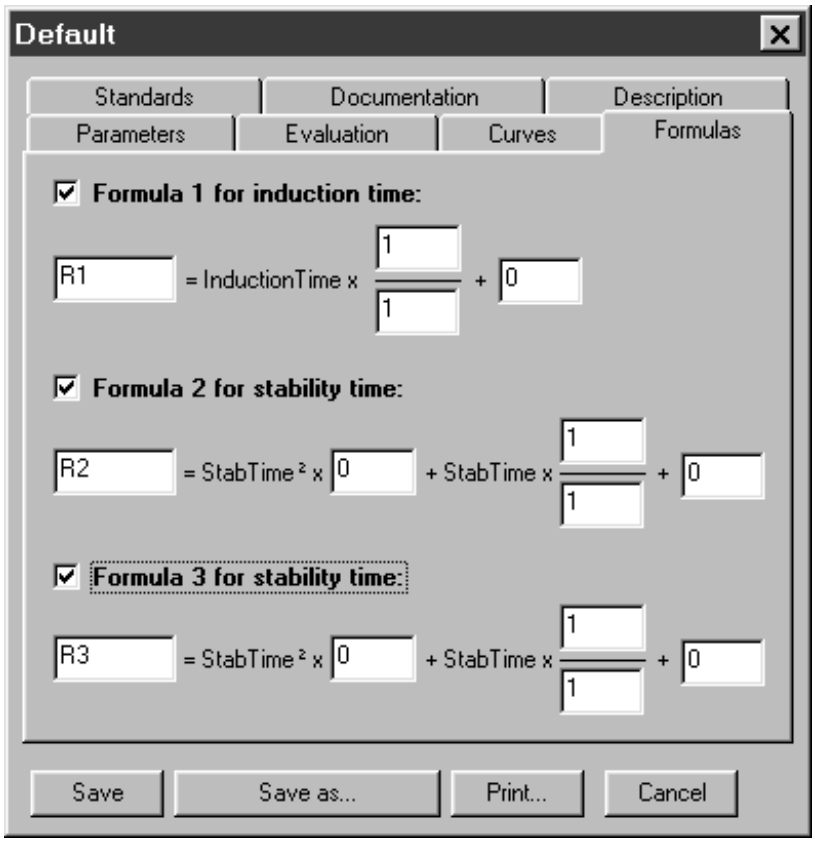
Auto scaling	Automatic fixing of the initial and final values of the time axis.
Manual scaling	Manual fixing of the final value of the time axis.
End value	Final value of the time axis. Range: **0.5...100 h**

Conductivity axis(y)

Auto scaling	Automatic fixing of the initial and final values of the conductivity axis.
Manual scaling	Manual fixing of the final value of the conductivity axis.
End value	Final value of the conductivity axis. Range: **10...400 mS/cm**

Formulas

Tab **Formulas** of the **Method parameters** window with parameters for entering equations for further calculations with the induction and stability times.



Formula 1 for induction time

If this option is switched on then the automatically determined **Induction time** will be calculated from the values entered in the equation by the operator and then stored as the **Formula 1** result under the name entered in the first field.

Formula 2 for stability time

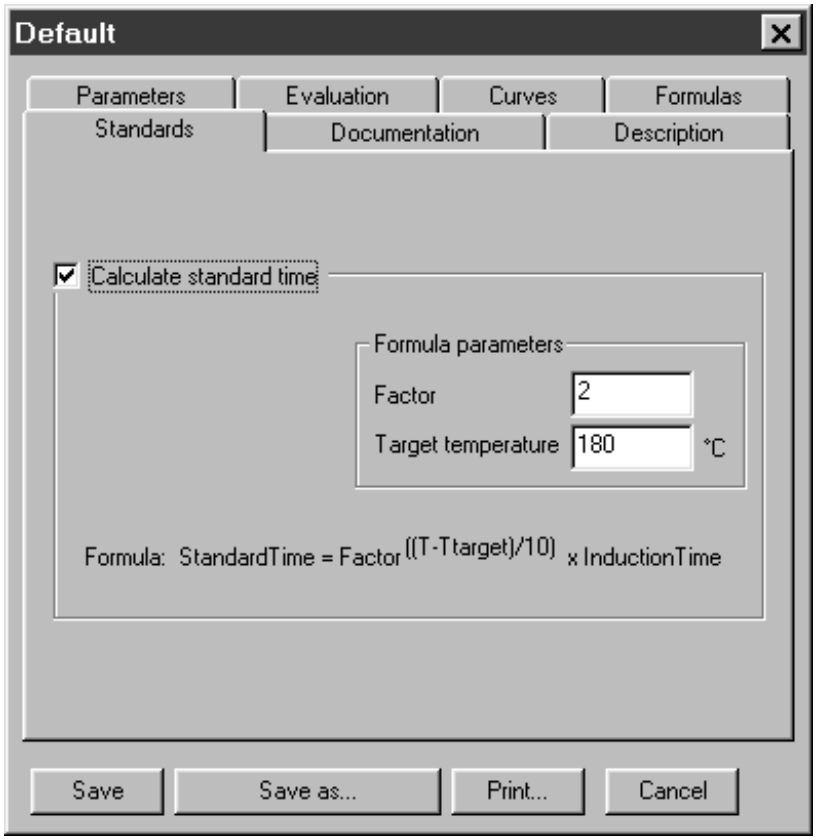
If this option is switched on then the automatically determined **Stability time** will be calculated from the values entered in the equation by the operator and then stored as the **Formula 2** result under the name entered in the first field.

Formula 3 for stability time

If this option is switched on then the automatically determined **Stability time** will be calculated from the values entered in the equation by the operator and then stored as the **Formula 3** result under the name entered in the first field.

Standards

Tab **Standards** of the **Method parameters** window with parameters for calculating the standard time.



Calculate standard time Switches the calculation of the standard time according to the following equation on/off:

$$\text{Standard time} = \text{Factor}^{(\text{Temperature} - \text{Target temperature})/10} \cdot \text{Induction time}$$

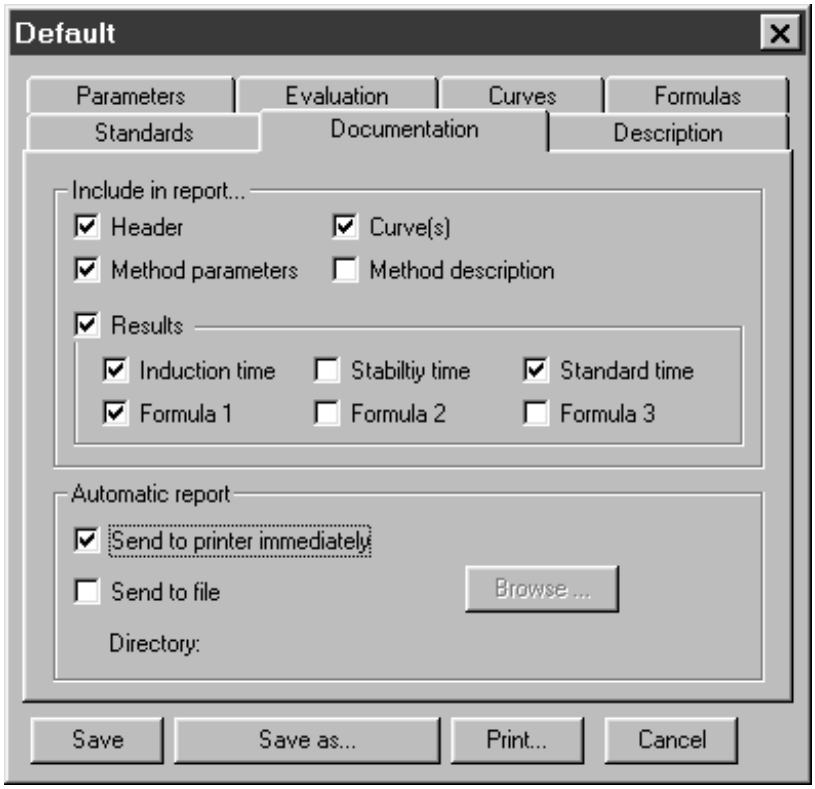
Formula parameters

Factor Parameter for calculating the standard time which gives the factor by which the time changes by 10 °C. This value can be determined by the **extrapolation** (see *section 4.7.8*) of results obtained at different temperatures.

Target temperature Target temperature for calculating the standard time.

Documentation

Tab **Documentation** of the **Method parameters** window with parameters for the report output.



Include in report

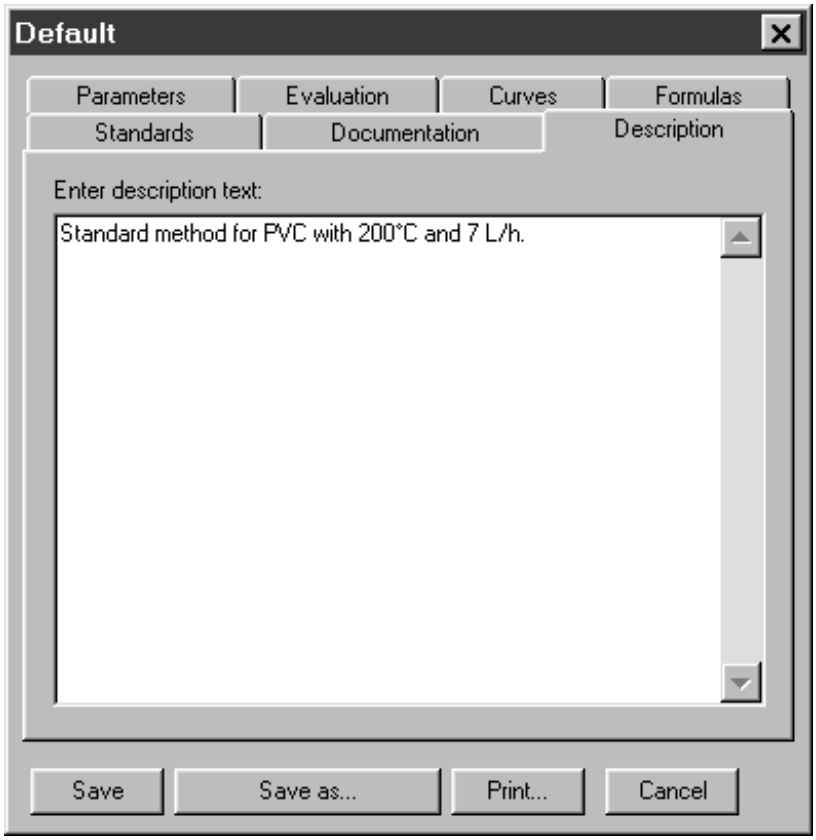
Header	Output of the header with company settings (if defined), printout date, sample identifications, instrument information, determination date, user, cell constant and serial number.
Method parameters	Output of the method parameters.
Curve(s)	Output of curves.
Method description	Output of the method description given on the **Description** tab.
Results	Output of the selected results: **Induction time**, **Stability time**, **Standard time**, **Formula 1...3**.

Automatic report

Send to printer immediately	Automatic printout of the reports defined under **Include in report** following the end of the determination.
Send to file	Automatic output of determination and method data following the end of the determination as a text file ***.txt** for further processing in other programs. The contents of this report does not depend on the report elements defined under **Include in report**.
<Browse>	Selection of the **Directory** for storing this file.

Description

Tab **Description** of the **Method parameters** window with freely-definable **comments about the method**.

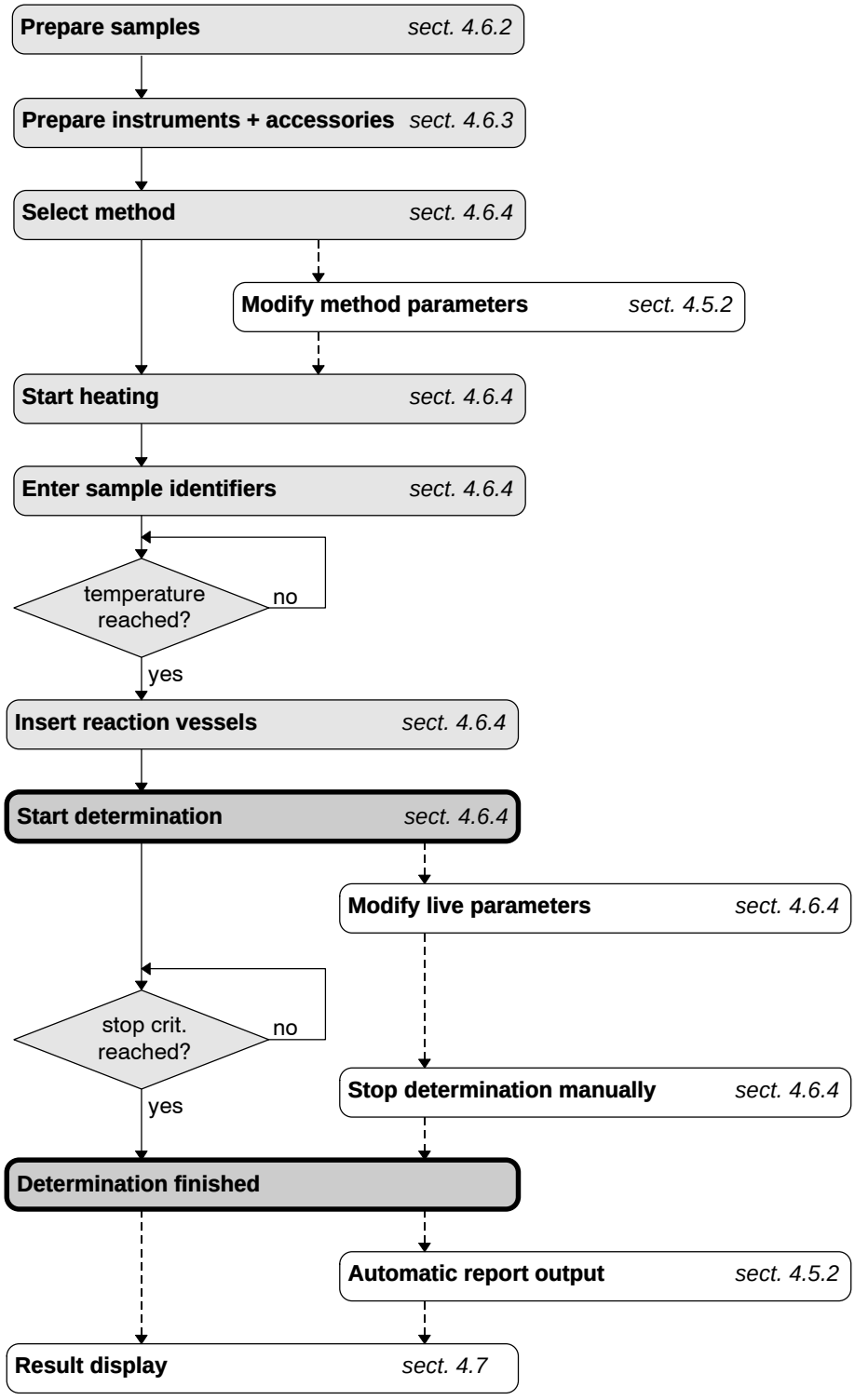


Enter description text Comments about the method.

4.6 Determinations

4.6.1 Flow chart

The following flow chart provides an overview of all steps for carrying out determinations of the oxidative stability.



4.6.2 Sample preparation

PVC or other plastics which decompose through the action of heat with cleavage of hydrogen halides are prepared in accordance with DIN 53'381 as follows:

1 Powdery samples

- Polymers, dry blends and coating powders need no special sample pretreatment.

2 Pastes

- Pastes are fused on a glass plate in a drying oven to a sheet with a thickness of about 0.5 mm. After cooling, the sheet is cut into pieces of edge length about 2 mm.

3 Solid samples

- Molding compounds in granular form, molded and finished parts, semi-finished products as well as coatings separated from the substrate are pulverized in accordance with DIN 53'733 and screened to separate the screening fraction between the analytical screens with wire mesh screen DIN 4188-1.4 (wire mesh of stainless steel with 1.4 mm inside diameter mesh size) and wire mesh screen DIN 4188-2 (wire mesh of stainless steel with 2 mm inside diameter mesh size) for the experiments.

4 Sheets

- Sheets are cut into pieces with an edge length of about 2 mm.

According to DIN 53'581, the samples are to be added directly into the prewarmed reaction vessels inserted in the heating block. Nevertheless, comparative measurements have shown identical results for sample addition to cold or hot reaction vessels.

4.6.3 Prepare instruments and accessories

Clean instruments and accessories

The **cleanness** of instruments and accessories is an absolute necessity for achieving reliable, reproducible and correct analytical results. Even minute contamination can catalytically accelerate the thermal decomposition and lead to completely false results. Make sure that the following checks and cleaning procedures are carried out before the start of a determination:

1 Openings for reaction vessels

- Check whether the openings for the reaction vessels are clean and empty. Blow out any dust in the openings with nitrogen.

- When the instrument is not in use always close the openings with stoppers.

2 Measuring vessels and accessories

- After the contents have been discarded clean used **measuring vessels 4** several times with alcohol and distilled water (**do not use acetone!**).
- Rinse the **measuring vessel cover 5** complete with electrodes **28** and PTFE tube **27** several times with distilled water and acetone. If necessary, remove the protective ring **29** for better access to the electrodes **28** (see *Fig. 4*).
- Rinse the **connecting tubing 3** between reaction vessel and measuring vessel several times with distilled water and acetone.

3 Reaction vessels and accessories

- It is recommended to use **new reaction vessels 1** and **gas inlet tubes 32** for each determination. Blow out new reaction vessels with nitrogen.
- Used and not too strongly contaminated reaction vessels and gas inlet tubes can be cleaned by immersion in boiling RBS solution for 1 h or a similar laboratory flushing agent. They must then be thoroughly rinsed with distilled water and acetone.

RBS attacks glass. Reaction vessels and gas inlet tubes therefore cannot be cleaned ad infinitum.

- Remove the gas inlet tube **32** from the **reaction vessel cover 2** and rinse it several times with distilled water and acetone. Dry the cover at 80 °C in a drying oven.

Reaction vessel covers which have not been dried properly can falsify the results of the following determinations.

Prepare measuring vessels

The cleaned measuring vessels must always be prepared as follows before the start of a determination (see *Fig. 4*):

1 Fill measuring vessels

- Fill each cleaned **measuring vessel 4** with **60 mL distilled water**. For analysis times >24 h more than 60 mL distilled water must be added to take the evaporation losses of approx. 7 mL/d into account and to ensure that the electrodes remain adequately immersed.

2 Insert measuring vessels

- Place the **measuring vessel cover 5** fitted with a gas inlet tube on the measuring vessel **4** (see *Fig. 4*).
- Place the measuring vessel **4** with its cover in one of the openings provided for it on the 763 PVC Thermomat and connect the electrode plugs **26** on the cover to the corresponding sockets **20** on the 763 PVC Thermomat (see *Fig. 3*).

Prepare reaction vessels

Before the start of a determination the reaction vessels are filled with the samples and prepared as follows (see *Fig. 4*):

1 Weighing in the samples

- Weigh out **0.5 g** of each of the prepared samples into the cold reaction vessels **1**.

According to DIN 53'581, the samples are to be added directly into the prewarmed reaction vessels inserted in the heating block. Nevertheless, comparative measurements have shown identical results for sample addition to cold or hot reaction vessels.

2 Mount accessories

- Take the upper rim of the reaction vessel **1** in your hand (e.g. in the space between thumb and index finger) and rotate the glass though 360°. This provides the degreased glass with a thin **grease film**, without which it is very difficult to remove the cover from the vessel after the determination.
- Insert an **gas inlet tube 32** (length = 98 mm) into the reaction vessel cover **2** and fasten it by screwing down the connection nipple **30**.
- Place the **reaction vessel cover 2** on the reaction vessel **1**. Rotate the cover so that the gas inlet tube **32** is as close as possible to the vessel wall.
- Fasten the **white connecting tubing 3** to the measuring vessel **4** on to the corresponding connection **31** on the reaction vessel cover **2**.
- Place the prepared reaction vessel **1** into the vessel holder.

4.6.4 Carry out determinations

Select method

The determination method is selected in the **Method** field in the control window.

Different methods with different temperatures can be selected for Block A and Block B. In contrast, the gas flow which is defined in the method loaded for Block A also applies to Block B.

By clicking on  the method parameters (see *section 4.5.2*) can be altered as required.

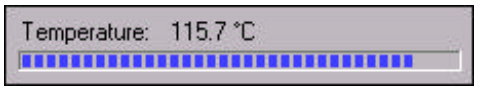
Switch on heating

The heating of the two blocks A and B to the temperature set in the method can be switched on individually for each block. This is done by clicking on the following button.

As soon as the heating is switched on the color of the frame of this button switches to red. At the same time the temperature display **9** "**TEMPERATURE"** on the 763 PVC Thermomat starts to blink for the selected block.

If this button is clicked on again during the heating-up phase then the heating will be switched off.

The actual temperature is shown digitally beside the button. A bar below shows the absolute variation of the actual temperature from the target temperature; the bar length corresponds to 50 °C.

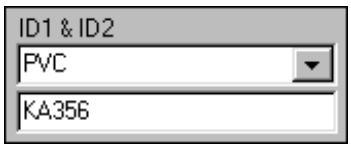


When the selected temperature has been reached the temperature display **9** "**TEMPERATURE"** stops blinking and the LED remains lit up. At the same time the color of the frame of the button switches to **green**. The block is now ready for receiving the samples and starting the determination.

If this button is clicked on the heating is switched off.

Enter sample identifiers

The sample identifiers **ID1** and **ID2** can be entered in the corresponding fields of the operating area in the control window before or after starting the determination.



The entry for **ID1** can be selected from the automatically formed list of identifiers which have already been entered. This list can be deleted again with **763 PVC THERMOMAT CONTROL / Options / Clear sample ID history**.

Insert reaction vessels

When the heating block reaches the temperature defined in the method (frame color of the **<Heat to...>** button turns **green**) the reaction vessels can be inserted into the PVC Thermomat 763 as follows (see *Fig. 4*):

1 Insert reaction vessels

- Place the prepared reaction vessels **1** in the heating block openings on the 763 PVC Thermomat. Close any openings which are not in use with stoppers or empty reaction vessels to prevent contamination.

2 Connect reaction vessels

- Connect the white connecting tubing **3** fastened to the reaction vessel cover **2** to the corresponding connection piece **22** on the measuring vessel cover **5**.
- Screw the brown gas supply tubing **6** onto the reaction vessel cover **2** and the corresponding connection **19** on the 763 PVC Thermomat (see *Fig. 3*).

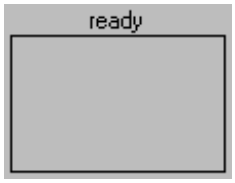
Start determinations

Determinations can be started by clicking on the following buttons in the operating area of the **763 PVC THERMOMAT CONTROL** window:

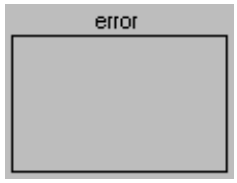
Start	Starts a single determination for the corresponding channel.
Start 1-2	Starts a double determination for channels 1 and 2.
Start 3-4	Starts a double determination for channels 3 and 4.
Start 1-4	Starts a multiple determination for channels 1 to 4.
▶A	Starts all Block A channels.
▶B	Starts all Block B channels.

Live curve

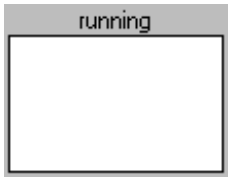
The live curve of a started determination is shown in the operating window beside the channel number. Either a status message or the actual measured value appears above the curve. A differentiation is made between the following conditions:



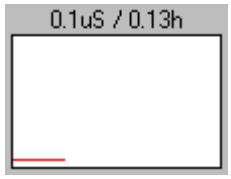
No active measurement.
The channel is ready for the start of a determination.



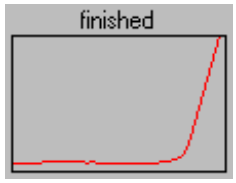
No active measurement.
Communication error between instrument and PC.



Determination started.
The first value is being measured.



Running measurement.
Measured value and time are shown above the live curve.

