873 Biodiesel Rancimat

Manual

8.873.8003EN

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8.873.8003EN 03.2009 jb/ars

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Although all the information given in this documentation has been checked with great care, errors cannot be entirely excluded. Should you notice any mistakes please send us your comments using the address given above.

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1 Introduction

1.1 Instrument description

The 873 Biodiesel Rancimat is a PC-controlled measuring device for determining the oxidation stability of biodiesel and biodiesel blends (mixture of biodiesel and conventional diesel fuel) according to the standard EN 14112.

It is equipped with two **heating blocks** each with 4 measuring positions (channels). Every block can be heated individually, i.e. 4 samples can each be measured at 2 different temperatures or 8 samples at the same temperature. The measurements at the individual measuring positions can be started individually for this.

Operation of the 873 Biodiesel Rancimat is realized completely via a PC connected to the RS-232 interface using the control and evaluation program **873 Biodiesel Rancimat**. Up to 4 instruments can be connected to each PC, hence allowing a maximum of 32 samples to be analyzed. The evaluation algorithm of the PC program determines the break point of the Rancimat curve fully automatically and hence the induction time. Besides the **induction time**, the so-called **stability time**, i.e. the time duration until attaining a defined conductivity change, can also be determined. In the case of conductivity changes (stages) which do not have anything to do with the autoxidation, the evaluation can be interrupted for definite time intervals. The results determined can be processed further by computer.

Each Rancimat curve can also be evaluated **manually**. A PC-supported tangential method is available for this, in which you can position the tangents anywhere on your curves. This makes evaluations possible in extreme cases as well.

The results of the determinations are saved in a **database** together with all methods and determination data. Determinations can be searched for, sorted, filtered, exported and printed in the program part for the results display. Besides the graphic display of single and multiple curves, recalculation with altered parameters and extrapolation of the results to a definite temperature is also possible.

GLP (Good Laboratory Practice) and instrument validation are becoming increasingly important. The 873 Biodiesel Rancimat enables GLP tests for temperature, conductivity and gas flow measurement. You determine whether and which tests have to be carried out. You can also specify the time interval between the tests as well as the requirements regarding accuracy. If the GLP function has been selected, each result report will receive a comment stating whether the GLP tests have been fulfilled. Metrohm offers

a GLP test set to carry out these tests *(see Optional accessories, page 180)*.

1.2 Rancimat method

Biodiesel, also referred to as RME (rape seed methyl ester) or FAME (fatty acid methyl esters), is usually obtained from oil seeds and used in the motor vehicle area either in pure form or as a mixture with conventional diesel fuel. As is the case with the natural oils, the fatty acid methyl esters obtained from them also exhibit a relatively low level of storage stability, as they are oxidized by atmospheric oxygen. These oxidation processes progressing slowly at ambient temperatures are referred to as **autoxidation**. They start with radical reactions on unsaturated fatty acids and undergo a process involving multiple stages resulting in diverse decomposition products, in particular peroxides as primary oxidation products and alcohols, aldehydes and carboxylic acids as secondary oxidation products.

With the **Rancimat method**, the sample is exposed to an air flow at a constant temperature between 50…220 °C *(see Figure 1, page 3)*. Highly volatile, secondary oxidation products (for the most part formic acid) are transferred into the measuring vessel with the air flow, where they are absorbed in the measuring solution (distilled water). Here the conductivity is continuously registered. The organic acids can thus be detected by increasing the conductivity. The time until occurrence of these secondary reaction products is referred to as the induction time or induction period, which is a good indicator for the oxidation stability.

The Rancimat method has been developed as an automated variant to the extremely complex AOM (active oxygen method) for determining the **induction time** of fats and oils. This method has become established over the course of time, also being used for derivatives of oils and fats. It has meanwhile become an integral element of various national and international standards for biodiesel and biodiesel mixtures, e.g. EN 14112 and ASTM 6751.

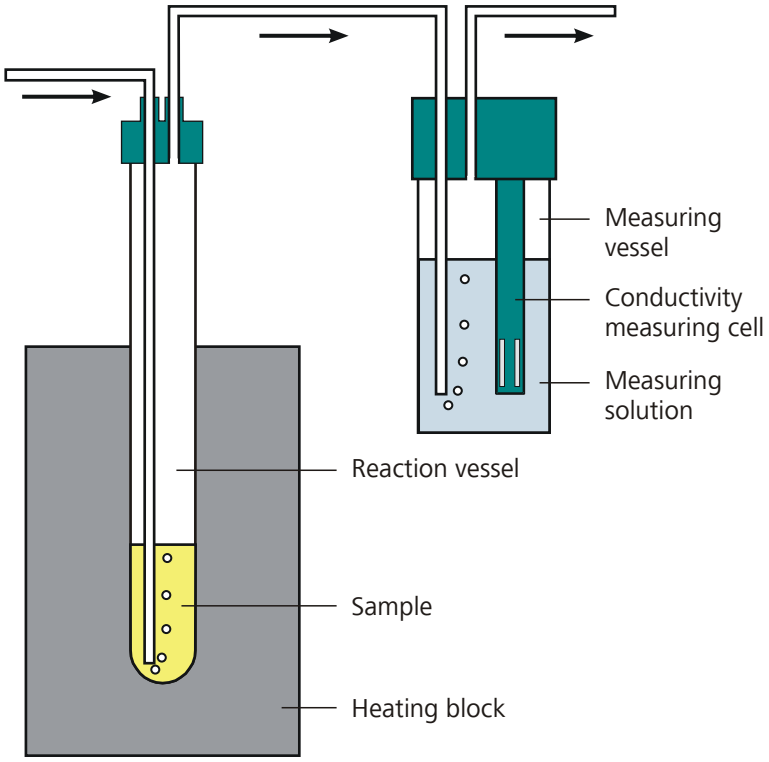


Figure 1 Measuring arrangement (schematic representation)

1.3 About the documentation

Caution

Please read through this documentation carefully before putting the instrument into operation. The documentation contains information and warnings which have to be followed by the user in order to ensure safe operation of the instrument.

1.3.1 Symbols and conventions

The following symbols and styles are used in this documentation:

*(5-**12**)*	**Cross-reference to figure legend** The first number refers to the figure number, the second to the instrument part in the figure.
1	**Instruction step** Carry out these steps in the sequence shown.
Method	**Dialog text**, **parameter** in the software
File ▸ New	Menu or menu item

[Next]	**Button** or **key**
	**Warning** This symbol draws attention to a possible life hazard or risk of injury.
	**Warning** This symbol draws attention to a possible hazard due to electrical current.
	**Warning** This symbol draws attention to a possible hazard due to heat or hot instrument parts.
	**Warning** This symbol draws attention to a possible biological hazard.
	**Caution** This symbol draws attention to a possible damage of instruments or instrument parts.
	**Note** This symbol marks additional information and tips.

1.4 Safety instructions

1.4.1 General notes on safety

Warning

This instrument may only be operated in accordance with the specifications in this documentation.

This instrument has left the factory in a flawless state in terms of technical safety. To maintain this state and ensure non-hazardous operation of the instrument, the following instructions must be observed carefully.

Hot reaction vessels

Warning

The reaction vessels can become very hot.

Avoid any contact with the hot reaction vessels. Place these in the vessel holders provided for cooling down.

Flammable substances

Warning

The oven of the 873 Biodiesel Rancimat can be heated to 220 °C.

Flammable substances may ignite at these temperatures.

Adjust the maximum heating temperature of the oven to the sample being examined.

Defective glass vessels

Warning

Caution with defective glass vessels.

An overflow of flammable samples into the heating block can be dangerous.

Check the glass vessels before each use.

1.4.2 Electrical safety

The electrical safety when working with the instrument is ensured as part of the international standard IEC 61010.

Warning

Only personnel qualified by Metrohm are authorized to carry out service work on electronic components.

Warning

Never open the housing of the instrument. The instrument could be damaged by this. There is also a risk of serious injury if live components are touched.

There are no parts inside the housing which can be serviced or replaced by the user.

Mains voltage

Warning

An incorrect mains voltage can damage the instrument.

Only operate this instrument with a mains voltage specified for it (see rear panel of the instrument).

Protection against electrostatic charges

Warning

Electronic components are sensitive to electrostatic charges and can be destroyed by discharges.

Always pull the mains cable out of the mains connection socket before connecting or disconnecting electrical appliances on the rear panel of the instrument.

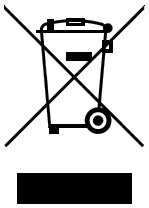
1.4.3 Flammable solvents and chemicals

Warning

All relevant safety measures are to be observed when working with flammable solvents and chemicals.

- Set up the instrument in a well-ventilated location (e.g. laboratory flue).
- Keep all sources of flame far from the workplace.
- Clean up spilled fluids and solids immediately.
- Follow the safety instructions of the chemical manufacturer.

1.4.4 Recycling and disposal



This product is covered by European Directive 2002/96/EC, WEEE – Waste from Electrical and Electronic Equipment.

The correct disposal of your old equipment will help to prevent negative effects on the environment and public health.

More details about the disposal of your old equipment can be obtained from your local authorities, from waste disposal companies or from your local dealer.

2 Overview of the instrument

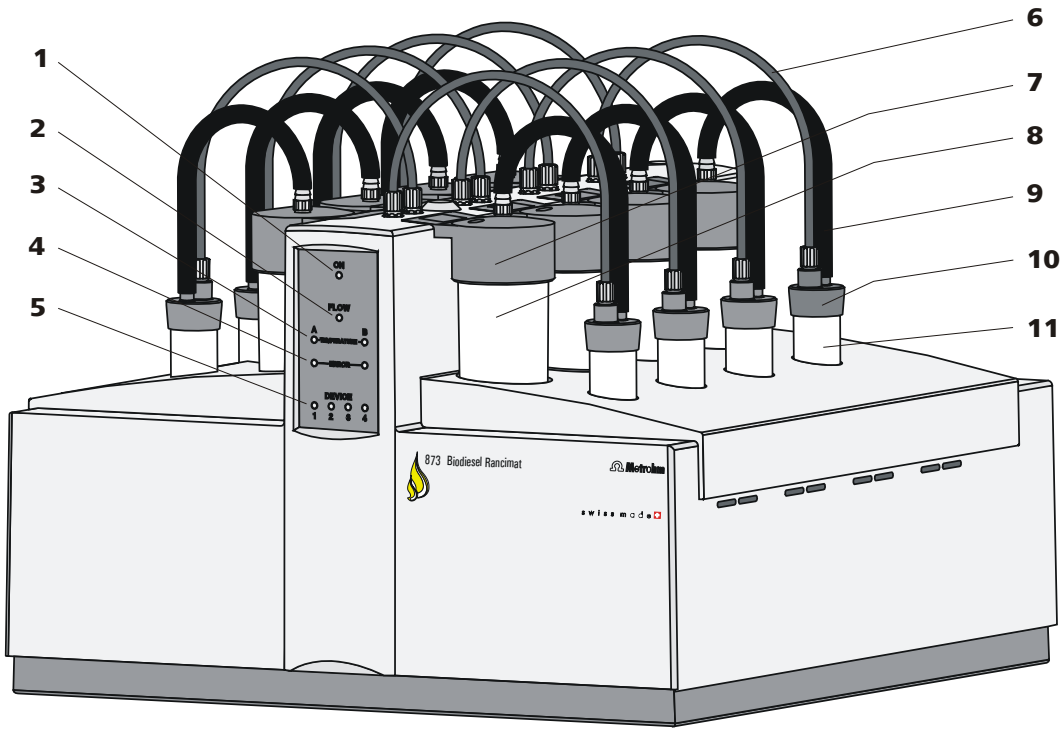


Figure 2 Front 873 Biodiesel Rancimat

1 Pilot lamp Lights up when the instrument is switched on.	**2 Gas flow display** Flashes when the gas flow is switched on. Lights up when the gas flow has been reached.
3 Temperature display Flashes when the heating is switched on. Lights up when the temperature has been reached.	**4 Error display (red)** Lights up or flashes when a fault has occurred in the instrument *(see Troubleshooting Chapter)*.
5 Instrument number display Indicates the number of the instrument. Lights up if the instrument is registered. Flashes (all LEDs) if the connection to the PC is interrupted.	**6 FEP tubing 250 mm (6.1805.080)** For supplying air into the reaction vessel.
7 Measuring vessel cover (6.0913.130) Contains an integrated conductivity measuring cell.	**8 Glass measuring vessel (6.1428.030)**
9 Iso-Versinic® tubing (6.1839.000) For connecting the reaction vessel to the measuring vessel. A fluoroelastomer.	**10 Reaction vessel cover (6.2753.107)**

11 Reaction vessel (6.1429.040)

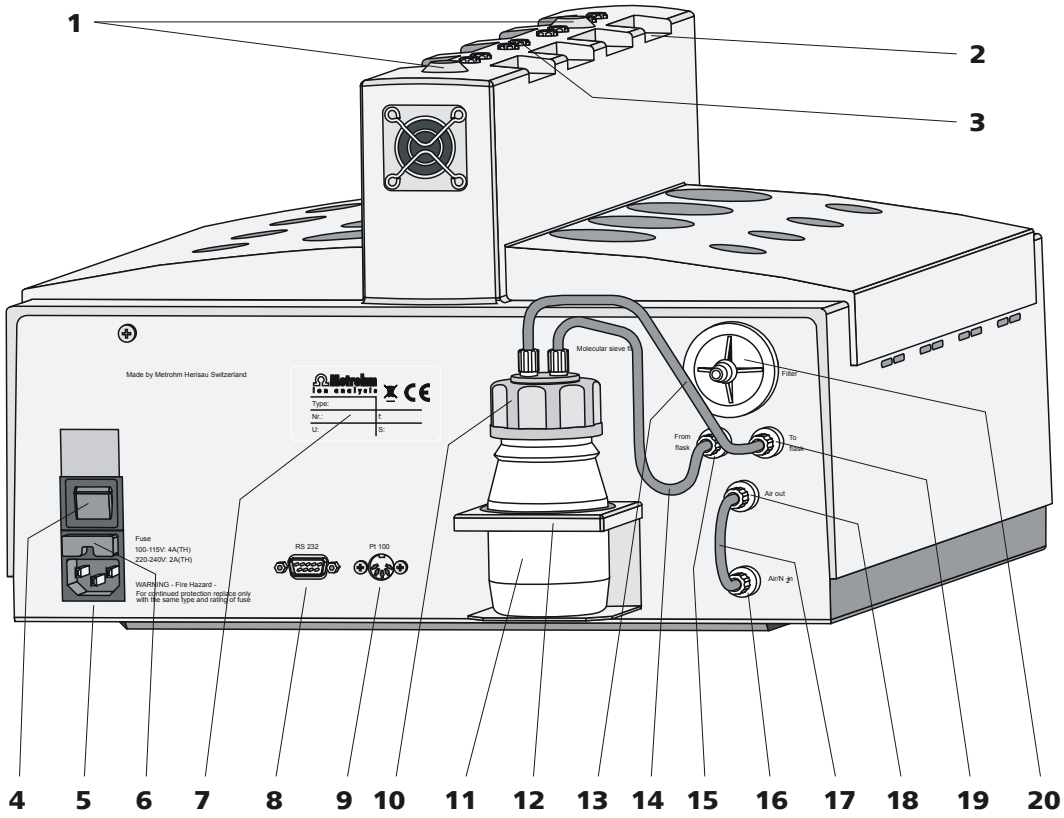


Figure 3 Rear 873 Biodiesel Rancimat

1 Collection tube holder
For fastening the optional exhaust collection tube (6.2757.000).

2 Electrode connector
For connecting the conductivity measuring cell integrated in the measuring vessel cover *(2-**7**)*.

3 Air supply connection
For connecting the FEP tubing 250 mm *(2-**6**)*.

4 Mains switch
For turning the instrument on and off.
I = ON / 0 = OFF.

5 Mains connection socket
For important information on the mains connection, see *Chapter 3.3*.

6 Fuse holder
Replacing fuses, *see Chapter 3.3.2*.

7 Type plate
Contains specifications concerning mains voltage and serial number.

8 RS-232 connector
For connecting the PC.

9 Pt100 connector
For connecting an external temperature sensor.

10 Drying flask cover (6.1602.145)
Cover for the drying flask.

11 Drying flask (6.1608.050)

12 Flask holder
For fastening the drying flask.

13 **FEP tubing 250 mm (6.1805.080)**
For supplying the air from the internal pump to the drying flask.

14 **FEP tubing 250 mm (6.1805.080)**
For supplying the air from the drying flask to the reaction vessel *(2-**11**)*.

15 **"From Flask" connection**

16 **"Air/N_2 in" connection**

17 **FEP tubing 130 mm (6.1805.010)**
For connecting the **Air out** connection to the **Air/N_2 in** connection during normal operation with the internal air pump.

18 **"Air out" connection**

19 **"To Flask" connection**

20 **Dust filter (6.2724.010)**

3 Installation

3.1 Setting up the instrument

3.1.1 Packaging

The instrument is supplied in highly protective special packaging together with the separately packed accessories. Keep this packaging, as only this ensures safe transportation of the instrument.

3.1.2 Checks

Immediately after receipt, check whether the shipment has arrived complete and without damage by comparing it with the delivery note.

3.1.3 Location

The instrument has been developed for operation indoors and may not be used in explosive environments.

Place the instrument in a location of the laboratory suitable for operation and free of vibrations, if possible protected from corrosive atmospheres and contamination by chemicals.

The instrument should be protected against excessive temperature fluctuations and direct sunlight.

Note

In order to improve accessibility for the measuring positions, the instrument can be placed on the optionally available 6.2059.000 turning ring.

3.2 Mounting accessories

3.2.1 Mounting accessories for the internal air supply

The gas in the Biodiesel Rancimat is normally supplied using the **internal air pump**, which sucks in **laboratory air**. For air supply and air purification, the following accessories must be mounted on the rear of the Biodiesel Rancimat:

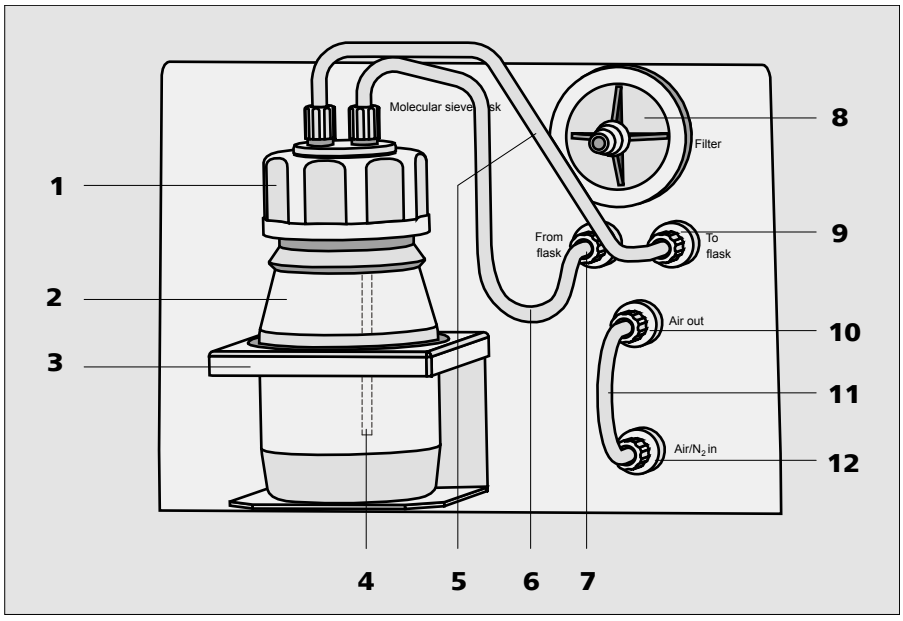


Figure 4 Mounting accessories for the air supply (rear of the instrument)

1	**Drying flask cover (6.1602.145)** Cover for the drying flask.	**2**	**Drying flask (6.1608.050)**
3	**Flask holder** For fastening the drying flask.	**4**	**Filter tube (6.1821.040)**
5	**FEP tubing 250 mm (6.1805.080)** For supplying the air from the internal pump to the drying flask.	**6**	**FEP tubing 250 mm (6.1805.080)** For supplying the air from the drying flask to the reaction vessel *(2-**11**)*.
7	**"From Flask" connection**	**8**	**Dust filter (6.2724.010)**
9	**"To Flask" connection**	**10**	**"Air out" connection**
11	**FEP tubing 130 mm (6.1805.010)**	**12**	**"Air/N_2 in" connection**

Mount the accessories for the air supply as follows:

1 Mount dust filter

- Insert the dust filter *(4-**8**)* on the connection marked with **Filter** on the rear of the Biodiesel Rancimat.
- If the laboratory air is heavily contaminated, a tubing for supplying fresh air can be connected to the dust filter.

Note

The dust filter serves for filtering the air sucked through the air pump and must be replaced at periodic intervals *(see Chapter 5.2, page 155)*.

2 Mount drying flask

Caution

Do **not** fill the hot molecular sieve directly into the drying flask after regeneration, as otherwise the plastic filter on the filter tube will melt.

Wait until the molecular sieve has cooled down before filling.

- Fill the molecular sieve into the drying flask *(4-**2**)*.
- Screw the filter tube *(4-**4**)* on the lower side of the drying flask cover *(4-**1**)* into the opening marked (above) with a dot.
- Screw the drying flask cover with mounted filter tube onto the drying flask and insert in the flask holder *(4-**3**)* on the rear of the Biodiesel Rancimat.
- Screw the one end of the FEP tubing 250 mm *(4-**6**)* onto that opening on the drying flask cover at the bottom of which the filter tube is located.
- Screw the other end of the FEP tubing onto the **From flask** connection *(4-**7**)* on the rear of the Biodiesel Rancimat.
- Screw the one end of the second FEP tubing 250 mm *(4-**5**)* onto the second opening on the drying flask cover.
- Screw the other end of the second FEP tubing onto the **To flask** connection *(4-**9**)*.

Note

The molecular sieve serves for adsorbing interfering oxidizing gases as well as water from the sucked in air. You can regenerate it in the drying oven at +140…+180 °C for 24 to 48 h *(see Chapter 5.3, page 156)*.

3 Mount the FEP tubing for the supply air

- Screw the one end of the FEP tubing 130 mm *(4-**11**)* onto the **Air out** connection *(4-**10**)*.
- Screw the other end of the FEP tubing onto the **Air/N_2 in** connection *(4-**12**)*.

3.2.2 Mounting accessories for the external air supply

If the laboratory air is heavily contaminated, an external gas supply with synthetic air can be provided. For this, the corresponding accessories must be mounted on the rear of the Biodiesel Rancimat.

Mount the accessories for the external air supply as follows:

1 Mount the FEP tubing

- Screw the one end of the FEP tubing 130 mm *(4-**11**)* onto the **Air/ N_2 in** connection *(4-**12**)* on the rear of the Biodiesel Rancimat.
- Screw the tubing adapter M6 / olive (6.1808.020) onto the other end of the FEP tubing.

2 Connect the gas supply

- Mount the gas supply from the bomb with synthetic air onto the tubing adapter M6 / olive (6.1808.020).

Note

With an external gas supply, the gas flow cannot be regulated in the PC program. The gas flow must be set manually using the reducing valve and the gas flow display *(see Chapter 4.2.4, page 39)*.

3.2.3 Equipping the reaction and measuring vessels

The following figure shows in detail, how the accessories parts for measuring the oxidation stability have to be mounted and connected to one another.

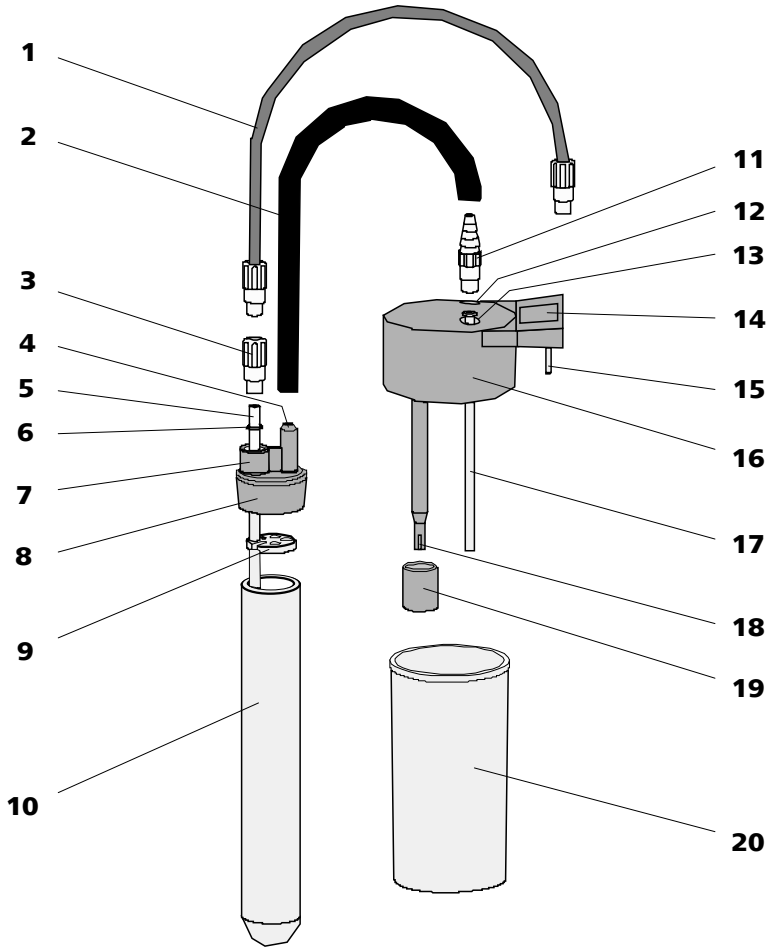


Figure 5 Equipping the reaction and measuring vessels

1	**FEP tubing 250 mm (6.1805.080)** For supplying air into the reaction vessel.	**2**	**Iso-Versinic® tubing (6.1839.000)** For connecting the reaction vessel to the measuring vessel. A fluoroelastomer.
3	**Thread adapter M8 / M6 (6.1808.090)**	**4**	**Tubing connector** For connecting the Iso-Versinic® tubing.
5	**Air tube (6.2418.130)**	**6**	**O-ring (6.1454.040)**
7	**Connection** For connecting the thread adapter M8 / M6.	**8**	**Reaction vessel cover (6.2753.107)**
9	**Foam barrier (6.1451.010)**	**10**	**Reaction vessel (6.1429.040)**
11	**Tubing adapter M8 / olive (6.1808.050)** For connecting the Iso-Versinic® tubing to the opening **In** *(5-**13**)*.	**12**	**Opening "Out"** For removing the air from the measuring vessel.
13	**Opening "In"** For supplying the air to the measuring vessel.	**14**	**Labeling field** For entering the cell constant.

15 **Connector plug**	**16** **Measuring vessel cover (6.0913.130)** Contains integrated conductivity measuring cell.
17 **PTFE tube (6.1819.080)** For supplying the air to the measuring solution.	**18** **Electrode**
19 **Protection ring**	**20** **Glass measuring vessel (6.1428.030)**

Proceed as follows to mount the measuring and reaction vessel:

1 Mount the measuring vessel cover

- Insert the PTFE tube *(5-**17**)* from above into the opening **In** *(5-**13**)* of the measuring vessel cover.
- Screw the tubing adapter M8 / olive *(5-**11**)* into the opening **In** of the measuring vessel cover.
- Place the measuring vessel cover *(5-**16**)* on the measuring vessel *(5-**20**)*.

2 Mount the reaction vessel cover

- Insert the air tube *(5-**5**)* from below into the connection *(5-**7**)* of the reaction vessel cover.
- Place the O-ring *(5-**6**)* over the upper end of the air tube.
- Screw the thread adapter M8 / M6 *(5-**3**)* gently into the connection *(5-**7**)* and, at the same time, press the air tube against the thread adapter M8 / M6.
- Now fix the air tube onto the reaction vessel cover by firmly pulling the thread adapter M8 / M6.
- If determinations are carried out with highly foaming samples, clamp the foam barrier *(5-**9**)* onto the air tube.
- Place the reaction vessel cover on the reaction vessel.

Warning

The foam barrier can melt if it projects too deeply into the heating block.

Ensure that the foam barrier *(5-**9**)* is **at least 7 cm** above the base of the reaction vessel *(5-**10**)*.

3.2.4 Inserting vessels / Establishing tubing connections

After you have assembled the reaction and measuring vessels, insert them in the Rancimat and establish the tubing connections as follows:

1 Insert the measuring vessel

- Fill the measuring vessel *(5-**20**)* with distilled water.
- Place the measuring vessel cover on the measuring vessel.
- Insert the measuring vessel into the openings provided on the Biodiesel Rancimat. At the same time, carefully insert the connector plugs *(5-**15**)* into the electrode connector *(3-**2**)*.
- Connect the black Iso-Versinic® tubing *(5-**2**)* to the tubing adapter M8 / olive *(5-**11**)* of the measuring vessel cover.

2 Mount the tubings for the air supply

- Screw the FEP tubings 250 mm onto the air supply connections *(3-**3**)* of the Biodiesel Rancimat.

3 Insert the reaction vessel

- Fill the reaction vessel *(5-**10**)* with the sample.
- After reaching the required temperature, insert the reaction vessel with the mounted reaction vessel cover in the openings provided on the Biodiesel Rancimat.

4 Establish the tubing connections

- Connect the Iso-Versinic® tubing *(5-**2**)*, which is fastened on the measuring vessel cover, to the tubing connector *(5-**4**)* of the reaction vessel cover.
- Screw the FEP tubing 250 mm *(5-**1**)*, which is fastened on the tubing adapter M8 / olive *(5-**11**)* of the Biodiesel Rancimat, onto the thread adapter M8 / M6 *(5-**3**)* of the reaction vessel cover.

3.2.5 Mounting the exhaust collection tube (optional accessories)

The optionally available exhaust collection tube 6.2757.000 can be mounted on the 873 Biodiesel Rancimat for targeted removal of the exhaust air. In addition to the exhaust collection tube, 8 Iso-Versinic® tubings 6.1839.000 also have to be ordered.

Proceed as follows to mount the collection tube:

1 Mount the exhaust collection tube

- Insert the exhaust collection tube with both nozzles into the collection tube holders *(3-**1**)* on the Biodiesel Rancimat in such a way that the connection to the exhaust air removal is located to the rear.

2 Connect the measuring vessels

- Screw the tubing adapter M8 / olive *(5-**11**)* into the opening **Out** *(5-**12**)* of the measuring vessel cover.
- Connect the one end of the Iso-Versinic® tubing *(5-**2**)* to the tubing adapter M8 / olive.
- Insert the other end of the Iso-Versinic® tubing into the corresponding opening on the collection tube.
- Seal the unused openings on the collection tube with the enclosed plugs E.400.0010.

3 Connect the exhaust collection tube

- Connect a suitable tubing to the connection of the exhaust collection tube and connect this to an active suction device (e.g. water jet pump).

3.3 Mains connection

Warning

There is a risk of fire if the instrument is operated with an incorrect mains fuse!

Follow the regulations below for the mains connection.

3.3.1 Checking the mains voltage

Before switching the 873 Biodiesel Rancimat on for the first time, check whether the mains voltage indicated on the type plate *(3-**7**)* corresponds to the mains voltage present. If this is not the case, please contact Metrohm Service.

3.3.2 Replacing fuses

Two fuses 4 A (slow-acting) for 115 V or 2 A (slow-acting) for 230 V are installed in the fuse holder *(3-**6**)* of the 873 Biodiesel Rancimat as standard.

Warning

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

Proceed as follows to replace defective fuses:

1 Pull out the mains cable

- Pull the mains cable out of the mains connection socket of the Biodiesel Rancimat.

2 Remove the fuse holder

- Use a screwdriver to loosen and completely pull out the fuse holder *(3-**6**)* above the mains connection socket.

3 Replace fuses

- Carefully take the defective fuses out of the fuse holder and replace them with two new fuses suitable for the set mains voltage of the type TH (slow-acting, 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 Insert the fuse holder

- Push the fuse holder back into the instrument until it latches into place.