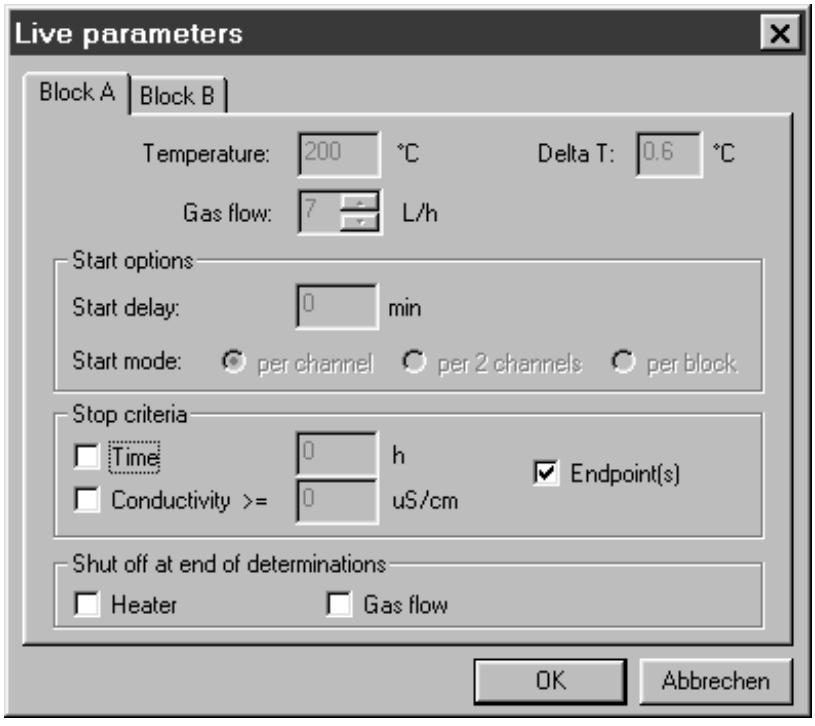


Determination finished.
The channel is ready for the start of a new determination.

Live parameters

763 PVC THERMOMAT CONTROL / View / Live parameters

The **Live parameters** window opens for altering method parameters during a running determination.



The following parameters for Block A or Block B can be altered live:

Stop criteria

Time	Time up to automatic termination of the determination. Range: **0.1...100 h**
Conductivity	Conductivity limit value. When this value is reached the determination is automatically terminated. Range: **1...400 mS/cm**
Endpoint(s)	Automatic termination of the determination when each active channel has reached all endpoints**.**

Shut off at end of determinations

Heater	Automatically switches off the heating at the end of the determination.
Gas flow	Automatically switches off the gas flow at the end of the determination.

*If the gas flow is automatically switched off, the **reaction vessels must be removed** from the PVC Thermomat within 1 h after the end of the determination. If the reaction vessels are not removed, a vacuum is generated in the course of the time by condensation of the further produced HCl in condensed water vapor from the measuring vessel and the measuring solution is siphoned back into the hot reaction vessel.*

Stop determinations

Determinations are normally terminated automatically when the stop criteria defined in the method have been achieved. Running determinations can also be stopped manually be clicking on the following buttons in the operating area of the **763 PVC THERMOMAT CONTROL** window:

Stop	Stops a single determination for the corresponding channel.
Stop 1-2	Stops a double determination for channels 1 and 2.
Stop 3-4	Stops a double determination for channels 3 and 4.
Stop 1-4	Stops a multiple determination for channels 1 to 4.

If a determination is stopped manually an inquiry **Do you want to save this determination?** appears. With **No** neither measuring data will be saved nor will results be automatically outputted.

4.7 Results

4.7.1 Determination overview

Open determination overview

The determination overview shows selected information in tabular form about all the determinations which are stored in the opened database. To open a determination overview the results window must be opened first and then the database opened.

763 PVC THERMOMAT CONTROL / File / Results

This menu item is used to open the **763 PVC THERMOMAT RESULTS** window. It is used for the display, output and recalculation of **results** of the determinations recorded by the 763 PVC Thermomat. The determination data are stored in the ***.mrd** database files and can be shown in this window as an overview table and as curves. The results window can run even when the control window is closed.

When the results window is opened the **Repos.mrd** database is automatically loaded; all the recorded determinations are loaded in this database as standard.

*The program part for display of the results window is organized as database and saved in the **Nachaus.prg** file, which normally has approx. 5 MB. If the size of this file exceeds 10 MB it should be newly optimized and compressed. To do this, close the program and select **Optimize / Program** from the PVC Thermomat program folder in the **Start / Program** menu.*

763 PVC THERMOMAT RESULTS / File / Open database

Opens an existing database. The **Open database** window opens for the selection of the database file ***.mrd**. When the database has been selected the corresponding **determination overview** is automatically opened. Only one database can be opened at any time.

*The **Repos.mrd** database has to be optimized and compressed from time to time, especially if data records are deleted frequently. To do this, close the program and select **Optimize / Database** from the PVC Thermomat program folder in the **Start / Program** menu.*

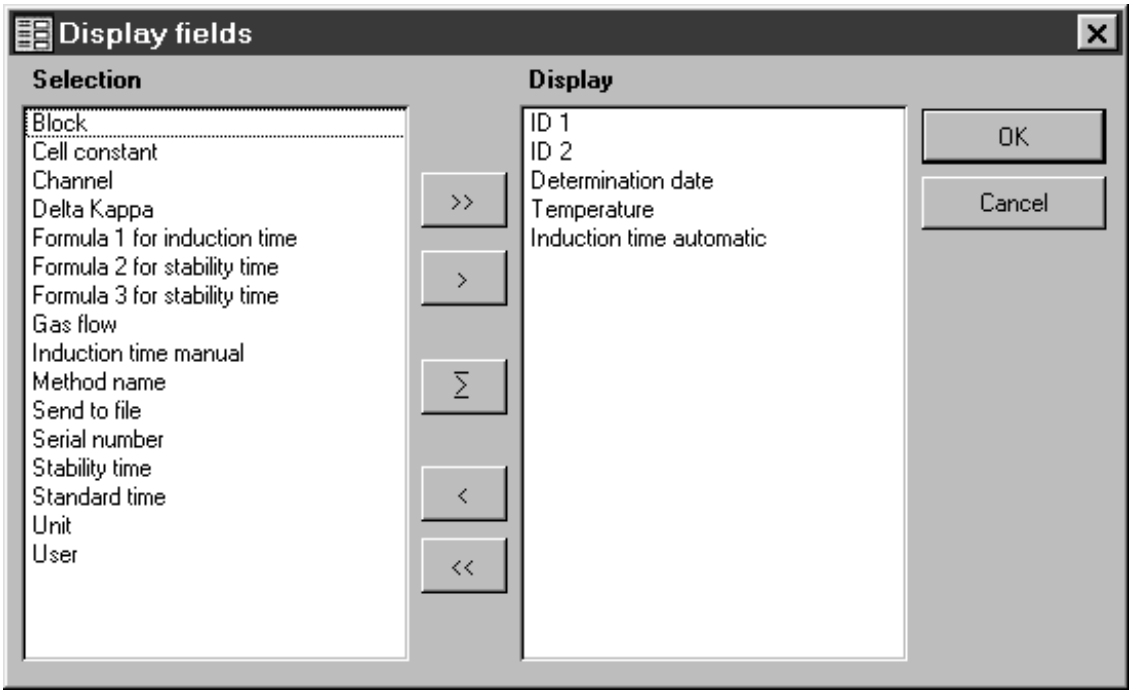
763 PVC THERMOMAT RESULTS / View / Determination overview

Displays an overview table with selected data fields of all the determinations contained in the opened database.

Format determination overview

763 PVC THERMOMAT RESULTS / Format / Select fields

The **Display fields** window opens for selection of the **fields** which are to be shown in the determination overview.



Selection — Selection of all the fields which can be used for the determination overview. The following fields are available:

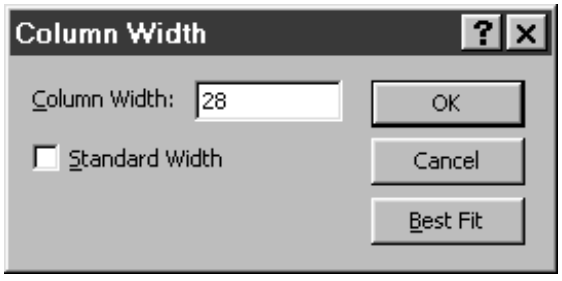
- **Block** — Number of the block used for the determination.
- **Cell constant** — Cell constant of the conductivity cell used for the determination.
- **Channel** — Number of the channel used for the determination.
- **Delta Kappa** — Alteration in conductivity for the determination of the **Stability time**.
- **Determination date** — Date and time at which the determination was started.
- **Formula 1 for induction time** — Result for **Formula 1**.
- **Formula 2 for stability time** — Result for **Formula 2**.
- **Formula 3 for stability time** — Result for **Formula 3**.
- **Gas flow** — Gas flow through sample.
- **ID 1** — Sample identifier 1.
- **ID 2** — Sample identifier 2.

Induction time automatic	Automatically determined **induction time**.
Induction time manual	**Induction time** manually determined with the re-evaluation.
Method name	Name of method.
Send to file	Folder in which the ***.txt** determination file is to be stored.
Serial number	Serial number of the instrument used for the determination.
Stability time	Automatically determined **stability time**.
Standard time	Automatically determined **standard time**.
Temperature	Set temperature for the method.
Unit	Number of the instrument used for the determination.
User	Name of the user logged in at the start of the determination.

Display	Fields which have been selected for the determination overview.
>>	Moves all fields in **Selection** to **Display**.
>	Moves the field selected in **Selection** to **Display**. The field is inserted at the end of the list.
∑	Moves the field selected in **Selection** to **Display**. The field is inserted above the field which is marked in **Display**.
<	Moves the field selected in **Display** to **Selection**.
<<	Moves all fields in **Display** to **Selection**.

763 PVC THERMOMAT RESULTS / Format / Adjust width

The **Column width** window opens in which the width of the columns selected for the determination overview can be fixed.

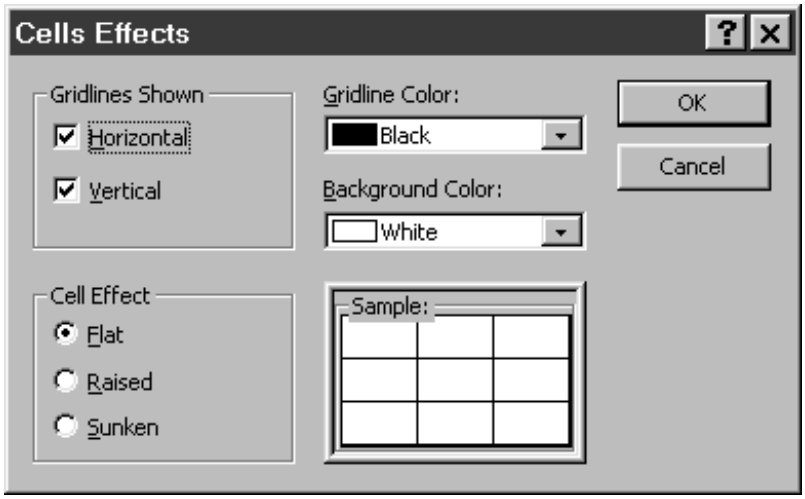


Column Width	Column width in characters.
Standard width	Resets the column width to the default value (18.5 characters).
<Best fit>	Adapts the column width to the longest field content (including title).

Alternatively the column width can be set directly by using the mouse. This is carried out by moving the cursor in the title line of the table between two columns until the cursor appears as ⇹. With the **left-hand mouse key** the column can then be drawn to the required size. A **double-click** on the selected column automatically adapts the column width to the longest field content (including title).

763 PVC THERMOMAT RESULTS / Format / Fields...

The **Cells effects** window opens in which the format of cells shown in the determination overview can be fixed.



Gridlines shown

Horizontal	Shows horizontal grid lines.
Vertical	Shows vertical grid lines.

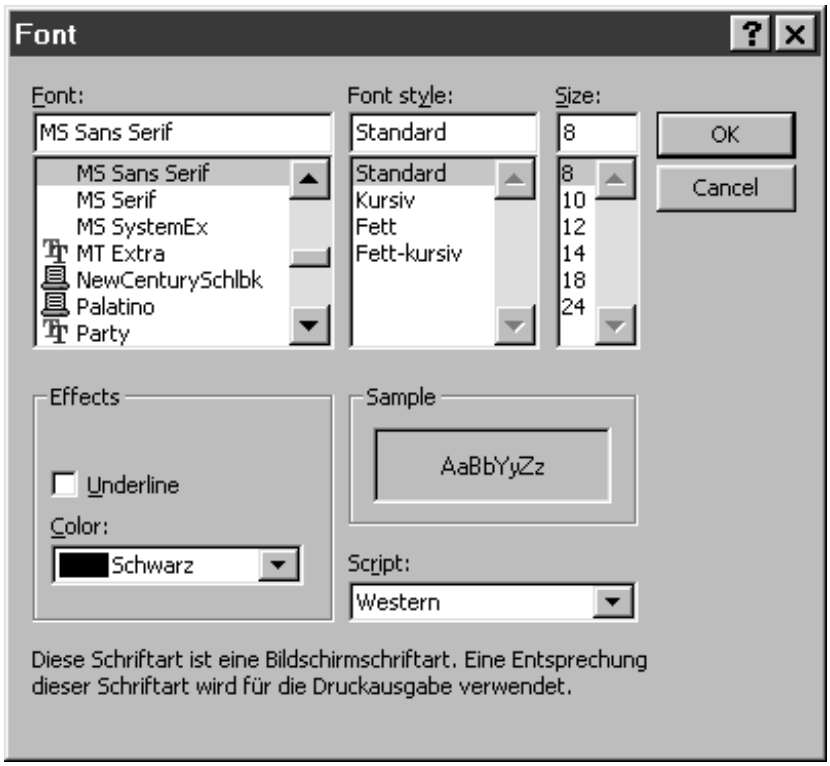
Cell effect

Flat	Shows flat cells.
Raised	Shows raised cells.
Sunken	Shows depressed cells.

Gridline color	Color of horizontal and vertical grid lines.
Background color	Color of cell background.
Sample	Display of the selected format for the cells.

763 PVC THERMOMAT RESULTS / Format / Fonts...

The **Font** window opens in which the font used for the determination overview can be fixed.



Font	Selects the font from those available in the PC.
Font style	Selects the typestyle for the selected font.
Size	Selects the type size for the selected font in points.
Effects	
Underline	Selects that the characters are to be underlined.
Color	Selects the color for the selected font.
Sample	Display of the selected font.

Mark determinations

763 PVC THERMOMAT RESULTS / Edit / Select record

Marks the record (line) in the determination overview in which the cursor is located.

Alternatively the record can be marked by using the **mouse**; this is done by clicking on the gray field to the left of the required line. If the left-hand mouse key is kept pressed down then several lines in sequence can be marked.

763 PVC THERMOMAT RESULTS / Edit / Select all records

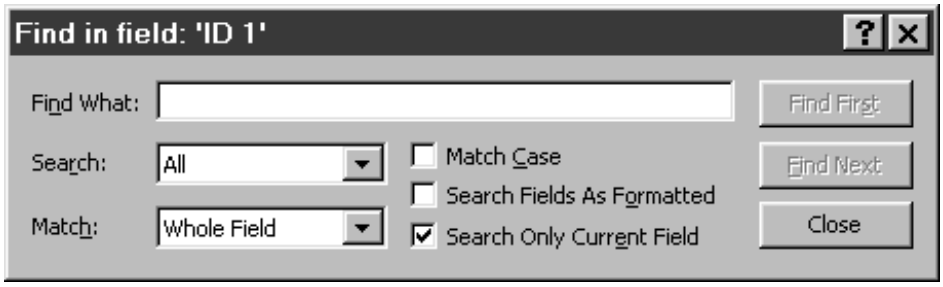
Marks all records (lines) in the determination overview.

This function can be triggered by clicking on the first empty field in the title line of the determination overview.

Find determination

763 PVC THERMOMAT RESULTS / Determination / Find

The **Find** window opens in which the search template for the search in the determination overview can be entered.



Find What	**Search template**. The following wildcards are also allowed: ***** any character sequence (e.g. ***ab**, ***ab***, **ab***) **?** individual character (e.g. **a?a**, **??a**, **ab?**) **#** individual number (e.g. **##20**, **ab###**, **#23##**)
Search	**Search mode**.
All	Searches through the whole table.
Down	Searches downwards to the end of the table.
Up	Searches upwards to the start of the table.
Match	**Search area**.
Whole field	The search is only successful when the whole field agrees with the search template.
Start of field	The search is successful when the start of the field contents agrees with the search template.
Any part of field	The search is successful when part of the field contents agrees with the search template.

Match Case	If this option is switched on then the search will only be successful when the upper and lower case characters of the search template and field contents coincide.
Search Fields As Formatted	If this option is switched on then, in searches for the whole field content in fields with units (e.g. °C, h), the unit will also be taken into account. If only the numerical value is being looked for then this option must be switched off (standard setting).
Search Only Current Field	If this option is switched on then the search will be limited to the selected field. The field name is shown in the window title. This option is not available if one or more complete lines are marked.
<Find First>	Begins the search at the start of the determination overview.
<Find Next>	Continues the search from the location of the cursor in the determination overview.

Delete determinations

763 PVC THERMOMAT RESULTS / Determination / Delete

Deletes the marked determinations from the database.

*This menu item is reserved for the **Administrator**.*

Sort and filter determinations

763 PVC THERMOMAT RESULTS / Determination / Sort / Ascending

Sorts the records in the determination overview according to the field contents in the selected column (marked column or column in which the cursor is located) in increasing sequence. If several columns are marked then the records will be sorted beginning with the first marked column from the left. The sorting conditions are stored under **Special filter/sorting**.

763 PVC THERMOMAT RESULTS / Determination / Sort / Descending

Sorts the records in the determination overview according to the field contents in the selected column (marked column or column in which the cursor is located) in decreasing sequence. If several columns are marked then the records will be sorted beginning with the first marked column from the left. The sorting conditions are stored under **Special filter/sorting**.

The ascending or descending sort can be removed again by clicking on  or **Determination / Filter / Remove filter/sorting**. If the sort is not removed then it is retained until a new sorting process is carried out.

If sorting has been switched off it can be used again by clicking on  or **Determination / Filter / Apply filter/sorting**.

For complex sorting processes with several columns a **Special sorting** must be used.

763 PVC THERMOMAT RESULTS / Determination / Filter / Selection based filter

The selection-based filter is a method for filtering records rapidly and conveniently. This is done by first marking a part of an entry or the complete entry in a field in the determination overview. After activating the selection-based filter the table is filtered so that only records which contain the marked entry are shown. The filtering conditions are stored under **Special filter/sorting**.

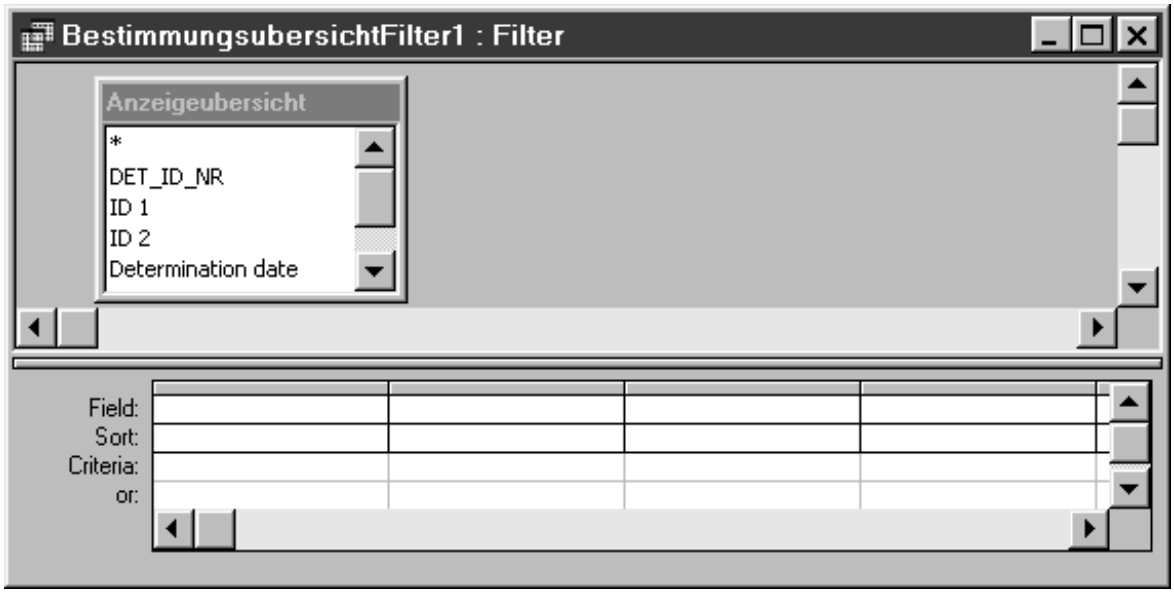
The filter can be removed again by clicking on  or **Determination / Filter / Remove filter/sorting**. If the filter is not removed then it is retained until a new filtering process is carried out.

If filtering has been switched off it can be used again by clicking on  or **Determination / Filter / Apply filter/sorting**.

For complex filters a **Special filter** must be used.

763 PVC THERMOMAT RESULTS / Determination / Filter / Special filter/sorting

The **Special filter/sorting** window opens in which the complex conditions for filtering and sorting records can be defined.



The lower, important part of the window contains a table for entry of conditions with the following lines:

Field	Selects a field (column) in the determination overview.
Sort	Sorts the selected field: **Ascending**, **Descending** or **(not sorted)**.
Criteria	Criteria for filtering records.
or	Further criteria for filtering records.

In the lines **Criteria** and **or** and in all following lines the number of records to be searched for can be limited by the use of expressions. An **expression** is a combination of operators, functions and field names for laying down a condition for filtering records. Only the most important elements and rules for entering expressions and a few selected examples are given below. For more information please refer to the documentation about Microsoft Access.

Operators, functions, field names	
Mathematical operators	**+**, **-**, **=**, *****, **/**
Comparison operators	**<**, **>**, **<>**, **<=**, **>=**, **Between ... And ...**
Logical operators	**And**, **Or**, **Not**
Wildcards	*****, **?**, **#** (see **Find**)
Current date	**Date()**
Current time	**Now()** (date and time)
Other fields	**[**"field name"**]**

Linkages with different fields

If conditions are fixed in different fields then these conditions can also be linked with **And** and **Or**. The following conditions apply:

Criteria in the **same line** are **And-conditions**, i.e. only those records are shown which fulfill all conditions.

Criteria in **different lines** are **Or-conditions**, i.e. all records which fulfill one of the conditions will be shown.

Examples

>10	Values larger than 10.
PVC	All data with "PVC" as part of their name.
##.##.1998	All data for 1998.
Between Date() And (Date() - 7)	all data of previous week.

Newly entered conditions are only saved when they are used with the **Special filter/sorting** window open by clicking on  or **Determination / Filter / Apply filter/sorting**.

By clicking on  or **Edit / Delete filter** all the conditions entered in the **Special filter/sorting** window are deleted again.

By clicking on  or **Edit / Undo** the last alterations made in the **Special filter/sorting** window are canceled.

The filter/sort can be removed again by clicking on  or **Determination / Filter / Remove filter/sorting**. If the filter/sort is not removed then it is retained until a new filtering/sorting process is carried out.

If filtering has been switched off it can be used again by clicking on  or **Determination / Filter / Apply filter/sorting**.

763 PVC THERMOMAT RESULTS / Determination / Filter / Apply filter/sorting

The conditions for filtering and sorting records stored under **Special filter/sorting** are applied to the determination overview.

The filter/sorting can be removed again by clicking on  or **Determination / Filter / Remove filter/sorting**. If the filter/sorting is not removed then it is retained until a new filtering/sorting process is carried out.

763 PVC THERMOMAT RESULTS / Determination / Filter / Remove filter/sorting

The filter/sorting applied to the determination overview is removed and all records are shown again.

If filter/sorting is switched off it can be switched on again by clicking on  or **Determination / Filter / Apply filter/sorting**.

763 PVC THERMOMAT RESULTS / Edit / Undo

The last alteration made in the **Special filter/sorting** window is canceled.

763 PVC THERMOMAT RESULTS / Edit / Delete filter

All the conditions entered in the **Special filter/sorting** window are deleted again.

763 PVC THERMOMAT RESULTS / Determination / Filter / Filter selection

The lines marked in the determination overview are incorporated in the filter, i.e. only those lines which have previously been marked will be shown.

763 PVC THERMOMAT RESULTS / Determination / Filter / Selection not in filter

The lines marked in the determination overview are removed from the filter, i.e. only those lines which were not previously marked will be shown.