3.3.3 Mains cable and mains connection

Mains cable

The mains cable optionally supplied for the instrument

- 6.2122.020 with plug SEV 12 (Switzerland)
- 6.2122.040 with plug CEE(7), VII (Germany, …)
- 6.2122.070 with plug NEMA 5-15 (USA, …)

is three-core and provided with a plug with grounding pin. If another plug has to be mounted, the yellow/green conductor (IEC standard) must be connected to the protective ground (protection class I).

Warning

Any interruption to the grounding within or outside the instrument can make it dangerous!

Mains connection

Plug the mains cable into the mains connection socket of the Biodiesel Rancimat.

3.3.4 Switching the instrument on/off

The Biodiesel Rancimat is switched on and off using the mains switch. When switching the instrument on, the pilot lamp **ON** lights up on the front of the instrument auf.

3.4 Connecting a PC

3.4.1 Connecting the 873 Biodiesel Rancimat and the PC

Caution

Always switch the Biodiesel Rancimat and PC off before you connect the two devices with the RS-232 cable 6.2134.100.

The PC program **873 Biodiesel Rancimat** allows control of max. 4 instruments. The following options are available for connecting the instruments to serial PC interfaces:

- Connection to integrated COM interfaces of the PC
- Connection to an additional integrated interface expansion card

Connect the RS-232 interface of the Biodiesel Rancimat to the required serial COM port on the PC using the RS-232 cable 6.2134.100 (9-pin/9-pin). For 25-pin COM ports, the optional RS-232 cable 6.2125.110 (not in the scope of delivery) or a commercially available adapter must be used.

3.4.2 Installing the software

3.4.2.1 System requirements

Operating system	Windows 2000, Windows XP Professional, Windows Vista
RAM	256 MB (Windows 2000 / Windows XP)
	1 GB (Windows Vista)
Processor	Pentium III or higher

Memory	approx. 20 MB for program files
RS-232 interface	one free RS-232 interface (COM)

The windows user must have administrator rights in order to be able to install 873 Biodiesel Rancimat.

3.4.2.2 Installing the program

Proceed as follows to install 873 Biodiesel Rancimat:

1 Start the installation program

- Place the installation CD in the CD drive. The installation program is started automatically.
  If this option is deactivated on your computer, double click the file **Setup.exe**.
  Windows Vista: Select the option "Approve".
- Click on **873**.

2 Select the dialog language

- Select the dialog language of the program.
- Click on the **[OK]** button.
- Click on the **[Next >]** button.

3 Accept the license agreement

- Read through the license agreement and accept it with **[Yes]**.

4 Define a target folder for the program

- If required, choose another folder than the default target folder for the program files. For that, click on the **[Browse...]** button.
- Confirm the target folder with **[Next >]**.

5 Define the components of the program

- Click on the **[Next >]** button.

6 Define a target folder for the program icon

- Choose or enter the program folder of the Start menu in which the program icon is to be inserted and confirm with **[Next >]**.

The program will be installed.

7 Complete the installation

- Click on the **[Finish]** button after the successfully completed installation.

The installation program will be exited.

3.4.2.3 Windows Vista

Caution

If you run the software 873 Biodiesel Rancimat on a computer with Windows Vista, you need a special update in order to be able to display the help. Due to licensing reasons you have to download this file from a Microsoft web page. On the installation CD the link "**Download WinHelp.url**" to the corresponding Microsoft web page can be found in the folder "**Vista Update for HLP help**". Download the necessary installation file and save it.

Proceed as follows to install the update:

1
- Exit 873 Biodiesel Rancimat if you have started the software after the installation.
- Start the installation file by double-clicking and follow the instructions of the installation program.

3.4.3 Carrying out basic settings

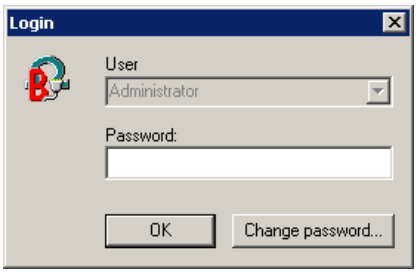
Setting the Administrator password

When starting the program for the first time, the Administrator password must be set. Proceed as follows:

1 **Switch on the instruments**
- Check whether the Biodiesel Rancimat is correctly connected to the PC *(see Chapter 3.4.1, page 19)*.
- Switch on the Biodiesel Rancimat using the mains switch.
- Switch on the PC.

2 **Start program**
- In the Windows Start menu under **Programs** ▸ **Metrohm** ▸ **Biodiesel Rancimat**, click on the menu item **Biodiesel Rancimat**.

The login window for the Administrator opens:

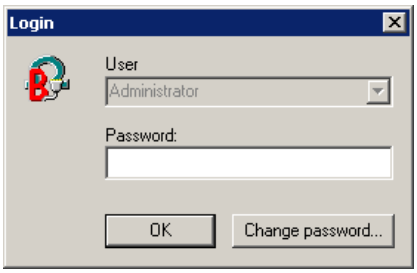


3 Set Administrator password

- Do **not** enter a password, instead click on **[OK]**.
- Confirm the displayed message with **[OK]**.
  The following dialog window appears:

- Leave the **Old password** field empty.
- Enter a new password for the Administrator in the **New password** field.
- Reenter the password in the **Confirm new password** field and confirm with **[OK]**.

The dialog window **Login** opens again:



4 Log in as Administrator

- Enter the Administrator password set before in the **Password** field and confirm with **[OK]**.
- Confirm the message **"There are no units configured yet!"** with **[OK]**.

Establishing the device communication

1 Follow the instructions in Section *4.2.1*.

4 Operation

This section describes the most important points for operation of the 873 Biodiesel Rancimat.

You can find more detailed information in the Online help of the software, which you can use to rapidly and easily call up the required information everywhere, via **[F1]**.

4.1 Fundamentals of operation

4.1.1 Starting and exiting the program

Proceed as follows to start and exit the program:

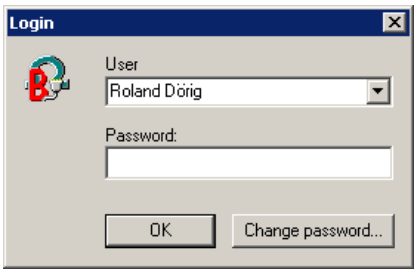
Note

If you are starting the program for the first time, you first have to set an Administrator password *(see Chapter 3.4.3, page 21)*.

1 Start program

- In the Windows Start menu under **Programs** ▸ **Metrohm** ▸ **Biodiesel Rancimat**, click on the menu item **Biodiesel Rancimat**.

The following dialog window appears:



2 Log in

- Select the required **User**.
- Enter the **Password** and confirm with **[OK]**.

3 Close program

- Click on the menu item **File** ▸ **Exit** *or* the symbol ✕ in the upper right corner of the dialog window.

For opening and closing the Results window, see *page 28*.

4.1.2 Terms

Control window — The main window **873 Biodiesel Rancimat Control** is called the control window. It contains all the functions for monitoring and controlling the Biodiesel Rancimats connected to the PC.

Method — A method comprises all parameters for carrying out and evaluating a determination.

Determination — Determination refers to the automatic determination of the induction time and/or stability time for a sample. In order to carry out a determination, a method suitable for the samples must be selected.

Results — The results of a determination are automatically saved in the database **Repos.mrd** and can be viewed in the **Results window**.

Reevaluation — Reevaluation refers to the subsequent revision of a determination, in particular the manual specification of the induction time using tangents.

Recalculation — The recalculation can be used to subsequently recalculate results, formulas and standards.

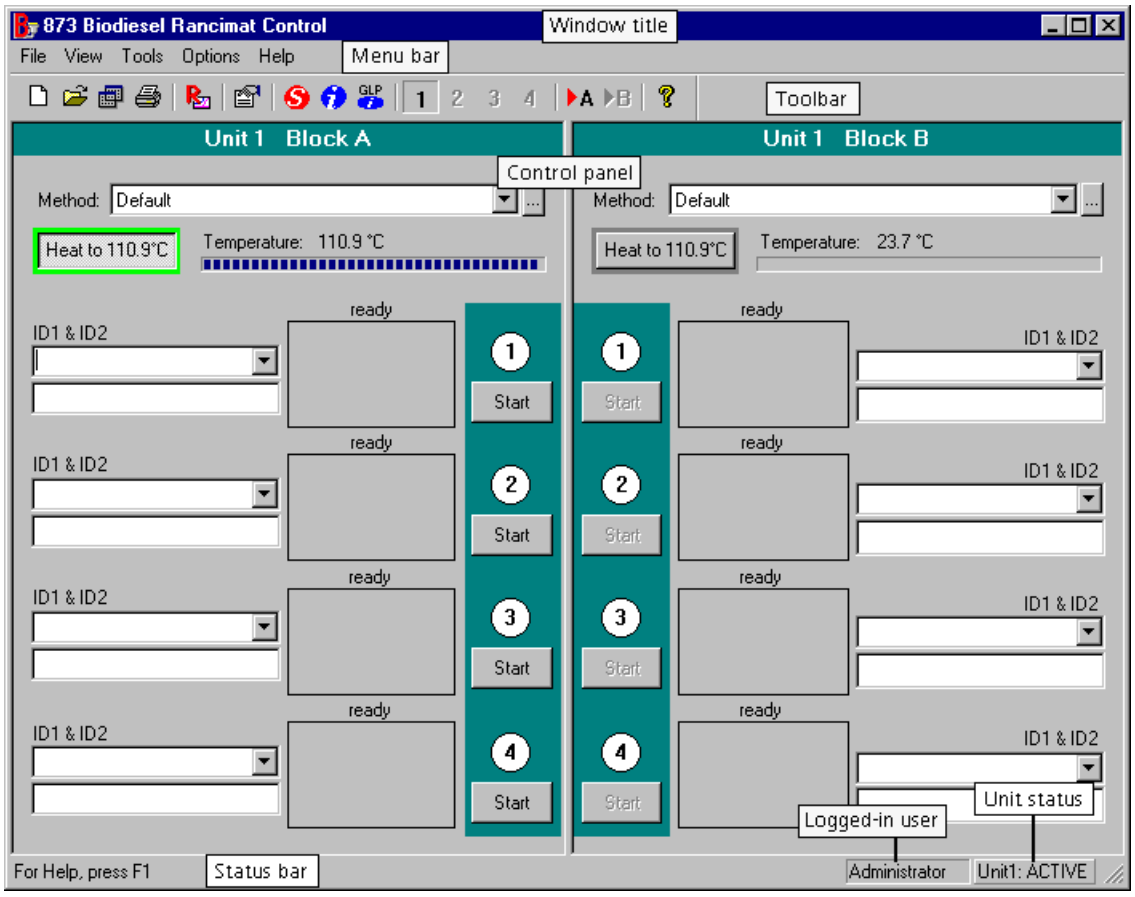
Extrapolation — Extrapolation can be used to convert the results measured at various temperatures to a required target temperature. In addition, the factor for converting the induction time to the standard time can be determined.

4.1.3 Control window

The dialog window **873 Biodiesel Rancimat Control** serves for controlling the devices, managing methods, displaying the live curves and accessing various program functions. The dialog window opens automatically when the program is started.

Structure

The elements of the Control window are the **menu bar**, the **toolbar**, the **control panel** and the **status bar**.



An image of the Biodiesel Rancimat is shown in the Control panel, which can be used for starting, displaying and stopping determinations.

The following information is shown in the status bar:

- a short description of the menu item highlighted in the menu bar
- the registered user
- the instrument status

Menus

The Control window contains the following main menus:

File — Create new methods, open existing methods, manage methods, open Results window, print, new login, close program.

View — Activate/Deactivate toolbar and status bar, display instrument information, change live parameters, display status overview, display event logging, select instrument.

Tools — Manage cell constants, perform GLP tests, set timer, set gas flow control, start all channels, determine Delta T, record temperature, perform service diagnosis.

Options	Carry out general settings, set instrument configuration, manage access rights.
Help	Call up program-specific Online help.

Symbols

New method	Create a new method *(see Chapter 4.5.1, page 55)*.
Open method	Open an existing method *(see Chapter 4.5.1, page 55)*.
Method Manager	Open, rename and delete methods *(see Chapter 4.5.1, page 55)*.
Print	Print results *(see Chapter 4.7.6, page 124)*.
Results	Open Results window *(see "Opening and closing the Results window", page 28)*.
Live parameters	Display live parameters which can be changed for a current determination *(see Chapter 4.6.5, page 81)*.
Status overview	Display status overview for the instruments connected *(see Chapter 4.3.2, page 44)*.
Instrument information	Instrument information *(see Chapter 4.3.1, page 42)*.
GLP status	Display GLP status *(see Chapter 4.8.3, page 140)*.
Instrument 1…4	Select instrument 1…4.
Start all channels Block A	Start all channels of Block A *(see Chapter 4.6.3, page 79)*.
Start all channels Block B	Start all channels of Block B *(see Chapter 4.6.3, page 79)*.
Help topics	Call up the Online help.

4.1.4 Results window

The dialog window **873 Biodiesel Rancimat Results** serves for displaying, outputting and recalculating results of the determinations recorded with the 873 Biodiesel Rancimat. The determination data is saved in database files ***.mrd** and can be displayed in this dialog window in the form of overview tables and curves. The Results window can also run if the Control window is closed.

Opening and closing the Results window

Proceed as follows to open and close the Results window:

1 Open Results window

- In the dialog window **873 Biodiesel Rancimat Control**, click on the menu item **File ▸ Results...** *or* the symbol .

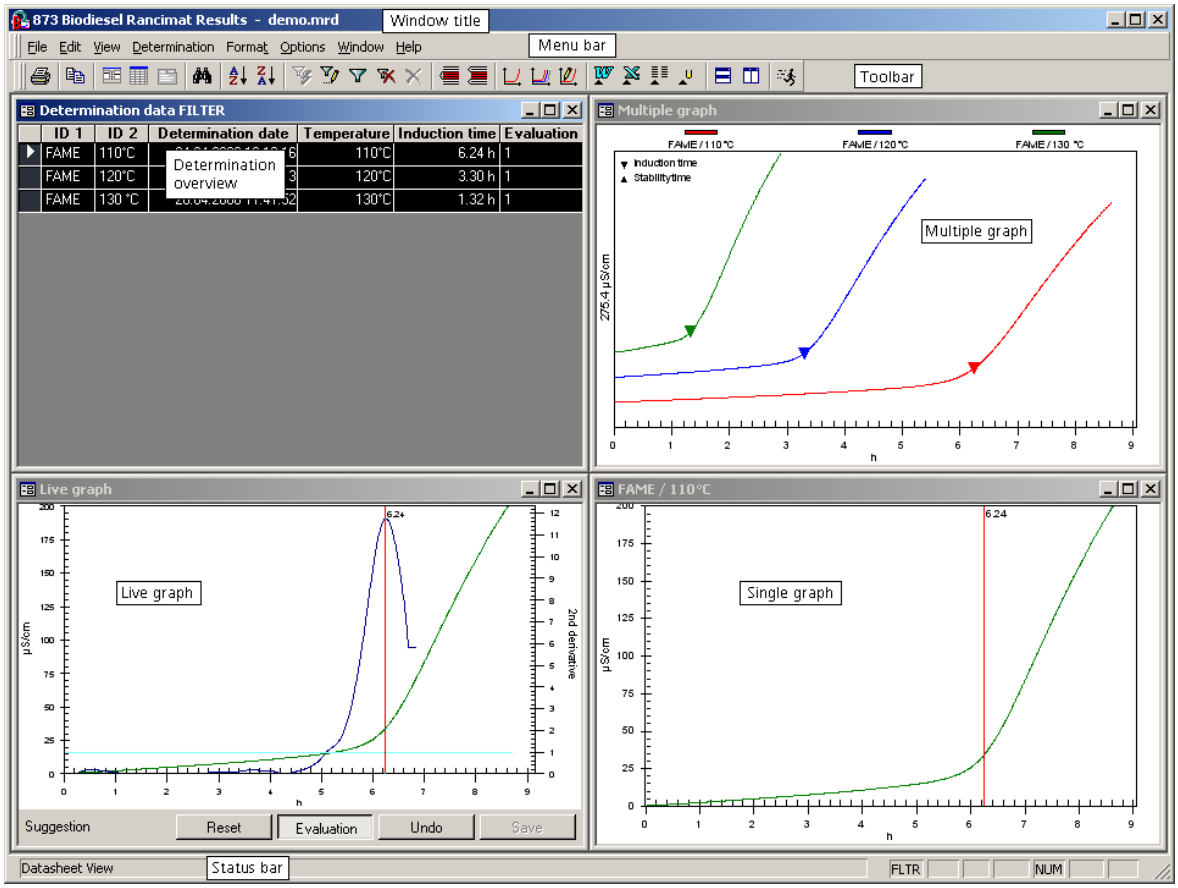
When opening the Results window, the database **Repos.mrd** is automatically loaded, in which all recorded determinations are saved by default.

2 Close Results window

- In the dialog window **873 Biodiesel Rancimat Results**, click on the menu item **File ▸ Back** *or* the symbol .

Structure

The elements of the Results window are the **menu bar**, the **toolbar**, the **subwindows** (determination overview, live, single and multiple graphs) and the **status bar**.



In the Results window subwindows with a determination overview, single, multiple or live graphs can be opened.

Menus

The Results window contains the following main menus:

File	Open database, print, close dialog window.
Edit	Copy, select, delete content of the filter.
View	View selection: determination overview, determination and method data, GLP.
Determination	Search, sort, filter, display graphs, perform extrapolation, perform recalculation, export determinations, delete determinations.
Format	Format determination overview.
Options	Carry out general program settings.
Window	Arrange subwindows.
Help	Display program-specific Online help.

Symbols

The toolbar of the Results window contains the following symbols:

Print	Print results, curves and overview lists *(see Chapter 4.7.6, page 124)*.
Copy	Copy data to clipboard.
Select fields for determination overview	Format determination overview *(see Chapter 4.7.1, page 83)*.
Display determination overview	Display determination overview *(see Chapter 4.7.1, page 83)*.
Display all methods and determination data	Display all methods and determination data of the selected determination *(see Chapter 4.7.2, page 99)*.
Search	Search in the database *(see Chapter 4.7.1, page 83)*.
Ascending	Sort column in ascending order *(see Chapter 4.7.1, page 83)*.
Descending	Sort column in descending order *(see Chapter 4.7.1, page 83)*.
Selection-based filter	Filter by selection *(see Chapter 4.7.1, page 83)*.

Special filter	Define filter *(see Chapter 4.7.1, page 83)*.
Apply filter	Apply filter *(see Chapter 4.7.1, page 83)*.
Remove filter	Remove filter *(see Chapter 4.7.1, page 83)*.
Delete filter	Delete content of the filter *(see Chapter 4.7.1, page 83)*.
Filter selection	Only display selected determinations *(see Chapter 4.7.1, page 83)*.
Selection not in filter	Hide selected determinations *(see Chapter 4.7.1, page 83)*.
Single graph	Display single graph *(see Chapter 4.7.3, page 108)*.
Multiple graph	Display multiple graph *(see Chapter 4.7.3, page 108)*.
Live graph with reevaluation	Reevaluate live graph *(see Chapter 4.7.3, page 108)*.
Export to Word	Export Determination overview to Word *(see Chapter 4.7.6, page 124)*.
Export to Excel	Export Determination overview to Excel *(see Chapter 4.7.6, page 124)*.
Export measured values	Export measured values to a TXT file *(see Chapter 4.7.6, page 124)*.
Export determination and method data	Export determination and method data to a TXT file *(see Chapter 4.7.6, page 124)*.
Tile vertically	Arrange subwindows on top of each other *(see Chapter 4.7.8, page 137)*.
Tile horizontally	Arrange subwindows beside each other *(see Chapter 4.7.8, page 137)*.
Return to main program	Close Results window and return to main program.

4.1.5 File types

The following file types are generated by the program 873 Biodiesel Rancimat:

***.mrd** **Database file**

This file contains the measured data and results of the determinations. The file **repos.mrd** is automatically saved in the directory **Database**. When exporting databases, the memory location can be freely selected.

***.mel** **Event file**

This file contains a protocol of all events which have occurred with the connected Biodiesel Rancimats. The file ***.mel** is automatically saved in the **Log** directory.

***.txt** **Text file**

Measured values, determination and method data as well as the data of the temperature log can be saved in ASCII format as a **TXT file**. The memory location can be freely selected, except for the temperature log. This file is saved in the **Log** directory.

4.1.6 Context-sensitive menus

Many menu functions of the Program window can also be selected by clicking with the right mouse button on the required dialog window or element. The menu options opened depend on the active dialog window or element selected.

4.1.7 Mouse functions

The standard functions for program control such as selecting menu items and fields can be executed using the mouse.

It also serves for magnifying a curve sector (Zooming).

Zooming

Proceed as follows:

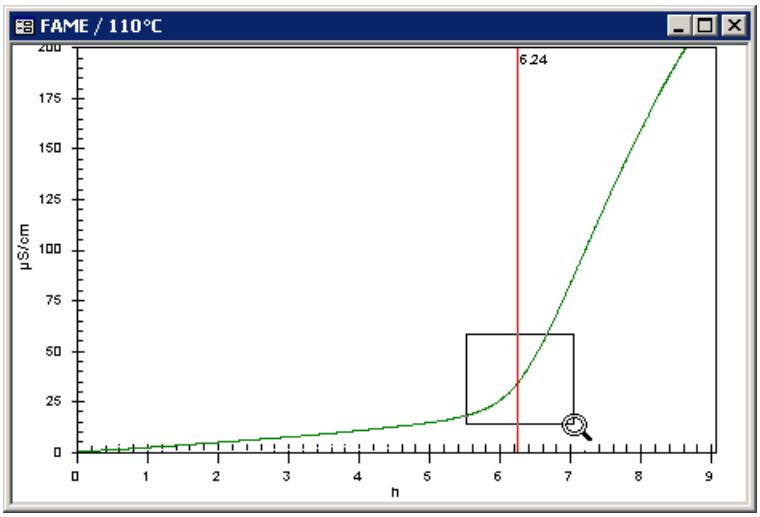
1 Drag zoom square

- Place the cursor on the upper left corner of the sector to be magnified.
- Keeping the left mouse button pressed down, pull the cursor to the lower right corner of the sector.

The cursor assumes the form of a magnifying glass.

2 Release mouse button

- Release the mouse button in order to magnify the selected area to the full size of the window.



3 Switch zoom off again

- Right click in the graphs window.
  The context-sensitive menu for graphs appears.
- Click on the menu item **Zoom Off**.

4.1.8 Help

You can call up help for the current topic anywhere with the symbol , the menu item **Help ▸ Help topics** or the key **[F1]**.

Green text	can be clicked on. This takes you to another help topic.
Violet text	marks menu items, parameters or buttons in the program.
Blue text	marks titles and important information.

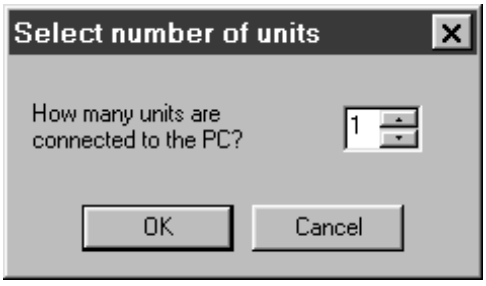
4.2 Instrument and Program Settings

4.2.1 Establishing the instrument communication

Up to four instruments can be controlled with the program **873 Biodiesel Rancimat**. Configure the communication between your PC and the 873 Biodiesel Rancimat as follows:

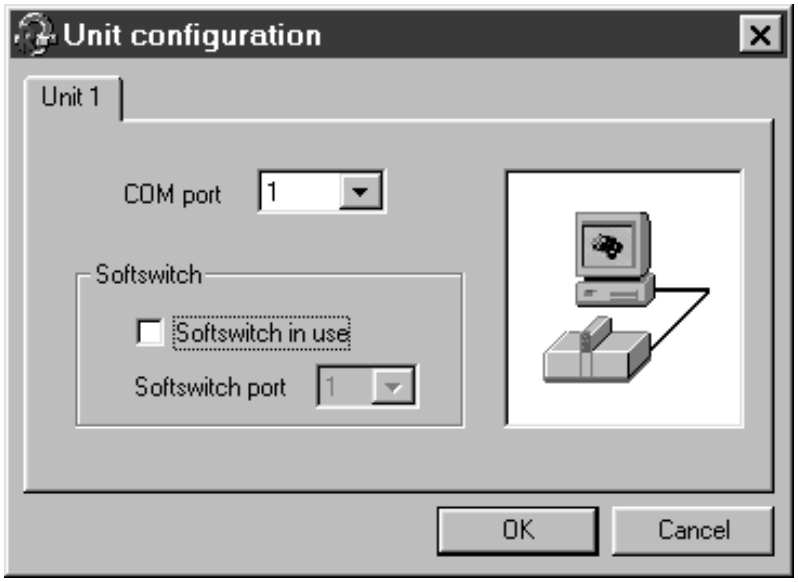
1 Select number of instruments connected

- In the **Control window**, click on the menu item **Options ▸ Communication...**.
  The following dialog window appears:



- Enter the number of connected instruments and confirm with **[OK]**.

The following dialog window appears:



2 Select interface

- Under **COM port**, select the serial interface of the PC to which the instrument is connected.

- If a softswitch is used, activate the option **Softswitch in use** and select the serial interface of the softswitch to which the instrument is connected under **Softswitch port**.
- Confirm the entry with **[OK]**.

3 Restart program

- Close the program with the menu item **File ▸ Exit**.
- In the Windows Start menu under **Programs ▸ Metrohm ▸ Biodiesel Rancimat**, click on the menu item **Biodiesel Rancimat**.
- Enter the Administrator password and confirm with **[OK]**.

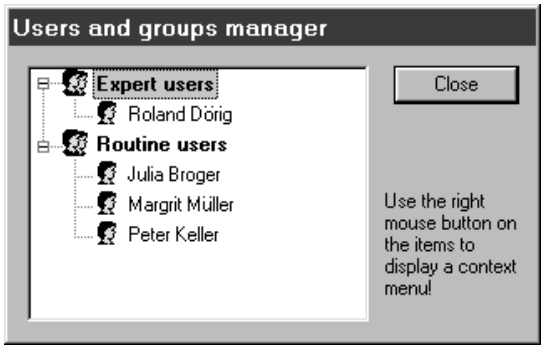
The program is opened. The message **Instr.#: Ready** now appears in the status bar for all connected instruments.

4.2.2 Managing access rights

The **873 Biodiesel Rancimat** program offers extensive password protection, allowing each menu item to be provided with individual access rights.

The dialog **Users and groups manager**, used to manage the access rights, is only accessible to the Administrator. The Administrator himself is not listed as a user, but has all access rights.

First of all, the access rights are given to different **Groups**. Then, individual **Users** can be assigned to a specific group.



Note

User lists are best created and passwords are best entered immediately after the first program start.

The setup for the users and their access rights is carried out in the following order:

1. Add a new group
2. Adjust rights for this group
3. Create users in this group

After having entered the access rights, the Login window for selecting the user and entering the password appears each time the program is started. All methods, determinations and reports are marked with the user name. It is possible to change the user at any time with the menu item **File ▸ New login...**.

Adding a new group

In order to add a new group, proceed as follows:

1 Open Users and groups manager

- In the **Control window**, click on the menu item **Options ▸ User permissions...**.

2 Create new group

- Right click on an existing group to select the menu item **Add group...**.
- Under **Enter new group name** enter the name for the group and confirm with **[OK]**.

Adjusting access rights for the group

Proceed as follows to set the access rights:

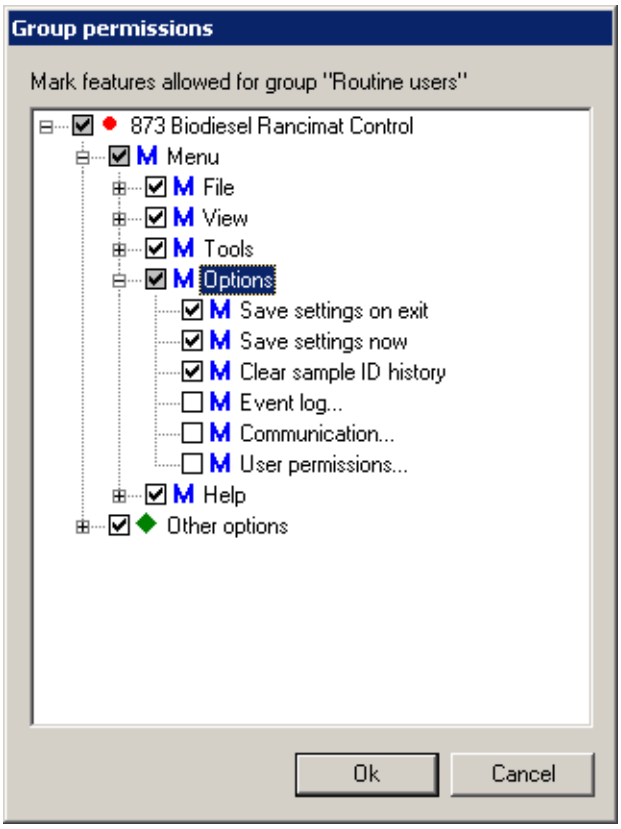
1 Open Users and groups manager

- In the **Control window**, click on the menu item **Options ▸ User permissions...**.

2 Open Properties dialog

- Right click on the relevant group whose access rights are to be adjusted to select the menu item **Properties...**.

The following dialog window appears:



3 Activate/Deactivate the rights

- Click on the symbol ⊞ to open the menu tree for the access rights..
- Activate or deactivate the required options by clicking on the symbol ☑.
- Confirm the changes with **[OK]**.

Adding a new user

Proceed as follows to add a new user:

1 Open Users and groups manager

- In the **Control window**, click on the menu item **Options ▸ User permissions...**.

2 Create new user

- Right click on an existing group to select the menu item **Add user...**.
- Under **Enter new user name** enter the name for the user and confirm with **[OK]**.

Deleting a user

Proceed as follows to delete an existing user:

1 Open Users and groups manager

- In the **Control window**, click on the menu item **Options ▸ User permissions...**.

2 Delete user

- Right click on the user to be deleted to activate the menu item **Delete user**.
- Confirm the message **Do you really want to delete user "user name"?** with **[Yes]**.

Deleting a group

Proceed as follows to delete an existing group:

1 Open Users and groups manager

- In the **Control window**, click on the menu item **Options ▸ User permissions...**.

2 Delete group

- Right click on the group to be deleted in order to select the menu item **Delete group**.
- Confirm the message **Do you really want to delete group "group name"?** with **[Yes]**.

4.2.3 Timer

The function **Timer** can be used to start the two heating blocks A and B, independently of one another, automatically and at a time specified by you. The Biodiesel Rancimat has to be switched on and the software must be running.

Two different examples will explain how to program the timer.

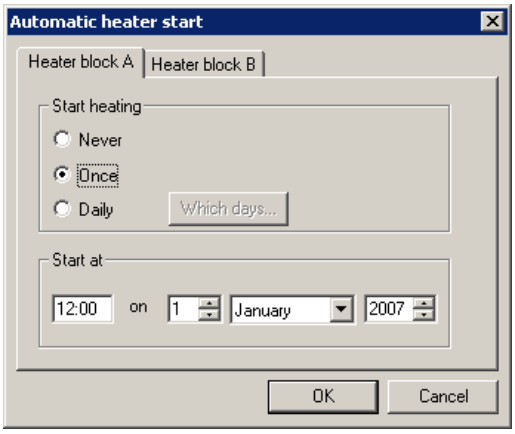
Starting heating block A once automatically

Proceed as follows to automatically start the heating of block A at a specific time:

1 Open dialog window

- In the **Control window**, click on the menu item **Tools ▸ Timer function...**.

The following dialog window appears:



2 Set automatic start time

- Under **Start heating** select the option **Once**.
- Under **Start at...** enter the required time and date.
- Confirm the entries with **[OK]**.