4.7.2 Determination and method data

Open view

763 PVC THERMOMAT RESULTS / View / Determination and method data

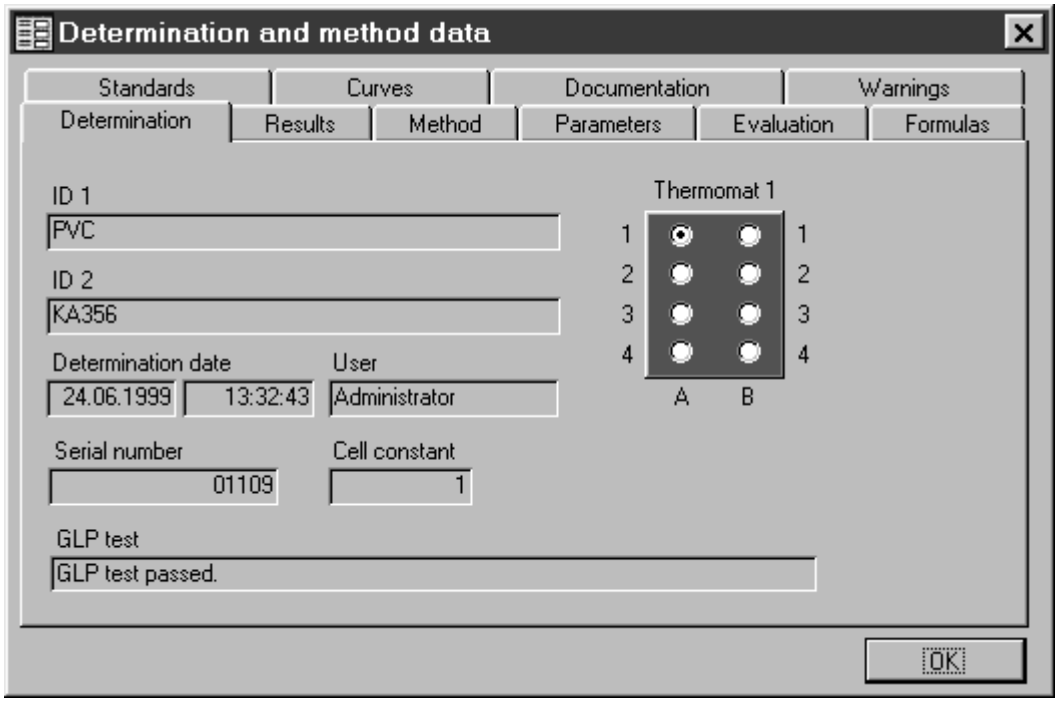
The **Determination and method data** window is opened which contains all parameters and results for the selected determination on the following tabs:

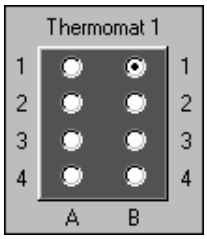
Determination	Information about the determination.
Results	Overview of results of determination.
Method	Information about the method.
Parameters	Measuring parameters.
Evaluation	Parameters for curve evaluation.
Formulas	Formula input for further calculation of induction and stability times.
Standards	Parameters for calculating the standard time.
Curves	Parameters for scaling the axes.
Documentation	Parameters for report output.
Warnings	Warnings about the determination.

If the determination in the determination overview is changed by marking a different determination then the parameters in the **Determination and method** window will be updated automatically.

Determination

Determination tab of the **Determination and method data** window with information about the determination.

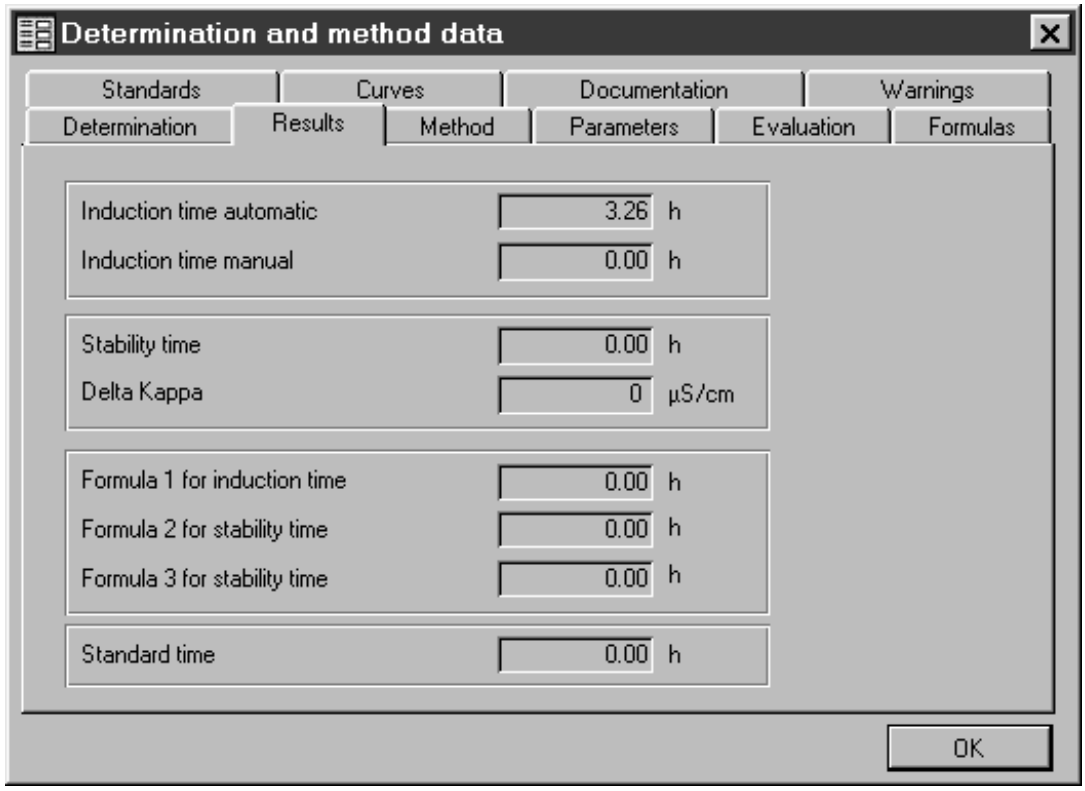


ID 1	Sample identifier 1.
ID 2	Sample identifier 2.
Determination date	Date and time at which the determination was started.
User	Name of the user logged in at the start of the determination.
Serial number	Serial number of the instrument used for the determination.
Cell constant	Cell constant of the conductivity cell used for the determination.
GLP test	GLP status at the time the determination was carried out. The following results are possible:
GLP test not switched on	GLP monitoring was not switched on for either temperature or conductivity or gas flow.
Due GLP test	The next GLP test was due.
GLP test not passed	The last GLP test carried out was failed.
GLP test passed	The last GLP test carried out was passed.
	Display of instrument, block and channel used for the determination

Results

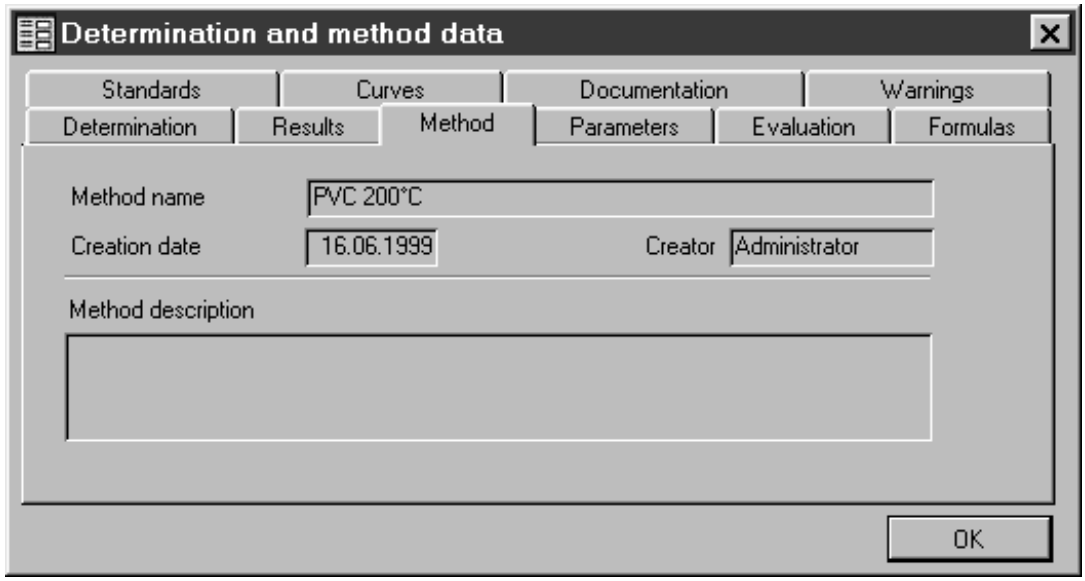
Results tab of the **Determination and method data** window with results overview for the determination.

Induction time automatic	Automatically determined **Induction time**.
Induction time manual	**Induction time** determined manually by re-evaluation.
Stability time	Determined **Stability time** for **Delta Kappa**.
Delta Kappa	Alteration in conductivity for the determination of the **Stability time**.
Formula 1 for induction time	Result for **Formula 1**.
Formula 2 for induction time	Result for **Formula 2**.
Formula 3 for induction time	Result for **Formula 3**.
Standard time	Result for **standard time**.



Method

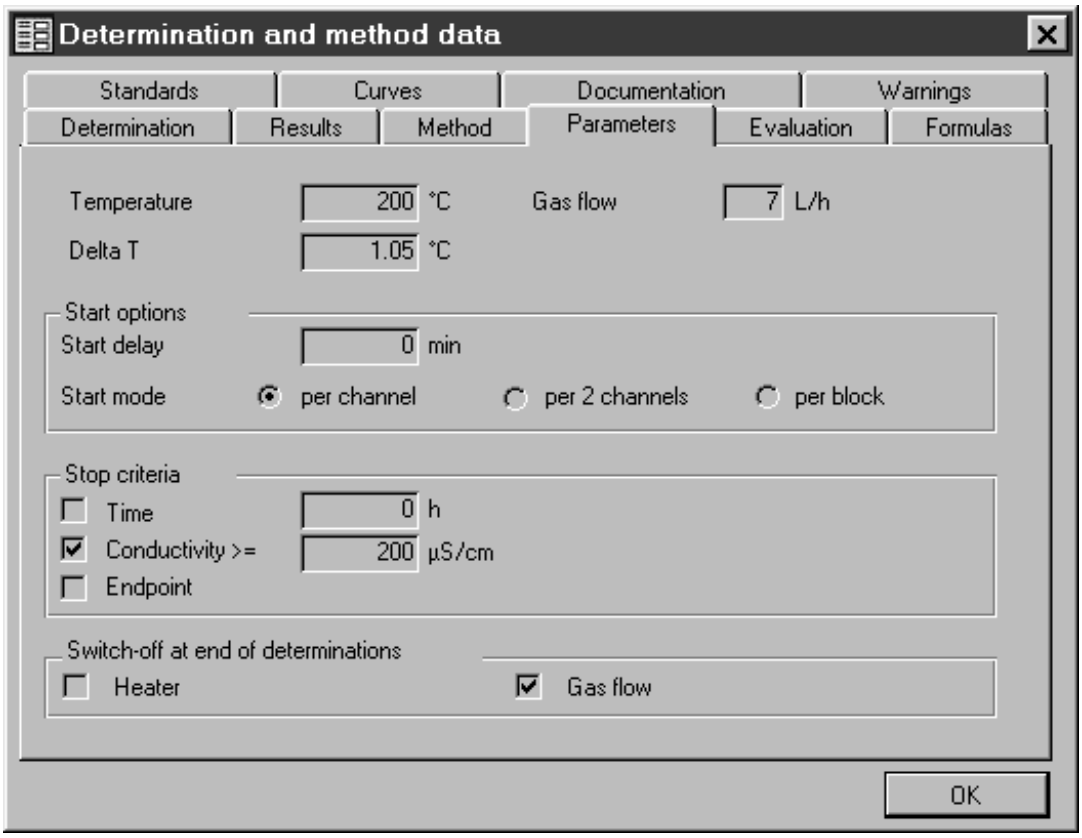
Method tab of the **Determination and method data** window with information about the method used for the determination.



Method name	Name of the method.
Creation date	Date on which the method was last saved.
Creator	Name of the logged-in used the last time that the method was saved.
Method description	**Description** of the method (see *section 4.5.2*).

Parameters

Parameters tab of the **Determination and method data** window with the display of the measuring parameters used in the determination.



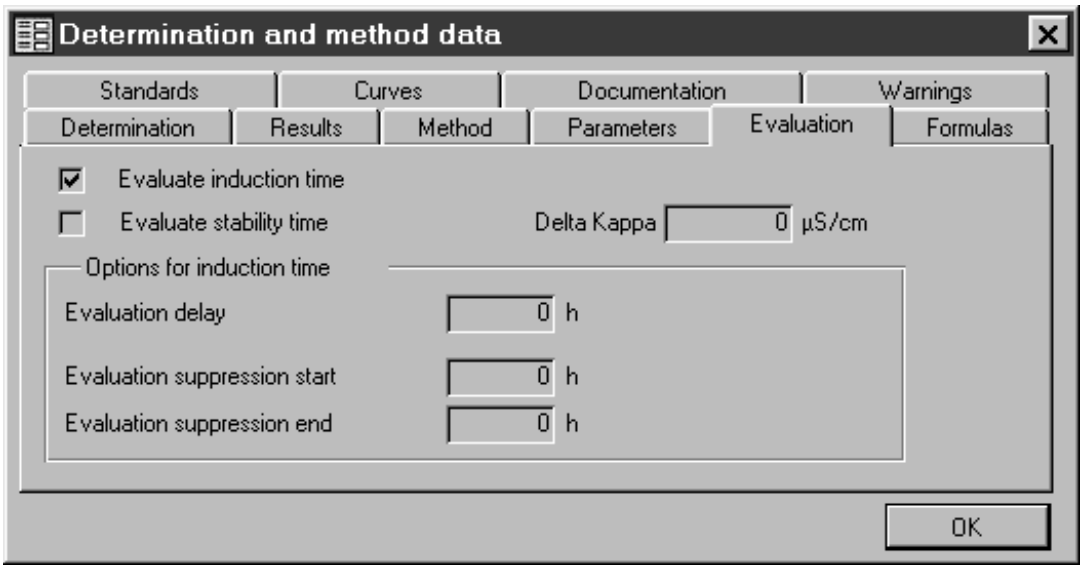
Temperature	**Set temperature** of sample.
Delta T	**Temperature correction**: Correction value for increasing the block temperature in order to reach the set temperature in the sample.
Gas flow	**Gas flow** through sample.
Start options	
Start delay	Delay time until start of measurement.
Start mode	Mode for starting the channels:
per channel	All channels have been started individually.
per 2 channels	Each 2 channels have been started together.
per block	4 channels in one block have been started together.
Stop criteria	
Time	Time until automatic termination of determination.
Conductivity	Conductivity limit. When this value is reached the determination is automatically terminated.
Endpoint(s)	Automatic termination of the determination when each active channel has reached all endpoints.

Shut off at end of determinations

Heater	Heating was switched off automatically at end of determination.
Gas flow	Gas flow was switched off automatically at end of determination.

Evaluation

Evaluation tab of the **Determination and method data** window with the display of the parameters used for curve evaluation.



Evaluate induction time	Determination of **induction time**.
Evaluate stability time	Determination of **stability time**.
Delta Kappa	Alteration in conductivity for the determination of the **stability time**.

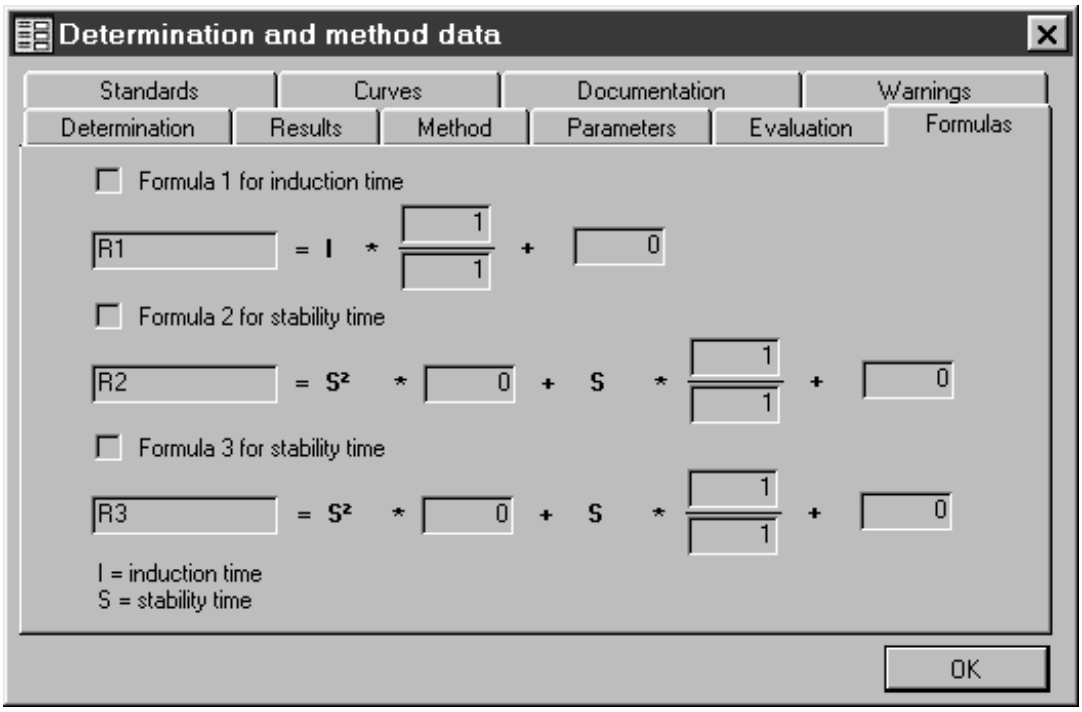
Evaluation delay (Option for induction time)
Delay time before the start of curve evaluation.

Evaluation suppression (Option for induction time)

Start	Time from start of determination until start of evaluation suppression.
End	Time from start of determination until end of evaluation suppression.

Formulas

Formulas tab of the **Determination and method data** window with the display of the formulas used for the calculation.



Formula 1 for induction time

The automatically determined **Induction time** was calculated from the values entered in the equation by the operator and then stored as the **Formula 1 result** under the name entered in the first field.

Formula 2 for induction time

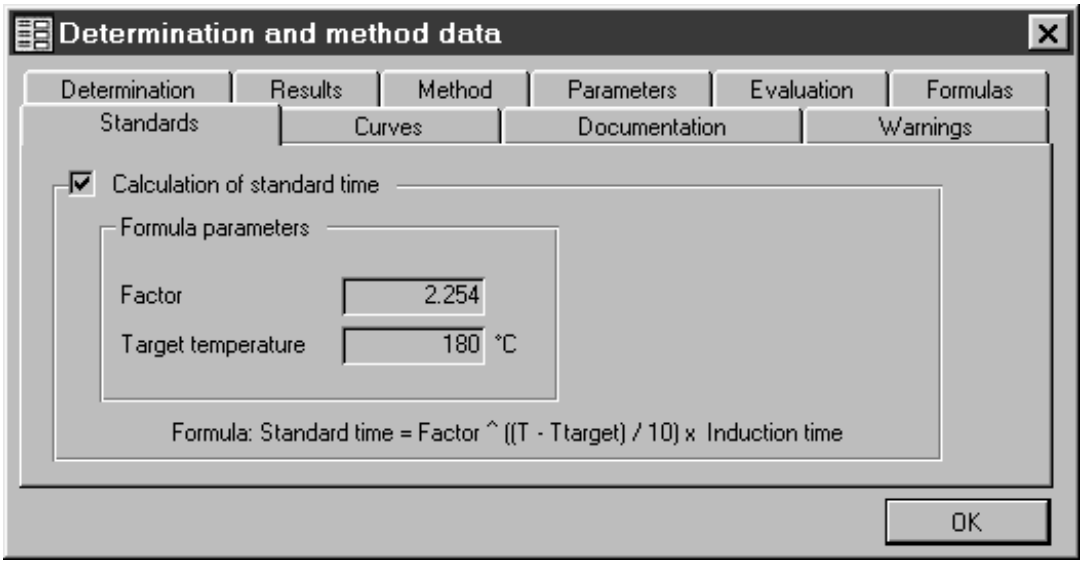
The automatically determined **Stability time** was calculated from the values entered in the equation by the operator and then stored as the **Formula 2 result** under the name entered in the first field.

Formula 3 for induction time

The automatically determined **Stability time** was calculated from the values entered in the equation by the operator and then stored as the **Formula 3 result** under the name entered in the first field.

Standards

Standards tab of the **Determination and method data** window with the parameters used for the calculation of the **standard time**.



Calculation of standard time

Shows whether the standard time was calculated or not.

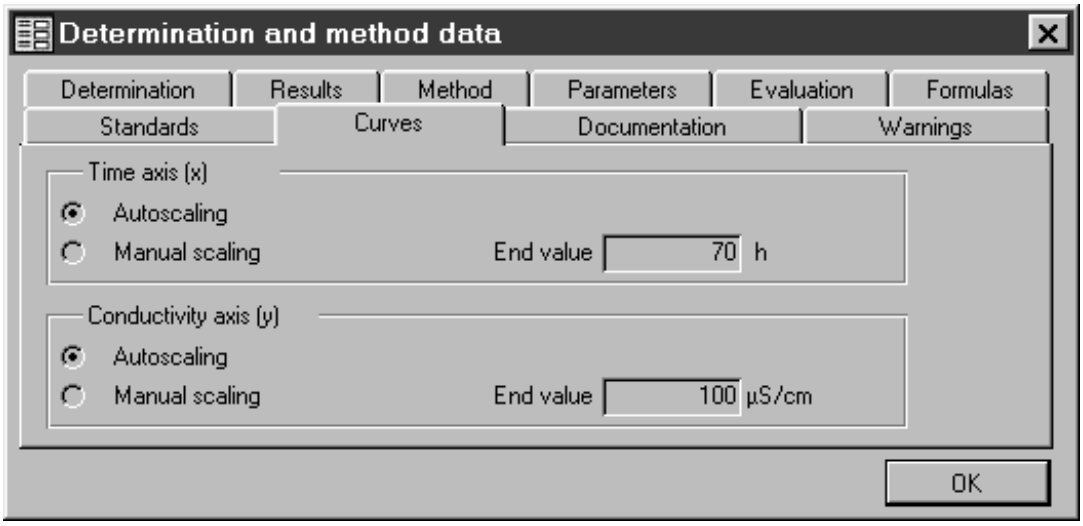
Formula parameters

Factor Parameter for calculating the standard time which gives the factor by which the time changes per 10 °C.

Target temperature Target temperature for calculating the standard time.

Curves

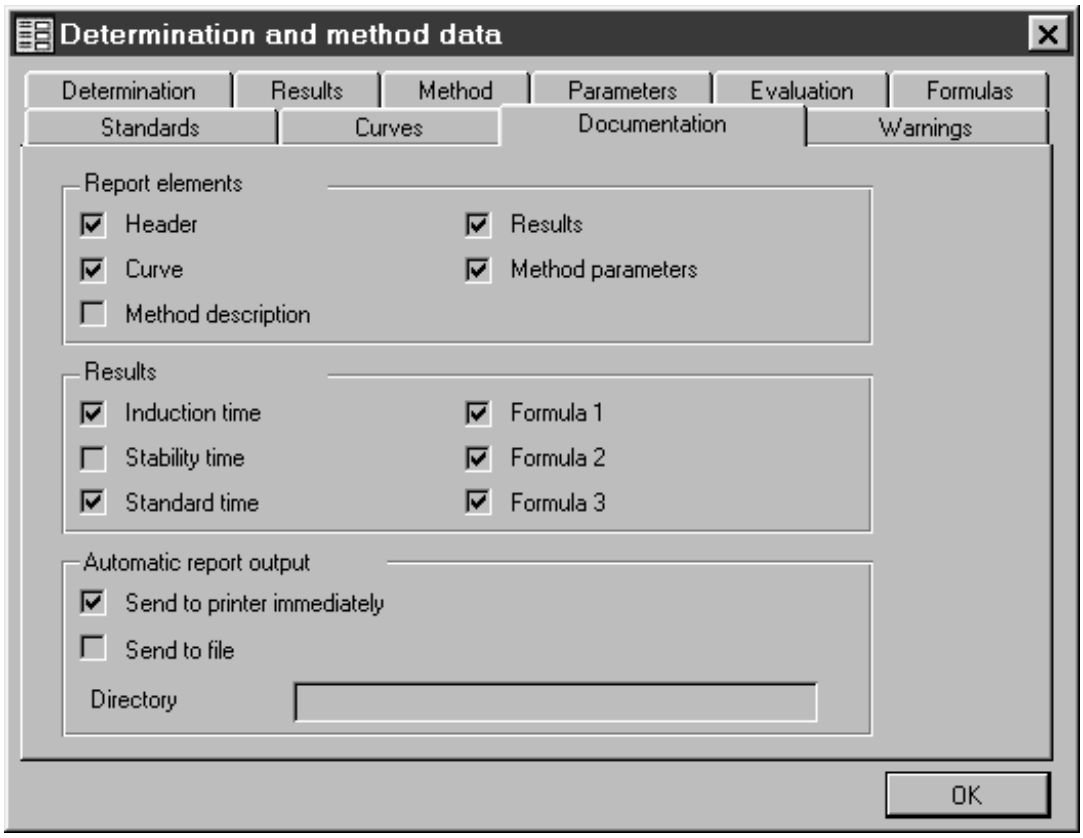
Curves tab of the **Determination and method data** window with the display of the parameters used for scaling the axes of the curve.



Time axis (x)	
Autoscaling	Automatic fixing of the initial and final values of the time axis.
Manual scaling	Manual fixing of the final value of the time axis.
End value	Final value of the time axis.
Conductivity axis (y)	
Autoscaling	Automatic fixing of the initial and final values of the conductivity axis.
Manual scaling	Manual fixing of the final value of the conductivity axis.
End value	Final value of the conductivity axis.

Documentation

Documentation tab of the **Determination and method data** window with display of the settings used for the report output.



Include in report	
Header	Output of the header.
Curve	Output of curves.
Method description	Output of the method description entered on the **Description** tab.
Results	Output of the selected results: **Induction time**, **Stability time**, **Standard time**, **Formula 1...3**.
Method parameters	Output of method parameters.

Automatic report output

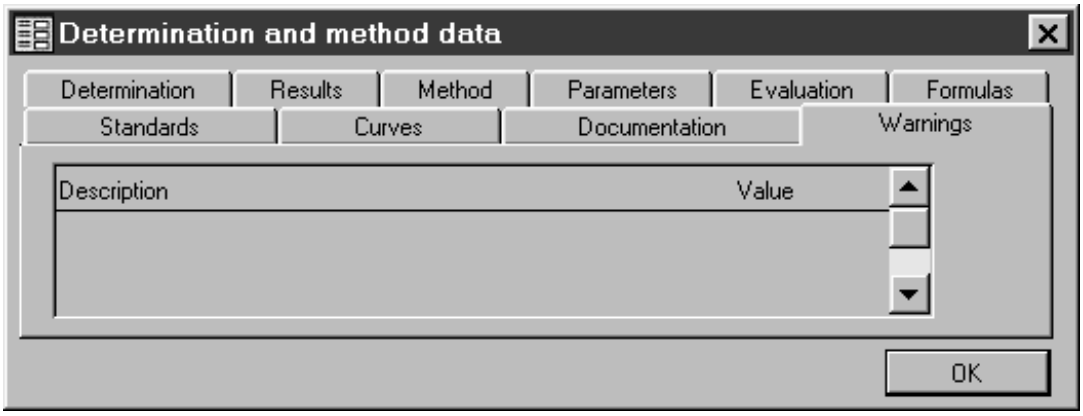
Send to printer immediately
Automatic printout of the reports defined under **Include in report** following the end of the determination.

Send to file
Automatic output of determination and method data following the end of the determination as a text file ***.txt**.

Directory
Directory in which the report will be stored.

Warnings

Warnings tab of the **Determination and method data** window with the display of warnings about the determination.



Description
If the determination was not carried out properly then the following warnings are possible:

Temperature not reached
Set temperature was not reached at start of determination.

Missing measuring points (start time)
Measuring points are missing from the given time (in s) onwards.

Missing measuring points (end time)
Measuring points are again present from the given time (in s) onwards.

Manual stop
The determination was stopped manually.

Abortion after communication error
Automatic determination stop because connection between the 763 PVC Thermomat and PC was interrupted.

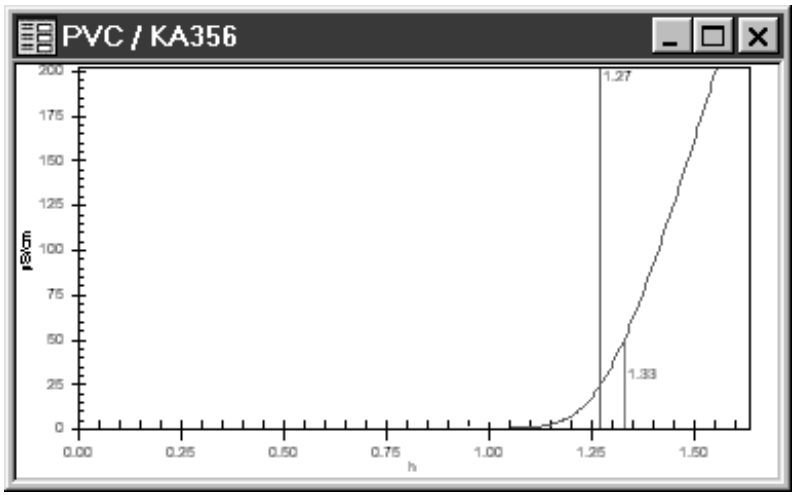
Value
Value of the warning (e.g. temperature, time).

4.7.3 Graphics and reevaluation

Single graph

763 PVC THERMOMAT RESULTS / Determination / Graphics / Single graph

Opens a new window in which the **single graph** for the determination selected in the determination overview is shown. The sample identifiers **ID 1** and **ID 2** are shown in the window title.



If present then the determined **induction time** (red vertical line and numbers) and **stability time** (black vertical line and numbers) will be shown in the graphics window as well as the **measuring curve** (green). A maximum of 16 single graph windows can be displayed.

By clicking the graph using the right mouse key the context-sensitive menu with the following menu items is opened:

Zoom off	Switch off zooming.
Copy	Copy graph to clipboard.
Autoscaling	Switch on/off autoscaling.

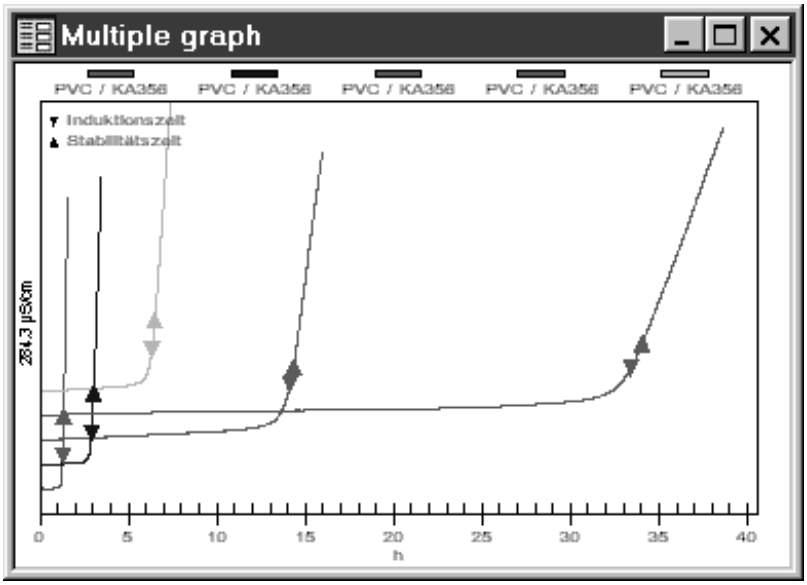
In contrast to the **Multiple graph** *and* **Live graph** *windows, the window with the single graph is* ***not*** *automatically updated when the determination in the determination overview is changed.*

Multiple graph

763 PVC THERMOMAT RESULTS / Determination / Graphics / Multiple graph

Opens a new **Multiple graph** window in which **all curves** for the determinations marked in the determination overview are shown.

The individual measuring curves are shown in color, the legends referring to the colors (sample identifiers **ID 1** and **ID 2**) are located in the graph header. If present the determined **induction time** (▼) and **stability time** (▲) will also be shown.



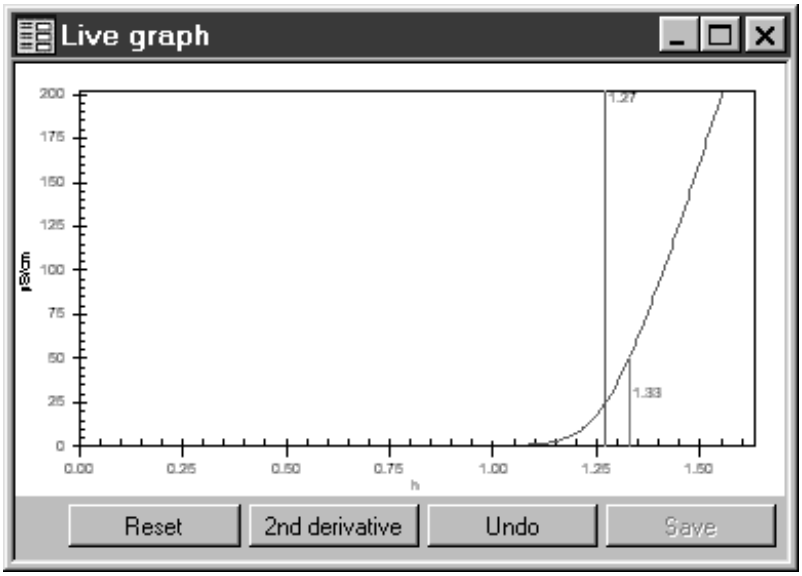
By clicking the graph using the right mouse key the context-sensitive menu with the following menu items is opened:

Offset	Switch on/off curve offset.
Zoom off	Switch off zooming.
Copy	Copy graph to clipboard.

Live graph

763 PVC THERMOMAT RESULTS / Determination / Graphics / Live graph with reevaluation

Opens a new **Live graph** window in which the **measuring curve** of the marked determination is shown. If a different determination is marked in the determination overview then the curve is automatically updated.



If present then the determined **induction time** (red vertical line and numbers) and **stability time** (black vertical line and numbers) will be shown in the graphics window as well as the **measuring curve**

(green). With **<2nd derivative>** the second derivative of the measuring curve can be shown as a blue line.

By clicking the graph using the right mouse key the context-sensitive menu with the following menu items is opened:

Reevaluation	Open reevaluation window (see below).
Zoom off	Switch off zooming.
Copy	Copy graph to clipboard.
Autoscaling	Switch on/off autoscaling.

Switch zoom on/off

Switch on zoom: move the cursor to the left-hand upper corner of the area to be enlarged, press the left-hand mouse key and move the cursor to the lower right-hand corner of the required zoom area. When the left-hand mouse key is released the selected area will be enlarged to fill the whole window.

Switch off zoom: click on the graph window with the right-hand mouse key and select the menu item **Zoom off** in the context-sensitive menu.

Switch autoscaling on/off

With the **Autoscaling** option in the context-sensitive menu of the graph window the manual scaling of the axes made under **Curves** can be canceled. If this option is switched on then the initial and final values of the axes will be fixed automatically so that all measuring points are visible.

Switch offset on/off

The **Offset** option in the context-sensitive menu of the **Multiple graph** window can be used to switch the offset for the displayed measuring curves on and off. If offset is switched on the curves will be shown offset from each other. In this case the difference of the whole axis is shown in µS/cm instead of the normal conductivity axis marking.

Copy graph to clipboard

The **Copy** option in the context-sensitive menu of the graph window can be used to copy the displayed graph into the clipboard.

Print graph

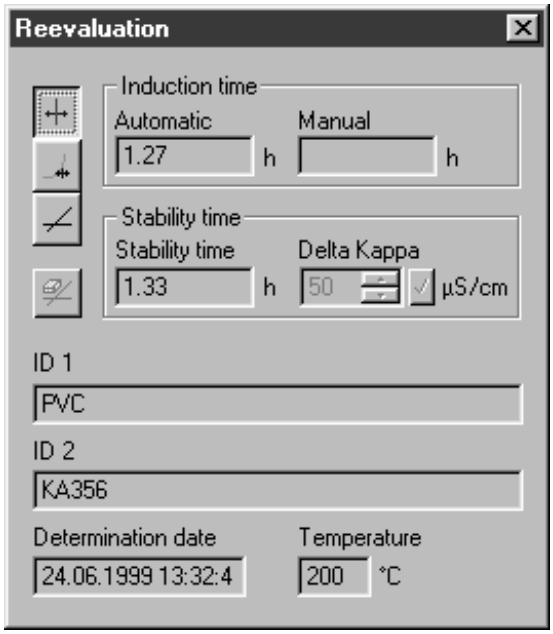
763 PVC THERMOMAT RESULTS / File / Print

If one of the **Single graph**, **Multiple graph** or **Live graph** windows is open and selected (active) then this menu item can be used to print out the graph shown in the graph window directly.

For printing several single or multiple graphs at the same time, see *section 4.7.6*.

Reevaluation

The **Reevaluation** option in the context-sensitive menu of the **Live graph** window opens the **Reevaluation** window for the manual reevaluation of measuring curves.



Induction time

Automatic	Automatically determined **Induction time**.
Manual	Induction time determined manually in the reevaluation.

Stability time

Stability time	Determined **Stability time** for **Delta Kappa**.
Delta Kappa	Alteration in conductivity for the determination of the **Stability time**.

ID 1	Sample identifier 1.
ID 2	Sample identifier 2.
Determination date	Date and time at start of determination.
Temperature	Sample temperature in the determination.

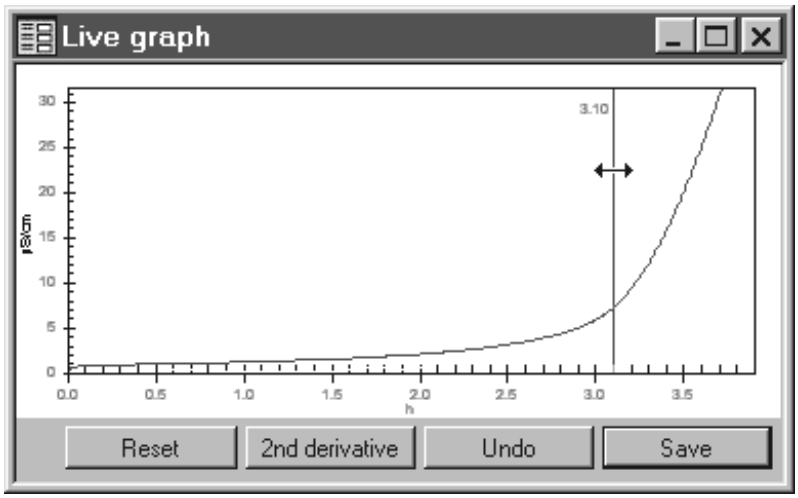
The alterations in the results achieved by means of the reevaluation can be stored, canceled or reset with the following buttons:

<Save>	Saves the results altered in the reevaluation.
<Undo>	Cancels all the alterations made in the reevaluation since the last time the results were saved.
<Reset>	Deletes all the alterations produced with the reevaluation. The determination will then only contain the results which were automatically determined during data recording.

The following reevaluation possibilities are offered:

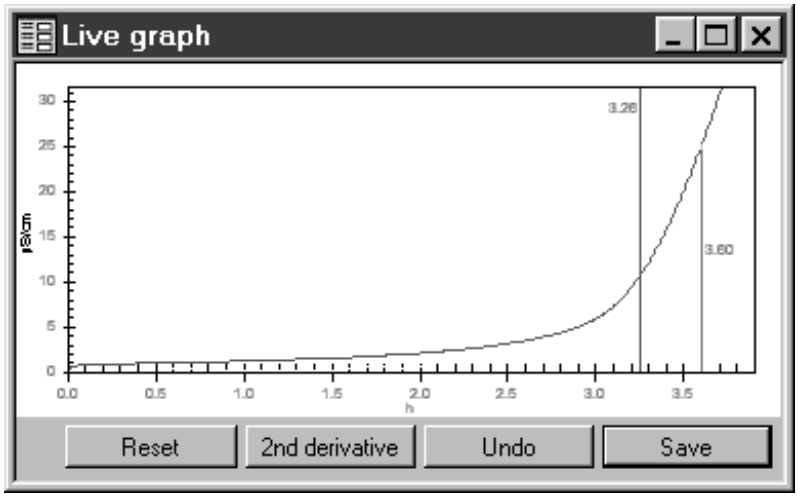
Set induction time manually

If this option is switched on then the **induction time** can be set manually. This is done by pressing the **[Ctrl] key** and at the same time moving the red vertical line in the **Live graph** window with **pressed-down left-hand mouse key** to the required location. The new manually set induction time is displayed in the **Manual** field and in the **Live graph** window.



Set stability time manually

If this option is switched on then the alteration in conductivity **Delta Kappa** for the calculation of the **stability time** can be altered manually. After alterations in the **Delta Kappa** field the newly calculated **Stability time** is shown both in the field with the same name and in the **Live graph** window.



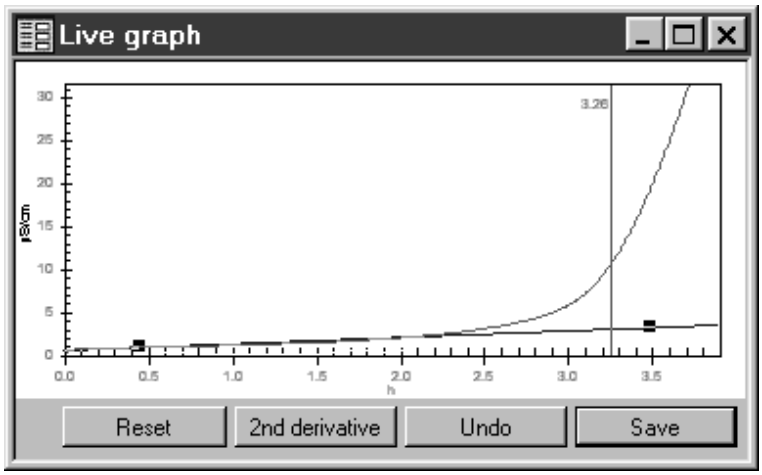
Set tangents manually

If this option is switched on then tangents can be set manually to determine the **induction time**. The induction time is then defined as being the intersection point of the tangents. The tangents are set as follows:

Set first tangent

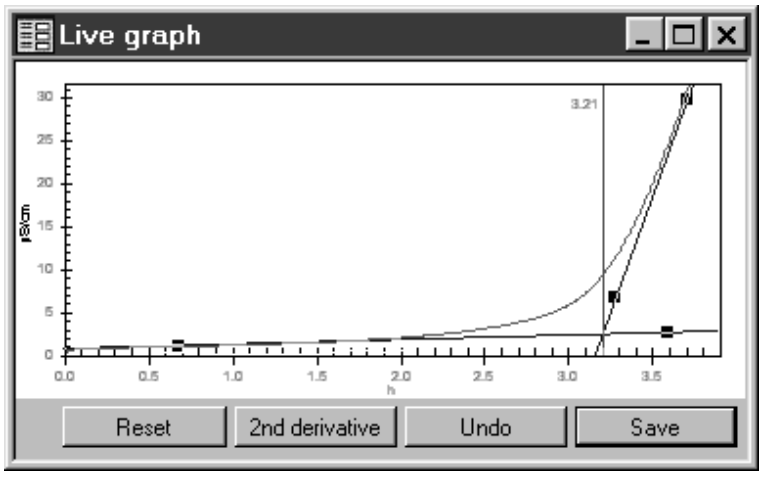
Press the **[Ctrl]** key and use the left-hand mouse key to

click on the required starting point of the first tangent on the measuring curve and then draw the tangent to the required location with the pressed-down left-hand mouse key.



Set second tangent

Press the **[Shift]** key and use the left-hand mouse key to click on the required starting point of the second tangent on the measuring curve and then draw the tangent to the required location with the pressed-down left-hand mouse key.



Alter tangents

Each of the two tangents has two points indicated by small black squares; these can be used as follows to alter the tangents after they have been created:

– Move the mouse close to such a point until the cursor appears as a crosswire.
– Press the **[Ctrl]** key and move tangent to the required location with the left-hand mouse key pressed down.

The new induction time determined with the tangents is displayed both in the **Manual** field and in the **Live graph** window.

Delete manually set tangents

Deletes the manually set tangents; the newly determined induction time is retained.

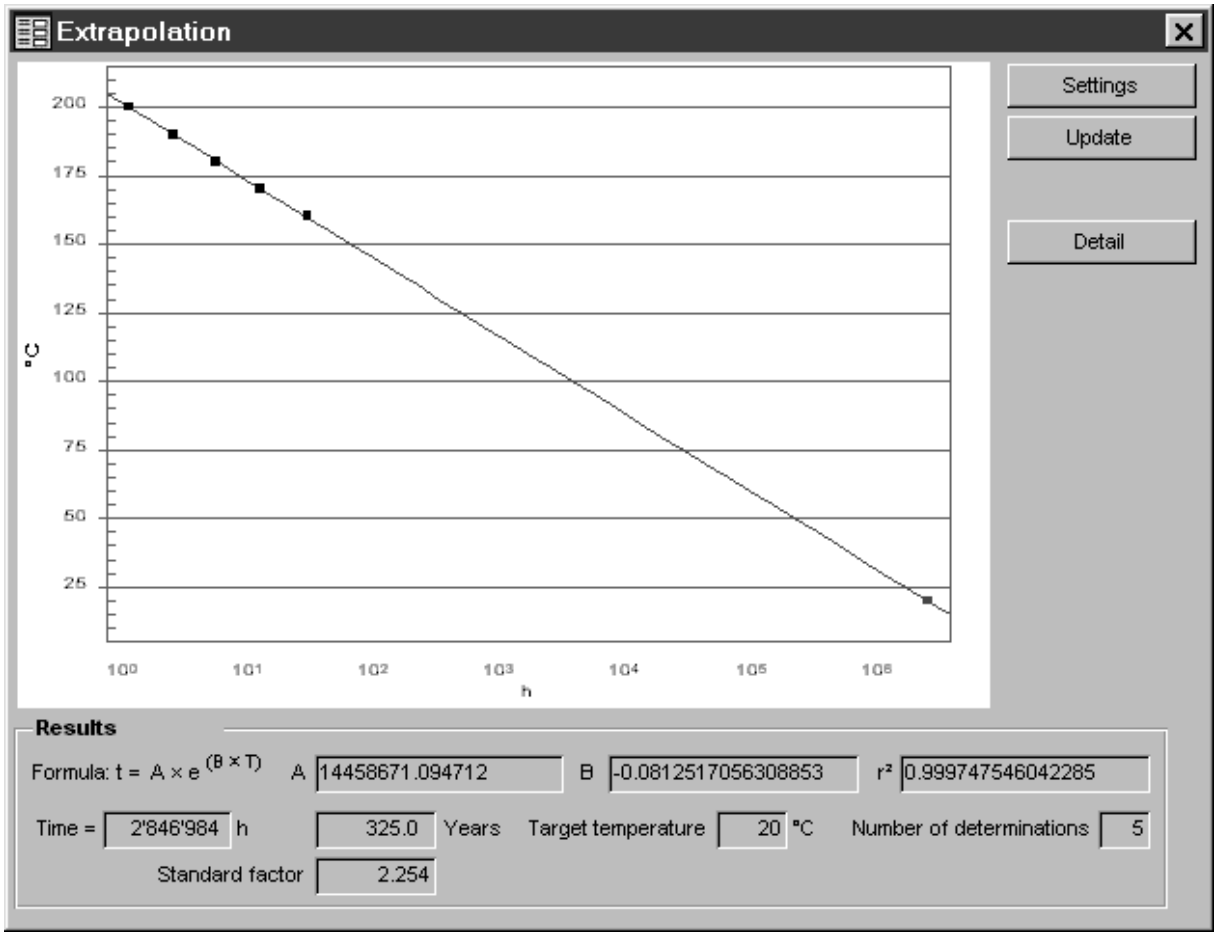
4.7.4 Extrapolation

Extrapolation window

763 PVC THERMOMAT RESULTS / Determination / Extrapolation

Opens the **Extrapolation** window in which the extrapolation values calculated from the results of the determinations marked in the determination overview are displayed numerically and graphically. The extrapolation is based on an exponential equation with the help of the result quantities defined under **<Settings>** and **Target temperature**. Extrapolation can be used to convert results measured at different temperatures to the required target temperature. In addition this method can be used to determine the **Factor** for converting the **induction time** into the **standard time**.

The time determined for a particular sample by extrapolation to low target temperatures (e.g. 25 °C) is a purely theoretical value. The relationship between this time and, for example, the working life of the sample must always be determined experimentally.



Extrapolation graph

In the **Temperature vs. Time** graph the line **T = ln(t)/B – ln(A)/B**, calculated with the help of a linear regression, is shown. The measured values of the selected determination are shown in **black**, the regression line in **green** and the time calculated for the **Target temperature** in **red**.