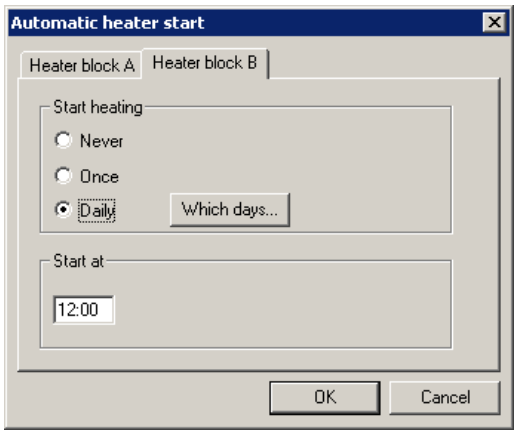
Starting heating block B daily automatically at the same time

Proceed as follows to automatically start the heating of block B on a specific day of the week, at a specific time:

1 Open dialog window

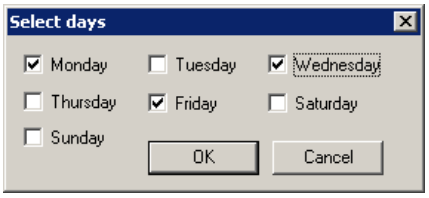
- In the **Control window**, click on the menu item **Tools ▸ Timer function...**.

The following dialog window appears:



2 Set automatic start time

- Under **Start heating** select the option **Daily**.
- Click on the button **[Which days...]**.
  The following dialog window appears:



- Select the required day(s) and confirm with **[OK]**.
- Under **Start at...** enter the required time and confirm with **[OK]**.

4.2.4 Gas flow control

The gas flow produced by the **internal pump** through the reaction vessels to the measuring vessels can be switched on and off manually and displayed in a separate window.

Switching the gas flow on and off

Proceed as follows:

1 Switch on

- Click on the menu item **Tools ▸ Gas flow control ▸ Gas flow on**.

2 Switch off

- Click on the menu item **Tools ▸ Gas flow control ▸ Gas flow off**.

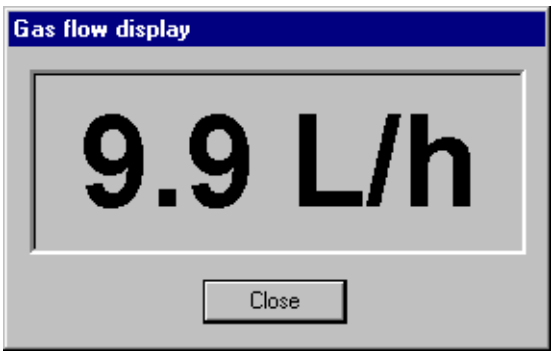
Displaying the gas flow

Proceed as follows:

1 Open dialog window

- Click on the menu item **Tools ▸ Gas flow control ▸ Display gas flow...**.

The following dialog window appears:



Note

This option is only available if no determination is running.

4.2.5 Recording the temperature

The temperature can be recorded at any time for each individual heating block.

The program writes the recorded measured values into a text file containing the following values:

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

The text file is saved under the name **U#X-YYMMDD-hhmmss.txt** in the folder **Log**, # denoting the instrument number (1...4), X denoting the block (A or B) and YYMMDD-hhmmss denoting the date and time when the data logging started.

Switching the temperature log on and off

Proceed as follows:

1 Switch on the recording for block A or B

- Click on the menu item **Tools ▸ Temp. logging ▸ Block A on**.

or

- Click on the menu item **Tools ▸ Temp. logging ▸ Block B on**.

2 Switch off the recording for block A or B

- Click on the menu item **Tools ▸ Temp. logging ▸ Block A off**.

or

- Click on the menu item **Tools ▸ Temp. logging ▸ Block B off**.

4.2.6 Optimizing program and database

The data which the program **Biodiesel Rancimat** requires for displaying the Results window is saved in the file **Nachaus.prg**. With the standard installation, this file is located at C:\Program Files\Metrohm\Biodiesel Rancimat\Template. As soon as the file is **greater than 10 MB**, you should optimize and compress the program, so that this file no longer has any superfluous data and the program runs faster again.

The **file size of the database Repos.mrd** can make access to existing determinations considerably slower. If you regularly work with the program and carry out a large amount of determinations, you should optimize the standard database **Repos.mrd** once a month.

Optimizing the program

Proceed as follows to optimize the program:

1 Quit program

- Close the program **Biodiesel Rancimat** *(see Chapter 4.1.1, page 24)*.

2 Optimize program

- In the windows Start menu, click on the menu item **Programs ▸ Metrohm ▸ Biodiesel Rancimat ▸ Optimize ▸ Program**.

Optimizing the database

Proceed as follows to optimize the standard database:

1 Export database

- Export determinations not currently required from the database **Repos.mrd** into a newly created database *(see "Exporting determinations to another database", page 129)*.
- Close the Results window and the program **Biodiesel Rancimat** *(see Chapter 4.1.1, page 24)*.

2 Optimize database

- In the Windows Start menu, click on the menu item **Programs ▸ Metrohm ▸ Biodiesel Rancimat ▸ Optimize ▸ Database**.

4.3 Program information

4.3.1 Instrument information

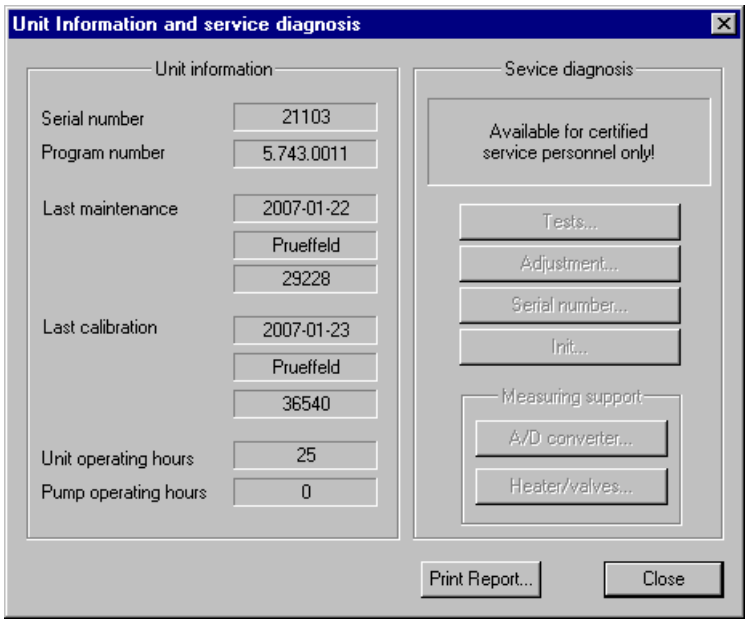
Displaying and printing the instrument information

Proceed as follows to display and print the instrument information of the currently selected Biodiesel Rancimat:

1 Open dialog window

- In the **Control window**, click on the menu item **View ▸ Unit information…**.

The following dialog window appears:



2 Print instrument information

- Click on the button **[Print report…]**.

The status report is printed on the printer which is defined on your PC as the default printer.

Meaning of the individual parameters

Serial number

Serial number of the selected instrument.

Program number

Number of the EEPROM program of the selected instrument.

Last maintenance

Date of the last maintenance with signature of the service technician who has carried out the service work.

Last calibration

Date of the last instrument adjustment with signature and code of the responsible body.

Unit operating hours

Number of hours during which the instrument was switched on.

Pump operating hours

Number of hours during which the internal pump was in operation.

Note

The block **Service diagnosis** is password-protected and only accessible for trained service personnel.

4.3.2 Status overview

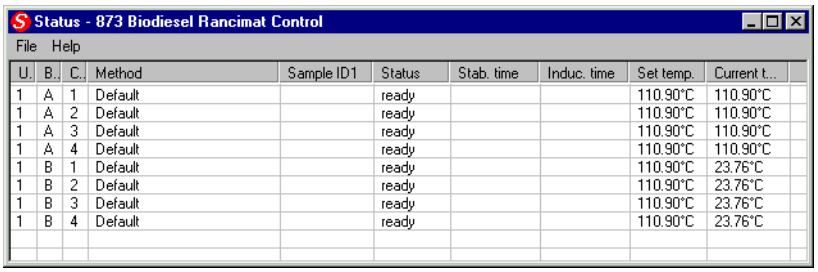
Displaying the status overview

Proceed as follows to display and adjust the status overview:

1 Open dialog window

- In the **Control window**, click on the menu item **View ▸ Status overview**.

The following dialog window appears:



U.	B.	C.	Method	Sample ID1	Status	Stab. time	Induc. time	Set temp.	Current t...
1	A	1	Default		ready			110.90°C	110.90°C
1	A	2	Default		ready			110.90°C	110.90°C
1	A	3	Default		ready			110.90°C	110.90°C
1	A	4	Default		ready			110.90°C	110.90°C
1	B	1	Default		ready			110.90°C	23.76°C
1	B	2	Default		ready			110.90°C	23.76°C
1	B	3	Default		ready			110.90°C	23.76°C
1	B	4	Default		ready			110.90°C	23.76°C

2 Adjust and save column width

- You can adjust the column width to the required size by dragging the right field border with the mouse.
- In the **Control window**, click on the menu item **Options ▸ Save settings now**.

Meaning of the columns in the status overview

Unit

Instrument number (1…4).

Block

Instrument block (A or B).

Channel

Channel (1…4).

Method

Name of the method loaded.

Sample ID1

Sample identification 1.

Status

Status of the channel.

ready: No active measurement. The channel is ready for starting a determination.

running: Instrument is measuring.

finished: Determination finished. The channel is ready for starting a new determination.

error: Communication error between instrument and PC.

Stab. time

Stability time determined.

Induc. time

Induction time determined.

Set temp.

Setpoint temperature (defined in the method).

Current temp.

Current block temperature.

4.3.3 Displaying, filtering and deleting event overview

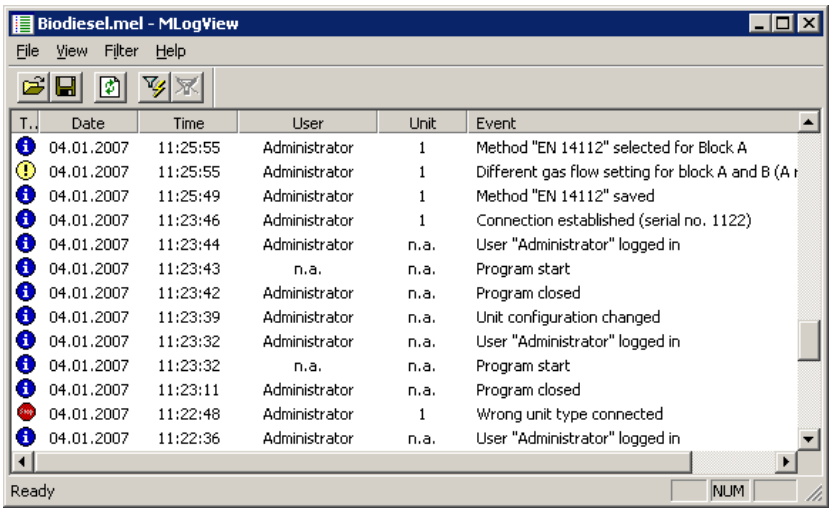
Opening and saving the event log file

Proceed as follows to display and save all events of the Biodiesel Rancimats connected to the PC:

1 Open current log file

- In the **Control window**, click on the menu item **View ▸ Event log overview**.

The following dialog window appears:



Note

You can find the meaning of the symbols and columns in the event overview at *"Meaning of the symbols and columns in the event overview", page 47*.

2 Update view

- Click on the symbol *or* the menu item **View ▸ Refresh** to view the current status of the log file.

3 Save log file

- Click on the symbol *or* the menu item **File ▸ Save as…**.
- In the dialog window **Save file as** enter a **file name** and confirm with **[OK]**.

4 Open saved log file

- Click on the symbol *or* the menu item **File ▸ Open…**.
- In the dialog window **Open**, select the required file and confirm with **[OK]**.

Filtering entries in the event overview

Proceed as follows to filter the entries in the event file:

1 Filter the entries

- Click on the required filter criterion (e.g.: date = "30.08.2006").

- Click on the symbol *or* the menu item **Filter ▸ Selection based filter**.

Only entries with the date "30.08.2006" are displayed in the dialog window.

2 Remove the filter again

- Click on the symbol *or* the menu item **Filter ▸ Remove filter**.

All entries are once again displayed in the dialog window.

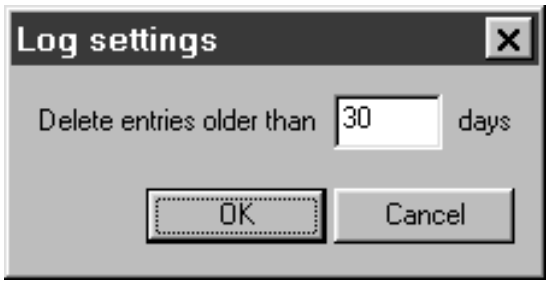
Deleting entries from the event overview

Proceed as follows to delete certain entries from the event file:

1 Open dialog window

- In the **Control window**, click on the menu item **Options ▸ Event log…**.

The following dialog window appears:



2 Delete entries

- Enter the maximum age in days, which the entries are allowed to have in the list. All older entries are deleted.
- Confirm the entry with **[OK]**.

Meaning of the symbols and columns in the event overview

Type

- Information on correctly executed events.

- Indication of a special event.

- Error message for error event.

Date

Date of the event. The format depends on the settings defined in Windows in **Control Panel ▸ Country settings ▸ Date**.

Time

Time of the event. The format depends on the settings defined in Windows in **Control Panel ▸ Country settings ▸ Time**.

User

Name of the user logged in at the time of the event.

Unit

Instrument number (1…4).

Event

Description of the event.

4.4 Calibration functions

4.4.1 Determining cell constants

Only the **change in conductivity** is measured an evaluated during the Rancimat measurement. As a result, the cell constants of the conductivity measuring cells used in the Biodiesel Rancimat, which are normally in the range from 1.00...1.20, only have to be entered exactly if the **absolute conductivity measured value** is to be displayed correctly.

The value also has to be entered before carrying out the **GLP test "Conductivity"**. The cell constants can thereby be entered manually or determined automatically using a defined standard solution *(see Optional accessories, page 180)*.

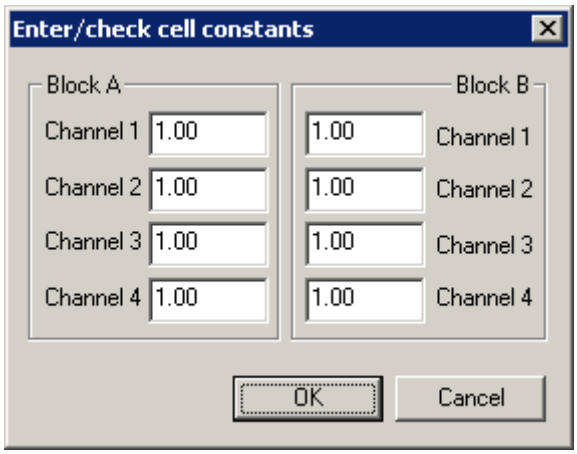
Entering cell constants manually

Proceed as follows if you wish to enter the cell constants manually:

1 Open dialog window

- Click on the menu item **Tools ▸ Cell constants ▸ Manual...**.

The following dialog window appears:



2 Enter cell constants

- Enter the cell constants of the conductivity measuring cells for the required channels and confirm with **[OK]**.

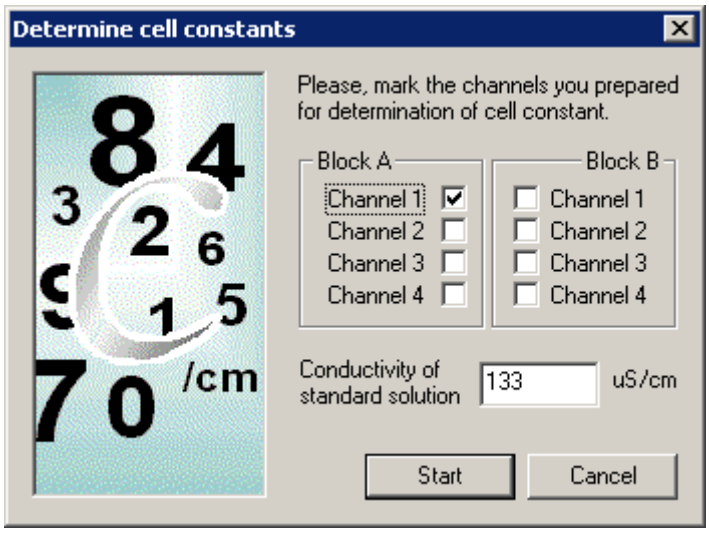
Determining cell constants automatically

Proceed as follows if you wish to enter the cell constants automatically:

1 Open dialog window

- Click on the menu item **Tools ▸ Cell constants ▸ Automatic...**.

The following dialog window appears:



2 Select channels and define conductivity

- Activate the check boxes of those channels, for which the cell constant is to be determined.

- Enter the conductivity of the standard used.

c(KCl) = 1 mmol/L can be used as a standard solution. This is derived from the optionally available conductivity standard 6.2301.060 (KCl 0.1 mol/L) through dilution with distilled water. The conductivity of this diluted 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

Alternatively, the conductivity standard 6.2324.000 (undiluted) can be used.

3 Start determination and check the values

- Click on **[Start]** to start the determination of the cell constant.

After the determination is finished, the dialog window **Enter/Check cell constants** appears again.

If you wish to accept the displayed values, click **[OK]**.

If you wish to maintain the old values, click **[Cancel]**.

4.4.2 Determining Delta T

The **temperature correction Delta T** indicates the deviation of the current temperature of the sample from the temperature in the heating block and belongs to the method as a parameter *(see Chapter 4.5.2, page 60)*. It can automatically be determined with the calibrated, external temperature sensor (part of the optionally available GLP test set, *see Optional accessories, page 180*).

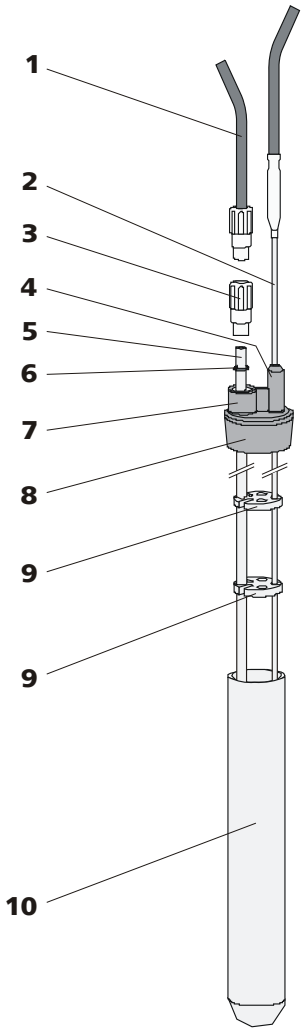


Figure 6 Equipping the reaction vessel for determining Delta T

1	**FEP tubing 250 mm (6.1805.080)** For supplying air into the reaction vessel.	**2**	**Pt100 temperature sensor**
3	**Thread adapter M8 / M6 (6.1808.090)**	**4**	**Opening for temperature sensor** For inserting the temperature sensor.
5	**Air tube**	**6**	**O-ring (6.1454.040)**
7	**Connector** For connecting the thread adapter M8 / M6.	**8**	**Reaction vessel cover (6.2753.107)**
9	**Spacer (6.2042.040)**	**10**	**Reaction vessel (6.1429.040)**

Preparing Delta T determination

Figure 6 shows in detail how the accessories for determining Delta T are mounted. Proceed as follows:

1 Prepare reaction vessel cover

- Mount the air tube on the reaction vessel cover *(see "Mount the reaction vessel cover", page 15)*.
- Clamp the first spacer at a distance of approx. 12 cm from the lower end onto the air tube.
- Clamp the second spacer at a distance of approx. 5 cm from the lower end onto the air tube.
- Insert the temperature sensor from above into the temperature sensor opening of the reaction vessel cover and fasten it in the corresponding openings of the spacers.

2 Prepare reaction vessel

- Fill the reaction vessel with 5 g silicone oil (e.g. FLUKA 85409).
- Place the reaction vessel cover with the temperature sensor on the reaction vessel.
- Push the temperature sensor fully downwards (the sensor must be on the vessel base).

3 Insert and connect reaction vessel

- Insert the reaction vessel with the mounted reaction vessel cover in channel 2 or 3 of the required heating block.
- Screw the one end of the FEP tubing 250 mm onto the thread adapter M8 / M6 of the reaction vessel cover.
- Screw the other end of the FEP tubing onto the corresponding connector *(3-**3**)* of the Biodiesel Rancimat.

4 Set method parameters

Note

Create a unique method for both blocks, so that you can separately enter the temperature correction for both blocks.

- In the **Control window**, select the required **method** for the heating block for which the temperature correction Delta T is to be determined *(see "Select method", page 77)*.

- Click on the symbol [...].
  The dialog window with the method parameters is opened.
- Under **Temperature** enter the required setpoint temperature for the sample and set **Delta T** to 0 °C.
- Set the **gas flow** to the required value and finalize with **[Save]**.

5 Start gas flow and heating

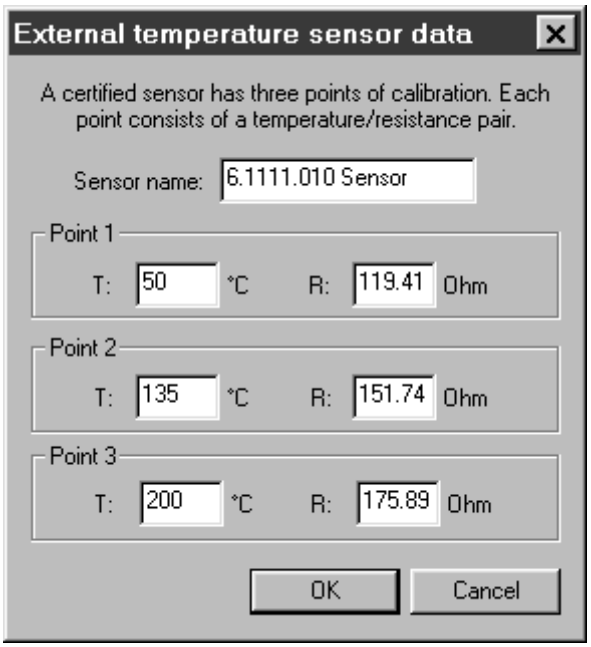
- Click on the menu item **Tools ▸ Gas flow control ▸ Gas flow on**.
- Click on the button **[Heat to xx °C]**.

Executing a Delta T determination

After you have prepared everything for determining Delta T *(see "Preparing Delta T determination", page 52)*, you can now execute the determination of Delta T as follows:

1 Enter sensor calibration data

- Click on the menu item **Tools ▸ Determine Delta T ▸ Block A** or **Block B**.
  The following dialog window appears:

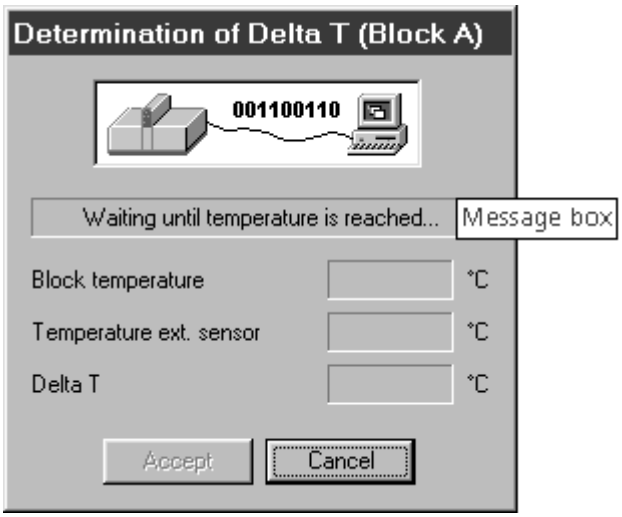


- Enter a **Sensor name**.
- Enter the calibration data (temperature and resistance) for **Points 1** to **3** and confirm with **[OK]**.

Note

You can find the calibration data (T1 to T3) for Metrohm sensors on the certificate enclosed with the sensor or on the label fastened on the sensor cable.

The following dialog window appears:



2 Wait for measurement

- Wait until the message **Measured temperature is now stable!** appears in the message box.

The following three values are displayed below the message box:

Block temperature	Temperature which has been measured in the heating block with the internal temperature sensor.
Temperature of ext. sensor	Temperature which has been measured in the reaction vessel with the external temperature sensor.
Delta T	Temperature correction which is determined from the difference between the block temperature and the temperature of the ext. sensor.

Note

If the temperature which is defined in the method, has not yet been reached, the heating is started automatically. At the same time the message **Waiting until temperature is reached...** appears in the message box.

As soon as the temperature defined in the method has been reached, the message **Measuring temperature. Please wait...** appears in the message box.

3 Accept measured values

- Click on the **[Accept]** button in order to accept the determined temperature correction Delta T in the method.

This is followed by the message that Delta T is now set to the corresponding method. It is automatically saved with the changed value of Delta T.

4.5 Methods

4.5.1 Managing methods

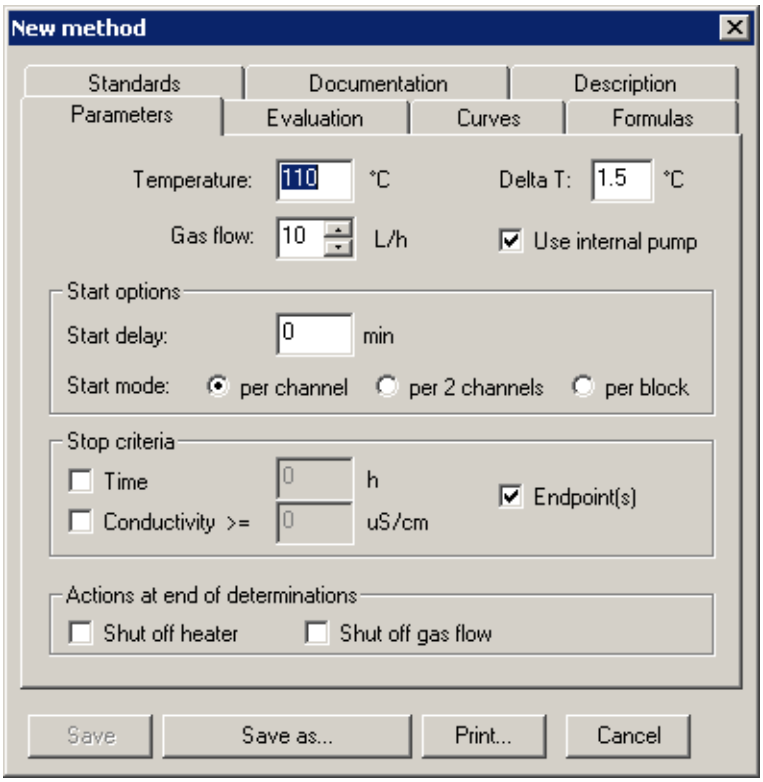
Creating a new method

Proceed as follows to create a new method:

1 Open empty method

- In the **Control window**, click on the symbol  *or* the menu item **File ▸ New method**.

The following dialog window appears:



2 Adjust parameters

- Adjust the required parameters.
- Click on the tab **Evaluation** and also adjust the parameters.
- Repeat for all remaining tabs.

Note

You can find a detailed description of all tabs and their parameters at *4.5.2 Parameter description*.

3 Save method

- Click on the **[Save as...]** button.
  The dialog window **Save method as...** opens.
- Enter the method name and confirm with **[Save]**.

Opening a selected method and adjusting parameters

Proceed as follows to open the method which is selected in the **Control window** and adjust its parameters:

1 Open method

- In the **Control window**, click on the symbol  in the required **Block A** or **B**.
  The dialog window **"Method name"** appears.

2 Adjust parameters and save changes

- Adjust the parameters *(see Chapter 4.5.2, page 60)*.
- Confirm the changes with **[Save]**.

Opening an existing method and adjusting parameters

Proceed as follows to open an existing method and adjust its parameters:

1 Open existing method

- In the **Control window**, click on the symbol  *or* the menu item **File ▸ Open method...**.
  The dialog window **Open method** appears.
- Click on the required method and confirm the selection with **[Open]**.

The dialog window **"Method name"** appears.

2 Adjust parameters and save changes

- Adjust the parameters *(see Chapter 4.5.2, page 60)*.
- Confirm the changes with **[Save]**.

Renaming an existing method

Proceed as follows to rename an existing method:

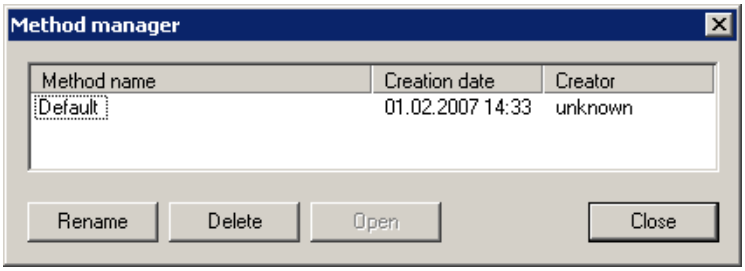
Note

A method which is still selected in one of the blocks A or B, cannot be renamed. Select another method first in order to rename the required method.

1 Open Method manager

- In the **Control window**, click on the symbol *or* the menu item **File ▸ Method manager...**.

The following dialog window appears:



2 Select and rename method

- Click on the required method and then the button **[Rename]**. The method name is highlighted in blue.
- Enter the new method name and click with the mouse on the free white area below the method name in order to accept the change.
- Click on the **[Close]** button.

Saving a method under a new name

Proceed as follows to save an existing method under a new name:

1 Open method

- In the **Control window**, click on the symbol *or* the menu item **File ▸ Open method...**.
  The dialog window **Open method** appears.
- Click on the required method and confirm the selection with **[Open]**.

The dialog window **"Method name"** appears.

2 Save it with a new name

- If necessary, adjust the parameters *(see Chapter 4.5.2, page 60)*.
- Click on the **[Save as...]** button.
  The dialog window **Save method as...** appears.
- Enter the method name and confirm with **[Save]**.

Deleting an existing method

Proceed as follows to delete an existing method:

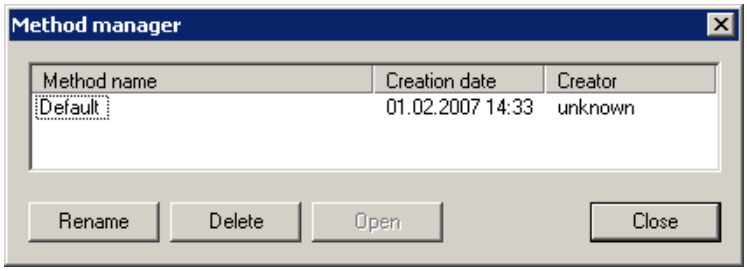
Note

A method which is still selected in one of the blocks A or B, cannot be deleted. Select another method first in order to delete the required method.

1 Open Method manager

- In the **Control window**, click on the symbol  *or* the menu item **File ▸ Method manager...**.

The following dialog window appears:



2 Select and delete method

- Click on the required method and then the button **[Delete]**.
  The safety prompt **"Do you really want to delete method "method name"?"** appears.
- Confirm the safety prompt with **[Yes]**.

Printing parameters of a method

Proceed as follows to print the parameters of an existing method:

1 Open method

- In the **Control window**, click on the symbol  *or* the menu item **File ▸ Open method...**.
  The dialog window **Open method** appears.
- Click on the required method and confirm the selection with **[Open]**.

The dialog window **"Method name"** appears.

2 Print parameters

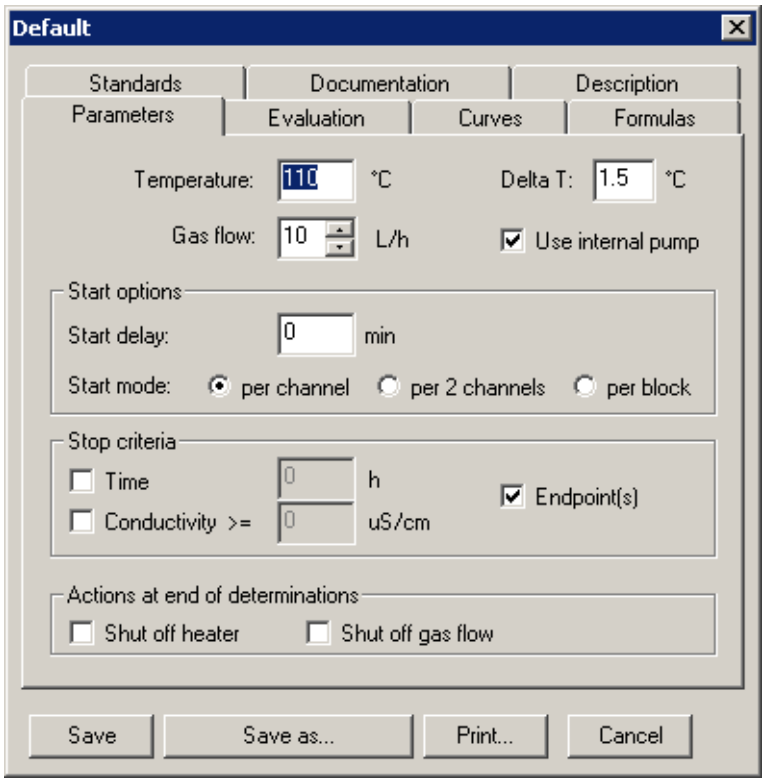
- Click on the **[Print...]** button.
  The dialog window **Print Setup** opens. All the printers installed on your PC appear under **Name**.
- Select the required printer and confirm with **[OK]**.

4.5.2 Parameter description

A method contains several parameters which are distributed on various tabs. This section explains all the parameters, arranged according to tab.

4.5.2.1 "Parameters" tab

The most important parameters (temperature, gas flow, Delta T) for carrying out the measurement are set using this tab. The settings affect the progression of the measurement curve recorded.



Temperature

Setpoint temperature of the sample.

Range	**50 ... 220 °C**

Delta T

Temperature correction: indication of how much the block temperature has to be increased so that the sample reaches the setpoint temperature. This value is determined through measurement of the deviation by the current temperature of the sample from the heating block temperature using a calibrated, external temperature sensor *(see Chapter 4.4.2, page 50)*.

Note

If no temperature sensor is available for determining Delta T, you can enter the approximate values listed in the table below, which have been determined with 5 g silicone oil and a gas flow of 10 L/h (air).

Range	**-9.9 ... 9.9 °C**

Table 1 Approximate values for determining Delta T

Setpoint temperature	Delta T
80 °C	+0.7 °C
90 °C	+0.8 °C
100 °C	+0.8 °C
110 °C	+0.9 °C
120 °C	+1.0 °C
130 °C	+1.1 °C
140 °C	+1.1 °C
150 °C	+1.2 °C
160 °C	+1.2 °C

Gas flow

Gas flow through the sample. The gas flow can only be set for both blocks jointly. The value of the method loaded for block A is always decisive.

Range	**7 ... 25 L/h**

Use internal pump

on | off (Default value: **on**)

Switch on/off gas supply with internal pump. If the internal pump is switched off, an external gas supply must be connected to the **Air/N_2 in** connection *(see Chapter 3.2.2, page 13)*.

Start options

Start delay

Waiting time until starting the measurement.

Range	**0 ... 1000 min**

Start mode

Mode for starting and stopping the channels.

Selection	**per channel** | **per 2 channels** | **per block**

per channel
All channels can be started and stopped individually.

per 2 channels
2 channels each can be started and stopped jointly. The **[Start]** button in the control window changes automatically for this.

per block
The 4 channels of a block can be started and stopped jointly.

Stop criteria

Time

on | off (Default value: **off**)

Time until the automatic stop of the determination.

Range	**0.1 ... 9999 h**

Conductivity

on | off (Default value: **off**)

Conductivity limit value. If this value is reached, the determination is ended automatically.

Range	**1 ... 400 µS/cm**

Endpoint(s)

on | off (Default value: **off**)

Automatic stop of the determination, as soon as every active channel has reached all endpoints (induction time and stability time).

Table 2 Overview of stop criteria

Time	Conductivity	Endpoint(s)	Analysis stops...
			not automatically.
✓			after the time entered, independently of reaching the induction/stability time (endpoint).
	✓		after reaching the conductivity entered, independently of reaching the induction/stability time (endpoint).
		✓	after reaching the induction and stability time (endpoint) defined on the **Evaluation** tab. If the induction and stability time are evaluated, both endpoints have to be found.
✓	✓		after reaching the first criterion (time or conductivity), independently of reaching the induction/stability time (endpoint).
✓		✓	after reaching the first criterion (time or endpoint). If the time entered has been reached, there is no further measuring until the induction/stability time (endpoint) has been reached.

Time	Conductivity	Endpoint(s)	Analysis stops...
	✓	✓	after reaching the first criterion (conductivity or endpoint). If the conductivity entered has been reached, there is no further measuring until the induction/stability time (endpoint) has been reached.
✓	✓	✓	after reaching the first criterion. If the time or conductivity entered has been reached, there is no further measuring until the induction/stability time (endpoint) has been reached.

Actions at end of determinations...

Shut off heater

on | off (Default value: **off**)

Automatic deactivation of the heating at the end of the determination.

Shut off gas flow

on | off (Default value: **off**)

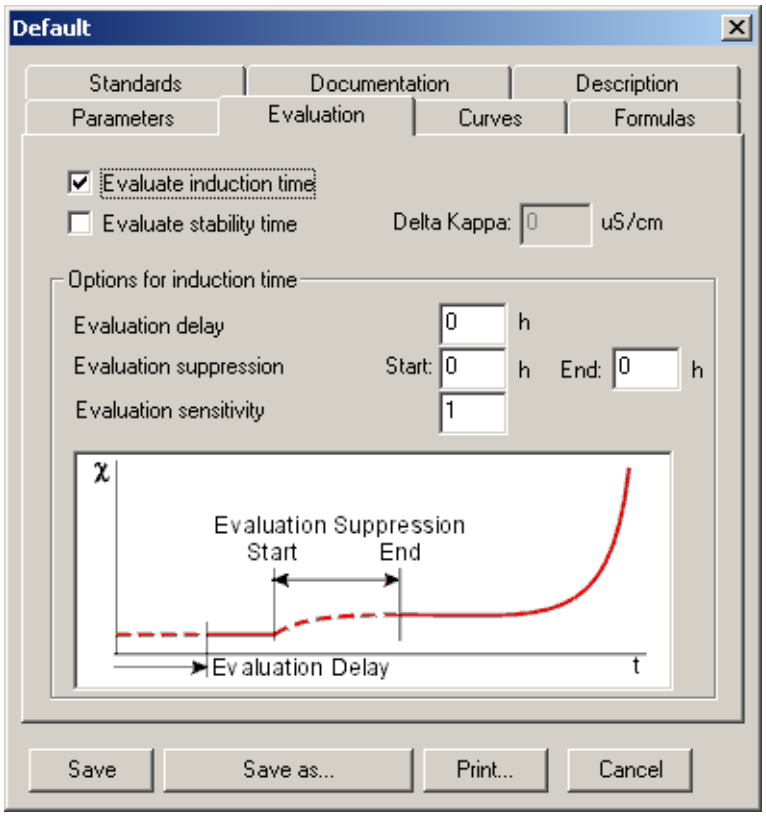
Automatic deactivation of the gas flow at the end of the determination.

Caution

If the gas flow is automatically switched off, the reaction vessels must be removed from the instrument within 30 to 60 min after the end of the determination. If the vessels are not removed, a vacuum could develop over the course of time, which would thus result in back suction of the measuring solution into the hot reaction vessel.

4.5.2.2 "Evaluation" tab

These settings serve for automatic evaluation of the curve. After finishing the measurement, curves can also be reevaluated in the database *(see Chapter 4.7.3, page 108)*.



Evaluate induction time

on | off (Default value: **on**)

Determination of the **induction time** *(see "Induction time", page 66)*.

Evaluate stability time

on | off (Default value: **off**)

Determination of the **stability time** *(see "Stability time", page 67)*.

Note

If you select this evaluation mode, you always need to calibrate the conductivity measuring cell beforehand.

Delta Kappa

Conductivity change for the determination of the stability time.

Range	**1 ... 400 µS/cm**

Options for induction time

Evaluation delay

Waiting time until starting the curve evaluation.

Range	**0 ... 100 h**

Evaluation suppression

Start

Time from the start of the determination to the start of the evaluation suppression.

Range	**0 ... 100 h**

End

Time from the start of the determination to the end of the evaluation suppression.

Range	**0 ... 100 h**

Evaluation sensitivity

This value defines the minimum value which the maximum of the curve of the second derivative has to reach in order to be accepted as a result. The result, i.e. the induction time, is the time until the first accepted maximum.

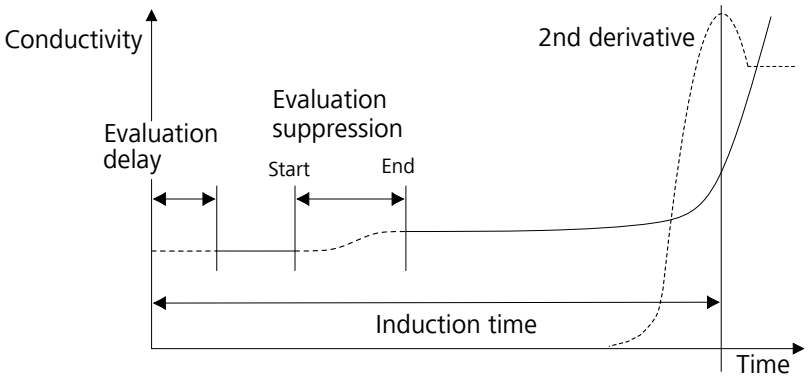
The default value "**1**" will lead to the correct result for most applications. Smaller values will cause the acceptance of smaller maximums of the second derivative. Higher values require a higher maximum.

Range	**0.1 ... 9999**

Induction time

The induction time is the **time to the break point** of the curve *conductivity* vs. *time* recorded with the Biodiesel Rancimat. The induction time is a parameter for the oxidation stability.

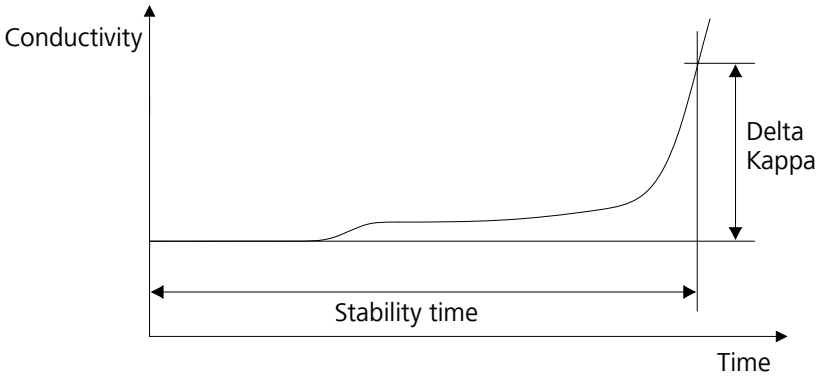
For automatic determination of the induction time, the second derivative of the measured curve is used, which exhibits a maximum at the break point and which can be displayed in the window **Live graph**. To enable detection of a break point, certain criteria must be fulfilled with regard to the height and width of the peak in the second derivative. The break point can also be determined manually as the intersection point of the two tangents at the extended straight branches of the curve *(see "Setting tangents manually", page 112)*.



With certain samples, it may occur that the conductivity rises in steps, to a small degree, long before the actual induction time (e.g. through side reactions or with volatile compounds). To prevent this rise being evaluated as an endpoint, the user can delay the evaluation or suppress it for a certain range.

Stability time

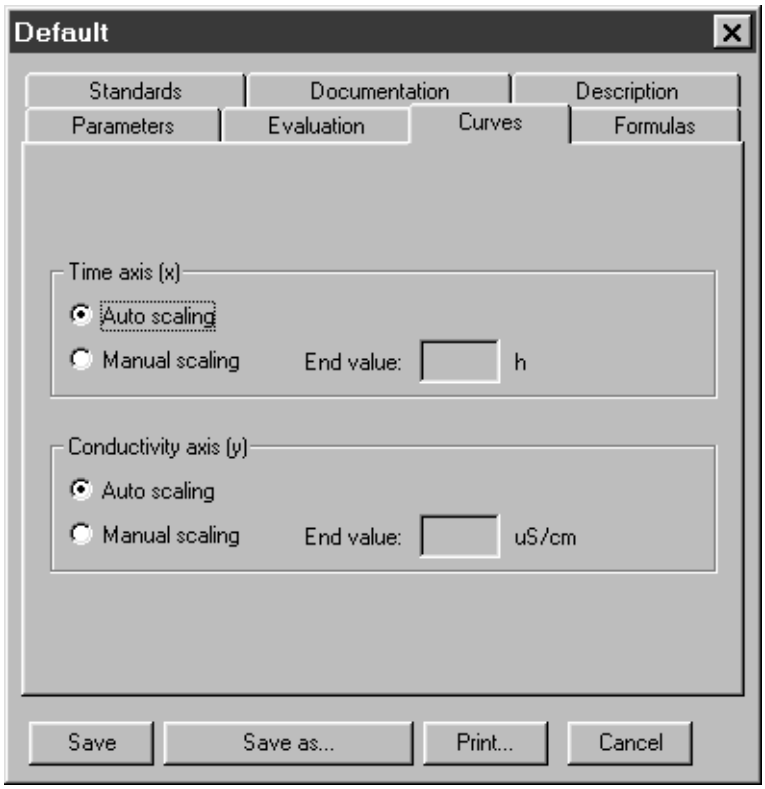
The stability time is the time which is necessary in order to attain a specified **conductivity change Delta Kappa**.



The entry of an evaluation delay or evaluation suppression does not have any effect on this evaluation mode. In all cases the time from the start point of the measurement is calculated.

4.5.2.3 "Curves" tab

The scaling of the x and y axis can be defined here. The settings have an effect on the expression of the curves, but not on the measurement and evaluation. After completing the determination, the scaling for single and live graphs can be changed again in the determination overview.



Time axis (x)

Selection	**Auto scaling** | **Manual scaling**

Auto scaling
Automatic definition of the start and end value of the time axis.

Manual scaling
Manual definition of the end value of the time axis.

End value

End value of the time axis with manual scaling.

Range	**0.5 ... 100 h**

Conductivity axis (y)

Selection	**Auto scaling** | **Manual scaling**

Auto scaling
Automatic definition of the start and end value of the conductivity axis.

Manual scaling
Manual definition of the end value of the conductivity axis.

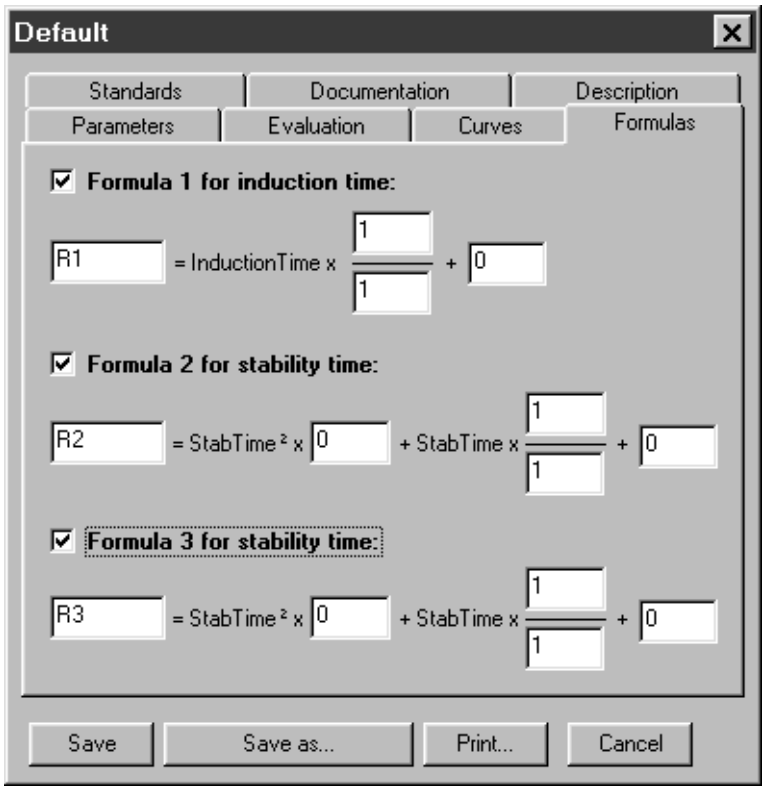
End value

End value of the conductivity axis with manual scaling.

Range	**10 ... 400 µS/cm**

4.5.2.4 "Formulas" tab

You can enter values for further automatic calculations with the induction and stability time on this tab. The settings here do not have any effect on the measuring sequence or the evaluation. After finishing the measurement, a post-calculation or recalculation is possible at any time in the determination overview *(see Chapter 4.7.5, page 117)*.



Formula 1 for induction time:

on | off (Default value: **off**)

If this option is switched on, the automatically determined induction time will be calculated with the entered values. The result will be saved under the name entered in the field "R1".

Formula 2 for stability time:

on | off (Default value: **off**)

If this option is switched on, the automatically determined stability time will be calculated with the entered values. The result will be saved under the name entered in the field "R2".

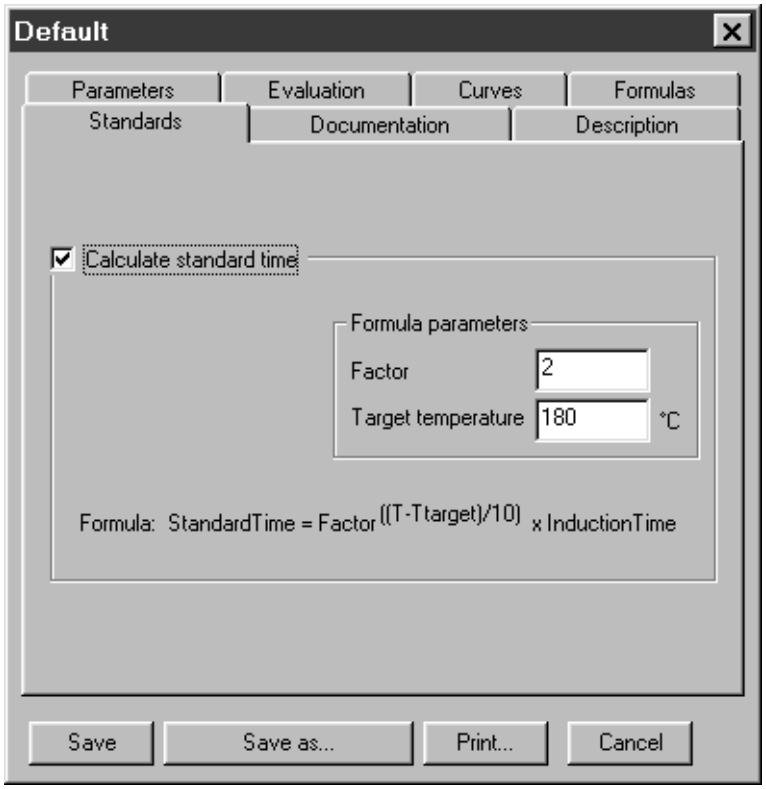
Formula 3 for stability time:

on | off (Default value: **off**)

If this option is switched on, the automatically determined stability time will be calculated with the entered values. The result will be saved under the name entered in the field "R3".

4.5.2.5 "Standards" tab

This function can be used to automatically calculate the induction time for a temperature deviating from the measuring temperature, the so-called standard time. The settings here do not have any effect on the measuring sequence or the evaluation. After finishing the measurement, a post-calculation or recalculation is possible at any time in the determination overview *(see Chapter 4.7.5, page 117)*.



Calculate standard time

on | off (Default value: **off**)

Activation/Deactivation of the calculation of the standard time according to the following formula:

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

Formula parameters

Factor

Parameter for the calculation of the standard time, which indicates the factor by which the time changes per 10 °C. This value can be determined through the extrapolation *(see Chapter 4.7.4, page 113)* of results, which have been recorded at different temperatures.

Range	**0.001 ... 100**

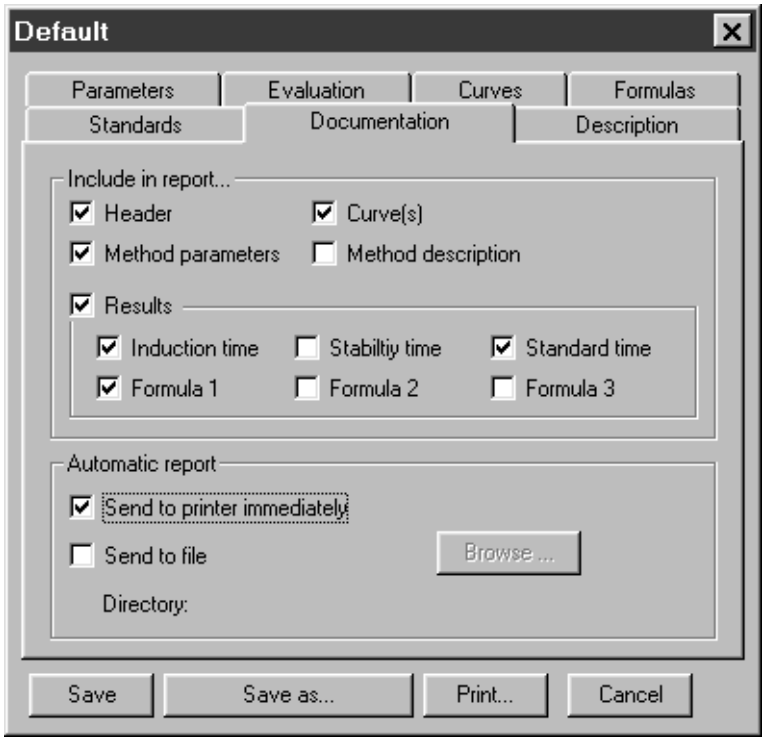
Target temperature

Target temperature for the calculation of the standard time.

Range	**20 ... 300 °C**

4.5.2.6 "Documentation" tab

This tab contains parameters for the automatic report output.



Include in report...

Header

on | off (Default value: **on**)

Output of the header with company settings *(see ""Company settings" tab", page 133)*, date of the printout, sample identifications, instrument information, determination date, user, cell constant and serial number.

Method parameters

on | off (Default value: **on**)

Output of the method parameters.

Curve(s)

on | off (Default value: **on**)

Output of the curves.

Method description

on | off (Default value: **off**)

Output of the method description entered on the tab **Description**.

Results

on | off (Default value: **on**)

Output of the selected results.

Selection	**Induction time** | **Stability time** | **Standard time** | **Formula 1** | **Formula 2** | **Formula 3**

Automatic report output

Send to printer immediately

on | off (Default value: **on**)

Automatic printout of the report defined under **Include in report...** after finishing the determination.

Send to file

on | off (Default value: **off**)

Automatic output of determination and method data after finishing the determination as TXT file for further processing in other programs. The content of this report is independent of the report elements defined at **Include in report...**.

Browse…

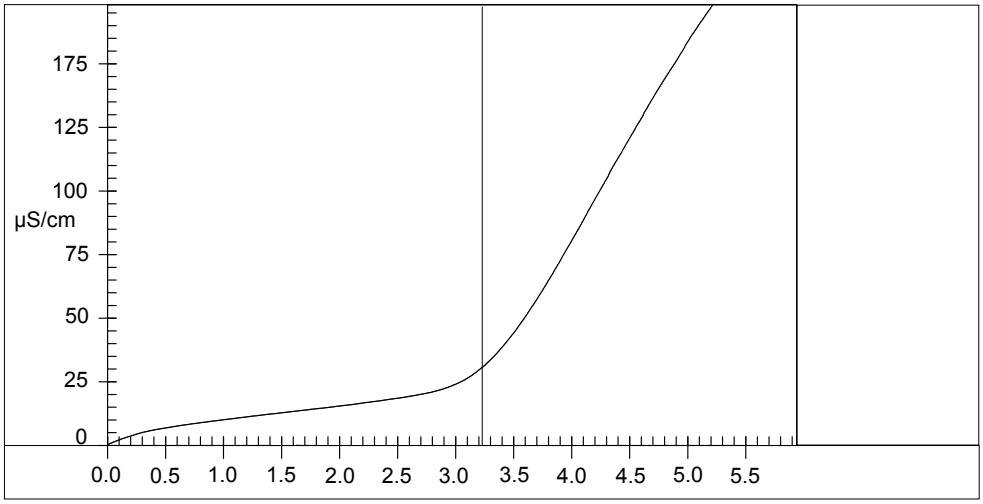
Selection of the directory for saving this file.

Example of an automatically printed report

Unit	1	**Serial number**	01109	**Printing date**	10.02.2007 09:34:03
Block	A	**Cell constant**	1	**Determination date**	08.02.2007 13:25:52
Channel	1	**User**	Administrator		

ID 1	**ID 2**
FAME	120 °C

Induction time 3.23 h



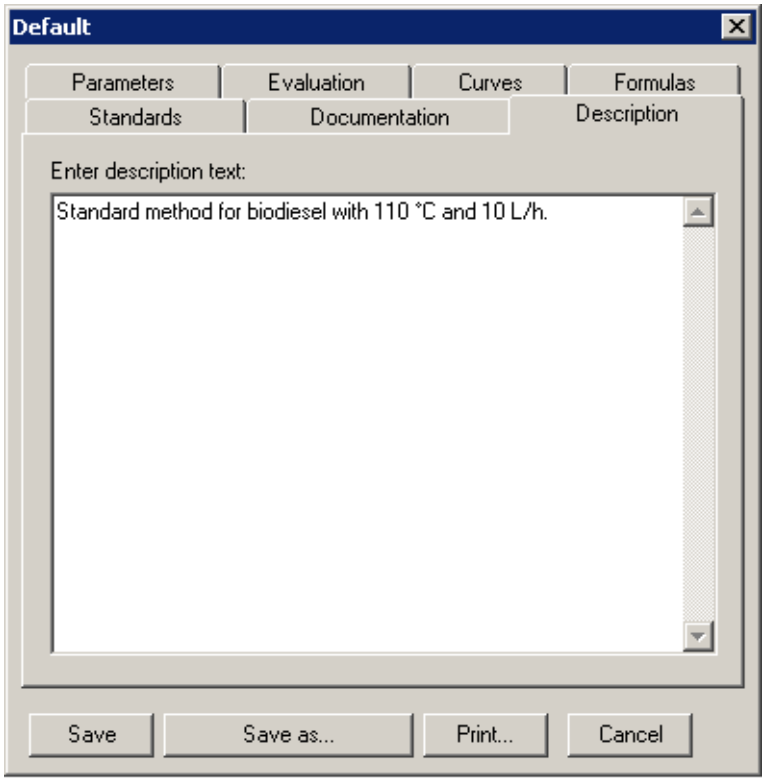
Method name	FAME		
Creator	Administrator	**Creation date**	08.02.2007 08:27:20
Temperature	120 °C	**Stop time**	0.00 h
Delta T	0.69 °C	**Stop at conductivity**	200 µS/cm
Gas flow	10 L/h	**Stop at endpoint**	☑
Start delay	0 min	**Delta Kappa**	0 µS/cm
Start mode	◉ per channel	**Evaluation delay**	0.00 h
	○ per 2 channels	**Evaluation delay start**	0.00 h
	○ per block	**Evaluation delay end**	0.00 h

Send to file

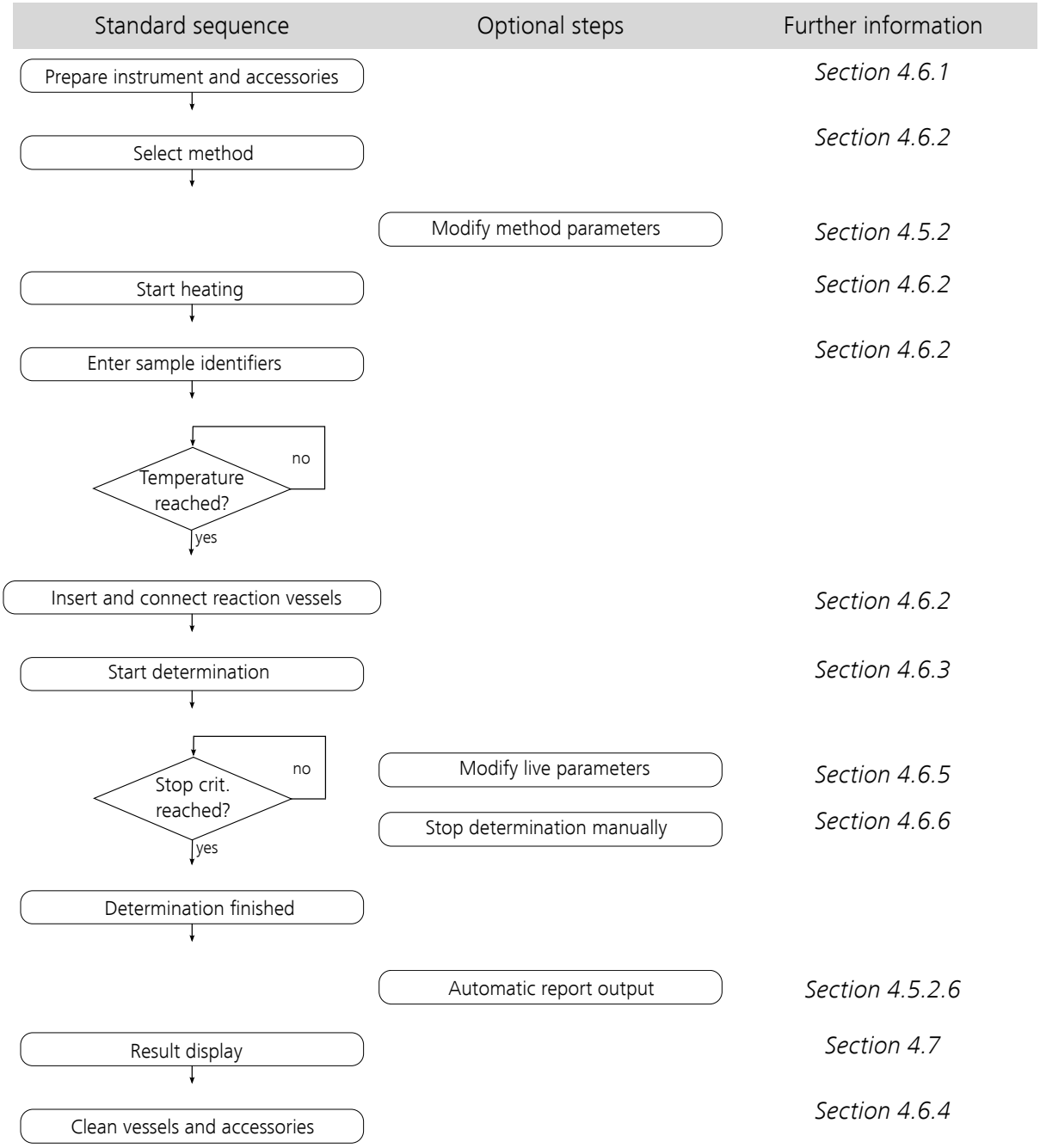
GLP test passed.

4.5.2.7 "Description" tab

You can enter a comment on the method in this tab.



4.6 Determinations



4.6.1 Preparing the instrument and the accessories

The cleanliness of instrument and accessories is an indispensable prerequisite for **reliable, reproducible and correct analysis results**. Even the slightest contamination could catalytically accelerate the oxidative decomposition and lead to completely incorrect results. Therefore always observe the instructions for use of measuring and reaction vessels in this section.

Check and prepare the instruments and vessels as follows *(see also figure 5, page 14)*:

1 Check openings for reaction vessels

- Check whether the openings are clean and empty. Blow the dust out of the openings with nitrogen.
  If the instrument is not used, always seal the openings with the sealing plugs.

2 Fill the measuring vessels

Note

Only use **measuring vessels** and accessories which are **absolutely clean** and in a **perfect condition**.

- Fill the cleaned measuring vessels with **60 mL distilled water** each.
  For analysis times > 24 h approx. 7 mL more distilled water must be added per day to compensate for the evaporation loss, so that the electrodes remain safely immersed.