With the **Copy** option in the context-sensitive menu, which is opened by clicking on the graph with the right-hand mouse key, the displayed extrapolation graph can be copied into the clipboard.

Extrapolation results

The results of the extrapolation calculation are displayed beneath the graph:

Formula	Exponential equation for calculating the extrapolated time for the target temperature from the measuring values.
A	Calculated coefficient **A** of the exponential equation.
B	Calculated coefficient **B** of the exponential equation.
r^2	Calculated coefficient of correlation. If this value is below the **limit** (see below) set by the administrator then a warning appears when the extrapolation window is opened.
Time	Result of the extrapolation for the target temperature in h and years.
Target temperature	Target temperature for the extrapolation.
Number of determinations	Number of determinations used for the extrapolation.
Standard factor	Value determined from the regression line which gives the factor by which the time changes per 10 °C. This value can be entered in the equation for determination of the **Standard time** as a **Factor** (see *section 4.5.2*).
<Settings>	Opens the **Program settings** window with the **Extrapolation** tab for the entry of parameters for extrapolation (see below).
<Update>	Updates the display of graph and results after an alteration in the selection of the determinations in the determination overview.
<Detail>	Shows only the measured values in the graph.

Print extrapolation results

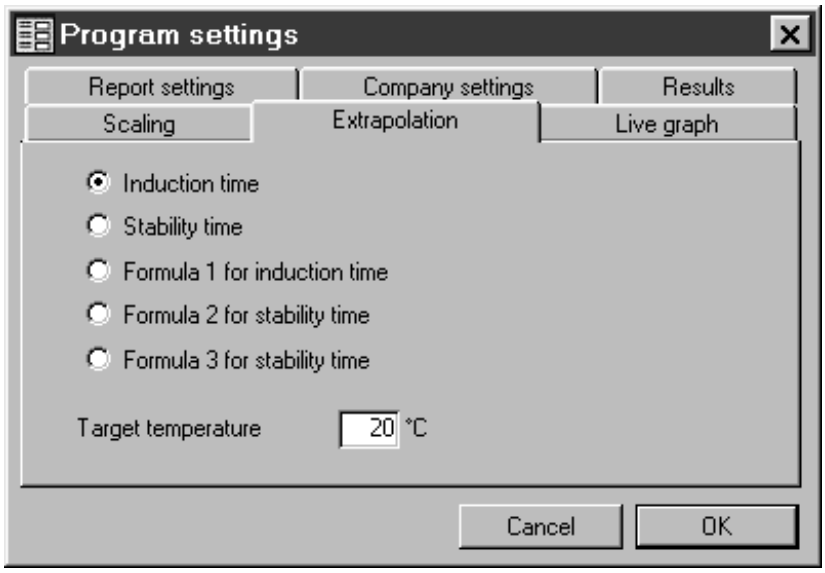
763 PVC THERMOMAT RESULTS / File / Print

If the **Extrapolation** window is open then this menu item prints out the extrapolation graphs (normal and detail), the extrapolation results and an overview of the determinations used for the extrapolation with the **ID 1**, **ID 2**, **Temperature** and **Results** fields.

Settings for extrapolation

763 PVC THERMOMAT RESULTS / Options / Settings / Extrapolation

Extrapolation tab of the **Program settings** window (see *section 4.7.7*) with settings for extrapolation.

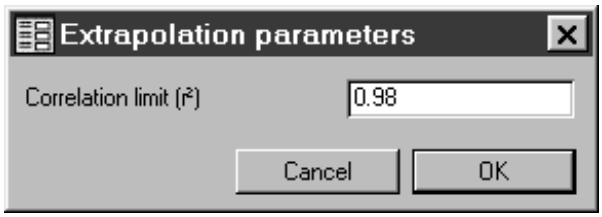


Induction time	Extrapolation with the results for the **induction time**.
Stability time	Extrapolation with the results for the **stability time**.
Formula 1..3 ...	Extrapolation with the **Formula results 1...3**.
Target temperature	Temperature to which the selected result is to be converted with the help of the regression line obtained from the extrapolation.

Parameters for extrapolation

763 PVC THERMOMAT RESULTS / Options / Administrator / Extrapolation parameters

The **Extrapolation parameters** window opens.



Correlation limit	Limit for the correlation coefficient **r^2** determined in the extrapolation. If this value is below the limit a warning will be given.

*This menu item is reserved for the **Administrator**.*

4.7.5 Recalculation

Recalculation window

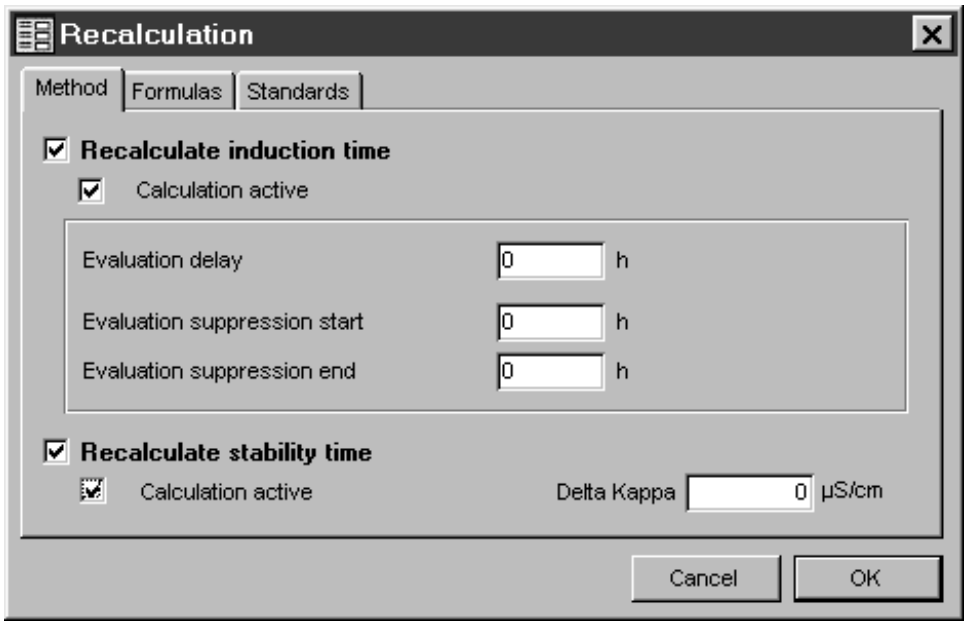
763 PVC THERMOMAT RESULTS / Determination / Recalculation

The **Recalculation** window opens in which the results of the determinations marked in the determination overview can be recalculated; it contains the following three tabs:

Method	Recalculation of the induction and stability times.
Formulas	Recalculation of formula 1...3.
Standards	Recalculation of standard time.

Method

Method tab of the **Recalculation** window for the recalculation of induction and stability times.



Recalculate induction time

Switches recalculation of the **induction time** on/off. If recalculation is switched off the original result is retained.

Calculation active If this option is **switched on** then the parameters for the calculation of the induction time can be newly entered.
If this option is **switched off** then no result will be produced (the original result is deleted).

Evaluation delay Delay time until start of the curve evaluation.

Evaluation suppression start

Time from start of determination until start of evaluation suppression.

Evaluation suppression end

Time from start of determination until end of evaluation suppression.

Recalculate stability time

Switches recalculation of the **stability time** on/off. If recalculation is switched off the original result is retained.

Calculation active

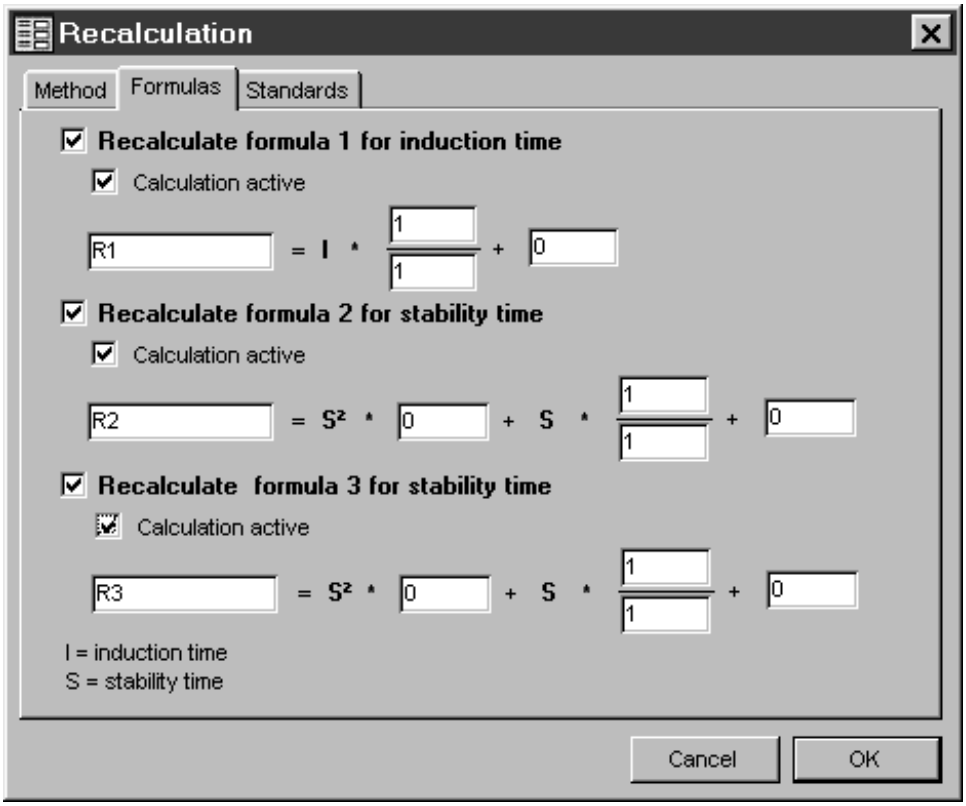
If this option is **switched on** then **Delta Kappa** can be newly entered to calculate the stability time.
If this option is **switched off** then no result will be produced (the original result is deleted).

Delta Kappa

Alteration in conductivity for the determination of the stability time in µS/cm.

Formulas

Formulas tab of the **Recalculation** window for the recalculation of formula results.



Recalculate formula 1

Switches recalculation of **Formula 1** for the **induction time** on/off. If recalculation is switched off the original result is retained.

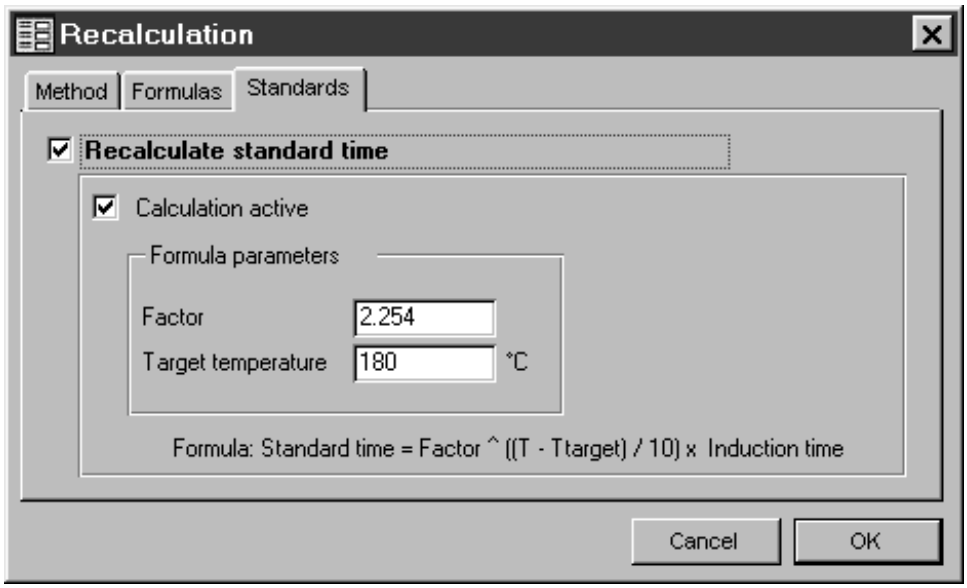
Calculation active

If this option is **switched on** then the parameters for formula 1 can be newly entered.
If this option is **switched off** then no result will be produced (the original result is deleted).

Recalculate formula 2	Switches recalculation of **Formula 2** for the **stability time** on/off. If recalculation is switched off the original result is retained.
Calculation active	If this option is **switched on** then the parameters for formula 2 can be newly entered. If this option is **switched off** then no result will be produced (the original result is deleted).
Recalculate formula 3	Switches recalculation of **Formula 3** for the **stability time** on/off. If recalculation is switched off the original result is retained.
Calculation active	If this option is **switched on** then the parameters for formula 3 can be newly entered. If this option is **switched off** then no result will be produced (the original result is deleted).

Standards

Standards tab of the **Recalculation** window for the recalculation of the standard time.



Recalculate standard time

Switches the recalculation of the **standard time** from the **induction time** on/off. If recalculation is switched off the original result is retained.

Calculation active

If this option is **switched on** then the parameters for calculating the standard time can be newly entered.
If this option is **switched off** then no result will be produced (the original result is deleted).

Formula parameters

Factor
Parameter for calculating the standard time which gives the factor by which the time changes per 10 °C. This value can be determined by the extrapolation of results obtained at different temperatures.

Target temperature
Target temperature for calculating the standard time.

4.7.6 Data export and printing

Copy to clipboard

763 PVC THERMOMAT RESULTS / Edit / Copy

Copies the marked lines or columns in the determination overview into the clipboard.

Export to other database

763 PVC THERMOMAT RESULTS / Determination / Export / Export to database

Exports the marked determinations from the opened database to a different database. The **Export data to...** window opens in which either an existing ***.mrd** database file can be selected or a new database can be created by entering a name in the **File name** field.

When a determination is exported all the current GLP tests (see *section 4.8.4*) which apply to the particular determination are also exported. The GLP results always remain stored in the original database.

Export to Microsoft Word

763 PVC THERMOMAT RESULTS / Determination / Export / Export to MS Word

Copies the lines or columns marked in the determination overview to **Microsoft Word**. The copied text is stored in an RTF file which is automatically opened by Microsoft Word.

Export to Microsoft Excel

763 PVC THERMOMAT RESULTS / Determination / Export / Export to MS Excel

Copies the lines or columns marked in the determination overview to **Microsoft Excel**. The copied text is stored in an XLS file which is automatically opened by Microsoft Excel.

Export of measured values

763 PVC THERMOMAT RESULTS / Determination / Export / List of measured values

Exports the measured values of the determinations marked in the determination overview. The **Save to export file** window opens for the selection of the folder and the entry of the **File name** for the export file. After clicking on **<Save>** the list of measurements for each of the marked determinations is stored in a ***.txt** text file. This text file contains the following values for each determination:

ID 1	Sample identifier 1.
ID 2	Sample identifier 2.
Determination date	Date of determination in format DD.MM.YYYY hh:mm:ss.
Output date	Output date in format DD.MM.YYYY hh:mm:ss.
Time [s]	Time for every measurement in s.
Measured value [mS/cm]	Measured conductivity in µS/cm.

Export of determination and method data

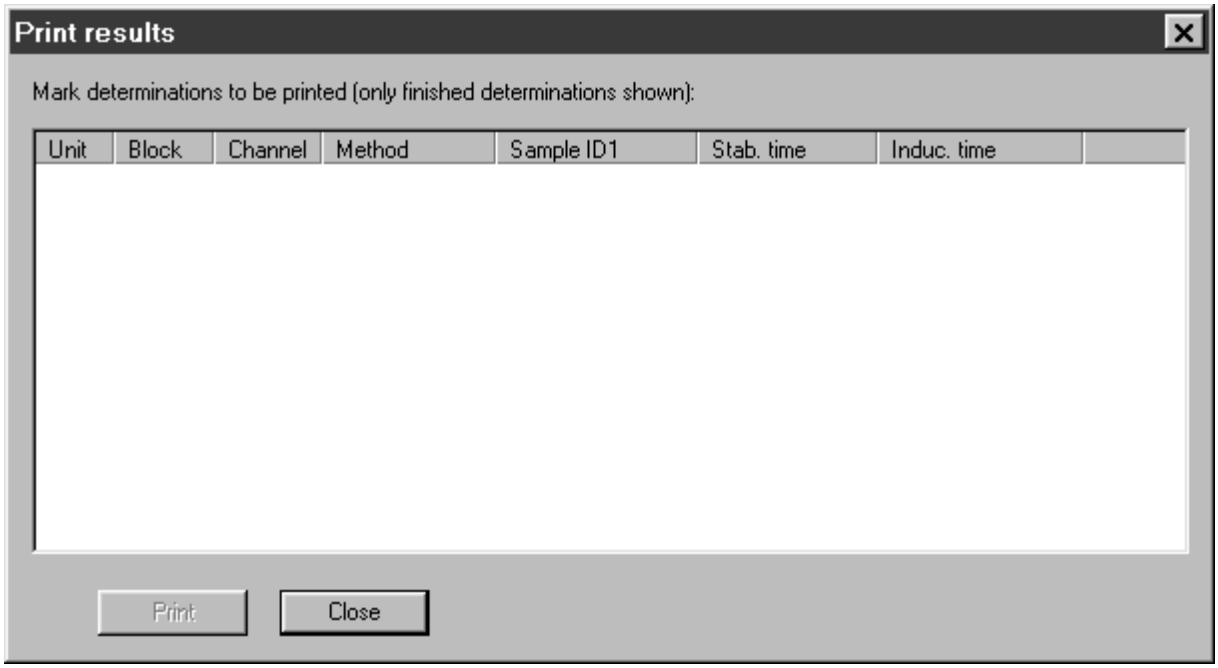
763 PVC THERMOMAT RESULTS / Determination / Export / Determination and method data

Exports all the determination and method data for the determinations marked in the determination overview. The **Save to export file** window opens for the selection of the directory and entry of the **File name** for the export file. After clicking on **<Save>** the determination and method data for each of the marked determinations is stored in a ***.txt** text file. The first line of this text file contains the **parameter title**, separated by tabulator characters. The following lines contain the **parameter values**, also separated by tabulator characters.

Printing of results in the control window

763 PVC THERMOMAT CONTROL / File / Print

The **Print results** window opens and provides an overview of all running and finished determinations of all channels of the connected units.



Unit	Instrument number (1...4).
Block	Instrument block (A, B).
Channel	Channel number (1...4).
Method	Method name.
Sample ID1	Sample identifier 1.
Stab. time	Determined stability time.
Induc. time	Determined induction time.

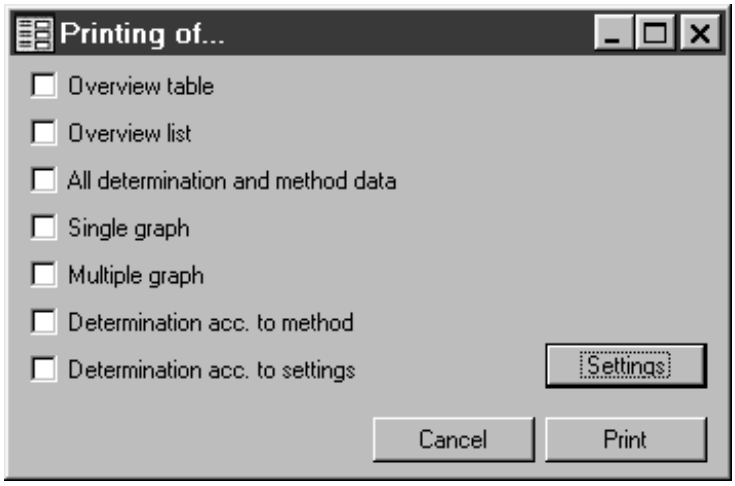
The width of the columns in the **Print results** window can be altered by pulling the right-hand field margin of the column title with the mouse. These settings are permanently stored when the window is closed.

The required determinations must be marked for the **printout of results**. After clicking on **<Print>** the results are printed out according to the settings defined in the method on the **Documentation** tab.

Printing of data in the results window

763 PVC THERMOMAT RESULTS / File / Print

If the **Determination data** window is open and selected (active) then the **Printing of...** window opens.



Overview table	Prints out the determination overview in **tabular form**. Only those columns which are within the maximum printing width (15 cm) will be printed out.
Overview list	Print out all fields for all determinations in the form of a **list**.
All determination and method data	Prints out all determination and method data for all the marked determinations. A two-page report is printed out for each determination.
Single graph	Prints out the single graphs for all marked determinations.
Multiple graph	Prints out the multiple graphs with all marked determinations.
Determination acc. to method	Prints out the report defined in the method (see *section 4.5.2*) for all marked determinations.
Determination acc. to settings	Prints out the report defined under **Settings** for all marked determinations.
<Settings>	Opens the **Program settings** window for fixing the report settings (see *section 4.7.7*).
<Print>	Prints all selected options for the determinations selected in the determination overview.

4.7.7 Options

Program settings

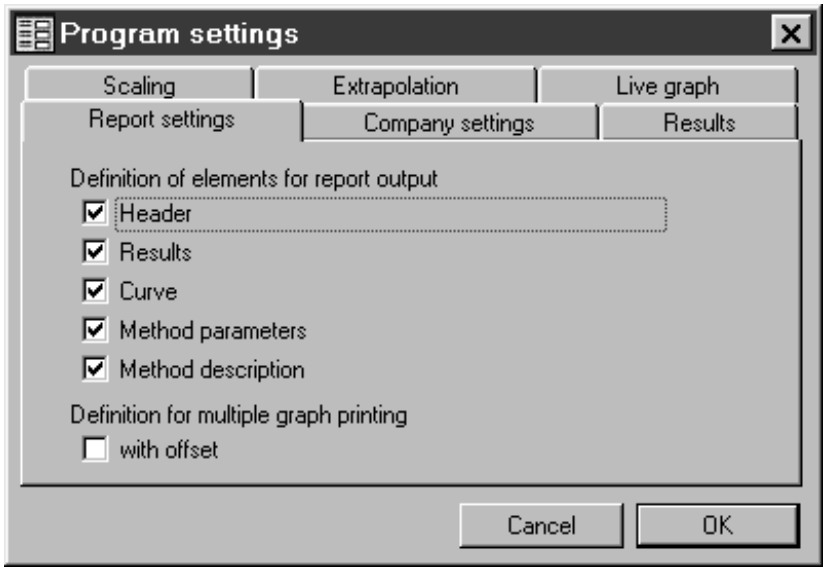
763 PVC THERMOMAT RESULTS / Options / Settings

The **Program settings** window opens; it contains the following tabs:

Report settings	Settings for report output.
Company settings	Settings for company logo and text.
Results	Settings for results output.
Scaling	Settings for scaling the curve axes.
Extrapolation	Settings for extrapolation.
Live graph	Settings for displaying the live graph.

Report settings

Tab of the **Program settings** window with settings for the report output.



Definition of elements for report output

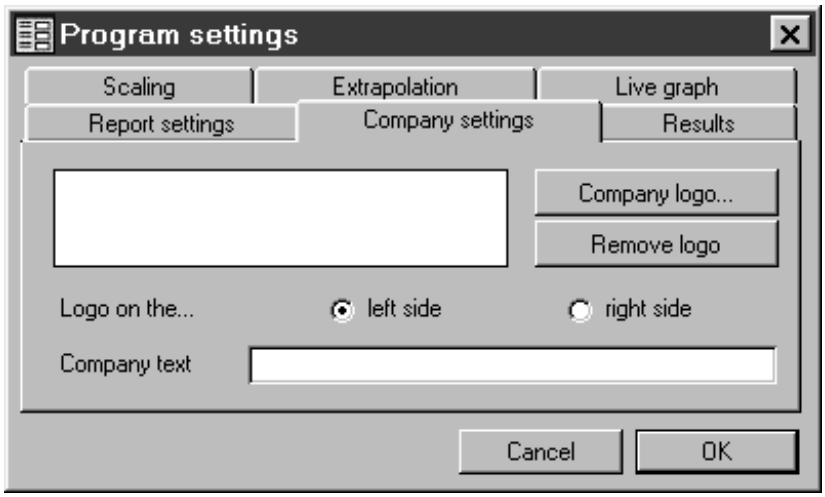
Header	Output of the header with company settings (if defined), date of printout, sample identifiers, instrument information, date of determination, user, cell constant and serial number.
Results	Output of the results selected on the **Results** tab.
Curve	Output of curves.
Method parameters	Output of method parameters.
Method description	Output of the method description entered under **Description**.

Definition for multiple printing

with offset If this option is switched on then in a printout of multiple graphs the curves will be shown offset from each other. In this case the difference of the whole axis is shown in µS/cm instead of the normal conductivity axis marking.