3 Insert the measuring vessels

- Place the clean measuring vessel covers equipped with a PTFE tube on the measuring vessels.
- Insert the measuring vessels with the measuring vessel covers into the openings provided for this on the 873 Biodiesel Rancimat and, at the same time, carefully guide the connection plugs *(5-**15**)* of the cover carefully into the electrode connectors *(3-**2**)*.

4 Weigh in the sample

Note

Use **new reaction vessels and air tubes** for each measurement. Blow the reaction vessels out with nitrogen before use so that any particles adhering are removed.

- Weigh in **7.5 g of the samples** in each of the reaction vessels.

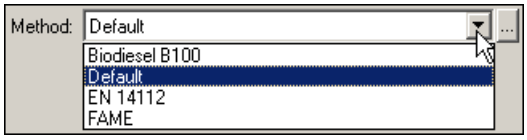
5 Mount the accessories

- Take the upper edge of the reaction vessel in your hand (e.g. in the gap between your thumb and index finger) and turn the glass round once.
  This serves for covering the degreased glasses with a light **fat film** so that the vessel covers can be removed more easily after the determination.
- Insert an air tube *(5-**5**)* in the connector *(5-**7**)* of the reaction vessel cover, fix it with the O-ring *(5-**6**)* and fasten it by screwing in the thread adapter M8 / M6 *(5-**3**)*.
- Place the reaction vessel cover on the reaction vessel. Turn the cover in such a way that the air tube is as close as possible to the vessel wall.
- Connect the Iso-Versinic® tubing to the tubing connector *(5-**4**)* of the reaction vessel cover.
- Place the prepared reaction vessel in the vessel holder.

4.6.2 Preparing the determination

1 Select method

- Under **Unit x Block A**, click on the symbol in order to select the method for Block A.
- If required, also select a method for Block B.



Note

Different methods with different temperatures can be selected for Block A and Block B. For the gas flow, on the other hand, the value which is defined in the method loaded for Block A applies for both blocks.

See also *"Opening a selected method and adjusting parameters", page 57*.

2 Start heating

- Under **Unit x Block A**, click on the button **[Heat to xx °C]**.
- If required, also switch on the heating for Block B.

The color of the button frame changes to red and the temperature display *(2-**3**)* on the instrument flashes. The current temperature is displayed directly next to the button.

Heating up duration for 120 °C: approx. 45 min.

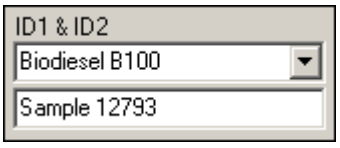
Heating up duration for 200 °C: approx. 60 min.

Note

If you wish to turn off the heating, click on the button **[Heat to xx °C]** again.

3 Enter sample identification

- Enter the sample identifications **ID1 & ID2** for all sample positions used.



The entry for **ID1** can be selected from the automatically created list of already entered identifications. This list can be deleted again in the **Control window** with the menu item **Options ▸ Clear sample ID1 history**.

4 Insert and connect reaction vessels

Note

The temperature defined in the method must be reached, before you insert the reaction vessels, i.e. the frame of the button **[Heat to xx °C]** must be **green**.

- Seal the channels not used with the sealing plugs or empty reaction vessels for protection against impurities.
- Insert the prepared reaction vessels into the openings of the heating block.
- Connect the Iso-Versinic® tubings, which are fastened to the reaction vessel cover, to the tubing adapter M8 / olive *(5-**11**)* of the measuring vessel covers.
- Screw the FEP tubings 250 mm *(5-**1**)* to the thread adapters *(5-**3**)* of the reaction vessel covers and the air supply connections *(3-**3**)* of the Biodiesel Rancimat.

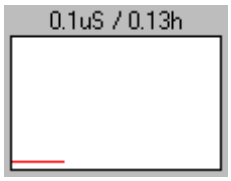
4.6.3 Starting the determination

Before you can carry out the determination, the channels you intend to use must be **ready***(see Chapter 4.6.7, page 82)*.

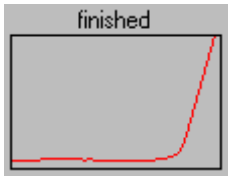
1 Start determination

- Start of an **individual determination**: Click on the button **[Start]** of the corresponding channel.
- Start **of all channels of Block A**: Click on the button A.
- Start **of all channels of Block B**: Click on the button B.

After the **determination has been started**, the live curve is displayed for every channel:



As soon as a stop criterion defined in the method *(see table 2, page 63)* has been reached, the **determination is ended**:



The determination is automatically saved in the database **Repos.mrd**. The channel is ready for starting a new determination.

2 Clean vessels and accessories

Note

Always clean the vessels and accessories **directly after each determination** *(see Chapter 4.6.4, page 80)*.

4.6.4 Cleaning the instrument and accessories

1 Clean the measuring vessels and accessories

- Clean used **measuring vessels** after pouring off the measuring solution with acetone or 2-propanol. Pre-clean with washing-up liquid in the case of severe contamination.
- Rinse the product thoroughly with distilled water.
- Clean the **measuring vessel covers** *(5-**16**)*, the **PTFE tubes** *(5-**17**)* and the **electrodes** *(5-**18**)* with acetone or 2-propanol and rinse with distilled water. Pre-clean with washing-up liquid in the case of severe contamination.
  Remove the protective ring *(5-**19**)* to be able to clean the electrodes.

2 Clean the reaction vessels and accessories

- Dispose of used **reaction vessels** and **air tubes** and use new reaction vessels and air tubes for the next measurement.
- Clean the **reaction vessel covers** with acetone or 2-propanol and rinse with distilled water. Pre-clean with washing-up liquid in the case of severe contamination.
- Then heat the **reaction vessel covers** in the drying oven for 2 hours at 80 °C.

Note

Replace the reaction vessel covers if they are not tightly on the reaction vessel anymore or the material is obdurate.

3 Clean the tubings

- Clean the **Iso-Versinic® tubings** *(5-**2**)* with acetone or 2-propanol and rinse with distilled water. Pre-clean with washing-up liquid in the case of severe contamination.

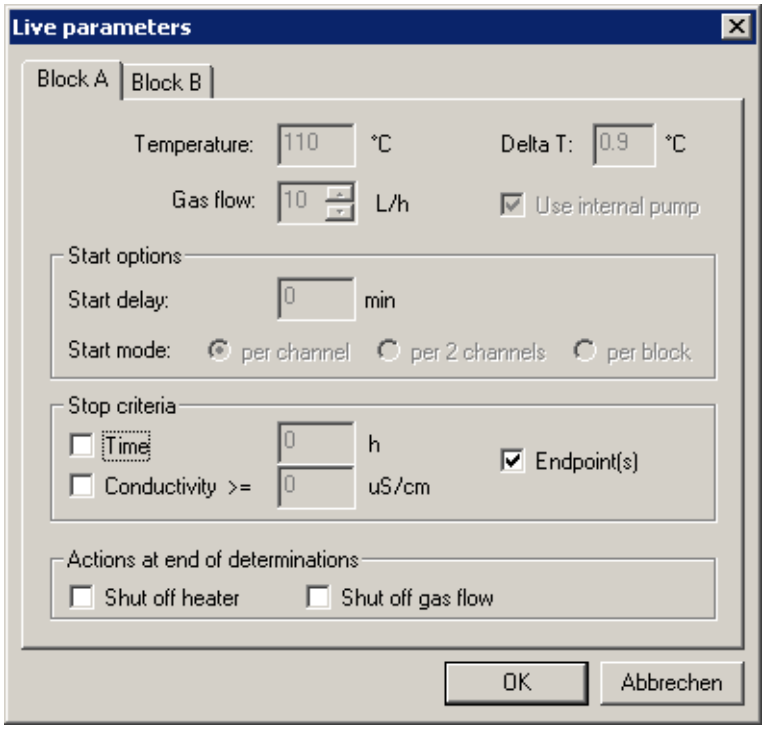
- Then heat the **Iso-Versinic® tubings** in the drying oven for 2 hours at 80 °C.

4.6.5 Adjusting the method parameters during the determination

1 Open the dialog window Live parameters

- In the **Control window**, click on the symbol or the menu item **View ▸ Live parameters...**.

The following dialog window appears:



2 Adjust the parameters

- Adjust all required parameters *(see Chapter 4.5.2.1, page 60)*.
- Confirm the changes with **[OK]**.

4.6.6 Stopping the determination manually

Determinations are normally automatically stopped after reaching the stop criteria defined in the method. Current determinations can also be stopped manually.

1 Stop determination

- In the **Control window**, click on the button **[Stop]**.

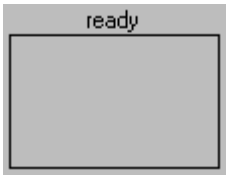
The message **Do you want to save this determination?** appears.

2 Save measured data and results

- Confirm the message with **[Yes]** to save all data of the determination.
- or confirm the message with **[No]** to reject all data.

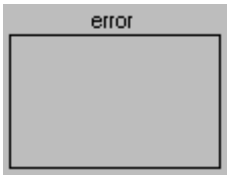
4.6.7 Status of the live curve

Before a determination:



No active measurement.

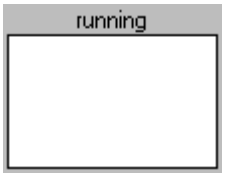
The channel is ready for starting a determination.



No active measurement.

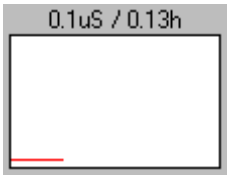
Communication error between instrument and PC.

During a determination:



Determination started.

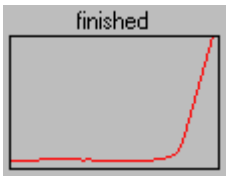
The first measured value is measured.



Current measurement.

Measured value and time are displayed above the live curve.

After a determination:



Determination finished.

The channel is ready for starting a new determination.

4.7 Results

4.7.1 Determination overview

The determination overview displays selected information on all determinations which are saved in the opened database, in tabular form. You can adjust this overview, i.e. you can for example hide fields, adjust the column width or change the font type.

You can also filter and sort the content of the determination overview according to specific criteria.

You will find step-by-step instructions on various actions, which you can execute in the determination overview, below. You can also execute many actions in addition to the described methods via the context menu.

Further below in this section you will find a detailed description of all parameters of the dialog windows of the determination overview *(see "Parameter description", page 91)*.

Displaying the determination overview

Proceed as follows to open a determination overview:

1 Open Results window

- In the **Control window**, click on the symbol  *or* the menu item **File ▸ Results...**.

The Results window is opened with the standard database **Repos.mrd**.

If you wish to open another database, carry out step 2.

2 Open database

- In the **Results window**, click on the menu item **File ▸ Open database...**.
  The dialog window **Open database** appears.
- Click on the required database and confirm the selection with **[OK]**.

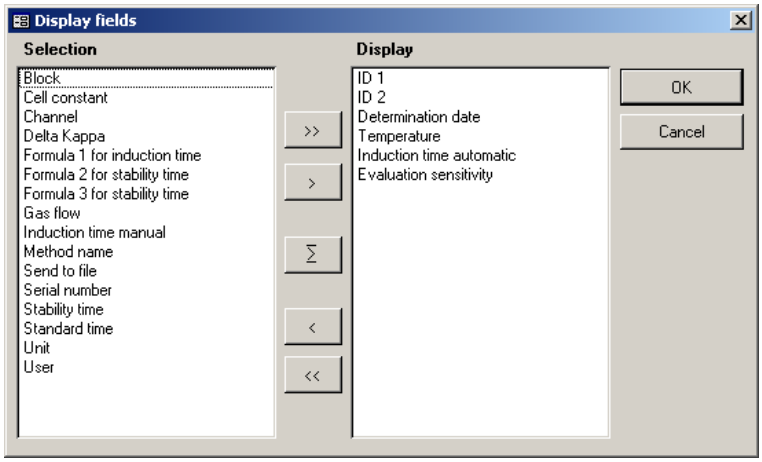
Formatting the determination overview

Proceed as follows to format the determination overview.

A detailed description of the parameters can be found at *"Parameter description", page 91*.

1 Select fields

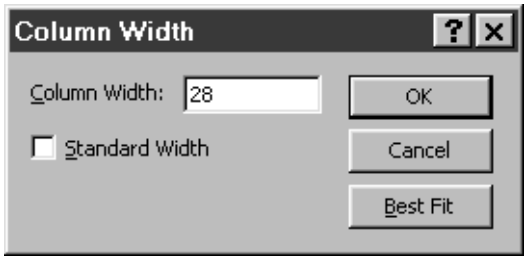
- Click on the symbol *or* the menu item **Format ▸ Select fields**.
  The following dialog window appears:



  Only those fields are shown in the determination overview which are listed in the right column under **Display**.
- In the column **Selection**, click on a field which is to be displayed in the determination overview.
- Click on the **[>]** button.
- In the column **Display**, click on a field which is **not** to be displayed in the determination overview.
- Click on the **[<]** button.

2 Adjust the column width

- Click in any cell of the column whose width is to be adjusted.
- Click on the menu item **Format ▸ Adjust width**.
  The following dialog window appears:



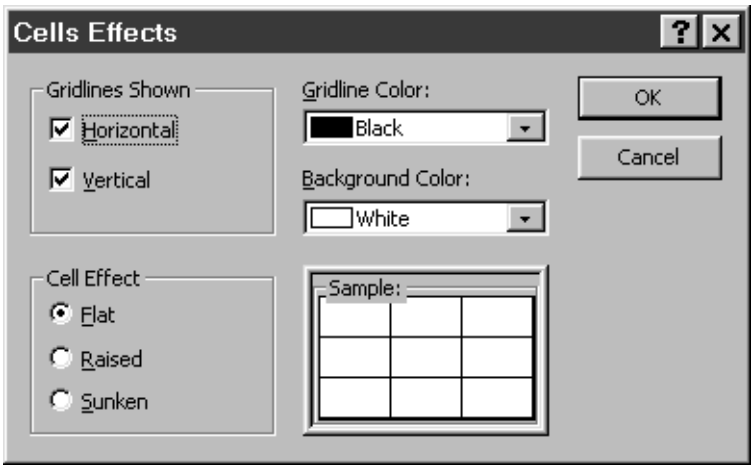
- Under **Column width**, enter the number of characters of a column and confirm with **[OK]**.

or

- Position the cursor in the title line of the table between two columns.
  The cursor assumes the following form: ✣
- Drag the column width to the required size keeping the left mouse button pressed down.

3 Adjust the cell format

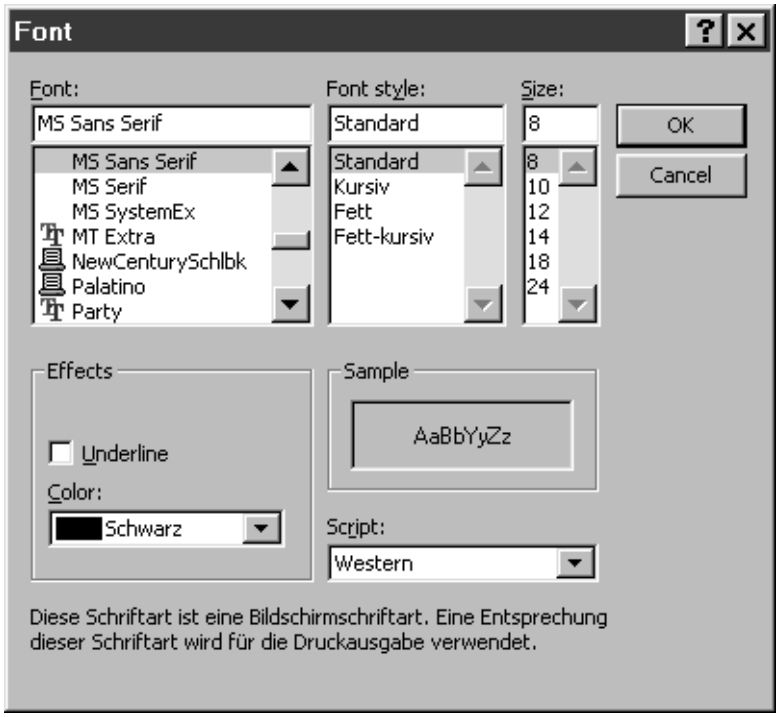
- Click on the menu item **Format ▸ Fields...**.
  The following dialog window appears:



- Carry out the required settings and confirm with **[OK]**.

4 Change the font

- Click on the menu item **Format ▸ Fonts...**.
  The following dialog window appears:



- Carry out the required settings and confirm with **[OK]**.

Selecting determinations

Proceed as follows to select one or more lines of the determination overview:

1 Select a line

- Place the mouse button on the left edge of the line to be selected.
  The cursor assumes the following form: ➡
- Click on the line.

2 Select several lines

- Place the mouse button on the left edge of the line to be selected.
  The cursor assumes the following form: ➡
- Keep the mouse button pressed down and move over the left edge of all lines to be selected.

3 Select all lines

- Click on the menu item **Edit ▸ Select all records**.

or

- Click on the upper left in the title line.

Searching for determinations

Proceed as follows to search for specific determinations.

A detailed description of the parameters can be found at *"Find What", page 96*.

1 Open search field

- Click on the symbol  *or* the menu item **Determination ▸ Find...**

The dialog window **Find in field: 'Field name'** appears.

2 Enter search criteria

- Enter the search term and the other search criteria.
- Click on the button **[Find first]** in order to search through the determination overview.

Sorting determinations

Proceed as follows to sort the columns of the determination overview:

1 Sort column ascending

- Click in the title line of the column to be sorted.
  The entire column is highlighted in black.
- Click on the symbol  *or* the menu item **Determination ▸ Sort ▸ Ascending**.

2 Sort column descending

- Click in the title line of the column to be sorted.
  The entire column is highlighted in black.
- Click on the symbol  *or* the menu item **Determination ▸ Sort ▸ Descending**.

3 Remove sorting again

- Click on the symbol  *or* the menu item **Determination ▸ Filter ▸ Remove filter/sorting**.

Displaying or hiding selected determinations

Proceed as follows to display or hide previously selected determinations only:

1 Select determinations

- *See "Selecting determinations", page 86*.

2 Display selected determinations only

- Click on the symbol  *or* the menu item **Determination ▸ Filter ▸ Filter selection**.

3 Remove the filter again

- Click on the symbol  *or* the menu item **Determination ▸ Filter ▸ Remove filter/sorting**.

All entries are once again displayed in the dialog window.

4 Hide selected determinations

- Click on the symbol  *or* the menu item **Determination ▸ Filter ▸ Selection not in filter**.

Filtering determinations with the "Selection based filter"

Proceed as follows to filter the entries of the determination overview according to a specific selection (cell or cell content):

1 Filter the entries

- Click in that cell which contains the required filter criterion (e.g. **Determination date** = "12.2005").
- Click on the symbol  *or* the menu item **Determination ▸ Filter ▸ Selection based filter**.

Only the entries with the **Determination date** "12.2005" are displayed in the dialog window.

2 Remove the filter again

- Click on the symbol  *or* the menu item **Determination ▸ Filter ▸ Remove filter/sorting**.

All entries are once again displayed in the dialog window.

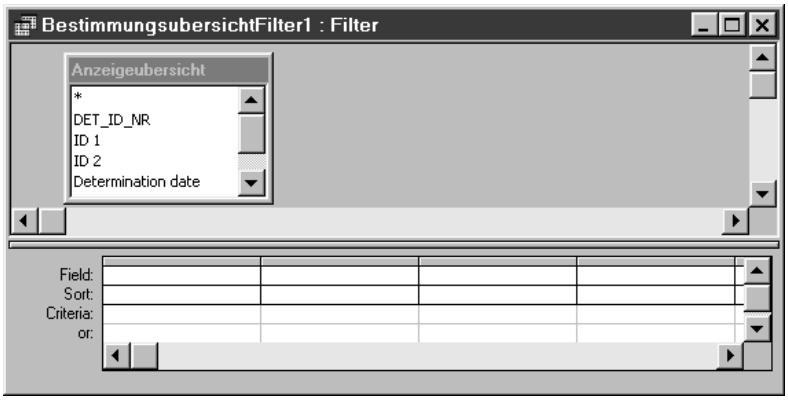
Filtering determinations with the special filter

With a special filter you can define complex conditions for filtering and sorting data sets. In the following section, you will see a simple example of a special filter. You will find a detailed description of all parameters under *"BestimmungsübersichtFilter", page 97*.

1 Open dialog window

- Click on the symbol  *or* the menu item **Determination ▸ Filter ▸ Special filter/sorting...**.

The following dialog window appears:



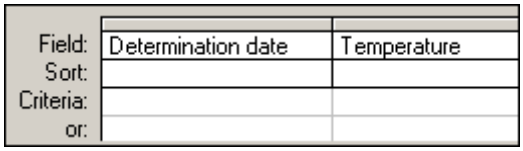
2 Select the fields which are to be filtered

- In the dialog window **Anzeigeübersicht**, double-click on the field **Determination date**.
- In the dialog window **Anzeigeübersicht**, double-click on the field **Temperature**.

Note

In the dialog window **Anzeigeübersicht**, only those field are available for selection which are displayed in the determination overview *(see "Formatting the determination overview", page 83)*.

The two fields are displayed in the lower part of the window under **Field**:



3 Specify the filter criteria

- In the column **determination date** in field **Criteria** enter the value **05.04.2000*** (incl. asterisk as wildcard).
- In the column **Temperature** and the field **Criteria** enter the value **> 110**.

Note

You will find a detailed description of the possible filter criteria under *"Criteria", page 98*.

4 Apply and remove the filter

- Click on the symbol *or* the menu item **Determination ▸ Filter ▸ Apply filter/sorting**.
  In the window **Determination data**, all determination data is now displayed that was carried out on April 5, 2000 at a temperature of over 110 °C. In addition, the dialog window **Determination data** uses the word **FILTER** to indicate that the displayed data is filtered.
- Click on the symbol *or* the menu item **Determination ▸ Filter ▸ Remove filter/sorting**.
  The special filter has been removed again.

Deleting determinations

Note

Only the **Administrator** can delete determinations.

Proceed as follows to delete specific entries from the determination overview:

1 Select the determination

- *See "Selecting determinations", page 86*.

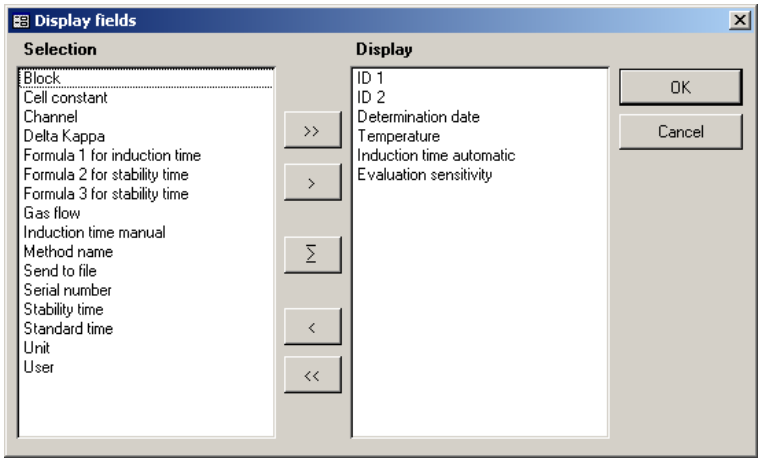
2 Delete the entry

- Click on the menu item **Determination ▸ Delete**.
  The safety prompt **Do you really want to delete the selected determinations?** appears.
- Confirm the security query with **[Yes]**.

Printing and exporting the determination overview

See Chapter 4.7.6, Page 124.

Parameter description



Selection

Selection of all fields, which can still be selected for the determination overview. The following **fields of the determination overview** are available:

Send to file

Directory in which the determination file (TXT file) has been saved.

Evaluation sensitivity

Evaluation sensitivity used for the determation of the induction time.

User

Name of the user who was logged in when recording the determination.

Determination date

Date and time at which the determination was started.

Block

Letter of the block used for the determination.

Delta Kappa

Conductivity change for the determination of the stability time.

Formula 1 for induction time

Result of the calculation with formula 1.

Formula 2 for stability time

Result of the calculation with formula 2.

Formula 3 for stability time

Result of the calculation with formula 3.

Gas flow

Gas flow through sample.

Unit

Number of the instrument used for the determination.

ID 1

Sample identification 1.

ID 2

Sample identification 2.

Induction time automatic

Automatically determined induction time.

Induction time manual

Induction time manually determined with the reevaluation.

Channel

Number of the channel used for the determination.

Method name

Name of the method with which the determination has been carried out.

Standard time

Automatically calculated standard time.

Serial number

Serial number of the instrument used to record the determination.

Stability time

Automatically or, with the reevaluation, manually determined stability time.

Temperature

Temperature as defined in the method.

Cell constant

Cell constant of the conductivity measuring cell used to record the determination.

Display

Fields which have been selected for the determination overview.

Selection	**Fields of the determination overview**

>>

Move all fields in **Selection** to **Display**.

>

Move selected field in **Selection** to **Display**. The field is inserted at the end of the list.

Σ

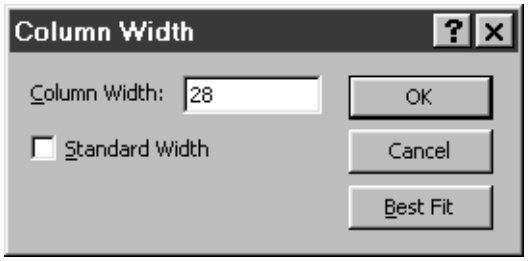
Move selected field in **Selection** to **Display**. The field is inserted above the field selected in **Display**.

<

Move selected field in **Display** to **Selection**.

<<

Move all fields in **Display** to **Selection**.



Column width

Width of the column in characters.

Input	**'Number of characters'**

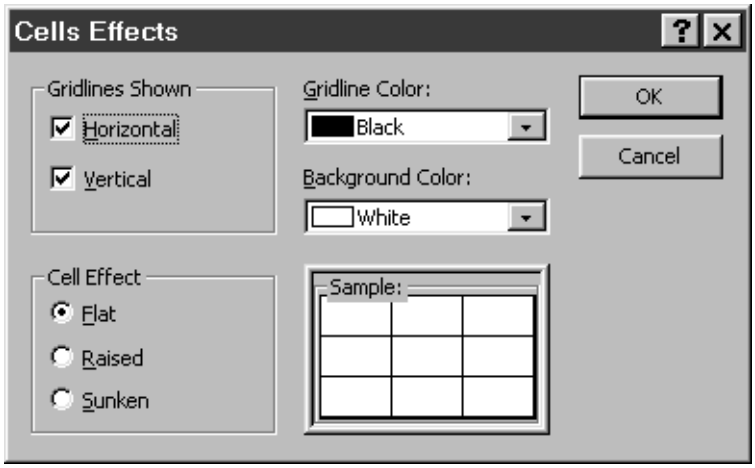
Standard width

on | off (Default value: **on**)

Reset the column width to standard width (18.5 characters).

[Best Fit]

Adjust the column width to the longest field content (including title).



Gridlines Shown

Horizontal

on | off (Default value: **on**)

Display horizontal grid lines.

Vertical

on | off (Default value: **on**)

Display vertical grid lines.

Cell Effect

Selection	**Flat** | **Raised** | **Sunken**

Flat
Flat view of the cells.

Raised
Raised view of the cells.

Sunken
Sunken view of the cells.

Gridline Color

Color of the horizontal and vertical grid lines.

Selection	**Color**

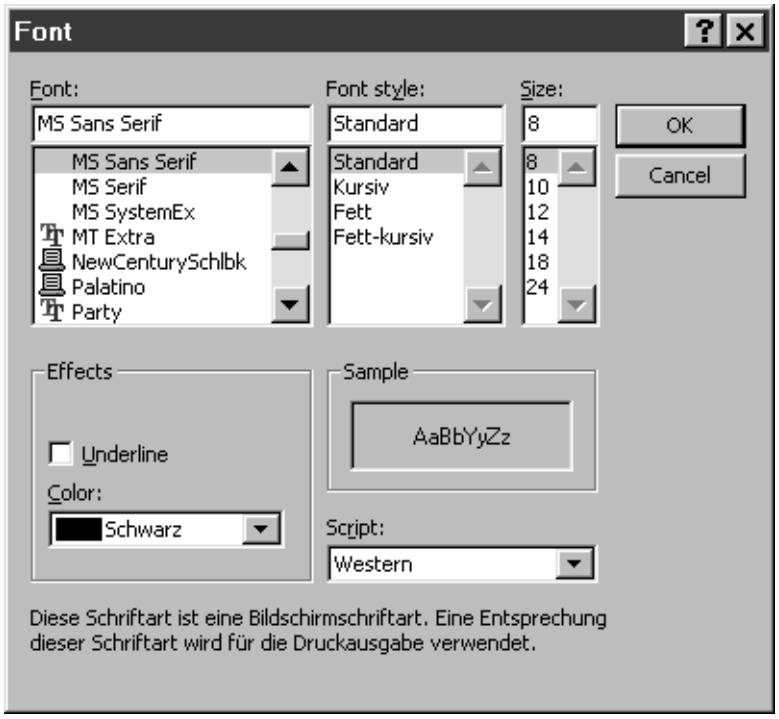
Background color

Color of the cell background.

Selection	**Color**

Preview

Display of the selected format for the cells.



Font

Selection of the font type from the fonts installed on the PC.

Selection	**Font type**

Effects

Underline

on | off (Default value: **off**)

Choice of underlining for characters.

Color

Color for the selected font type.

Selection	**Color**

Font style

Choice of font style for the selected font type.

Selection	**Standard** | **Italic** | **Bold** | **Bold italic**

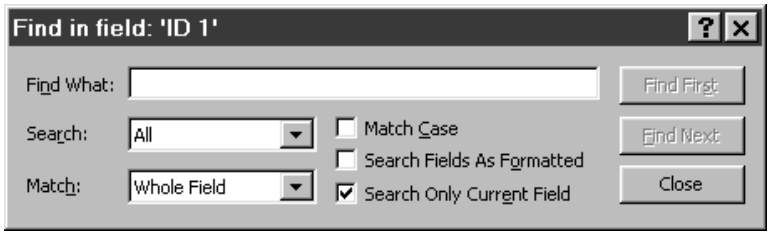
Sample

Display of the selected font type.

Size

Font size in points for the selected font type.

Selection	**Font size**



Find What

Required search term.

Input	**'Search term'**

The following characters can be used as wildcards:
*****: any character sequence (e.g. *ab, *ab*, ab*)
?: single character (e.g. a?a, ??a, ab?)
#: single numeral (e.g. ##20, ab###, #23##)

Search

Search mode

Selection	**All** | **Down** | **Up**

All
Search in the entire table.

Down
Search downwards as far as the table end.

Up
Search upwards as far as the table start.

Match

Search area where the entered text is to be searched for.

Selection	**Whole field** | **Start of field** | **Any part of field**

Whole field
The search is only successful if the entire field matches the search term.

Start of field
The search is successful if the start of the field content matches the search term.

Any part of field
The search is successful if the field content partly matches the search term.

Match case

on | off (Default value: **off**)

If this option is activated, the search is only successful if the uppercase and lowercase of the search term and field content match.

Search Fields As Formatted

on | off (Default value: **off**)

If this option is activated, the unit is also matched in the search for the entire field content in fields with units (e.g. °C, h). If only the numerical value is to be searched for, this option must be deactivated.

Search Only Current Field

on | off (Default value: **off**)

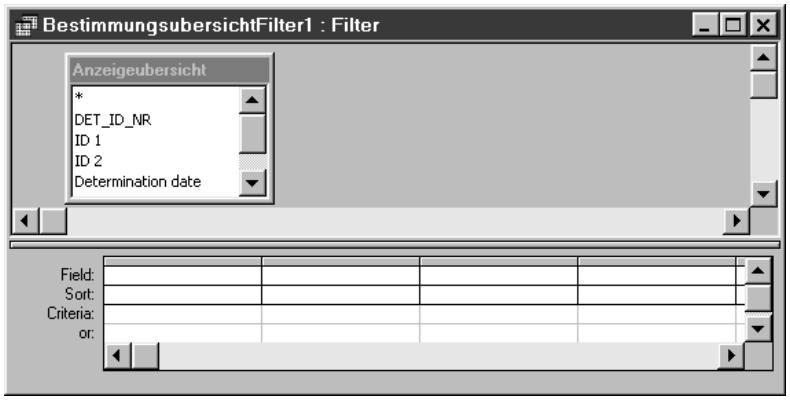
If this option is activated, the search is restricted to the selected field. The field name is displayed in the window title. This option is not available if one or more entire lines are selected.

[Find first]

Start search at the start of the determination overview.

[Find next]

Continue with the search at the cursor location in the determination overview.



Field

Selection of a field (column) from the determination overview.

Selection	**Field from the determination overview**

Sort

Sorting for the selected field.

Selection	**Ascending** | **Descending** | **(not sorted)**

Criteria

Criteria for filtering data sets.

or

Further criteria for filtering data sets.

In the lines **Criteria** and **or** and in all following lines, data sets can be filtered using expressions. An **expression** is a combination of operators, functions and field names for determining a condition for filtering data sets. In the following only the most important elements and rules are described and a few selected examples are given. For more extensive information please refer to the documentation on Microsoft Access.

Operators, functions, field names

Mathematical operators	+, −, =, *, /
Comparison operators	<, >, <>, <=, >=, Between…, And…
Logical operators	AND, OR, NOT
Wildcards	* (any character string), ? (single character), # (single numeral)
Current date	Date()
Current time	Now() (date and time)
Other fields	["Field name"]

Links with various fields

If conditions are specified in various fields, these conditions can also be linked with **AND** and **OR**. The following applies:

Criteria in the same line apply as AND conditions, i.e. only those data sets are displayed which satisfy all conditions.

Criteria in various lines apply as OR conditions, i.e. all data sets which fulfill one condition are displayed.

Examples

> 10	Values greater than 10.
oil	All data with "oil" as part of the name.
2007	All data of the year 2007.
Between Date() And (Date() - 7)	All data of the previous week.

4.7.2 Determination and method data

For each determination you can view all the determination data (name, determination date etc.) and method data (name, parameters etc.) and print or export these.

A detailed description of the parameters can be found at *"Parameter description", page 99*.

Displaying determination and method data

Proceed as follows to display all determination and method data for a selected determination:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

2 Open dialog window

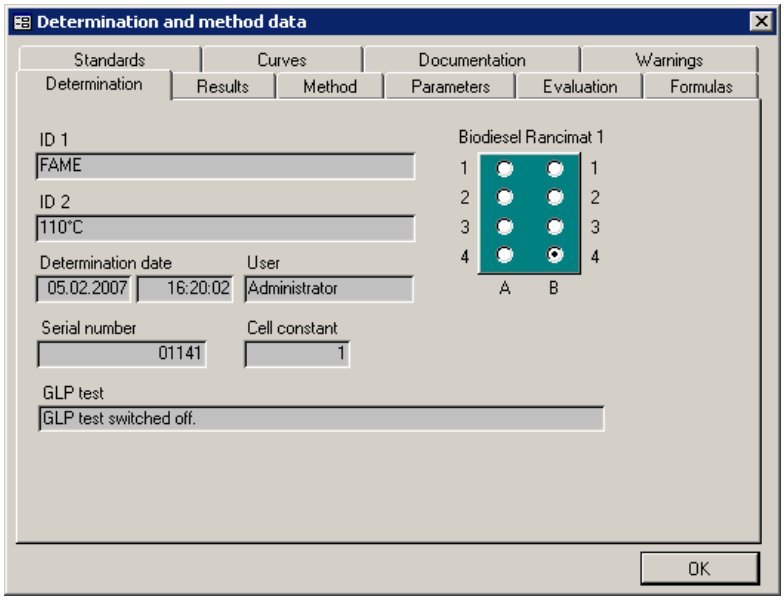
- Select the desired determination *(see "Selecting determinations", page 86)*.
- In the Results window, click on the symbol  *or* the menu item **View ▸ Determination and method data**.

The dialog window **Determination and method data** appears.

Printing and exporting determination and method data

See Chapter 4.7.6, Page 124.

Parameter description



ID 1

Sample identification 1.

ID 2

Sample identification 2.

Determination date

Date and time at which the determination was started.

User

Name of the user logged in when recording the determination.

Serial number

Serial number of the instrument used to record the determination.

Cell constant

Cell constant of the conductivity measuring cell used to record the determination.

GLP test

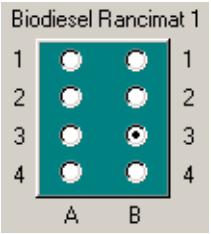
GLP status at the time of recording the determination. The following displays are possible:

GLP test switched off: The GLP monitoring was not activated for either temperature or conductivity or gas flow.

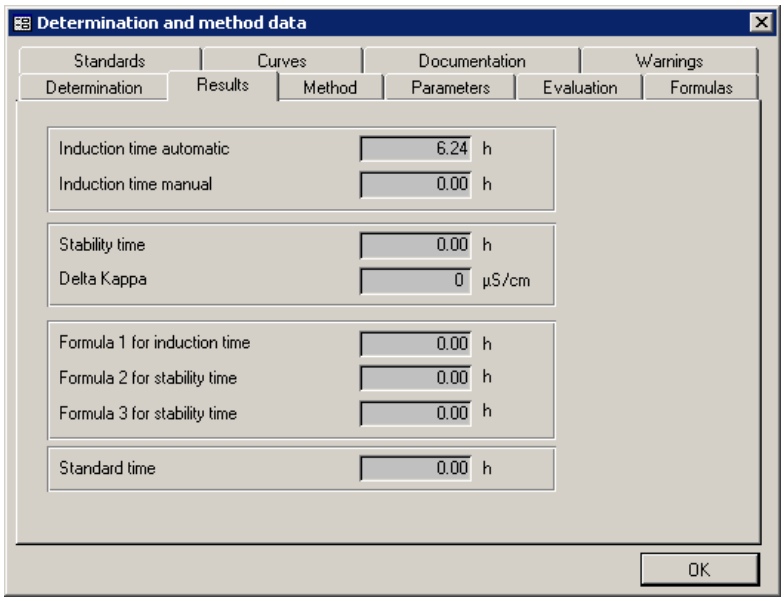
GLP test due: The next GLP test was due.

GLP test not passed: The last GLP test carried out was not passed.

GLP test passed: The last GLP test carried out was passed.



Display of the instrument, block and channel on which the determination was carried out.



Induction time automatic

Automatically determined induction time.

Induction time manual

Induction time manually determined with the reevaluation.

Stability time

Automatically or, with the reevaluation, manually determined stability time.

Delta Kappa

Conductivity change for the determination of the stability time.

Formula 1 for induction time

Result of the calculation with formula 1.

Formula 2 for stability time

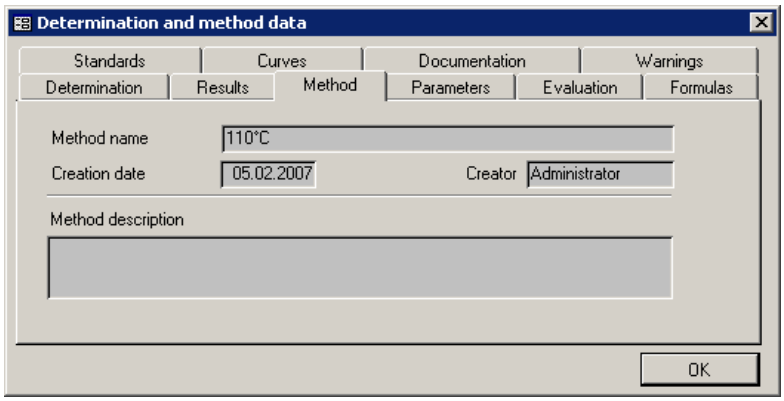
Result of the calculation with formula 2.

Formula 3 for stability time

Result of the calculation with formula 3.

Standard time

Automatically calculated standard time.



Method name

Name of the method.

Creation date

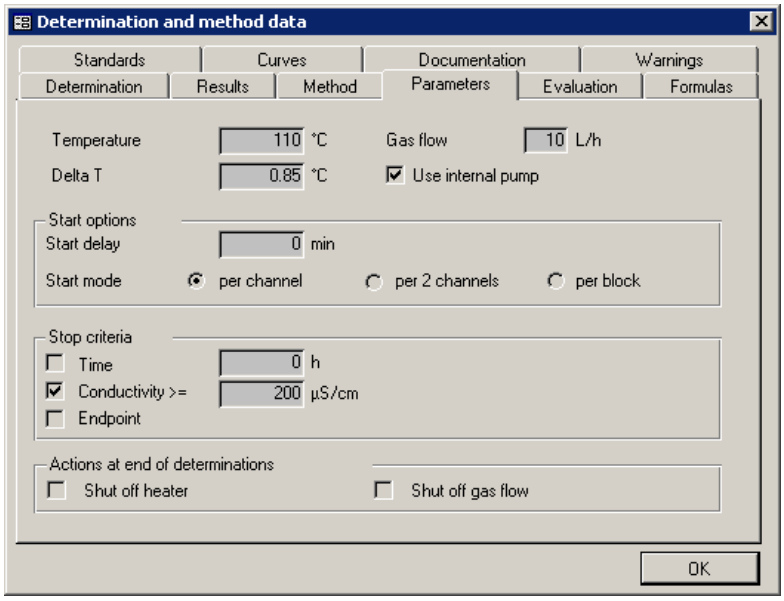
Date when the method was last saved.

Creator

Name of the logged in user who last saved the method.

Method description

Description of the method.



Temperature

Setpoint temperature of the sample.

Delta T

Temperature correction: indication of how much the block temperature has been increased so that the sample reaches the setpoint temperature.

Gas flow

Gas flow through sample.

Use internal pump

Gas supply with internal pump switched on/off.

Start options

Start delay

Waiting time until starting the measurement.

Start mode

Mode for starting the channels:

per channel: all channels were started individually.

per 2 channels: 2 channels each have been started jointly.

per block: the 4 channels of a block were started jointly.

Stop criteria

Time

Time until the automatic stop of the determination.

Conductivity

Conductivity limit value. The determination was automatically ended upon reaching this value.

Endpoint

Automatic stop of the determination, as soon as every endpoint (induction and stability time) has been reached.

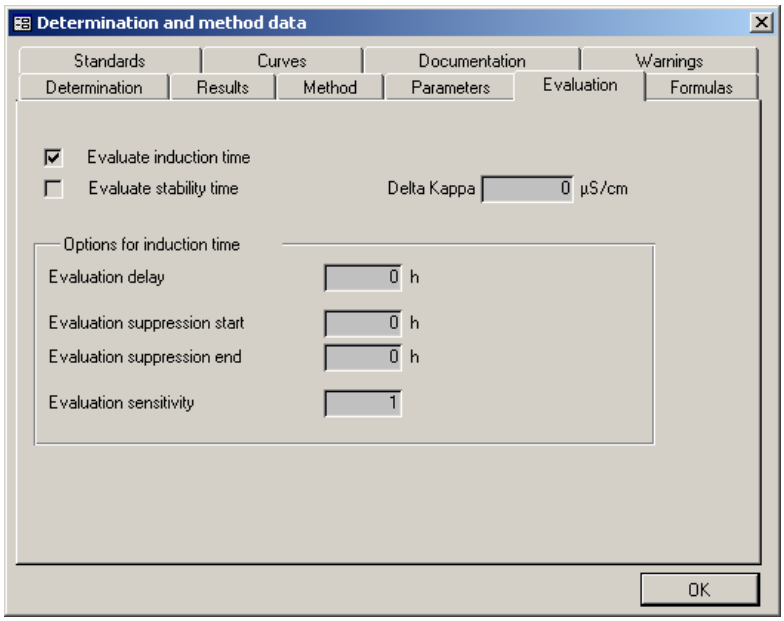
Actions at end of determinations...

Shut off heater

The heating was automatically deactivated at the end of the determination.

Shut off gas flow

The gas flow was automatically deactivated at the end of the determination.



Evaluate induction time

The induction time has been automatically determined *(see "Induction time", page 66)*.

Evaluate stability time

The stability time has been automatically determined *(see "Stability time", page 67)*.

Delta Kappa

Conductivity change for the determination of the stability time.

Options for induction time

Evaluation delay

Waiting time until starting the curve evaluation.

Evaluation suppression

Start

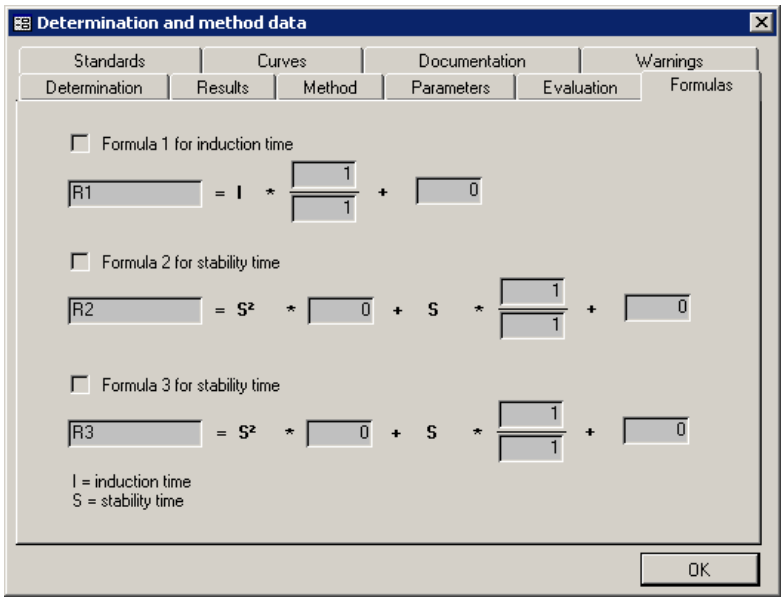
Time from the start of the determination to the start of the evaluation suppression.

End

Time from the start of the determination to the end of the evaluation suppression.

Evaluation sensitivity

Evaluation sensitivity used for the determation of the induction time.



Formula 1 for induction time

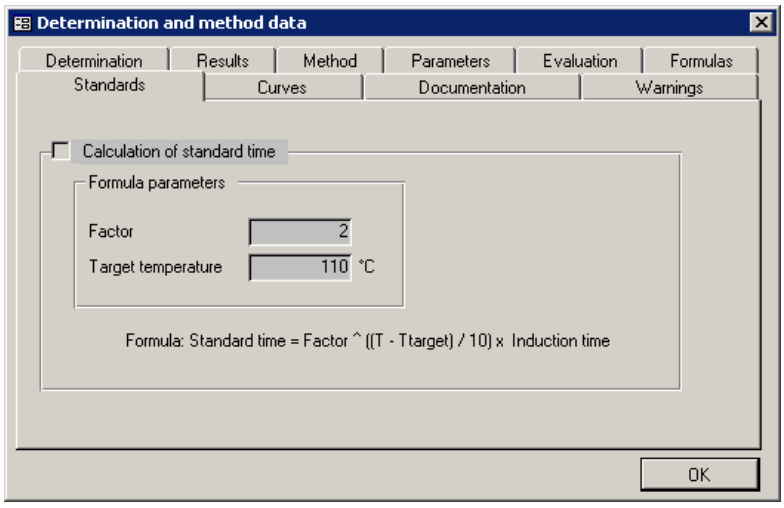
The automatically determined induction time was calculated with these values and saved under the name shown in the field "R1".

Formula 2 for stability time

The automatically determined stability time was calculated with these values and saved under the name shown in the field "R2".

Formula 3 for stability time

The automatically determined stability time was calculated with these values and saved under the name shown in the field "R3".



Calculation of standard time

Indicates whether the standard time has been calculated or not.

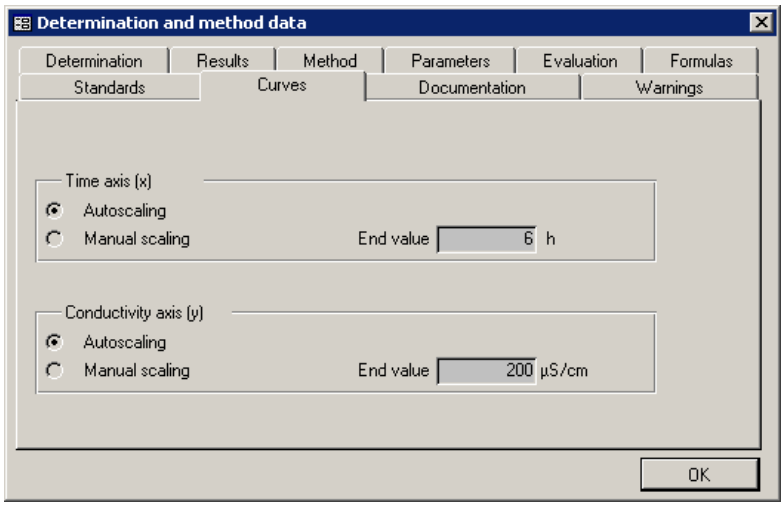
Formula parameters

Factor

Parameter for the calculation of the standard time. It indicates by which factor the time changes per 10 °C.

Target temperature

Target temperature for the calculation of the standard time.



Time axis (x)

Autoscaling

Automatic definition of the start and end value of the time axis.

Manual scaling

Manual definition of the end value of the time axis.

End value

End value of the time axis with manual scaling.

Conductivity axis (y)

Autoscaling

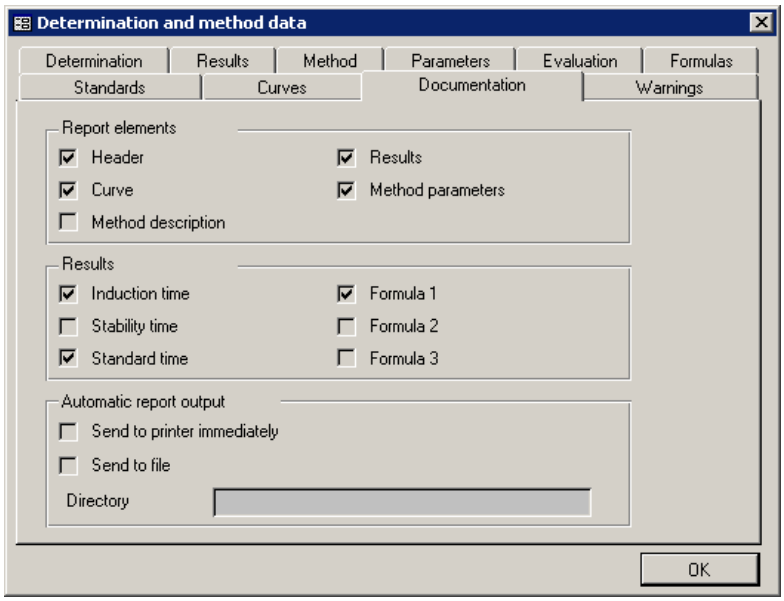
Automatic definition of the start and end value of the conductivity axis.

Manual scaling

Manual definition of the end value of the conductivity axis.

End value

End value of the conductivity axis with manual scaling.



Report elements

Header

Output of the header.

Curve

Output of the curves.

Method description

Output of the method description entered on the tab **Description**.

Results

Output of the results selected below.

Method parameters

Output of the method parameters.

Results

Output of the selected results: **Induction time**, **Stability time**, **Standard time**, **Formula 1...3**.

Automatic report output

Send to printer immediately

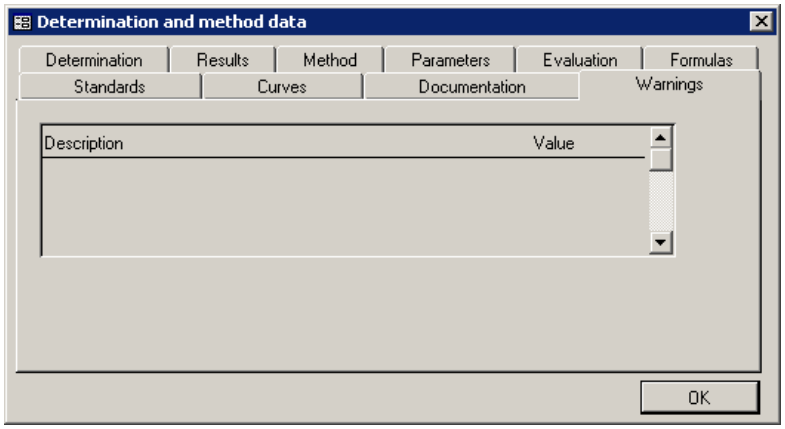
Automatic printout of the report after finishing the determination.

Send to file

Automatic output of determination and method data after finishing the determination as a TXT file.

Directory

Directory in which the report has been saved.



Description

The following warning messages are possible for incorrectly executed determinations:

Temperature not reached: setpoint temperature at start of the determination not reached.

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

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

Manual Stop: the determination was stopped manually with **[Stop]**.

Stop after communication error: Automatic stop of the determination because of interrupted connection between the Biodiesel Rancimat and the PC.

Value

Value for warning (e.g. temperature, time).

4.7.3 Graph and reevaluation

You can either open a graph of an individual determination (**single graph**) or have graphs of several determinations displayed above one another (**multiple graph**). In addition, you can open the so-called **live graph** of a determination, in the dialog window of which you can manually perform reevaluations of measuring curves.

You have the following options for reevaluation of graphs:

- Setting the induction time manually
- Setting the stability time manually
- Setting tangents manually

Single graph, multiple graph or live graph

Proceed as follows to open a single graph or several graphs:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

2 Open single graph

- Select the determination for which the graph is to be displayed *(see "Selecting determinations", page 86)*.
- Click on the symbol  *or* the menu item **Determination ▸ Graphics ▸ Single graph**.

The dialog window with the single graph appears (**ID 1** / **ID 2**).

If present, the determined **induction time** (red, vertical line and numerical value) and **stability time** (black, vertical line and numerical value) are displayed in the graphics window next to the **measurement curve** (green). A maximum of 16 single graph windows can be opened.

Note

In contrast to the windows *Multiple graph* and *Live graph*, the window with the single graph is **not** automatically updated upon selecting another determination in the determination overview

3 Open multiple graph

- Select the determinations for which the graphs are to be displayed *(see "Selecting determinations", page 86)*.
- Click on the symbol  *or* the menu item **Determination ▸ Graphics ▸ Multiple graph**.

The dialog window **Multiple graph** appears.

The individual measurement curves are displayed in color, the legends for the color (sample identifications **ID 1** and **ID 2**) are located in the header of the graph. If present, the determined **induction time** (▼) and **stability time** (▲) are displayed as well.

4 Open live graph

- Select the determination for which a reevaluation of the measurement curve is to be performed *(see "Selecting determinations", page 86)*.

- Click on the symbol  *or* the menu item **Determination ▸ Graphics ▸ Live graph with reevaluation**.

The dialog window **Live graph** appears.

If present, the determined **induction time** (red, vertical line and numerical value) and **stability time** (black, vertical line and numerical value) are displayed in the graphics window next to the **measurement curve** (green).

With the button **[Evaluation]** you can display the second derivative of the measuring curve as well as the used evaluation sensitivity.

Zooming graphs, switching on/off offset and autoscaling

Proceed as follows to adjust the graph display:

1 Zoom graph

- *See "Zooming", page 31*.

2 Switch on/off offset for multiple curves

- Right click in the dialog window of a multiple graph.
  The context-sensitive menu for graphs appears.
- Click on the menu item **Offset**.

The offset for the displayed measurement curve is switched on or switched off.

In the case of an activated offset, the curves are arranged with an offset above one another. Instead of the usual lettering of the conductivity axis, the difference of the entire axis is in this case displayed in µS/cm.

3 Switch on/off autoscaling for single and live graphs

- Right click in the dialog window of a single graph or live graph.
  The context-sensitive menu for graphs appears.
- Click on the menu item **Autoscaling**.

The autoscaling for the displayed measurement curves is now activated or the manually performed scaling *(see Chapter 4.5.2.3, page 67)* is canceled.

If this option is activated, the start and end values of the axes are automatically specified in such a way that all measured values are visible.

Copying graphs and pasting them into other programs

Proceed as follows to copy a graph to the clipboard and then paste it into another program:

1 Copy graph

- Right click in the dialog window of a graph.
  The context-sensitive menu for graphs appears.
- Click on the menu item **Copy**.

2 Paste graph

- Open program (e.g. Word, Excel, any graphics program, etc.)
- Paste the graph using the key combination **[Ctrl]** + **[V]**.

Printing graphs

See "Printing graphs", page 126.

Setting the induction time manually

Proceed as follows to subsequently and manually set the induction time:

1 Open dialog window

- Right click in the dialog window of a live graph and click on the entry **Reevaluation** in the context-sensitive menu.

The dialog window **Reevaluation** appears.

2 Set induction time

- Click on the symbol  .
- Place the cursor on the red line.
  The cursor assumes the following form: ⟷
- With the **[Ctrl]** key and **left mouse button pressed down**, drag the line to the required location.

The value of the manually set induction time is displayed both in the dialog window **Live graph** at the top right beside the red line and also in the dialog window **Reevaluation** under **Manual**.

3 Save changes

- In the dialog window **Live graph** click on the button **[Save]**.

The new induction time is accepted in the determination overview.

Setting the stability time manually

In order to set the stability time manually, you need to adjust the conductivity change **Delta Kappa**. After this, the stability time is recalculated automatically. Proceed as follows:

1 Open dialog window

- Right click in the dialog window of a live graph and click on the entry **Reevaluation** in the context-sensitive menu.

The dialog window **Reevaluation** appears.

2 Adjust Delta Kappa

- Click on the symbol  .
- Under **Delta Kappa** enter the required value and confirm with the symbol  .

The value of the calculated stability time is displayed both in the dialog window **Live graph** at the top right beside the gray line and also in the dialog window **Reevaluation** under **Stability time**.

3 Save changes

- In the dialog window **Live graph** click on the button **[Save]**.

The new stability time is accepted in the determination overview.

Setting tangents manually

For determining the **induction time**, you can manually set the tangents. The induction time is then defined as the **intersection of the tangents**. Set the tangents as follows:

1 Open dialog window

- Right click in the dialog window of a live graph and click on the entry **Reevaluation** in the context-sensitive menu.

The dialog window **Reevaluation** appears.

2 Set first tangent

- Click on the symbol  .
- Keep the **[Ctrl]** key and **left mouse button pressed down** to set the first tangent.

3 Set second tangent

- Keep the **[Shift]** key and **left mouse button pressed down** to set the second tangent.

4 Adjust the position of the tangents

- Position the cursor on a black square on the tangent.
  The cursor assumes the form of a cross hair.
- Keep the **[Ctrl]** key and the **left mouse button pressed down** to move the point to the required position.

After you have adjusted the position of both tangents, you can read the value of the new induction time both in the dialog window **Live graph** at the top right beside the red line and also in the dialog window **Reevaluation** under **Manual**.

5 Save changes

- In the dialog window **Live graph** click on the button **[Save]**.

The new induction time is accepted in the determination overview.

Deleting manually set tangents

Proceed as follows to delete the previously set tangents:

1 Delete tangents

- Click on the symbol  .

Resetting all reevaluations

1 Reset induction and stability time to the original value

- Click on the **[Reset]** button.

4.7.4 Extrapolation

Extrapolation can be used to extrapolate the results measured at various temperatures to the required target temperature. In addition, the **factor** for converting the **induction time** to the **standard time** can be determined.

A detailed description of the parameters can be found at *"Parameter description", page 116*.

Extrapolation of several results

Proceed as follows to carry out an extrapolation of several results:

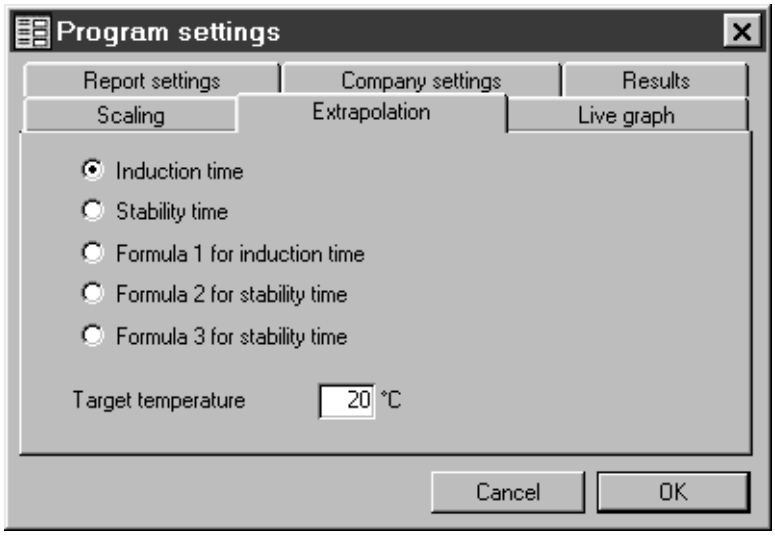
1 Open dialog window

- Open the Results window and the database *(see "Displaying the determination overview", page 83)*.
- Select the required determinations *(see "Selecting determinations", page 86)*.
- Click on the menu item **Determination ▸ Extrapolation**.

The dialog window **Extrapolation** with the extrapolation values which the program has calculated from the selected results (determinations) appears.

2 Select result and target temperature

- Click on the **[Settings]** button.
  The following dialog window appears:



- Select the result which is to be used for extrapolation (**Induction time**, **Stability time**, etc.).
- Enter the **Target temperature**.
- Confirm the entries with **[OK]**.

Note

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

3 Update the display

- Click on the **[Update]** button.

The graph and all results are updated.

Note

For extrapolation of the induction times, the manually changed values of the selected determinations (if present) are used, otherwise the automatically determined values are used.

Setting the correlation limit

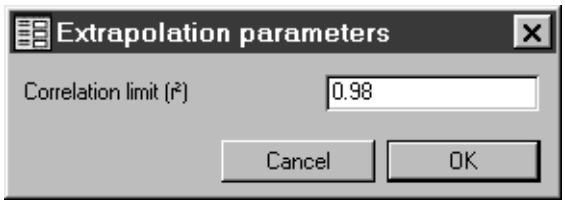
Note

The correlation limit for the **correlation coefficient r^2** can only be set by an Administrator.

1 Open dialog window

- In the Results window, click on the menu item **Options ▸ Administrator ▸ Extrapolation parameter**.

The following dialog window appears:



2 Adjust limit

- Under **correlation limit (r^2)** enter a value between 0 and 1 and confirm with **[OK]**.

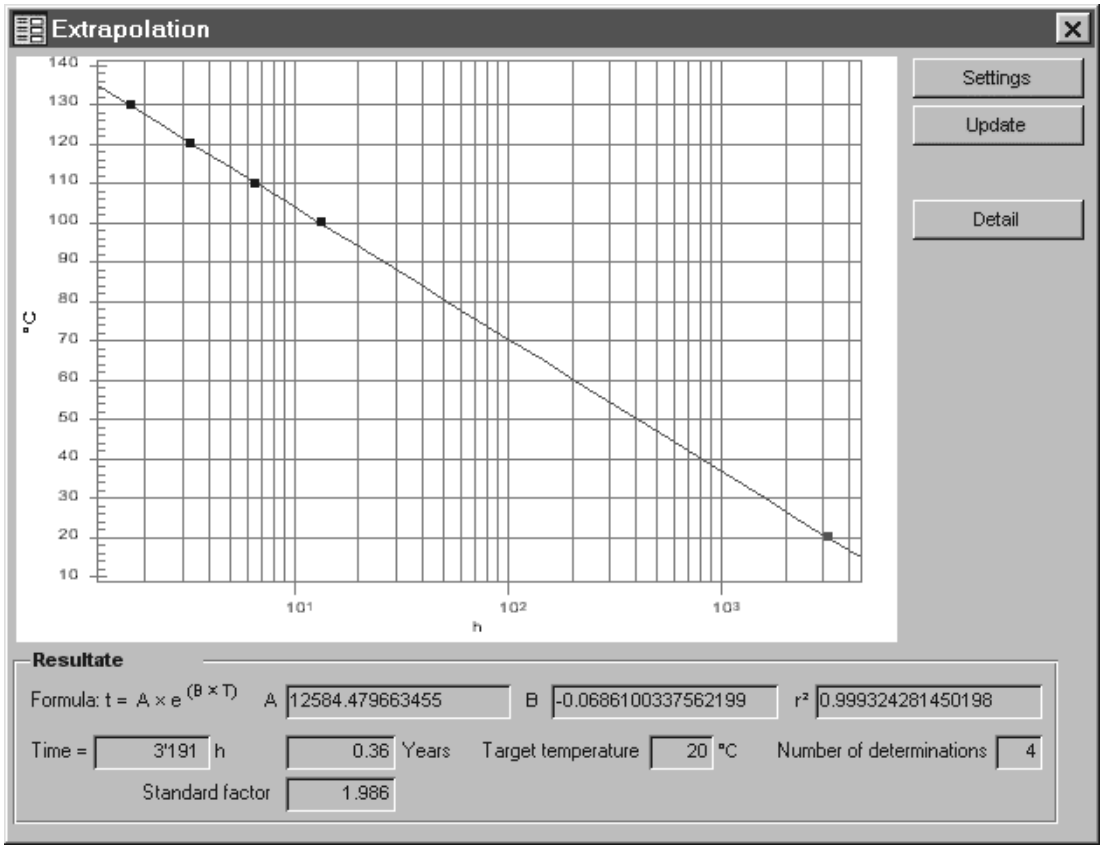
Copying a graph and pasting it into other programs

See "Copying graphs and pasting them into other programs", page 111.