Company settings

Tab of the **Program settings** window with settings for the output of the company logo and company header in the report header.



<Company logo...> The **Find company logo** window appears in which the required graphics file (***.wmf** or ***.bmp**) with the company logo can be selected. After clicking on **<Open>** the logo is shown in the adjacent window.

<Remove logo> The included company logo is removed.

Logo on the...

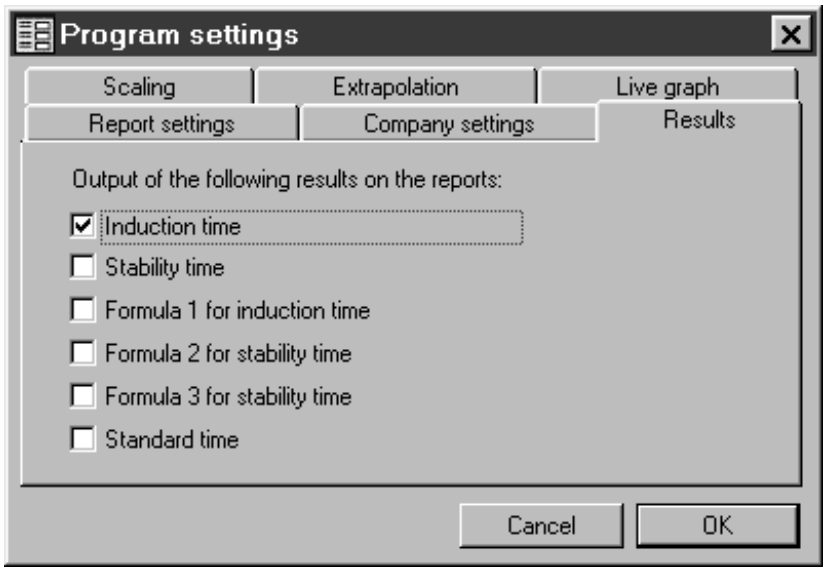
left side The company logo is printed out at the left-hand margin of the header.

right side The company logo is printed out at the right-hand margin of the header.

Company text The text entered here will be printed out at the opposite side of the header to the company logo.

Results

Tab of the **Program settings** window with settings for the output of results.

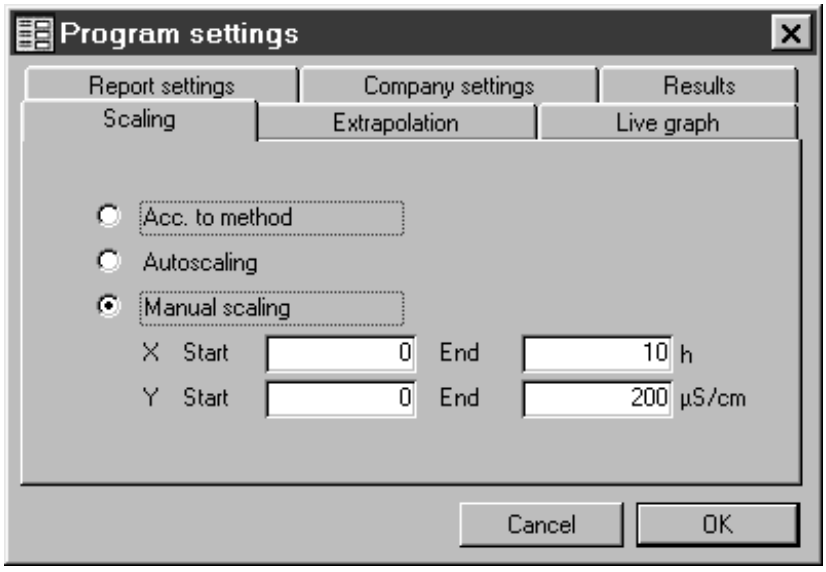


Output of the following results on the reports

Induction time	Induction time.
Stability time	Stability time.
Formula 1..3 ...	Formula results.
Standard time	Standard time.

Scaling

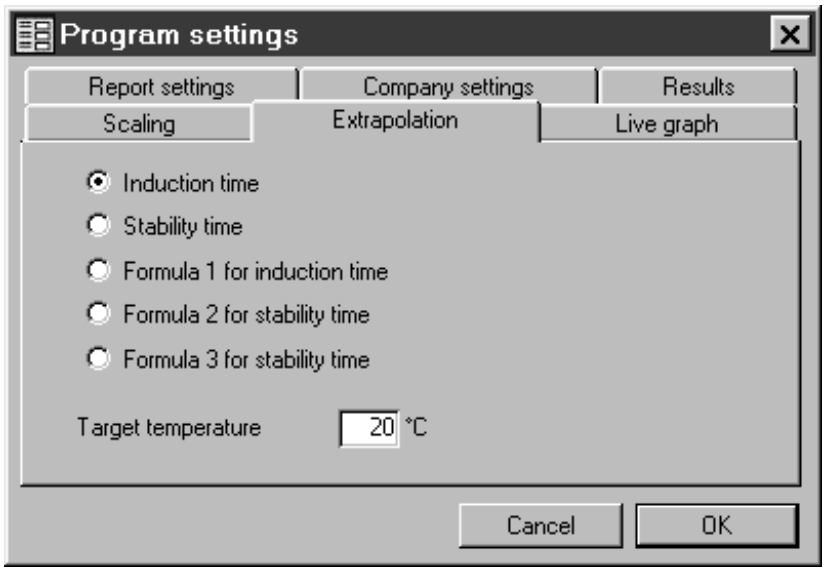
Tab of the **Program settings** window with settings for scaling the curve axes.



Acc. to method	Axis scaling according to the settings defined in the method under **Curves**.
Autoscaling	Automatic fixing of the initial and final values of the curve axes.
Manual scaling	Manual fixing of the initial and final values of the curve axes:
X Start	Starting value of time axis in h.
X End	Finishing value of time in h.
Y Start	Starting value of conductivity axis in µS/cm.
Y End	Finishing value of conductivity axis in µS/cm.

Extrapolation

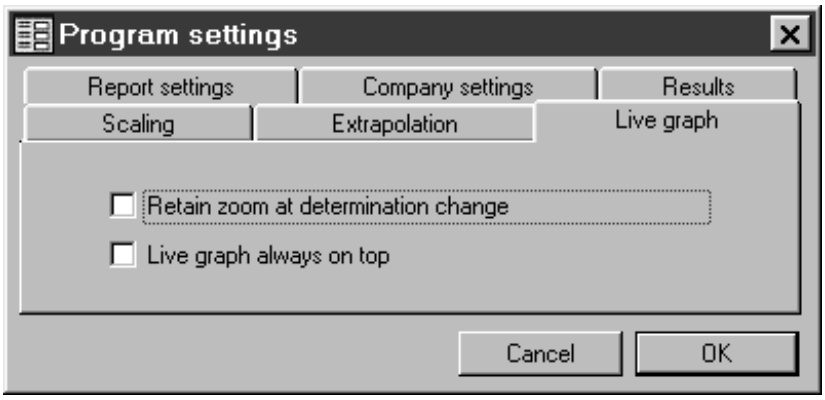
Tab of the **Program settings** window with settings for extrapolation.



Induction time	Extrapolation with the results for the **induction time**.
Stability time	Extrapolation with the results for the **stability time**.
Formula 1..3 ...	Extrapolation with the **Formula results 1...3**.
Target temperature	Temperature to which the selected result is to be converted with the help of the regression line obtained from the extrapolation.

Live graph

Tab of the **Program settings** window with settings for the display of the **Live graph**.



Retain zoom at determination change

The selected zoom range is retained after a change in determination in the determination overview.

Live graph always on top

The **Live graph** window is always shown in the foreground.

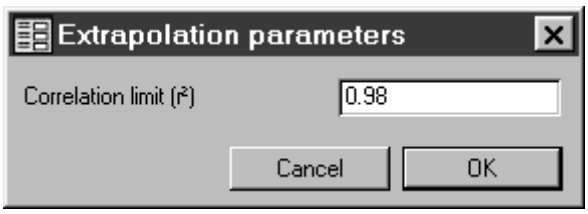
Options for the administrator

763 PVC THERMOMAT RESULTS / Options / Administrator / Database password

The **Open** window opens in which the required ***.mrd** database file can be selected. After clicking on **<Open>** the **Password** window appears in which the **Password** for opening the database with **Microsoft Access 97** is given.

763 PVC THERMOMAT RESULTS / Options / Administrator / Extrapolation parameters

The **Extrapolation parameters** window opens.



Correlation limit Limit for the correlation coefficient r^2. If this value is below the limit a warning will be given.

Save settings

The current window settings can be saved using the following two menu items:

763 PVC THERMOMAT RESULTS / Options / Save settings on exit

If this option is switched on then the current window settings will be saved automatically each time the results window is closed.

763 PVC THERMOMAT RESULTS / Options / Save settings now

The current window settings are saved immediately.

4.7.8 Windows

Tile windows vertically

763 PVC THERMOMAT RESULTS / Window / Tile vertically

All the subwindows opened in the results window (determination overview, curves, GLP window) are arranged one above the other.

Tile windows horizontally

763 PVC THERMOMAT RESULTS / Window / Tile horizontally

All the subwindows opened in the results window (determination overview, curves, GLP window) are arranged side by side.

Cascade windows

763 PVC THERMOMAT RESULTS / Window / Cascade

All the subwindows opened in the results window (determination overview, curves, GLP window) are arranged so that they slightly overlap horizontally and vertically.

Arrange icons

763 PVC THERMOMAT RESULTS / Window / Arrange icons

Arranges all the minimized subwindows contained in the results window (determination overview, curves, GLP window) so that all are visible side by side.

Close all windows

763 PVC THERMOMAT RESULTS / Window / Close all

Closes all the subwindows contained in the results window (determination overview, curves, GLP window).

4.8 GLP

4.8.1 General information about GLP and validation

The requirements of **GLP** (**G**ood **L**aboratory **P**ractice) include a periodic check of analytical measuring instruments with regard to their reproducibility and accuracy using **S**tandard **O**perating **P**rocedures, **SOP**. Under the title **«Application Bulletin No. 278 – Validation of Metrohm PVC Thermomats»** an example of such a standard operating procedure for the 763 PVC Thermomat is available from Metrohm.

The operating software for the 763 PVC Thermomat contains prepared **GLP tests** for temperature, conductivity and gas flow measurements. The operator can determine whether and which tests are to be carried out; in addition the intervals between the tests and the required accuracy can also be set down. If the GLP function is activated then each result report will contain a remark stating whether the GLP test requirements have been fulfilled. Metrohm offers the **6.5616.000 GLP test set** as an option for carrying out the most important tests.

Further information on the subjects of QA, GLP and validation can also be found in the brochure «**Quality management with Metrohm**», which is available from your local Metrohm agency.

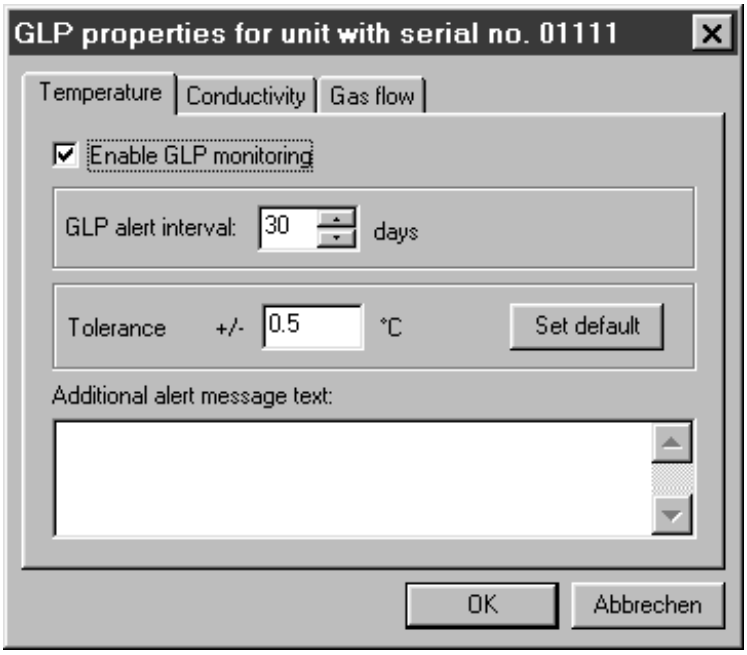
Testing of the electronic and mechanical function groups of Metrohm instruments can and should be performed as part of a regular service by trained personnel of the manufacturing company (see *section 5.1.1*). All Metrohm instruments are equipped with start-up-test routines which check for perfect functioning of the relevant assemblies when the instrument is switched on. If no error message is displayed, it may be assumed the instrument is operating without faults.

The Metrohm company also supplies its instruments with an integrated diagnostic program which, in the case of possible malfunctions or faulty behavior, allows the service technician to check the functioning of certain assemblies and localize the fault.

4.8.2 GLP properties

763 PVC THERMOMAT CONTROL / Tools / GLP test / Properties

This menu item is used to open the **GLP properties** window for the selected instrument; this contains the tabs for temperature, conductivity and gas flow.



Enable GLP monitoring	Activates automatic GLP monitoring.
GLP alert interval	Number of days before next GLP test is due.
Tolerance	States the tolerance values for the particular GLP test. If the result is within the **Set value** ± **Tolerance** range then the test has been passed. By clicking on **<Set default>** the preset default value is entered in this field.

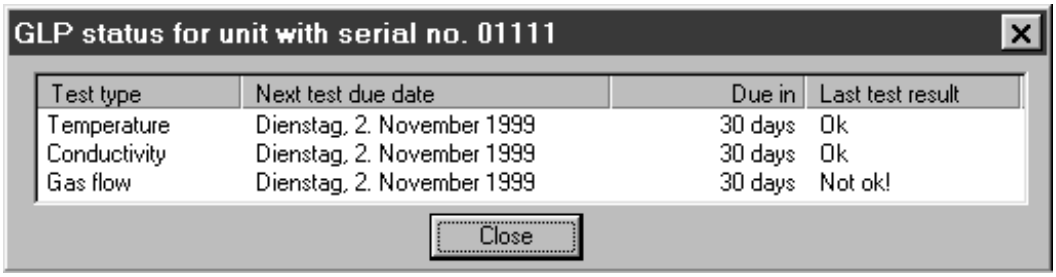
Additional alert message text

Additional text which is to be added to the request to carry out the next GLP test.

4.8.3 GLP status

763 PVC THERMOMAT CONTROL / Tools / GLP test / Status

Opens the **GLP status** window for the selected instrument.



Test type	Type of GLP test (**Temperature**, **Conductivity** or **Gas flow**).
Next test due date	Date on which the next GLP test is to be carried out. If GLP monitoring is not switched on then **Disabled** will appear in this column.
Due in	Number of days before next GLP test is due.
Last test result	States whether the previous test was passed (**Ok**) or not (**Not Ok!**).

4.8.4 GLP tests

GLP temperature test

763 PVC THERMOMAT CONTROL / Tools / GLP test / Temperature

This menu item is used to start the automatic GLP test for **temperature measurement**. This test requires the use of the 6.1111.010 calibrated temperature sensor and the 6.1253.000 GLP insert; these are contained in the 6.5616.000 GLP test set available from Metrohm as an option. The temperature sensor must be connected to the "**Pt100**" connection on the rear panel of the 763 PVC Thermomat.

Before starting the GLP test, the temperature of the GLP insert must have been stabilized. If the GLP insert is placed into a cold heating block, this condition is fulfilled after the normal heating time of heating block and GLP insert. On the other hand, if the GLP insert is placed in an already heated heating block, wait at least 30 min before starting the GLP test.

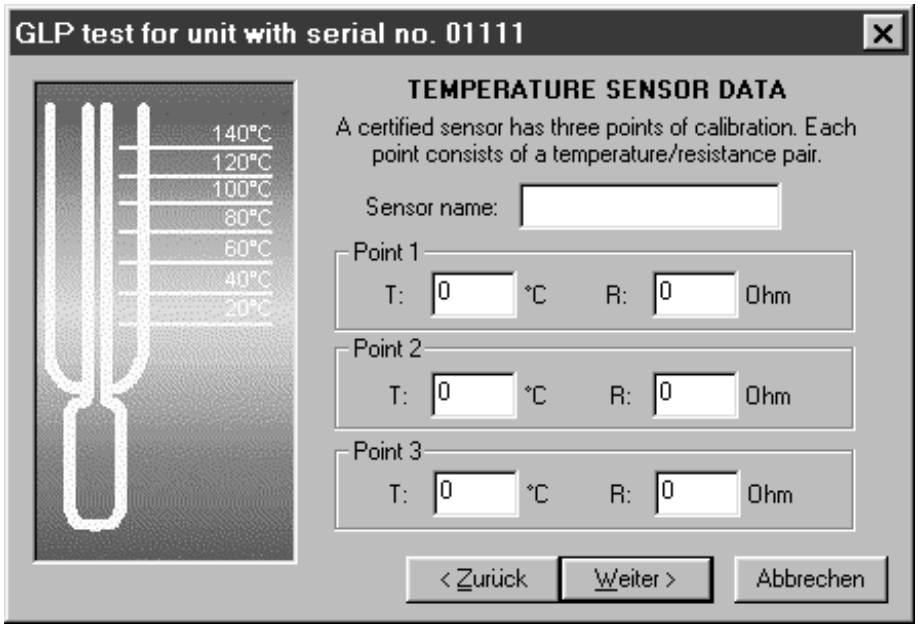
In the GLP test a separate window opens which guides you through the test step-by-step. The next step is reached with **<Next>,** the previous step with **<Back>** and the test is aborted with **<Cancel>**. Proceed as follows to carry out the GLP temperature test:

1 Select instrument and method

- Select unit 1...4 in the control window for which the GLP temperature test has to be carried out.
- Select the determination method whose temperature has to be used for the GLP test in the control window (see *section 4.6.4*) and alter the temperature if necessary.

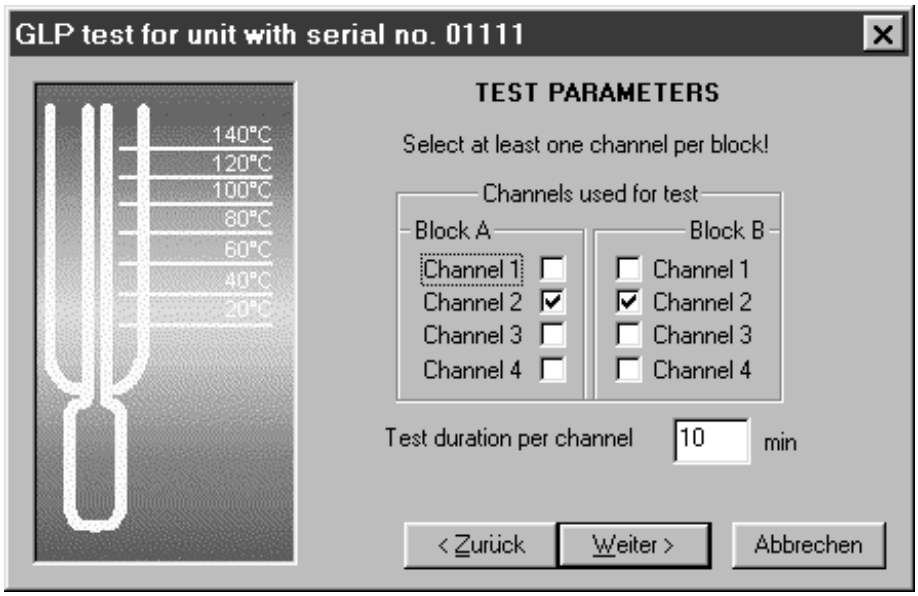
2 Start GLP test

- Click on **763 PVC THERMOMAT CONTROL / Tools / GLP test / Temperature** and click on **<Next>** in the GLP test window.



Sensor name	Name of the temperature sensor.
Point 1...3	Calibration points 1...3 of the temp. sensor.
T	Temperature at calibration point in °C.
R	Resistance at calibration point in ohms.

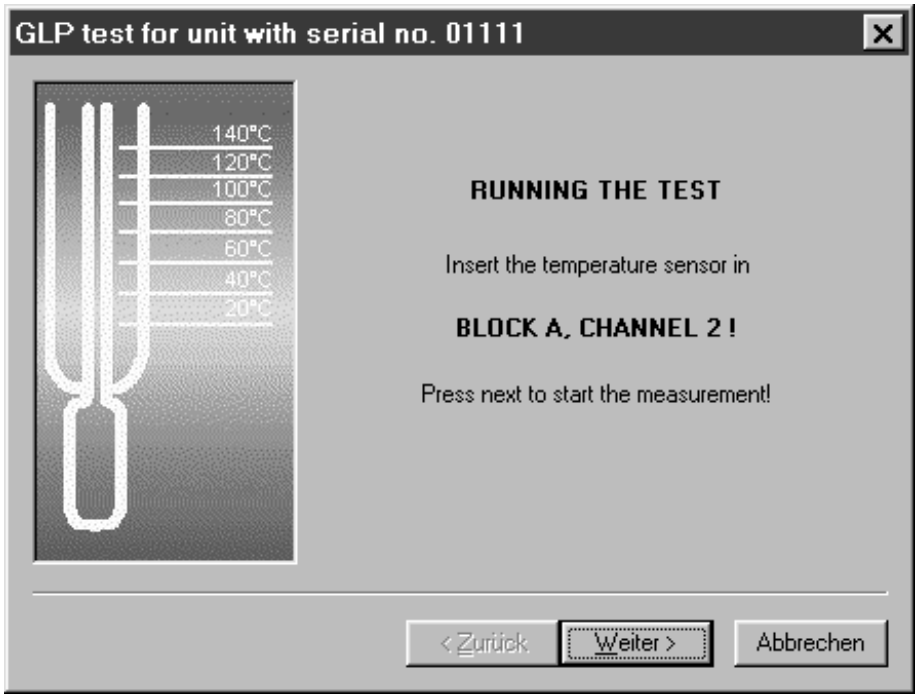
- Enter the calibration data of the temperature sensor used (once data have been entered they are retained) and click on **<Next>**.



Channels used for test

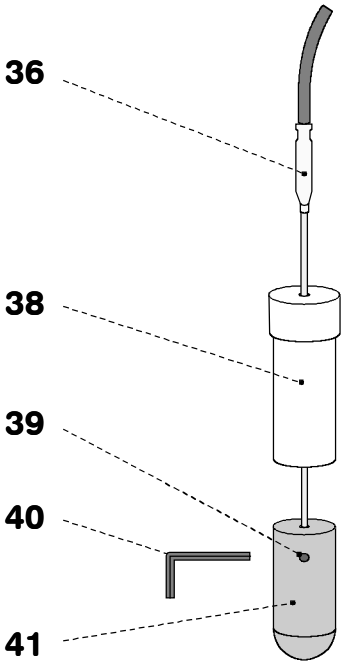
Block A	At least one channel must be selected for Block A.
Block B	At least one channel must be selected for Block B.
Test duration per channel	Measuring time per channel in min.

- Select the channels, for which the temperature test has to be carried out. Enter the desired **Test duration per channel** and click on **<Next>**.



3 Insert GLP test set

- Insert the temperature sensor **36** into the opening of the PTFE cylinder **38** from above (see *Fig. 6*).
- Position aluminium cylinder **41** vertically and introduce temperature sensor **36** into the aluminium cylinder from above until it reaches the stop.
- Fasten temperature sensor **36** to aluminium cylinder **41**, by gently tightening hexagonal screw **39** with the help of Allen key **40**.
- Place temperature sensor **36** together with PTFE and aluminium cylinder in the first channel selected for Block A.
- Connect temperature sensor **36** to connection **17** "Pt100" on the rear panel of the 763 PVC Thermomat and click on **<Next>** to start the measurement.

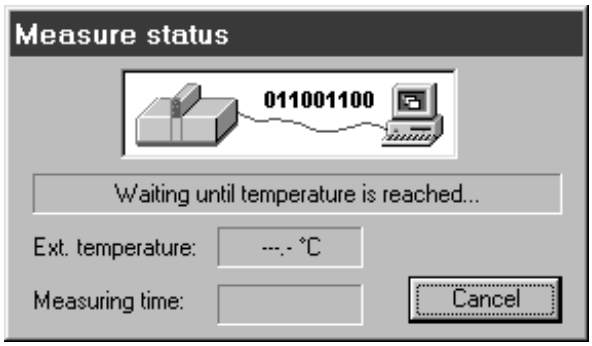


36	**Temperature sensor Pt100 (6.1111.010)**
38	**PTFE cylinder (4.422.1370, part of 6.1253.000)**
39	**Hexagonal screw**
40	**Allen key (6.2042.040)**
41	**Aluminium cylinder (3.422.1400, part of 6.1253.000)**

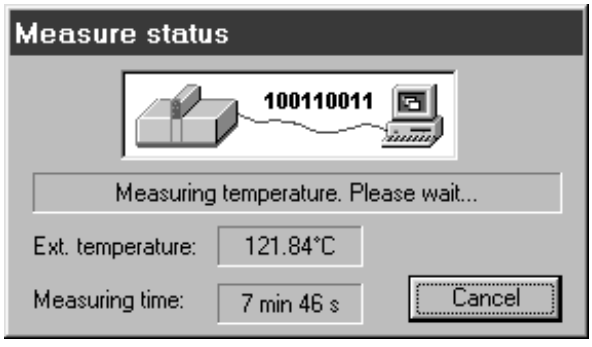
Fig. 6: Accessories for GLP temperature test

4 Carry out GLP temperature test

- If the temperature defined in the method has not been reached then the heating is started automatically. The message **Waiting until temperature is reached...** appears in the **Measure status** window.