Printing extrapolation results

See "Printing extrapolation results", page 127.

Parameter description



In the graphic **temperature vs. time** the linear regression is used to represent the calculated straight line **T = ln(t)/B – ln(A)/B**. 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**.

Results

Formula

Exponential formula for calculating the time extrapolated from the measured values for the target temperature.

A

Calculated coefficient A of the exponential formula.

B

Calculated coefficient B of the exponential formula.

r^2

Calculated correlation coefficient. If this value is below the limit value *(see "Setting the correlation limit", page 115)* specified by the administrator a warning appears when opening the extrapolation window.

Time

Result of the extrapolation for the target temperature in hours 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 straight line which indicates the factor by which the time changes per 10 °C. This value can be entered in the formula for the determination of the standard time as a factor *(see "Calculation of standard time", page 105)*.

[Settings]

Open the dialog window **Program settings**. The extrapolation parameters are to be entered in the tab **Extrapolation** *(see ""Extrapolation" tab", page 135)*.

[Update]

Update graphics and result display. This function has to be carried out if the selection of determinations in the determination overview or the settings for the extrapolation have been changed.

[Detail]

Display only the area with the results of the determinations in the graph.

4.7.5 Recalculating a determination

For a determination, you can recalculate the **Induction time**, the **Stability time**, the **Formulas** and the **Standard time**.

Recalculating the induction time

Proceed as follows to recalculate the induction time:

1 Open dialog window

- Select the determination which is to be recalculated *(see "Selecting determinations", page 86)*.
- Click on the menu item **Determination ▸ Recalculation**.

The dialog window **Recalculation** appears.

2 Adjust the parameters

- Activate the options **Recalculate induction time** and **Calculation active**.
- Under **Evaluation delay** and **Evaluation suppression start/end**, enter the required values.
- Under **Evaluation sensitivity**, enter the required value.
  The software suggests a value with which the highest maximum of the second derivative is accepted as the induction time. If there is no suggestion then the highest maximum has already been accepted.
  The default value "**1**" will lead to the correct result for most applications. Smaller values will cause the acceptance of smaller maximums of the second derivative. Higher values require a higher maximum.
- Confirm the entries with **[OK]**.

Note

A detailed description of the parameters can be found at *"Recalculate induction time", page 120*.

Recalculating the stability time

Proceed as follows to recalculate the stability time:

1 Open dialog window

- Select the determination which is to be recalculated *(see "Selecting determinations", page 86)*.
- Click on the menu item **Determination ▸ Recalculation**.

The dialog window **Recalculation** appears.

2 Adjust the parameters

- Activate the options **Recalculate stability time** and **Calculation active**.
- Under **Delta Kappa** enter the required value and confirm with **[OK]**.

Note

A detailed description of the parameters can be found at *"Recalculate stability time", page 121*.

Recalculating formulas

Proceed as follows to recalculate the results of the formulas:

1 Open dialog window

- Select the determination which is to be recalculated *(see "Selecting determinations", page 86)*.
- Click on the menu item **Determination ▸ Recalculation**.
  The dialog window **Recalculation** appears.
- Click on the tab **Formulas**.

2 Adjust the parameters

- Activate the options **Recalculate formula 1 for induction time** and **Calculation active**.
  or
  Activate the options **Recalculate formula 2 for stability time** and **Calculation active**.
  or
  Activate the options **Recalculate formula 3 for stability time** and **Calculation active**.
- Re-enter the parameters for the formula and confirm with **[OK]**.

Note

A detailed description of the parameters can be found at *"Recalculate formula 1 for induction time", page 122*.

Recalculating the standard time

Proceed as follows to recalculate the standard time:

1 Open dialog window

- Select the determination which is to be recalculated *(see "Selecting determinations", page 86)*.
- Click on the menu item **Determination ▸ Recalculation**.
  The dialog window **Recalculation** appears.

- Click on the tab **Standards**.

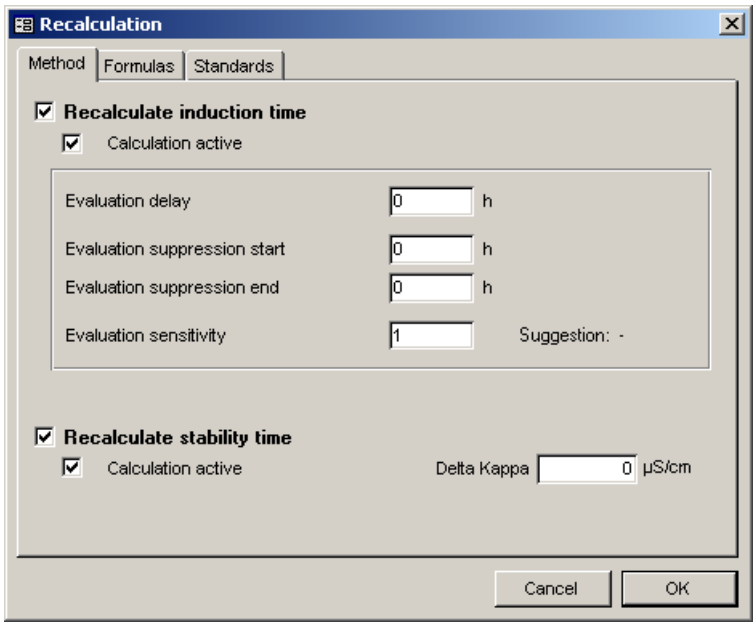
2 Adjust the parameters

- Activate the options **Recalculate standard time** and **Calculation active**.
- Under **Formula parameters**, enter the required values and confirm with **[OK]**.

Note

A detailed description of the parameters can be found at *"Recalculate standard time", page 123*.

Parameter description



Recalculate induction time

on | off (Default value: **off**)

Activate/Deactivate recalculation of the induction time. If the recalculation is deactivated, the original result is kept.

Calculation active

on | off (Default value: **on**)

If this option is **activated**, the parameters can be re-entered for the calculation of the induction time.

If this option is **deactivated**, the original result is deleted.

Evaluation delay

Waiting time until starting the curve evaluation.

Evaluation suppression start

Time from the start of the determination to the start of the evaluation suppression.

Evaluation suppression end

Time from the start of the determination to the end of the evaluation suppression.

Evaluation sensitivity

This value defines the minimum value which the maximum of the curve of the second derivative has to reach in order to be accepted as a result. The result, i.e. the induction time, is the time until the first accepted maximum.

The default value "**1**" will lead to the correct result for most applications. Smaller values will cause the acceptance of smaller maximums of the second derivative. Higher values require a higher maximum.

The software suggests a value with which the highest maximum of the second derivative is accepted as the induction time. If there is no suggestion then the highest maximum has already been accepted.

Recalculate stability time

on | off (Default value: **off**)

Activate/Deactivate recalculation of the stability time. If the recalculation is deactivated, the original result is kept.

Calculation active

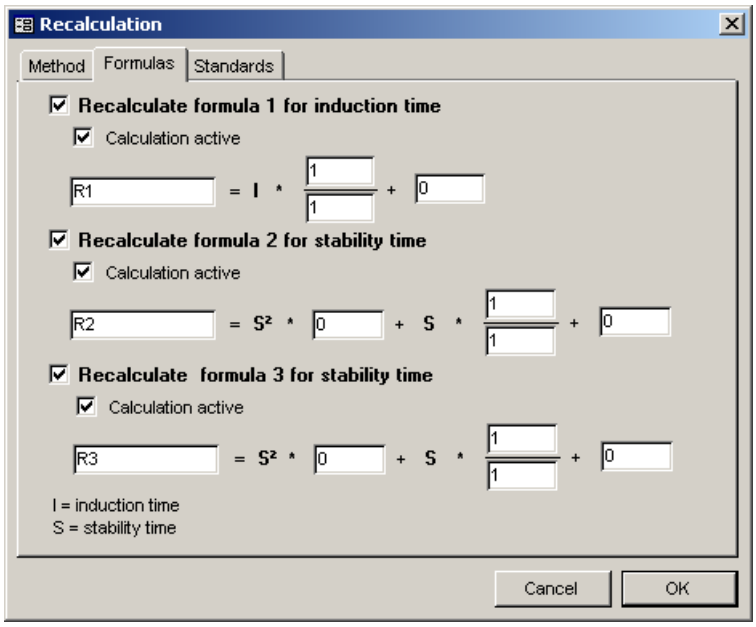
on | off (Default value: **off**)

If this option is **activated**, **Delta Kappa** can be re-entered for the calculation of the stability time.

If this option is **deactivated**, the original result is deleted.

Delta Kappa

Conductivity change for the determination of the stability time in µS/cm.



Recalculate formula 1 for induction time

on | off (Default value: **off**)

Activate/Deactivate the recalculation of the result calculated with formula 1. If the recalculation is deactivated, the original result is kept.

Calculation active

on | off (Default value: **off**)

If this option is **activated**, the parameters for formula 1 can be re-entered.

If this option is **deactivated**, the original result is deleted.

Recalculate formula 2 for stability time

on | off (Default value: **off**)

Activate/Deactivate the recalculation of the result calculated with formula 2. If the recalculation is deactivated, the original result is kept.

Calculation active

on | off (Default value: **off**)

If this option is **activated**, the parameters for formula 2 can be re-entered.

If this option is **deactivated**, the original result is deleted.

Recalculate formula 3 for stability time

on | off (Default value: **off**)

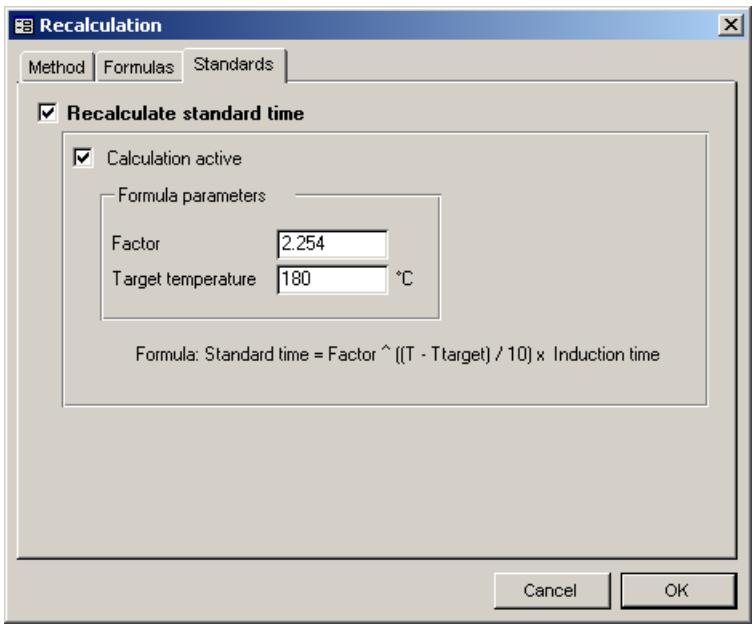
Activate/Deactivate the recalculation of the result calculated with formula 3. If the recalculation is deactivated, the original result is kept.

Calculation active

on | off (Default value: **off**)

If this option is **activated**, the parameters for formula 3 can be re-entered.

If this option is **deactivated**, the original result is deleted.



Recalculate standard time

on | off (Default value: **off**)

Activate/Deactivate the recalculation of the **standard time** from the **induction time**. If the recalculation is deactivated, the original result is kept.

Calculation active

on | off (Default value: **off**)

If this option is **activated**, the parameters for the calculation of the standard time can be re-entered.

If this option is **deactivated**, the original result is deleted.

Formula parameters

Factor

Parameter for the calculation of the standard time, which indicates the factor by which the time changes per 10 °C.

Target temperature

Target temperature for the calculation of the standard time.

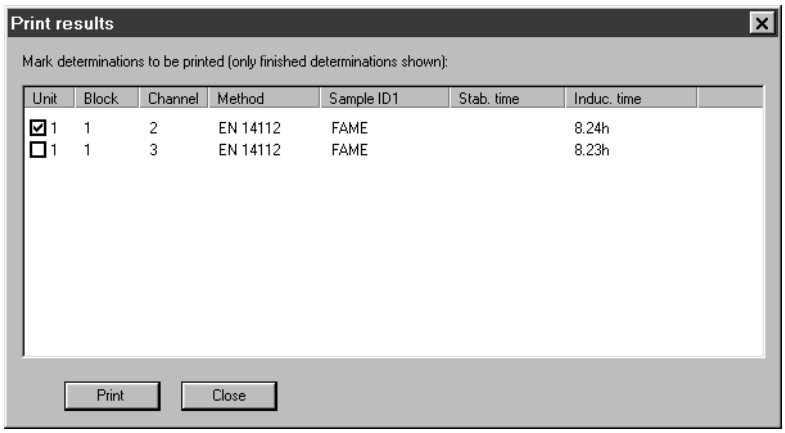
4.7.6 Printing and exporting data

Printing results from the Control window

Proceed as follows to print results of the completed determinations shown in the Control window:

1 Open dialog window

- In the **Control window**, click on the symbol *or* the menu item **File ▸ Print...**.
  The following dialog window appears:



2 Print results

- If required, adjust the column width *(see "Formatting the determination overview", page 83)*.
- Select the required determinations and click on **[Print]**.

The results are printed according to the settings which have been defined in the method on the tab **Documentation**.

Printing determination overview

You can print a determination overview either in the form of a table or a list. Proceed as follows:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

2 Select type of output (table or list)

- Click on the symbol *or* the menu item **File ▸ Print...**.
  The dialog window **Printing of...** appears.

- Click on the option **Overview table** and/or **Overview list**.

> **Note**
>
> In the overview table, only those columns are printed which are within the maximum print width (15 cm).

3 Print determination overview

- Click on the **[Print]** button.
  The dialog window **Print** appears.
- Select the required printer and click on **[Print]**.

The determination overview will be printed.

Printing determination and method data

Proceed as follows to print the determination and method data of one or several determinations:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

2 Select determination

- Select the determination of which the data is to be printed *(see "Selecting determinations", page 86)*.

3 Select data

- Click on the symbol *or* the menu item **File ▸ Print...**.
  The dialog window **Printing of...** appears.
- Select the option **All determination and method data** and confirm with **[Print]**.

The dialog window **Print** appears.

4 Print data

- Select the required printer and click on **[Print]**.

The determination and method data will be printed.

Printing graphs

Proceed as follows to print a single graph, multiple graph or live graph:

1 Open graph

- *See "Single graph, multiple graph or live graph", page 109.*

2 Print graph

- Click on the symbol  *or* the menu item **File ▸ Print...**.
  The dialog window **Print** appears.
- Select the required printer and click on **[Print]**.

The graph will be printed.

Printing the report defined in the method

Proceed as follows to print the report of one or several determinations, which has been defined in the method *(see Chapter 4.5.2.6, page 71)*:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

2 Select determination

- Select the determination of which the report is to be printed *(see "Selecting determinations", page 86)*.

3 Select data

- Click on the symbol  *or* the menu item **File ▸ Print...**.
  The dialog window **Printing of...** appears.
- Select the option **Determination according to method** and confirm with **[Print]**.

The dialog window **Print** appears.

4 Print report

- Select the required printer and click on **[Print]**.

The report will be printed.

Printing the report defined under "Settings"

Proceed as follows to print the report of one or several determinations, which has been defined under **Settings**:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

2 Select determination

- Select the determination of which the report is to be printed *(see "Selecting determinations", page 86)*.

3 Select data

- Click on the symbol  *or* the menu item **File ▸ Print...**.
  The dialog window **Printing of...** appears.
- Select the option **Determination according to settings**.

4 Carry out settings

- Click on the **[Settings]** button.
  The dialog window **Program settings** appears.
- Carry out the required settings *(see tab "Report settings", page 132)* and confirm with **[OK]**.

The dialog window **Printing of...** appears.

5 Print report

- Click on the **[Print]** button.
  The dialog window **Print** appears.
- Select the required printer and click on **[Print]**.

The report will be printed.

Printing extrapolation results

Proceed as follows to print the **extrapolation graph** (normal and detailed view), the **extrapolation results** and the overview of the **determinations** used for the extrapolation with the fields ID 1, ID 2, temperature and result:

1 Display extrapolation results

- *See "Extrapolation of several results", page 114*.

2 Print extrapolation results

- Click on the symbol  *or* the menu item **File ▸ Print...**.
  The dialog window **Print** appears.
- Select the required printer and click on **[Print]**.

The extrapolation results will be printed.

Exporting the determination overview to Word or Excel

Proceed as follows to export the determination overview to Word or Excel:

1 Select determinations

- *See "Selecting determinations", page 86*.
  If you do not select any determinations, the entire overview is exported.

2 Select export program

- Click on the symbol  *or* the menu item **Determination ▸ Export ▸ Export to MS Word**.
  The data is copied to a RTF file to be opened automatically by Microsoft Word.

or

- Click on the symbol  *or* the menu item **Determination ▸ Export ▸ Export to MS Excel**.
  Microsoft Excel will be opened automatically.

Exporting determination and method data to a TXT file

Proceed as follows to export the determination and method data of one or several determinations to a TXT file:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

2 Select determination

- Select the determination of which the data is to be exported *(see "Selecting determinations", page 86)*.

3 Export data

- Click on the symbol  *or* the menu item **Determination ▸ Export ▸ Determination and method data**.
  The dialog window **Save to export file** appears.
- Select memory location, enter file name and confirm with **[Save]**.

The determination and method data will be exported.

Exporting determinations to another database

You can export determinations from a database to another existing database or a newly created database. Proceed as follows:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

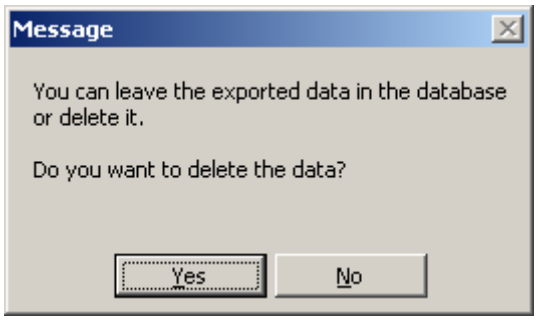
2 Select determination

- Select the determination to be exported *(see "Selecting determinations", page 86)*.

3 Select database

- Click on the menu item **Determination ▸ Export ▸ Export to database**.
  The dialog window **Export data to...** appears.
- Select an existing database.
  or
  Under **File name** enter the name for the new database.
- Confirm the selection or entry with **[Save]**.

The following message appears (only for the Administrator):



4 Keep or delete determination

- Click on **[No]** to also **keep** the exported determination in the original database.

- Click on **[Yes]** to **delete** the exported determination in the original database.

Opening the database in Microsoft Access 97

Note

Each database is password-protected. The password is generated automatically by the program and can only be read by the Administrator.

The databases are saved in the installation directory of the 873 Biodiesel Rancimat program at **C:\Program Files\Metrohm\Biodiesel Rancimat\Database**.

1 Open Results window

- In the **Control window**, click on the symbol  *or* the menu item **File ▸ Results...**.

The **Results window** appears.

2 Read out database password

- In the **Results window**, click on the menu item **Options ▸ Administrator ▸ Database password**.
  The dialog window **Open** appears.
- Select the required database and confirm with **[Open]**.

The dialog window **Password** appears with the password for the previously selected database.

3 Open database in Microsoft Access 97

- Start Microsoft Access 97.
- Open the required database by entering the previously read out password.

Exporting the measured value list

Proceed as follows to export a list of all measured values to a TXT file:

1 Display determination overview

- *See "Displaying the determination overview", page 83*.

2 Select determination

- Select the determination of which the data is to be exported *(see "Selecting determinations", page 86)*.

3 Export data

- Click on the symbol  *or* the menu item **Determination ▸ Export ▸ List of measured values**.
  The dialog window **Save to export file** appears.
- Select the memory location, enter the file name and confirm with **[Save]**.

The measured values list will be exported.

4.7.7 Program settings

Carrying out settings for the representation and output of results

You can carry out settings for the representation and output of results as follows:

1 Open dialog window

- In the **Results window**, click on the menu item **Options ▸ Settings**.

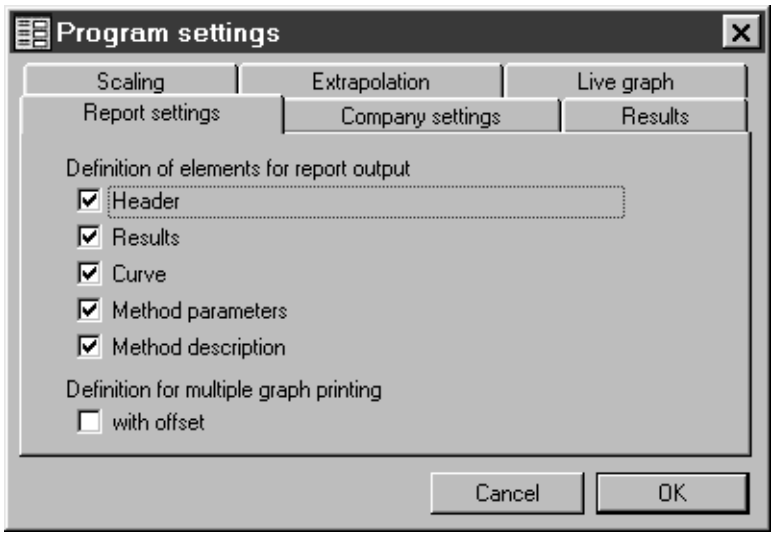
2 Carry out settings

- *See the following "Report settings" tab*.

4.7.7.1 Parameter description

"Report settings" tab

On the **Report settings** tab, you can carry out the settings for the report output.



Definition of elements for report output

Header

on | off (Default value: **on**)

Output of the header with company settings (if defined, *see "Company settings" tab*), date of the printout, sample identifications, instrument information, determination date, user, cell constant and serial number.

Results

on | off (Default value: **on**)

Output of the results selected in the **Results** tab *(see page 134)*.

Curve

on | off (Default value: **on**)

Output of the curves.

Method parameters

on | off (Default value: **on**)

Output of the method parameters.

Method description

on | off (Default value: **on**)

Output of the method description entered in the tab **Description**.

Definition for multiple graph printing

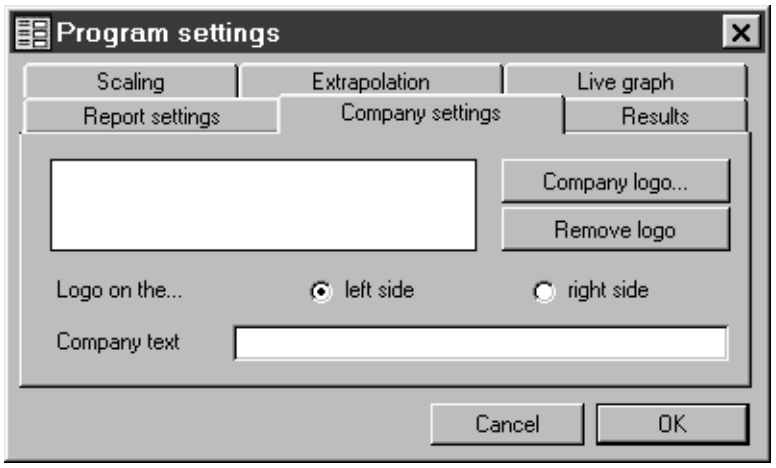
with offset

on | off (Default value: **off**)

If this option is activated, the curves are arranged with an offset above one another in the case of an output of multiple graphs. Instead of the usual lettering of the conductivity axis, the difference of the entire axis is, in this case, printed in µS/cm.

"Company settings" tab

On the **Company settings** tab, you can carry out the settings for the output of company logo and company text in the header.



[Company-logo…]

Open the window **Find company logo** for selecting the graphics file with the company logo. Only WMF or BMP files can be used. After clicking on **[Open]**, the logo is displayed in the adjoining window.

[Remove logo]

Remove the embedded company logo.

Logo on the...

Selection	**left side** | **right side**

left side
The company logo is printed out on the left edge of the header.

right side
The company logo is printed out on the right edge of the header.

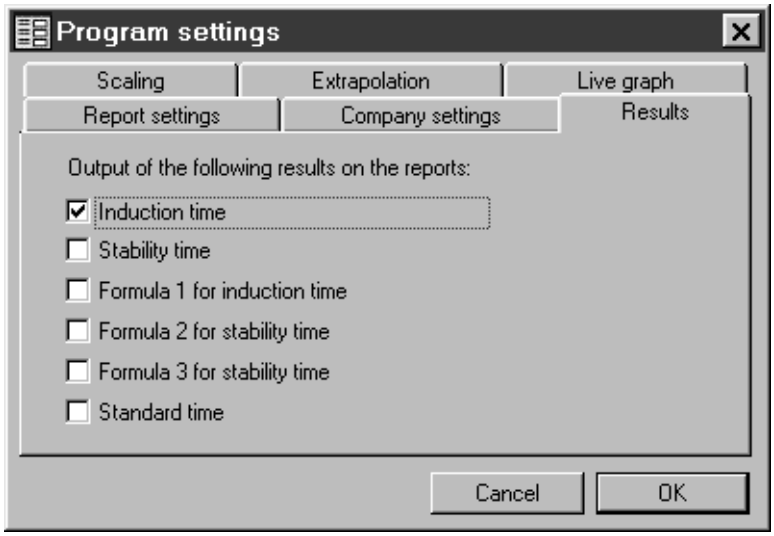
Company text

The text entered here is printed out on the side of the header opposite the company logo.

Input	**Characters**

"Results" tab

On the **Results** tab, you can carry out the settings for the result output.



Output of the following results on the reports:

Induction time

on | off (Default value: **on**)

Stability time

on | off (Default value: **off**)

Formula 1 for induction time

on | off (Default value: **off**)

Formula 2 for stability time

on | off (Default value: **off**)

Formula 3 for stability time

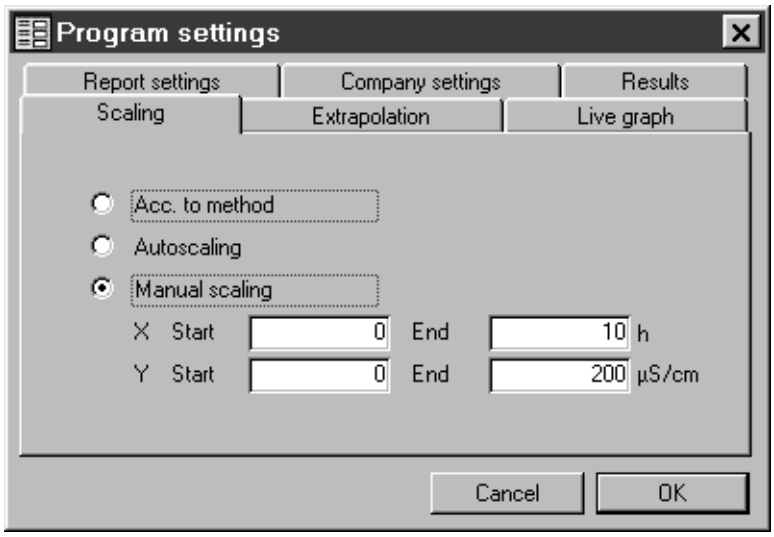
on | off (Default value: **off**)

Standard time

on | off (Default value: **off**)

"Scaling" tab

On the **Scaling** tab, you can carry out the settings for scaling the curve axes.



Acc. to method

Axis scaling in accordance with the settings defined in the method under **Curve representation** *(see Chapter 4.5.2.3, page 67)*.

Autoscaling

Automatic definition of the start and end values of the curve axes.

Manual scaling

Manual definition of the start and end values of the curve axes.

X Start

Start value of the time axis in hours.

X End

End value of the time axis in hours.

Y Start

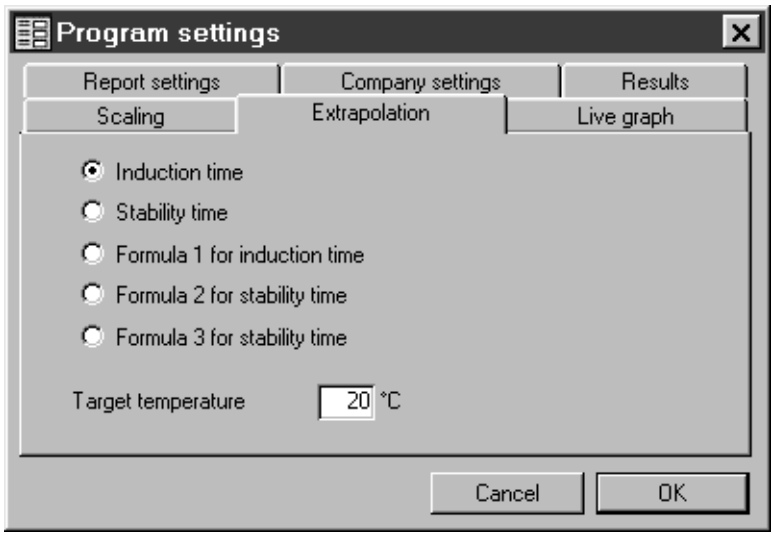
Start value of the conductivity axis in µS/cm.

Y End

End value of the conductivity axis in µS/cm.

"Extrapolation" tab

On the **Extrapolation** tab, you can carry out the settings for the extrapolation.



Induction time

The induction time is used for extrapolation. If the induction time has been determined manually, this value will be used and not the automatically determined induction time.

Stability time

The stability time is used for extrapolation.

Formula 1 for induction time

The result calculated with formula 1 is used for extrapolation.

Formula 2 for stability time

The result calculated with formula 2 is used for extrapolation.

Formula 3 for stability time

The result calculated with formula 3 is used for extrapolation.

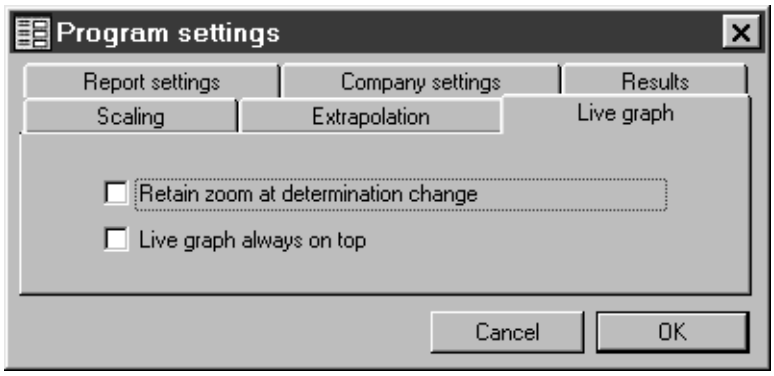
Target temperature

Temperature, for which the selected result is to be converted. The conversion is realized using the regression straight line (from the extrapolation).

Range	**freely selectable ... °C**

"Live graph" tab

On the **Live graph** tab, you can carry out the settings for displaying the live graph.



Retain zoom at determination change

on | off (Default value: **off**)

The selected zoom range is retained when changing the determination in the determination overview.