- When the temperature defined in the method has been reached the message **Measuring temperature. Please wait...** appears in the window.

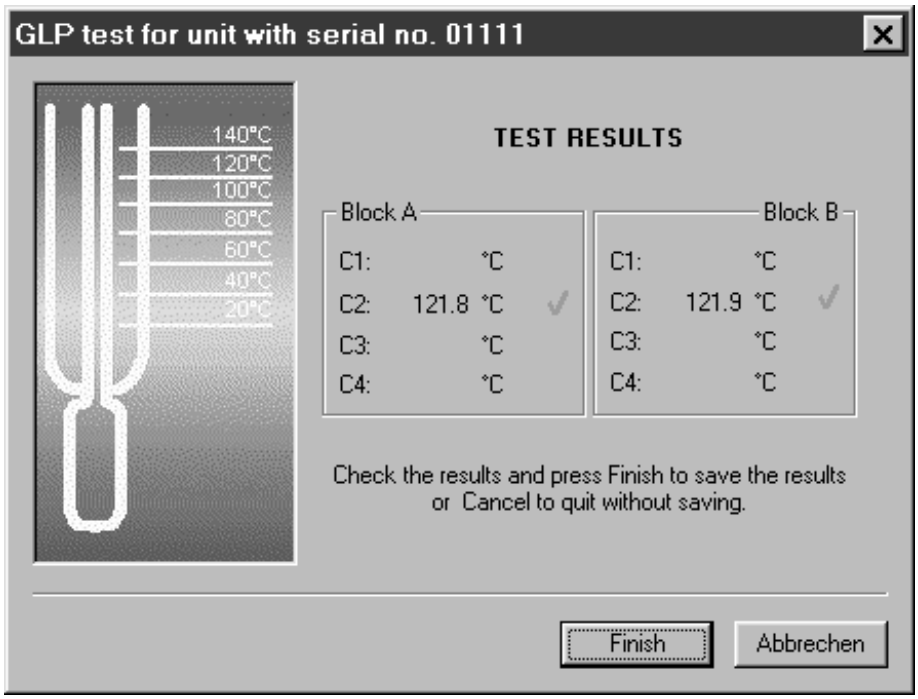


Ext. temperature	The temperature measured by the external temperature sensor.
Measuring time	The elapsed time since the start of the temperature measurement.

- When the measurement has been carried out you are requested to insert the external temperature sensor together with the GLP insert in the next selected channel and to start the measurement. The change must be carried out quickly.
- Carry out the GLP temperature measurement for each selected channel in sequence.

5 Confirm/cancel test results

- When the last temperature measurement has been completed the **TEST RESULTS** for the measured channels are shown.



✓	**Test passed**: The variation of the measured temperature is within the preset tolerance.
✗	**Test failed**: The variation of the measured temperature is larger than the preset tolerance.

- Check the results and click on **<Finish>** to save the GLP test results or on **<Cancel>** to abort the GLP test without saving the results.

GLP conductivity test

763 PVC THERMOMAT CONTROL / Tools / GLP test / Conductivity

This menu item is used to start the automatic GLP test for **conductivity measurement**. This test can either be carried out with a standard solution with known conductivity or with the 6.2109.030 Test resistor which is included in the 6.5616.000 GLP test set available from Metrohm as an option. The test resistor must be connected to the electrode connections **20** of the 763 PVC Thermomat.

In the GLP test a separate window opens which guides you through the test step-by-step. The next step is reached with **<Next>,** the previous step with **<Back>** and the test is aborted with **<Cancel>**.

*The **cell constants** of the conductivity cells have to be determined before carrying out the GLP conductivity test with standard solutions for the first time (see section 4.4.1).*

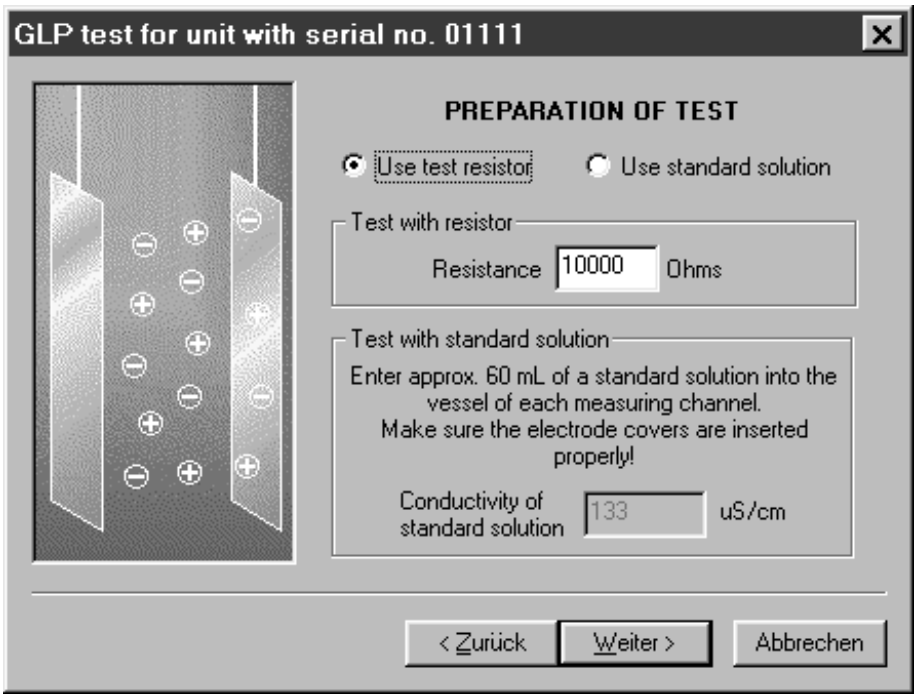
Proceed as follows to carry out the GLP conductivity test:

1 Select instrument

- Select unit 1...4 in the control window for which the GLP conductivity test has to be carried out.
- If the GLP test is carried out with the test resistor carry on from step **2**, otherwise continue with step **3**.

2 Carry out GLP test with test resistor

- Click on **763 PVC THERMOMAT CONTROL / Tools / GLP test / Conductivity** and click on **<Next>** in the GLP test window.

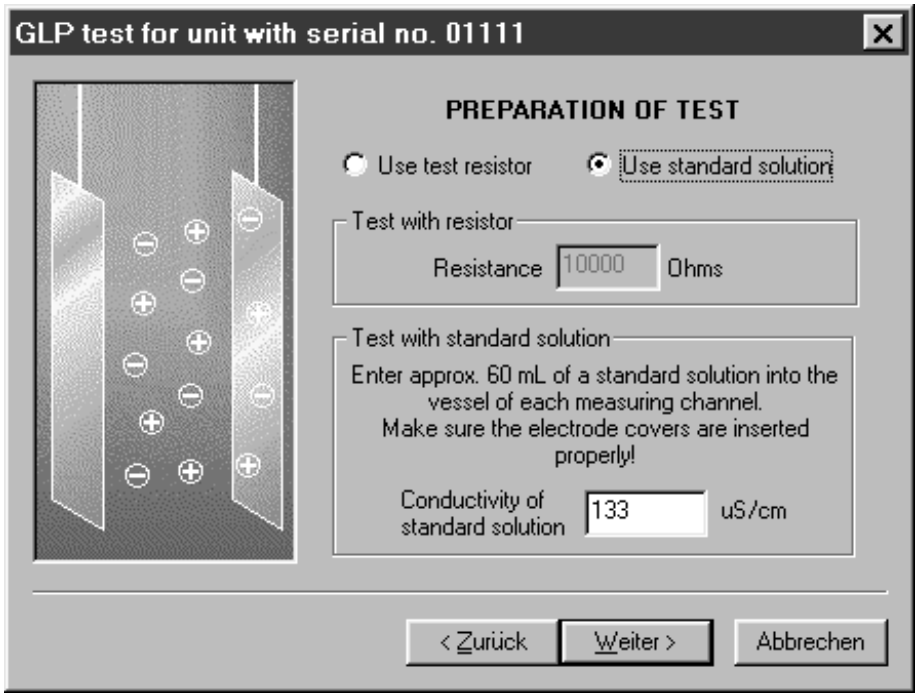


Use test resistor	Carries out the GLP test with 6.2109.030 Test resistor.
Resistance	Resistance in ohms (10 kΩ for 6.2109.030).

- Enable the **Use test resistor** option and enter the **Resistance** of the 6.2109.030 Test resistor in ohms. Afterwards, click on **<Next>**.
- For each channel in sequence, connect the 6.2109.030 Test resistor to the electrode connections **20** and click on **<Next>**.
- Continue with step **4**.

3 Carry out GLP test with standard solution

- Insert a measuring vessel filled with 60 mL distilled water together with a measuring vessel cover into all channels (see *section 4.6.3*).
- Click on **763 PVC THERMOMAT CONTROL / Tools / GLP test / Conductivity** and click on **<Next>** in the GLP test window.



Use standard solution	Carries out the GLP test with standard solutions.

Conductivity of standard solution

Conductivity of the standard solution filled into the measuring vessels in µS/cm.

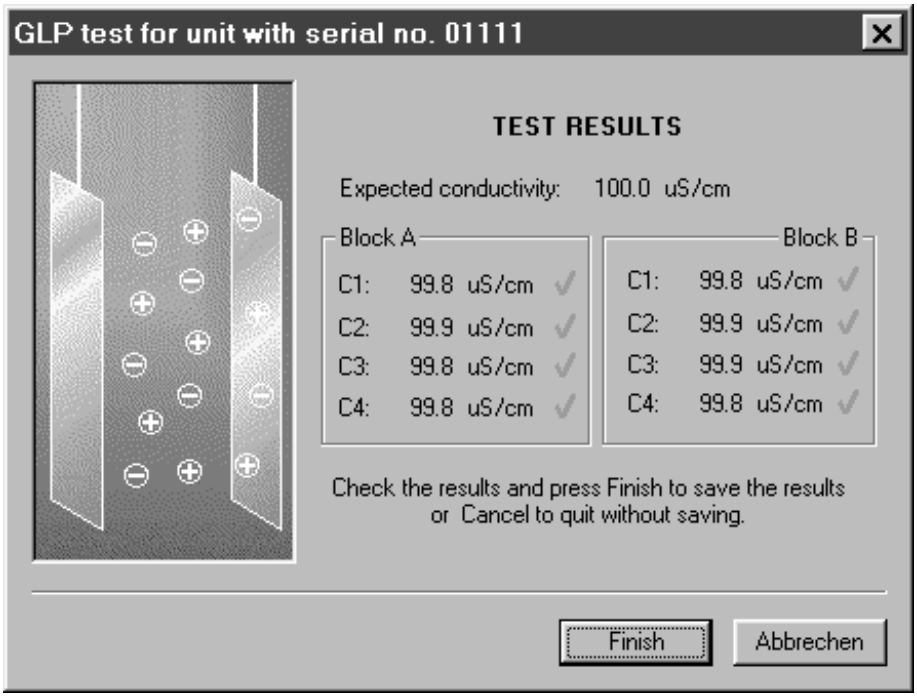
A solution of c(KCL) = 1 mmol/L, prepared from the 6.2301.060 Conductivity standard (KCl 0.1 mol/L) available as an option, can be used as standard solution. The conductivity of this standard solution is:

Temperature	Conductivity
18 °C	127 µS/cm
19 °C	130 µS/cm
20 °C	133 µS/cm
21 °C	136 µS/cm
22 °C	138 µS/cm
23 °C	141 µS/cm
24 °C	144 µS/cm
25 °C	147 µS/cm

- Click on **<Next>** in the GLP test window. The conductivity of each channel is measured in sequence.

4 Confirm/cancel test results

- When the last measurement has been completed the **TEST RESULTS** (set values and measured values) for the measured channels will be shown.



Test passed: The variation of the measured conductivity is within the preset tolerance.

Test failed: The variation of the measured conductivity is larger than the preset tolerance.

- Check the results and click on **<Finish>** to save the GLP test results or on **<Cancel>** to abort the GLP test without saving the results.

GLP gas flow test

763 PVC THERMOMAT CONTROL / Tools / GLP test / Gas flow

This menu item is used to start the automatic GLP test for **gas flow measurement**. A gas flow meter is required for this test (not available from Metrohm) so that the gas flow can be measured at each channel.

In the GLP test a separate window opens which guides you through the test step-by-step. The next step is reached with **<Next>,** the previous step with **<Back>** and the test is aborted with **<Cancel>**.

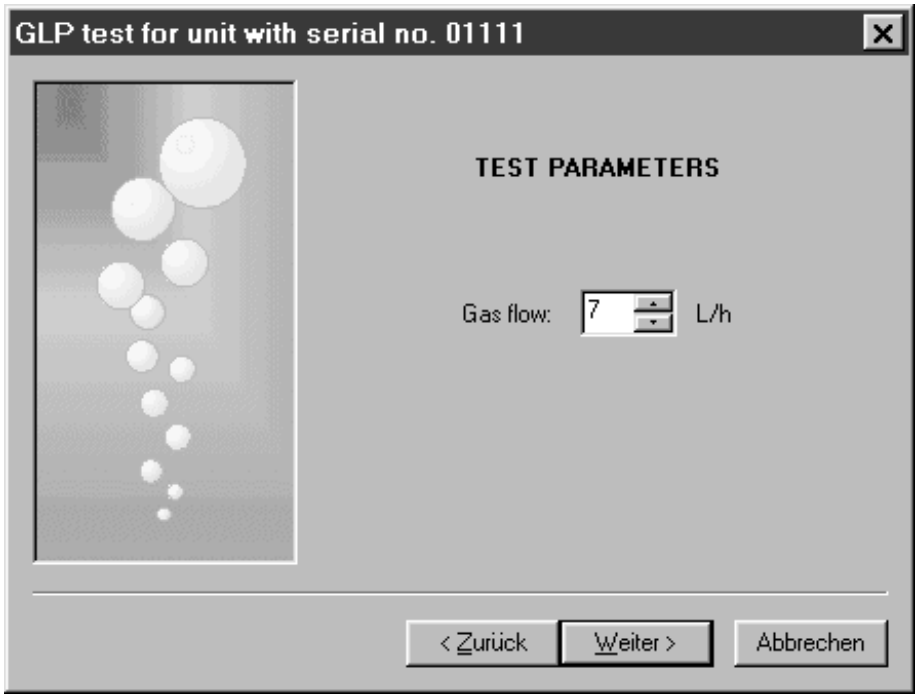
Proceed as follows to carry out the GLP gas flow test:

1 Select instrument

- Select unit 1...4 in the control window for which the GLP gas flow test has to be carried out.

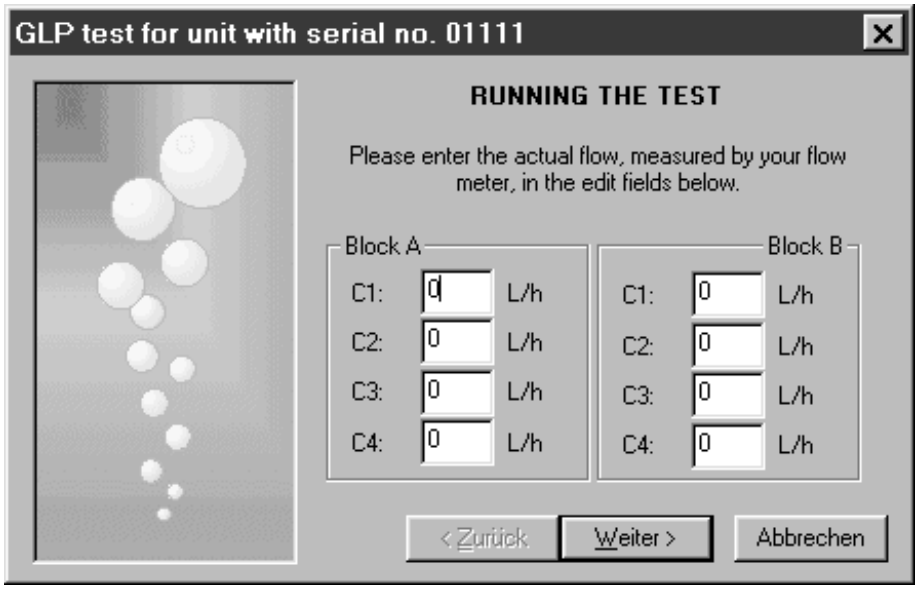
2 Start GLP test

- Click on **763 PVC THERMOMAT CONTROL / Tools / GLP test / Gas flow** and click on **<Next>** in the GLP test window.

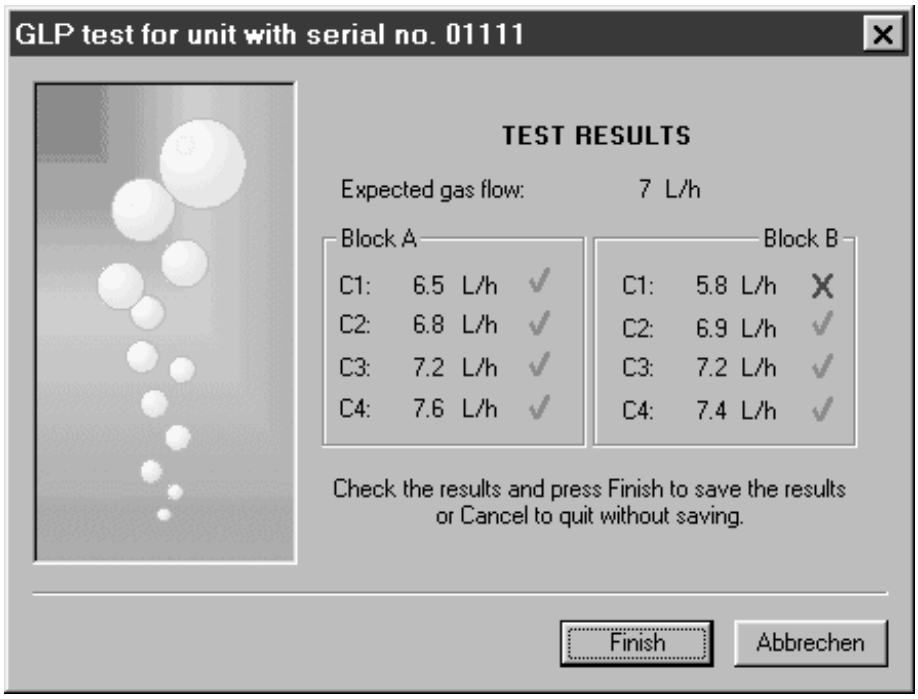


Gas flow rate Gas flow in L/h.

- Enter the **Gas flow rate** and click on **<Next>**.



- Measure the gas flow for each channel in sequence and enter the measured values in the corresponding fields.
- Afterwards, click on **<Next>**.



✓	**Test passed**: The variation of the measured gas flow is within the preset tolerance.
✗	**Test failed**: The variation of the measured gas flow is larger than the preset tolerance.

- Check the results and click on **<Finish>** to save the GLP test results or on **<Cancel>** to abort the GLP test without saving the results.

4.8.5 GLP results

Print GLP certificate

763 PVC THERMOMAT CONTROL / Tools / GLP test / Print GLP results

This menu item is used to print out the **GLP certificate** for the selected instrument; this contains the printout date, the **Serial number** of the instrument and the results of the previous GLP tests carried out on this instrument. Each of the GLP tests for temperature, conductivity and gas flow contains the following information:

Date	Date of the last GLP test carried out.
Tester	Operator who carried out the GLP test.
Test result	Information about whether the test was passed or failed.

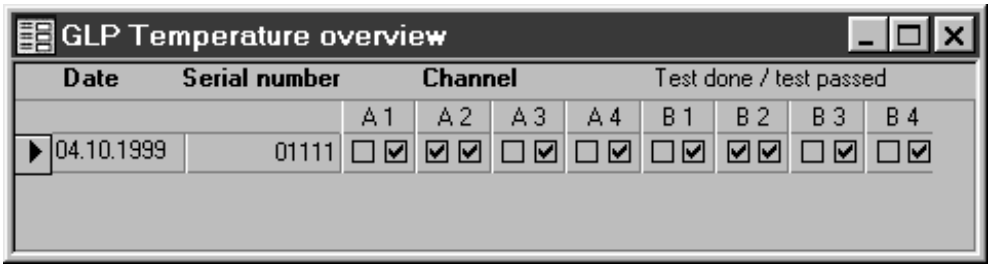
*In order to print out the test results **GLP monitoring** must be enabled (see section 4.8.2).*

View GLP results

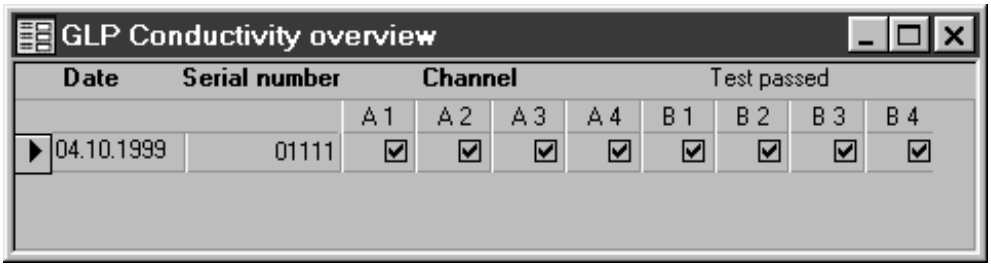
763 PVC THERMOMAT RESULTS / View / GLP

The **GLP** selection window opens to provide three possible selections:

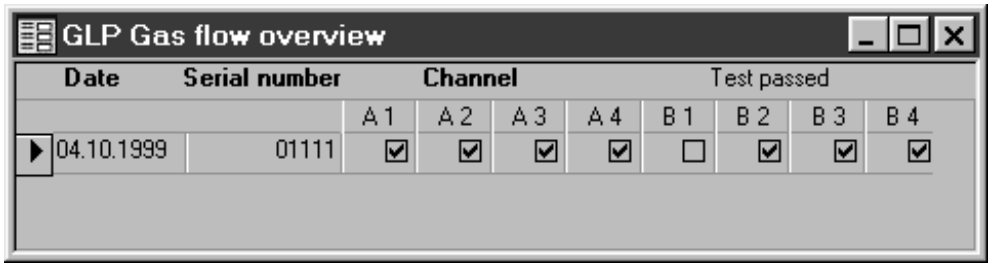
<Temperature> Opens the overview of the results contained in the current database of the **GLP test for temperature measurement**.



<Conductivity> Opens the overview of the results contained in the current database of the **GLP test for conductivity measurement**.



<Gas flow> Opens the overview of the results contained in the current database of the **GLP test for gas flow measurement**.



Date	Date on which the GLP test was carried out.
Serial number	Serial number of the instrument on which the GLP test was carried out.
Channel	Whether the GLP test was carried out and whether it was passed or failed is shown for each of the 8 channels **A1...A4** and **B1...B4**.

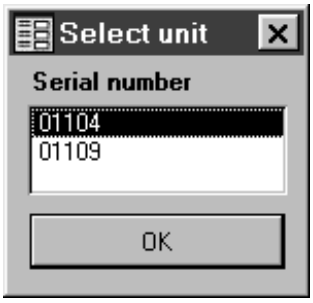
The **GLP Temperature**, **GLP Conductivity** or **GLP Gas flow** window is opened by a double-click on a single GLP test within the **Date**, **Serial number** or **Channel** fields; the detailed results of the GLP test are listed in this window.

*If you require an overview of all the GLP tests carried out with the connected instruments then the **Repos.mrd** database must be open. If a different database is opened then only those GLP tests will be shown which were exported to this database at the export of the determinations (see section 4.7.6).*

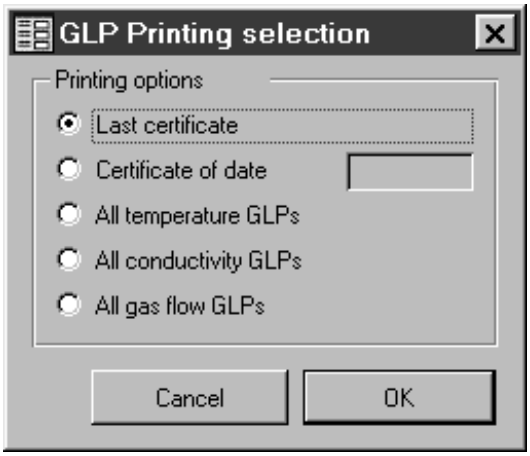
Print GLP results

763 PVC THERMOMAT RESULTS / File / Print

If a GLP window is open then the GLP results can be printed out with this menu item. If the database contains GLP tests carried out on different instruments then the **Select unit** window opens first in which the **Serial number** of the instrument for which the GLP results are to be printed out must be selected.



One of the following options for the printout can then be selected in the **GLP Printing selection** window.



Last certificate	Prints out the last GLP certificate which in addition to the **Serial number** of the instrument contains the **Date**, the **Tester** and the **Test result** (**Test passed** or **Test not passed**).
Certificate of date	Prints out the GLP certificate for the given **Date** which in addition to the **Serial number** of the instrument contains the **Date**, the **Tester** and the **Test result** (**Test passed** or **Test not passed**).
All temperature GLP's	Prints out the detailed results of all GLP tests for temperature measurement.
All conductivity GLP's	Prints out the detailed results of all GLP tests for conductivity measurement.
All gas flow GLP's	Prints out the detailed results of all GLP tests for gas flow measurement.

5 Maintenance – Faults

5.1 Maintenance and servicing

5.1.1 General information

Care

The 763 PVC Thermomat requires proper care and attention. Excessive contamination of the instrument could possibly lead to malfunctions and a shorter service life of the inherently rugged mechanical and electronic parts.

Spilled chemicals and solvents should be wiped up immediately. It is especially important to protect the plug connections at the rear of the instrument (particular the mains plug) against contamination.

Although constructional measures have been designed to virtually eliminate such a situation, should corrosive media penetrate the interior of the instruments the mains plug of the 763 PVC Thermomat must be immediately disconnected to prevent extensive damage to the instrument electronics. Inform Metrohm service if your instrument(s) have been damaged in such a way.

The instrument must not be opened by untrained personnel. Please comply with the safety notes in section 1.5.1.

5.1.2 Maintenance by Metrohm service

Maintenance of the 763 PVC Thermomat is best done as part of an annual service performed by specialists from the Metrohm company. If work is frequently performed with caustic and corrosive chemicals, it may be necessary to shorten the interval between servicing.

The Metrohm service department is always willing to offer expert advice on the maintenance and servicing of all Metrohm instruments.

5.2 Faults and malfunctions

5.2.1 Error messages in the PC program

If any type of malfunction occurs during operation of the 763 PVC Thermomat, this is shown by error messages in the PC program, which appear in an **error window**, e.g.:

Follow the instructions given in the **error window** and close this window with **<OK>**.

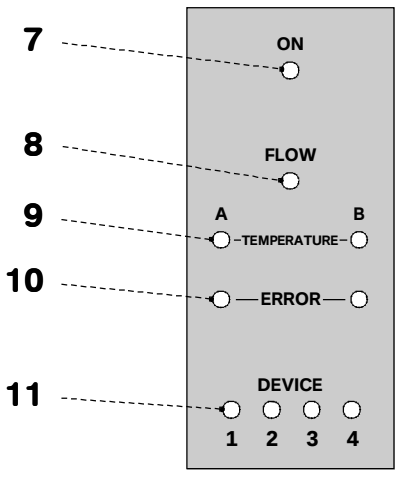
5.2.2 Status and error messages on the instrument

The 763 PVC Thermomat has 10 different LEDs to indicate the status of the instrument (see *Fig. 7*). The status and error messages which could occur on the 763 PVC Thermomat are listed in the following tables. The symbols have the following meanings:

○ LED switched off

● LED switched on

✳ LED flashes



7	**Mains pilot lamp** lit up when instrument switched on
8	**Gas flow display** lit up when gas flow switched on
9	**Temperature display** flashes when heating switched on lit up when temperature reached
10	**Error display (red)** lit up at error
11	**Instrument display** indicates numbers (1…4) of units connected

Fig. 7: Indicator lamps

Operating status

Display	Status/Error	Rectification
ON ○	Instrument switched on.	
ON ●	Instrument switched off.	

Self-test following switch-on

After the instrument has been switched on an internal hardware self-test is carried out. Each of the 7 test steps only lasts about a second and is shown on instrument display **11** by a particular combination pattern of the 4 display lamps. If there is a fault then the corresponding LEDs will flash.

Display	Status/Error	Rectification
DEVICE ● ● ● ●	LED test. If all LEDs do not light up during this test then an LED is faulty.	Inform Metrohm service.
DEVICE ○ ✳ ✳ ✳	RAM test error.	Switch instrument off and on again. If the error reoccurs please contact Metrohm service.
DEVICE ✳ ○ ✳ ✳	PROM check sum test error.	Switch instrument off and on again. If the error reoccurs please contact Metrohm service.
DEVICE ✳ ✳ ○ ✳	BUSY test error.	Switch instrument off and on again. If the error reoccurs please contact Metrohm service.
DEVICE ✳ ✳ ✳ ○	ADC test error.	Switch instrument off and on again. If the error reoccurs please contact Metrohm service.
DEVICE ○ ✳ ✳ ○	EEPROM test error.	Switch instrument off and on again. If the error reoccurs please contact Metrohm service.
DEVICE ✳ ○ ✳ ○	Incorrect mains voltage (Variation > 10%).	Check power supply.

Gas flow display during operation

Display	Status/Error	Rectification
FLOW ○	Gas flow switched off.	
FLOW ✳	Gas flow control active, set gas flow not yet reached.	
FLOW ●	Gas flow control active, set gas flow reached.	

Temperature display during operation

The heating status is shown separately for Block A and Block B.

Display	Status/Error	Rectification
A B ○–TEMPERATURE–○	Heating switched off.	
A B ☼–TEMPERATURE–☼	Heating active, set temperature not yet reached.	
A B ●–TEMPERATURE–●	Heating active, set temperature reached.	

Error displays during operation

Errors are shown separately for Block A and Block B.

Display	Status/Error	Rectification
○ —ERROR— ○	System working properly.	
☼ —ERROR— ☼	Operating error. Possible causes: • Heating fault · Maximum temperature (220°C) exceeded by 10 °C.	 • Switch instrument off and on again. If the error reoccurs please contact Metrohm service. · Switch off instrument and contact Metrohm service.
● —ERROR— ●	Self-test error (see above).	Switch instrument off and on again. If the error reoccurs please contact Metrohm service.

Instrument displays during operation

The status of each connected instrument is shown separately.

Display	Status/Error	Rectification
DEVICE ○ ○ ○ ○	Instrument must be logged in to PC.	Set up instrument communication (see *section 2.4.3*).
DEVICE ☼ ☼ ☼ ☼	Connection to PC interrupted.	Check connection between PC and PVC Thermomat.
DEVICE ● ○ ○ ○	Instrument 1 is logged in and connected to the PC.	
DEVICE ○ ● ○ ○	Instrument 2 is logged in and connected to the PC.	
DEVICE ○ ○ ● ○	Instrument 3 is logged in and connected to the PC.	
DEVICE ○ ○ ○ ●	Instrument 4 is logged in and connected to the PC.	

6 Appendix

6.1 Technical data

6.1.1 General information

No. of instruments which can be connected	1 … 4
No. of samples per instrument	1 … 8 (4 per heating block)
Sample quantity	a few grams / a few milliliters

6.1.2 Temperature regulation and measurement

Temperature setting range	50 … 220 °C
Temperature correction (Delta T)	0 … ± 9.9 °C (can be entered manually or determined automatically with the help of 6.1111.010 Temperature sensor)
Temperature measuring range	0 … 250 °C
Resolution	0.1 °C
Max. variation of heating block temperature from set value (50 … 220 °C)	± 0.3 °C
Reproducibility of set temperature	typ. < 0.2 °C
Max. temperature difference between different measuring positions in one block	typ. < 0.3 °C
Temperature variation (when operating temperature has been reached, with inserted and identically filled reaction vessels with an air flow of 20 L/h)	typ. < 0.1 °C
Switch-off temperature	260 ± 11 °C (if this temperature is exceeded the heating is switched off, it switches on again at 210 ± 11 °C)
Heating-up time of instrument from 20 °C	(up to a temperature stability of ± 0.1 °C)
to 120 °C	approx. 45 min
to 220 °C	approx. 60 min
External instrument temperature	< 50 °C (at operating temperature 220 °C)

6.1.3 Conductivity measurement

Sensor	6.0913.130 Conductivity cell built into measuring vessel cover
Construction	Conductivity measurement cell with 2 steel electrodes
Measurement principle	Alternating current measurement with 1 kHz frequency and approx. 1.7 V amplitude (peak to peak).
Cell constant	1.00 … 1.20 (the exact value can be manually entered or automatically determined)
Measurement range	0 … 400 µS/cm
Resolution	0.1 µS/cm
Maximum error	± 1 % ± 0.5 µS/cm

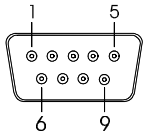
6.1.4 Gas flow regulation

Gas flow supply	External supply from N_2 cylinder
Adjustable range	7 … 25 L/h
Max. variation from set range	± 10 %

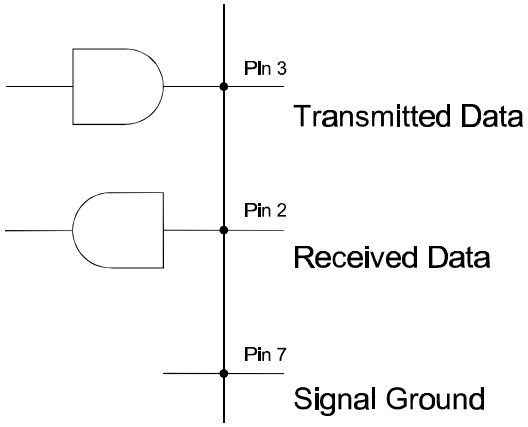
6.1.5 GLP test set

External temperature sensor	
Type	Pt100 jacketed element 1.9 mm dia., with 4-lead technology; DIN IEC 751 class B
Calibration accuracy	0 … 90 °C: ± 0.02 °C 90 … 220 °C: ± 0.03 °C
Max. variation of measured temperature from true value (50 … 220 °C)	± 0.3 °C
Test resistor	
Resistance	10 kΩ

6.1.6 RS232 interface

Connector	Dsub 9-pin (male) 
Function	TxD and RxD signal for connection with software handshake
Default settings	9600 baud, 8 bit, 1 stop bit, no parity, XON/XOFF

Pin assignment



6.1.7 Mains connection

Voltage	2.763.0015: 115 V: 100 ... 120 V ± 10 % 2.763.0014: 230 V: 220 ... 240 V ± 10 %
Frequency	50 ... 60 Hz
Power consumption	450 VA
Fuse	5 mm dia., 20 mm length 100 … 120 V: 2.0 ATH (slow-blow) 220 … 240 V: 4.0 ATH (slow-blow)

6.1.8 Safety specifications

Construction/testing	According to IEC 1010 / EN 61010 / UL 3101-1, protection class 1, degree of protection IP20
Safety directions	The Instructions for Use include information and warnings which must be heeded by the user to assure safe operation of the instrument.

6.1.9 Electromagnetic compatibility (EMC)

Emitted interference	Standards met: EN 55011 (class B) EN 55022 (class B) EN 50081-1 EN 50081-2 EN 61000-3-2 (class A) EN 61000-3-3
Immunity to interference	Standards met: EN/IEC 61000-4-2 (class 3) EN/IEC 61000-4-3 (class 3) EN/IEC 61000-4-4 (class 4) EN/IEC 61000-4-5 (class 2/3) EN/IEC 61000-4-6 (class 3) EN/IEC 61000-4-11 (class 3) ENV 50204

6.1.10 Ambient temperature

Nominal operating range	+5…+45°C (at 20…80 % atmospheric humidity)
Storage	–20…+70°C
Transport	–40…+70°C

6.1.11 Housing

Material of cover	Polyurethane rigid foam (PUR) with fire protection for fire class UL94VO, CFC-free
Material of base	Steel, enameled
Width	405 mm
Height	268 mm (without accessories) 353 mm (with accessories)
Depth	402 mm
Weight	19.4 kg (without accessories) 26.2 kg (with accessories)

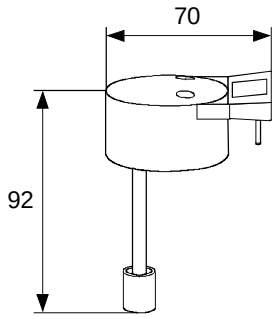
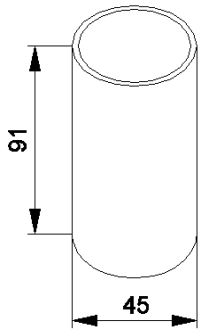
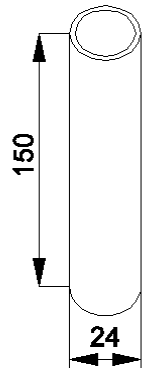
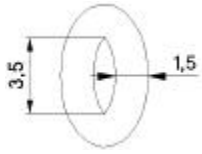
6.2 Standard equipment

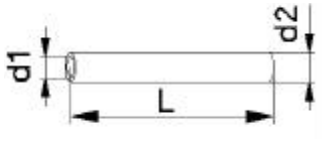
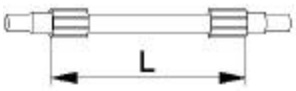
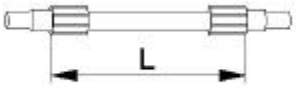
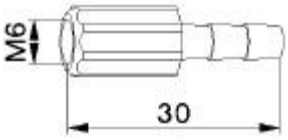
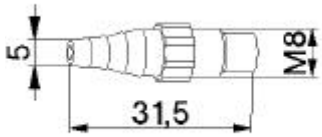
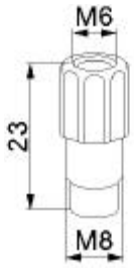
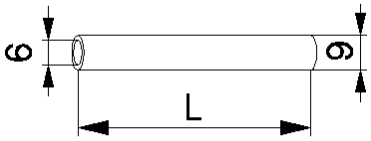
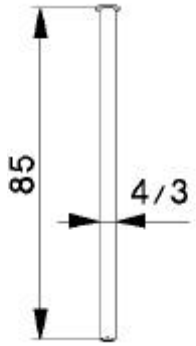
Subject to changes !
All dimensions are given in mm.

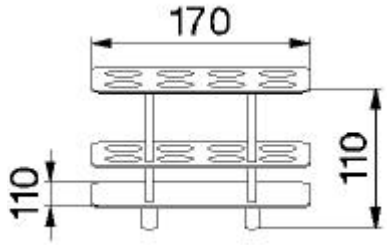
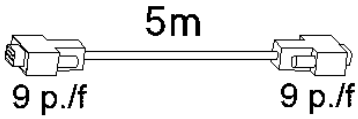
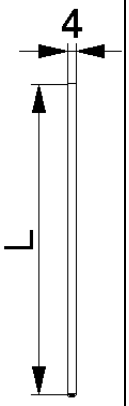
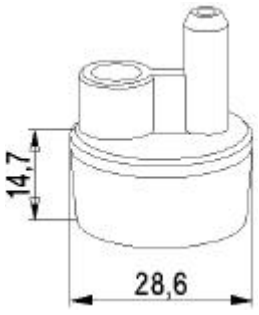
The 763 PVC Thermomat is available in two versions:

- **2.763.0014** PVC Thermomat for mains voltage 230 V
- **2.763.0015** PVC Thermomat for mains voltage 115 V

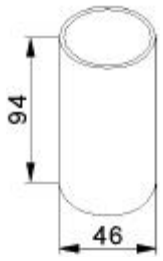
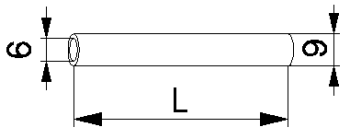
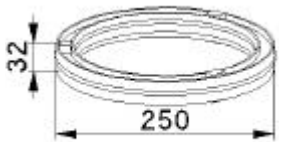
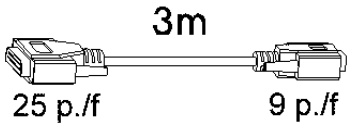
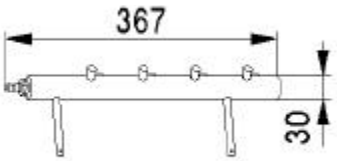
These instruments include the following parts:

Quant.	*Order No.*	*Description*
8	**6.0913.130**	**Measuring vessel cover** with integrated conductivity measurement cell 
8	**6.1428.100**	**Measuring vessel** made of polycarbonate 
1	**6.1429.040**	**Reaction vessel** made of clear glass Set of 117 
2	**6.1454.040**	**Sealing ring for air inlet tube** made of nitrile rubber Set of 6 

Quant.	Order No.	Description
1	6.1801.000	**PVC tubing** for connection of nitrogen L = 1 m, d1 = 4 mm, d2 = 7 mm 
1	6.1805.030	**FEP tubing** with two screw nipples with M6 thread, Length = 150 cm 
8	6.1805.080	**FEP tubing** with two screw nipples with M6 thread, Length = 25 cm 
1	6.1808.020	**Connection piece** with M6 thread and tubing stub 
16	6.1808.050	**Connection piece** with M8 thread and tubing stub 
8	6.1808.090	**Connection nipple** with M6 and M8 threads 
8	6.1816.010	**Connecting tubing** made of silicone Length = 22 cm 
8	6.1819.080	**PTFE tube** for air supply to the measuring vessel 

Quant.	Order No.	Description
1	6.2041.190	**Reaction vessel holder** for holding 8 6.1429.040 Reaction vessels 
1	6.2122.0X0	**Mains cable** to customer's specifications: Cable socket — Cable connector Type IEC 320/C 13 — Type SEV 12 (CH…) ..... 6.2122.020 Type IEC 320/C 13 — Type CEE (7), VII (D…) ..... 6.2122.040 Type CEE (22), V — Type NEMA 5-15 (USA…) 6.2122.070
1	6.2134.100	**Connecting cable** Connecting cable 763 PVC Thermomat (RS232) – PC 
1	6.2418.110	**Gas inlet tube** for 6.1429.040 Reaction vessel Length = 98 mm Set of 117 
24	6.2753.100	**Reaction vessel cover** for 6.1429.040 Reaction vessel 
1	6.6037.000	**Software CD "Metrodata 763 PC Software 1.0"**
1	8.763.1003	**Instructions for Use (English)** for 763 PVC Thermomat
1	8.763.8007	**Registration card (German/English)** for PC program «Metrodata 763 PVC Thermomat 1.0»

6.3 Optional accessories

Order No.	Description
2.145.001X	**Softswitch** for connection of 4 PVC Thermomats to 1 COM interface 2.145.0014: for 230 V 2.145.0015: for 115 V
6.1428.020	**Measuring vessel** made of clear glass 
6.1816.010	**Silicone tubing** for connection of measuring vessels to the 6.2757.000 Exhaust collection tube Length = 22 cm 
6.2059.000	**Rotation ring** for rotatable storage of a 763 PVC Thermomat. 
6.2125.110	**Connecting cable** for connection of the 763 PVC Thermomat to a 25-pin COM interface at the PC. 
6.2301.060	**Conductivity standard 250 mL** c(KCl) = 0.1 mol/L
6.2757.000	**Exhaust collection tube** for leading off waste air from the measuring vessels. With E.400.0010 Stoppers for closing the openings. 
6.5616.000	**GLP test set** for carrying out GLP tests, incl. the following accessories: 1 × 6.1111.010 Temperature sensor Pt100 1 × 6.1253.000 GLP insert, incl. the following accessories: 1 × 3.422.1400 Aluminium cylinder 1 × 4.422.1370 PTFE cylinder 2 × 6.2042.040 Distance piece 1 × 6.2109.030 Test resistor 10 kΩ 1 × 6.2418.000 Gas inlet tube, length = 148 mm, set of 12 1 × 6.2621.120 Hexagon key 1.5 mm

6.4 Warranty and conformity

6.4.1 Warranty

The warranty on our products is limited to defects that are traceable to material, construction or manufacturing error which occur within 12 months from the day of delivery. In this case, the defects will be rectified in our workshops free of charge. Transport costs are to be paid by the customer. For day and night operation, the warranty is limited to 6 months.

Glass breakage in the case of electrodes or other parts is not covered by the warranty. Checks which are not a result of material or manufacturing faults are also charged during the warranty period. For parts of outside manufacture insofar as these constitute an appreciable part of our instrument, the warranty stipulations of the manufacturer in question apply.

With the regard to the guarantee of accuracy, the technical specifications in the instruction manual are authoritative.

Concerning defects in material, construction or design as well as the absence of guaranteed features, the orderer has no rights or claims except those mentioned above.

If damage of the packaging is evident on receipt of a consignment or if the goods show signs of transport damage after unpacking, the carrier must be informed immediately and a written damage report demanded. lack of an official damage report releases Metrohm from any liability to pay compensation.

If any instruments and parts have to be returned, the original packaging should be used if at all possible. This applies above all to instruments, electrodes, burette cylinders and PTFE pistons. Before embedment in wood shavings or similar material, the parts must be packed in a dust-proof package (for instruments, use of a plastic bag is imperative). If open assemblies are enclosed in the scope of delivery that are sensitive to electromagnetic voltages (e.g. data interfaces etc.) these must be returned in the associated original protective packaging (e.g. conductive protective bag). (Exception: assemblies with built-in voltage source belong in a non-conductive protective packaging). For damage which arises as a result of non-compliance with these instructions, no warranty responsibility whatsoever will be accepted by Metrohm.

6.4.2 Software license

As the purchaser of the «763 PVC Thermomat 1.0» PC software you also receive the license to use this software on your PC. The PC software has been validated by Metrohm in accordance with the requirements of the ISO 9001 quality system concerning its functioning, analytical performance and accuracy of results. The software functions are documented in the 'Instructions for Use' provided.

Please send us your 8.763.8007 Registration card as soon as possible so that we can register you as an official purchaser. As a registered purchaser you will receive any updated program versions for a preferential price.

6.4.3 EU Declaration of conformity

CE

EU Declaration of Conformity

The METROHM AG company, Herisau, Switzerland hereby certifies, that the instrument:

763 PVC Thermomat

meets the requirements of EC Directives 89/336/EEC and 73/23/EEC.

Source of the specifications:

EN 50081-1	Electromagnetic compatibility, basic specification; Emitted Interference
EN 50082-1	Electromagnetic compatibility, basic specification; Interference Immunity
EN 61010	Safety requirements for electrical laboratory measurement and control equipment

Description of the instrument:

Instrument for automatic determination of the thermal stability of PVC samples.

Herisau, September 21, 1999

Dr. J. Frank	Ch. Buchmann
Development Manager	Production and Quality Assurance Manager

6.4.4 Certificate of conformity and system validation

Certificate of Conformity and System Validation

This is to certify the conformity to the standard specifications for electrical appliances and accessories, as well as to the standard specifications for security and to system validation issued by the manufacturing company.

Name of commodity:	763 PVC Thermomat
System software:	Stored in ROMs
Name of manufacturer:	Metrohm Ltd., Herisau, Switzerland
Principal technical information:	Voltages: 100…120, 220…240 V Frequency: 50…60 Hz

This Metrohm instrument has been built and has undergone final type testing according to the standards:

EN/IEC 61000-4-2 (class 3), EN/IEC 61000-4-3 (class 3),
EN/IEC 61000-4-4 (class 4), EN/IEC 61000-4-5 (class 2/3),
EN/IEC 61000-4-6 (class 3), EN/IEC 61000-4-11, ENV50204
— *Electromagnetic compatibility*

IEC1010, EN61010, UL3101-1 — *Security specifications*

It has also been certified by the Swiss Electrotechnical Association (SEV), which is member of the International Certification Body (CB/IEC).

The technical specifications are documented in the instruction manual.

The system software, stored in Read Only Memories (ROMs) has been validated in connection with standard operating procedures in respect to functionality and performance. The features of the system software are documented in the instruction manual.

Metrohm Ltd. is holder of the SQS-certificate of the quality system ISO 9001 for quality assurance in design/development, production, installation and servicing.

Herisau, September 21, 1999

Dr. J. Frank	Ch. Buchmann
Development Manager	Production and Quality Assurance Manager

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