Live graph always on top

on | off (Default value: **off**)

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

4.7.8 Arranging windows

In the Results window, you can arrange the opened dialog windows either **above one another**, **beside one another** or **overlapping**. Proceed as follows:

1 Open windows

- Open all required dialog windows.
  The following dialog windows can be opened in the Results window:
  - Determination overview (**View ▸ Determination overview**)
  - Graphs (**Determination ▸ Graphics ▸ Single/Multiple/Live graph**)
  - GLP window (**View ▸ GLP**)

2 Arrange windows

- Click on the menu item **Window ▸ Tile vertically** *or* **Window ▸ Tile horizontally** *or* **Window ▸ Cascade**.

The opened dialog windows are correspondingly arranged.

4.8 GLP functions

4.8.1 General information on GLP and validation

GLP (**G**ood **L**aboratory **P**ractice) requires, amongst other things, the periodical testing of analytical measuring devices for their reproducibility and correctness on the basis of **Standard Operating Procedures** (**SOP**).

An example of such a standard operating procedure for the 873 Biodiesel Rancimat is available from Metrohm under the title *Application Bulletin No. 278 – Validation of Metrohm Rancimats*.

The operating software for the 873 Biodiesel Rancimat contains prepared **GLP tests** for temperature, conductivity and gas flow measurement. The user can determine whether and which tests have to be carried out. In addition, the time interval between the tests and the accuracy requirements can be specified. If the GLP function is activated, each results report receives a comment whether the GLP tests are fulfilled. Metrohm offers a GLP test set to carry out these tests *(see Optional accessories, page 180)*.

Further information on the subject of QA, GLP and validation can also be found in the brochure *Quality Management with Metrohm* available from your Metrohm representative.

Electronic and mechanical functional groups in Metrohm instruments can and should be checked as part of regular service by specialist personnel of the manufacturing company. All Metrohm instruments are provided with start-up test routines which check the perfect functioning of the relevant assemblies when switching on the instrument. If no error message is displayed, it can be assumed that the instrument functions without fault.

The 873 Biodiesel Rancimat also contains an integrated diagnostic program which enables the service technician to check certain assemblies if any faults or incorrect functioning occur and localize the fault.

4.8.2 GLP monitoring

Activating/Deactivating GLP monitoring

Proceed as follows to either activate or deactivate the GLP monitoring for the **temperature**, the **conductivity** and the **gas flow**:

1 Open dialog window

- If several instruments are connected: In the **Control window**, use the menu item **View ▸ Unit x** to select the required instrument.
- Click on the menu item **Tools ▸ GLP test ▸ Properties...**.

The dialog window **GLP properties for unit with serial no. x** appears.

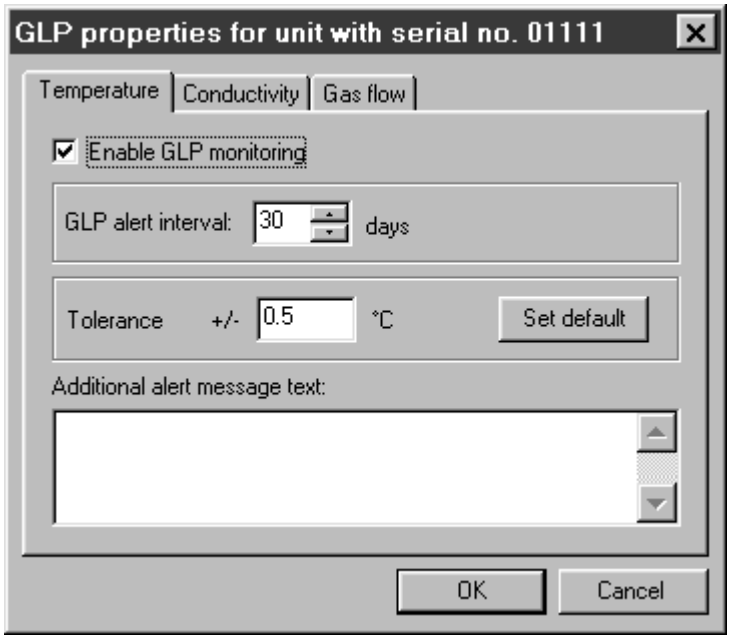
2 Switch on monitoring

- In the **Temperature** tab, activate the option **Enable GLP monitoring**.
- Carry out the remaining settings *(see "Parameter description", page 139)* and confirm with **[OK]**.

3 Switch off monitoring

- Open the dialog window **GLP properties for unit with serial no. x** (see step 1).
- In the required tab (**Temperature**, **Conductivity**, **Gas flow**), deactivate the option **Enable GLP monitoring** and confirm with **[OK]**.

Parameter description



Enable GLP monitoring

on | off (Default value: **off**)

Switch on/Switch off automatic GLP monitoring.

GLP alert interval

Number of days until the implementation of the next GLP test.

Tolerance

Indication of the tolerance value for the relevant GLP test. If the result is within the range **Setpoint ± Tolerance**, the test is regarded as passed. If you click on **[Set default]**, the specified standard value is entered in this field.

Additional alert message text

Additional message text which is to be added during the prompt for carrying out the next GLP test.

4.8.3 GLP status

Displaying the GLP status

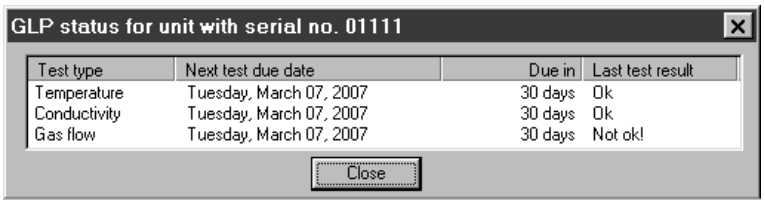
Proceed as follows to display the status of the GLP monitoring:

1 Open dialog window

- If several instruments are connected: In the **Control window**, use the menu item **View ▸ Unit x** to select the required instrument.
- Click on the menu item **Tools ▸ GLP test ▸ Status...**.

The dialog window **GLP status for unit with serial no. x** appears.

Parameter description



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 the GLP monitoring is not switched on, **Disabled** appears in this column.

Due in

Number of days until the next GLP test.

Last test result

Indication whether the test has been passed (**Ok**) or not (**Not Ok!**).

4.8.4 Carrying out GLP tests

4.8.4.1 Temperature

For the **GLP test for temperature measurement** the calibrated temperature sensor and the measuring insert are required (part of the optionally available GLP test set, *see Optional accessories, page 180*). For this, the temperature sensor must be connected to the Pt100 connector on the rear of the 873 Biodiesel Rancimat.

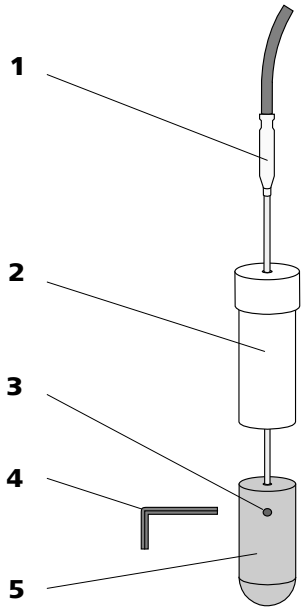


Figure 7 Accessories for GLP test Temperature

1	**Pt100 temperature sensor**	**2**	**PTFE cylinder**
3	**Hexagon screw**	**4**	**Hexagon key (6.2621.120)**
5	**Aluminum cylinder**		

Preparing the GLP test set

Proceed as follows in order to prepare the GLP test set according to *Figure 7*:

1 Assemble accessories

- Insert the temperature sensor from above into the opening of the PTFE cylinder.
- Place the PTFE cylinder on the aluminum cylinder in such a way that the temperature sensor fits into this opening.
- Push the temperature sensor into the aluminum cylinder as far as the limit stop.

2 Screw on accessories

- Use the hexagon key to lightly tighten the hexagon screw.

Carrying out the GLP test for the temperature measurement

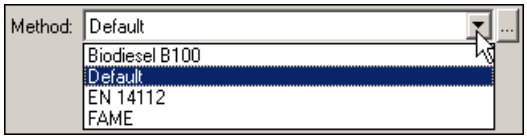
Proceed as follows to carry out the GLP test:

Note

Before starting the GLP test, ensure that the temperature of the inserted GLP test set is stable. If the GLP test set is used in a cold heating block, this condition is fulfilled after the normal heating up time of the heating block and the GLP test set. If the GLP test set, on the other hand, is used in an already heated heating block, it is necessary to wait at least 30 min before starting the GLP test.

1 Select instrument and method

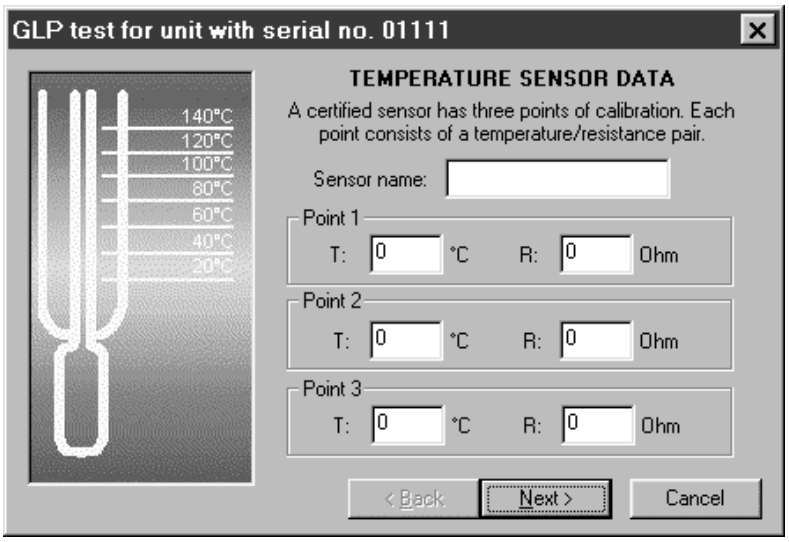
- In the **Control window**, use the menu item **View ▸ Unit x** to select the instrument for which the GLP test is to be carried out.
- Under **Unit x Block A** and **B**, click on the symbol in order to select the method whose temperature is required for the test.



- If required, adjust the temperature *(see "Opening a selected method and adjusting parameters", page 57)*.

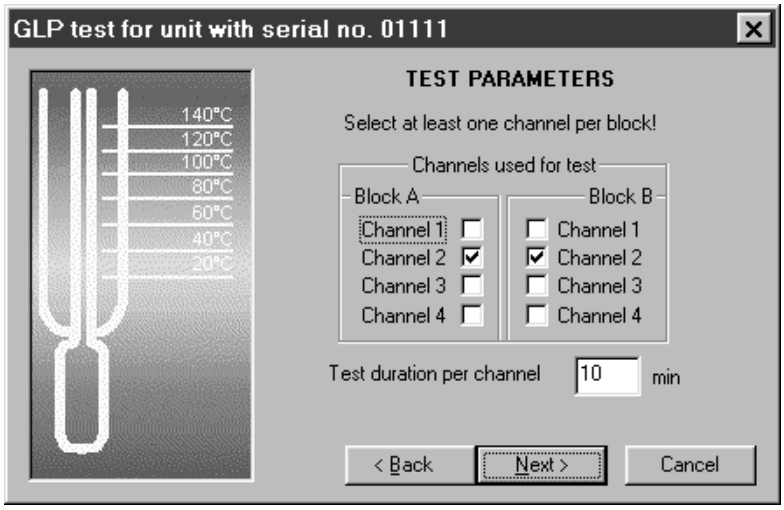
2 Enter calibration points of the temperature sensor

- Click on the menu item **Tools ▸ GLP test ▸ Temperature...** and confirm with **[Next >]**.
  The following dialog window appears:



- Enter a **Sensor name** for the temperature sensor.
- Under **Point 1...3**, enter the **Temperature** and the **Resistance** of the calibration point.
- Confirm the entry with **[Next >]**.

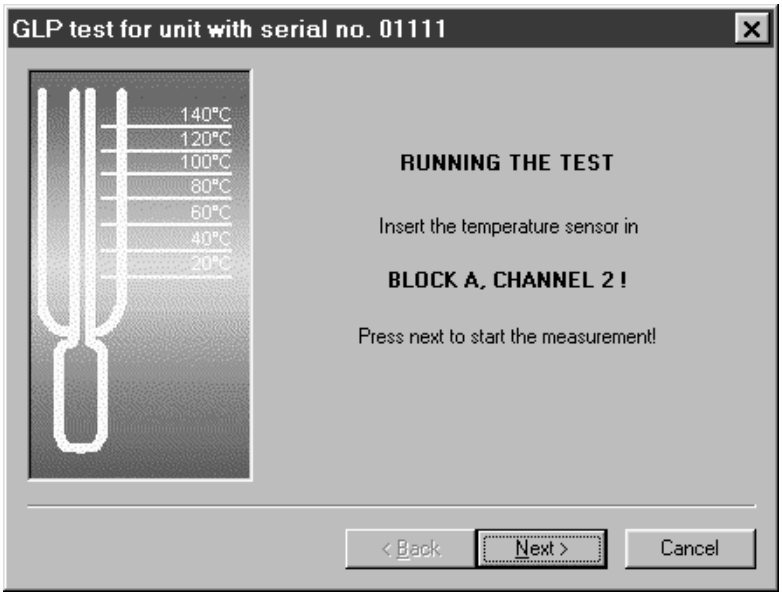
The following dialog window appears:



3 Enter test parameters

- Under **Channels used for test**, select the required channels. One channel per block has to be selected at least.
- Under **Test duration per channel**, enter the required test duration and confirm with **[Next >]**.

The following dialog window appears:

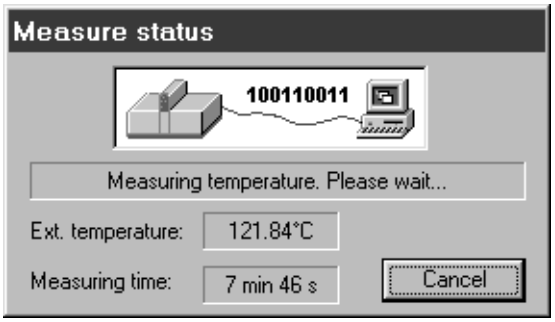


4 Insert GLP test set and start measurement

- Insert the prepared test set *(see "Preparing the GLP test set", page 141)* in the channel of Block A to be checked.
- Connect the temperature sensor to the Pt100 connector on the rear of the 873 Biodiesel Rancimat.
- In the dialog window **GLP test for unit with serial no. x**, click on **[Next >]**.

The test measurement is started.

As soon as the temperature defined in the method has been reached, the following message appears:



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

5 Carry out measurement for further channels

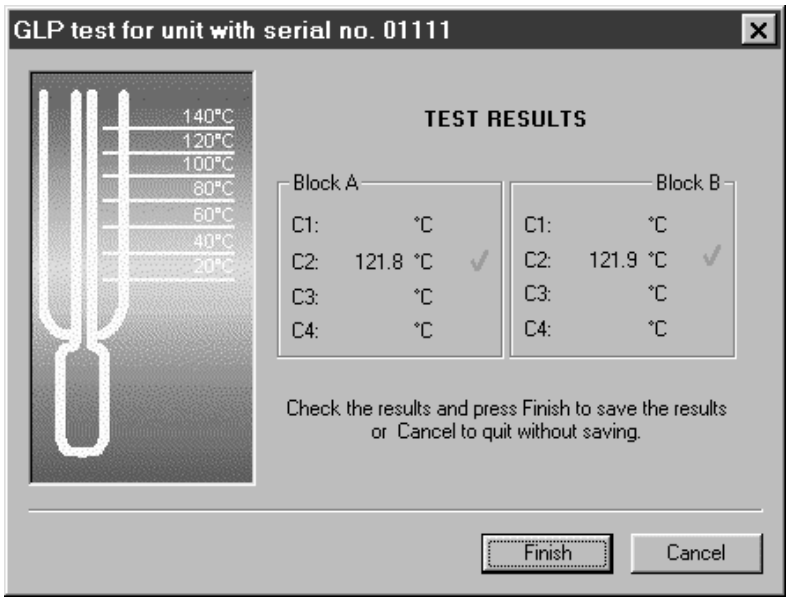
Warning

The aluminum cylinder of the GLP test set is very hot. Do not touch!

- As soon as the message appears with the prompt to insert the external temperature sensor together with the measuring insert in the next selected channel, **rapidly carry out the change**.
- Carry out measurement for each of the selected channels.

After the last measurement is completed, the dialog window with the **test results** appears.

6 Confirm or reject results



✓	**Test passed**: The deviation of the measured temperatures is within the specified tolerance.
✗	**Test failed**: The deviation of the measured temperatures is greater than the specified tolerance.

- Confirm the results with **[Finish]**.

or

- Reject the results with **[Cancel]**.

4.8.4.2 Conductivity

The **GLP test for conductivity measurement** can be carried out either with a **standard solution** with a known conductivity or with the **test resistor 6.2109.030**. The test resistor is part of the optionally avaiable GLP test set *(see Optional accessories, page 180)*. The test resistor must be connected during measurement at the electrode connector *(3-**2**)* of the 873 Biodiesel Rancimat.

Carrying out the GLP test for the conductivity measurement

Proceed as follows to carry out the GLP test:

Note

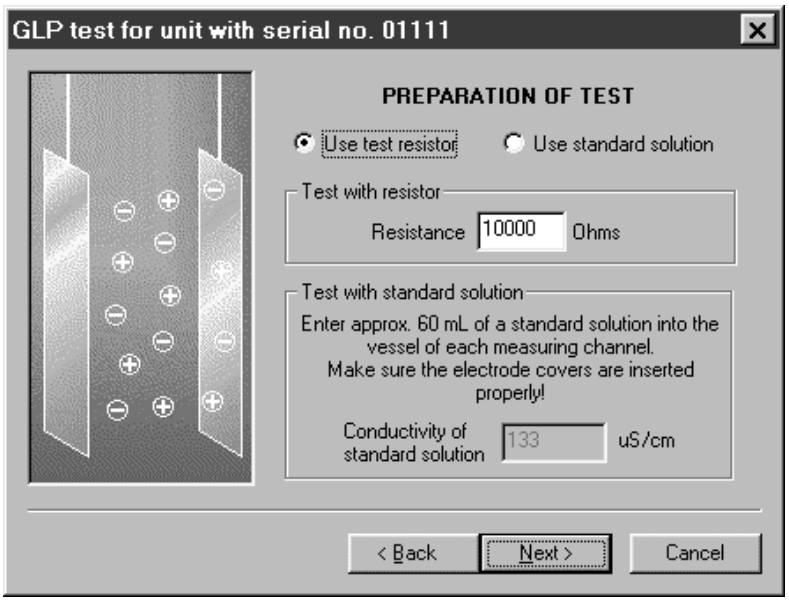
Before carrying out the GLP test **with standard solution** for the first time, the cell constants of the conductivity measuring cells must be determined *(see Chapter 4.4.1, page 48)*.

1 Select instrument

- In the **Control window**, use the menu item **View ▸ Unit x** to select the instrument for which the GLP test is to be carried out.
- Carrying out the GLP test with the **test resistor**: continue with step 2.
  Carrying out the GLP test with the **standard solution**: continue with step 3.

2 Carry out GLP test with test resistor

- Click on the menu item **Tools ▸ GLP test ▸ Conductivity...** and confirm with **[Next >]**.
  The following dialog window appears:

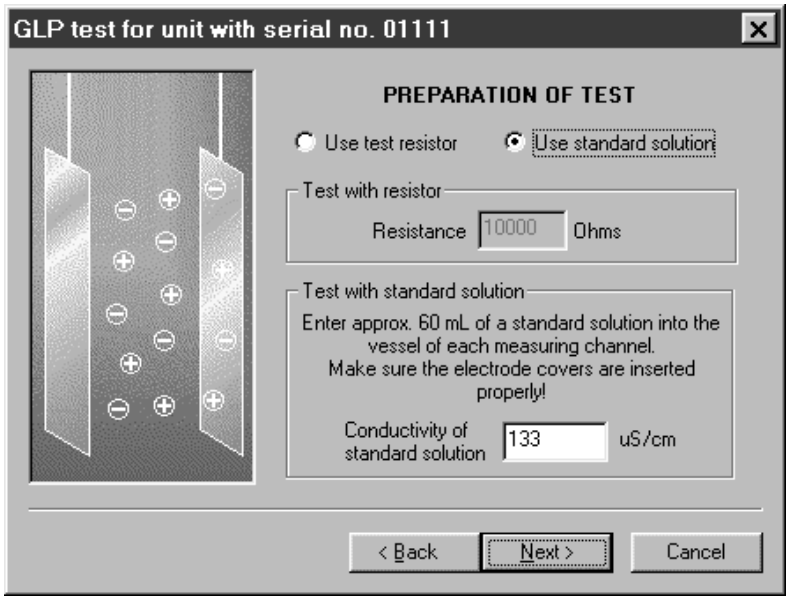


- Select the option **Use test resistor**.
- Under **Resistance**, enter the value of the test resistor in Ohms (10000 Ω for 6.2109.030) and confirm with **[Next >]**.
- Connect the test resistor to the electrode connector *(3-**2**)* indicated in the dialog window and confirm with **[Next >]**.
- Continue with step 4.

3 Carry out GLP test with standard solution

- Insert a measuring vessel, each filled with 60 mL standard solution, together with the measuring vessel cover at all measuring position *(see Chapter 4.6, page 75)*.

- Click on the menu item **Tools ▸ GLP test ▸ Conductivity...** and confirm with **[Next >]**.
  The following dialog window appears:



- Select the option **Use standard solution**.
- Under **Conductivity of standard solution**, enter the conductivity in µS/cm and confirm with **[Next >]**.

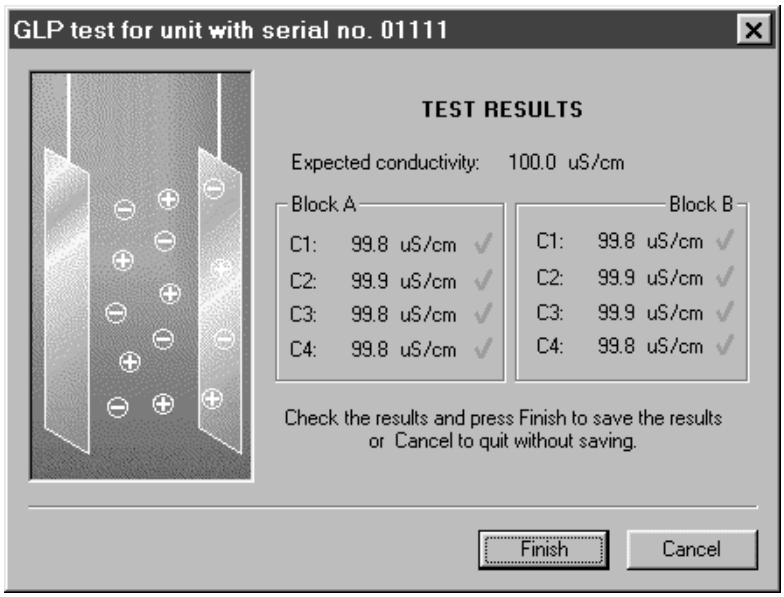
c(KCl) = 1 mmol/L can be used as a standard solution. This is derived from the optionally available conductivity standard 6.2301.060 (KCl 0.1 mol/L) through dilution with distilled water. The conductivity of this diluted solution is:

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

The conductivity is measured sequentially at all channels.

After the last measurement is completed, the dialog window with the **test results** appears.

4 Confirm or reject results



✓	**Test passed**: The deviation of the measured conductivities is within the specified tolerance.
✗	**Test failed**: The deviation of the measured conductivities is greater than the specified tolerance.

- Confirm the results with **[Finish]**.

or

- Reject the results with **[Cancel]**.

4.8.4.3 Gas flow

For the **GLP test for gas flow measurement**, you require a gas flow measuring device (not available from Metrohm), which can be used to measure the gas flow at every channel.

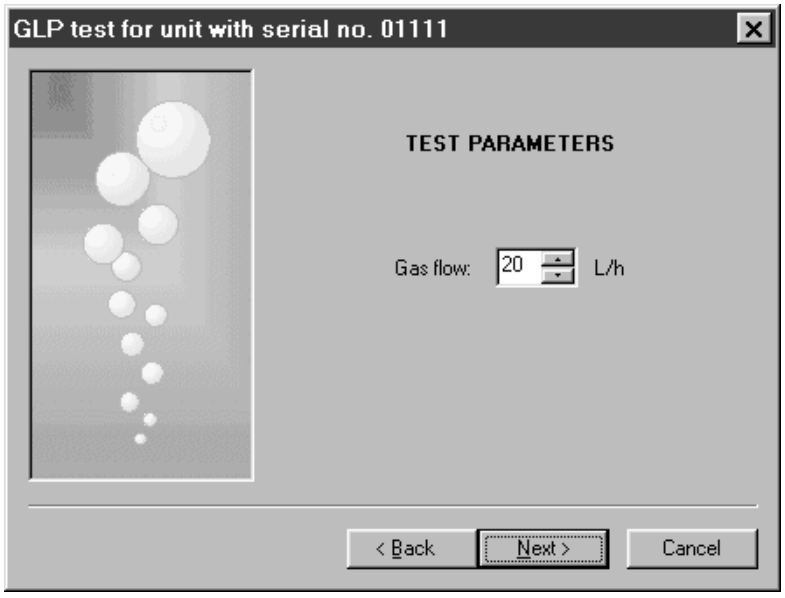
Carrying out the GLP test for the gas flow measurement

Proceed as follows to carry out the GLP test:

1 Select instrument and start test

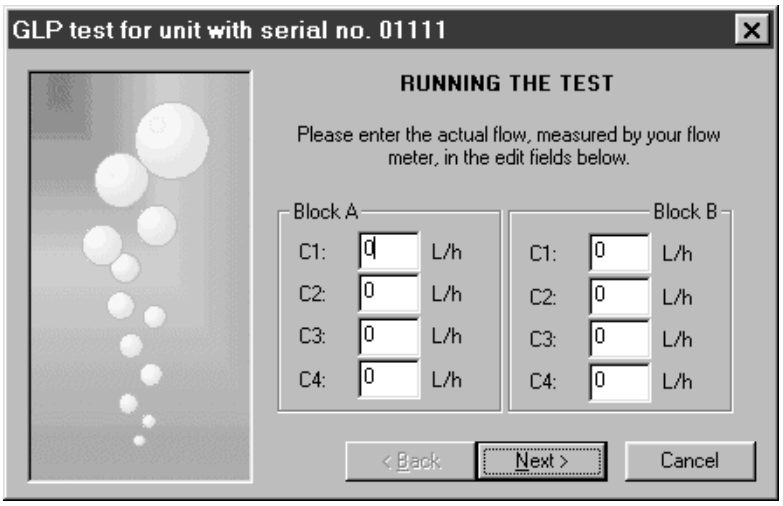
- In the **Control window**, use the menu item **View ▸ Unit x** to select the instrument for which the GLP test is to be carried out.

- Click on the menu item **Tools ▸ GLP test ▸ Gas flow...** and confirm with **[Next >]**.
  The following dialog window appears:



- Enter the **Gas flow** and confirm with **[Next >]**.

The following dialog window appears:

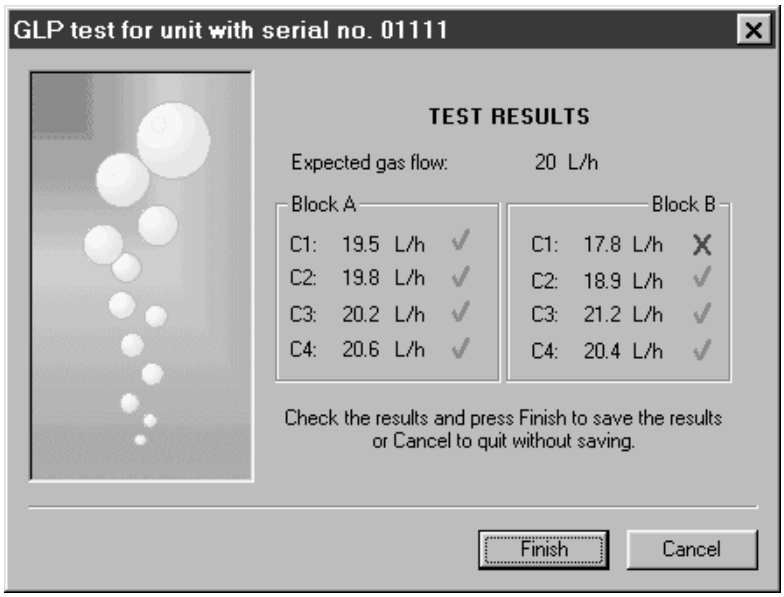


2 Test all channels

- Measure the gas flow sequentially for all channels and enter the values in the corresponding fields K1…K4 in L/h.
- After finishing the measurement, confirm the entries with **[Next >]**.

The dialog window with the **test results** appears.

3 Confirm or reject results



✓	**Test passed**: The deviation of the measured gas flow values is within the specified tolerance.
✗	**Test failed**: The deviation of the measured gas flow values is greater than the specified tolerance.

- Confirm the results with **[Finish]**.

or

- Reject the results with **[Cancel]**.

4.8.5 GLP results

Printing the GLP certificate

Note

To be able to print a test certificate, you have to switch on the GLP monitoring *(see "Activating/Deactivating GLP monitoring", page 138)*.

For the selected instrument, you can print a GLP certificate, which contains the following information:

Print date, serial number of the instrument, results of the last GLP tests (with date, test person and test results).

Proceed as follows to print out a test identification:

1 Print GLP certificate

- In the **Control window**, click on the menu item **Tools ▸ GLP test ▸ Print GLP results...**.

Displaying the GLP results

Note

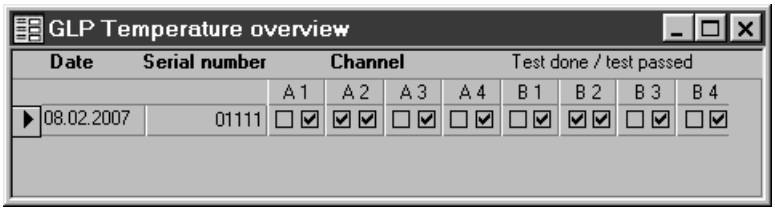
If you want an overview of all GLP tests carried out with the connected units, the database **Repos.mrd** must be open. If another database is open, only those GLP tests are displayed which have also been exported into this database during export of the determinations.

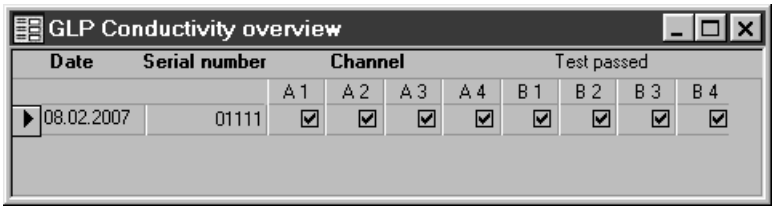
Proceed as follows to display the GLP results:

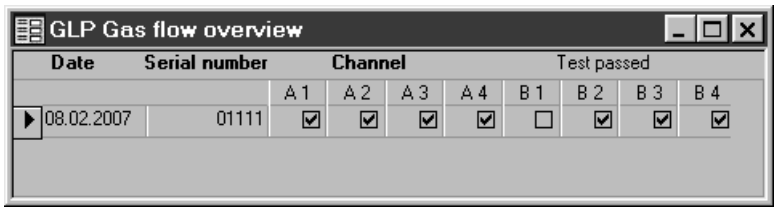
1 Open dialog window with overview

- Open the Results window *(see "Opening and closing the Results window", page 28)*.
- In the **Results window**, click on the menu item **View ▸ GLP** and select the required test results (**Temperature**, **Conductivity**, **Gas flow**).

The following dialog windows appear, depending on the selection previously made.







2 Display detailed results

- Double click on the required GLP test within the fields **Date**, **Serial number** or **Channel**.

The dialog window **GLP Temperature**, **GLP Conductivity** or **GLP Gas flow** appears.

Printing GLP results

Note

To be able to print out the GLP results, you have to switch on the GLP monitoring *(see "Activating/Deactivating GLP monitoring", page 138)*.

Proceed as follows to print the GLP results:

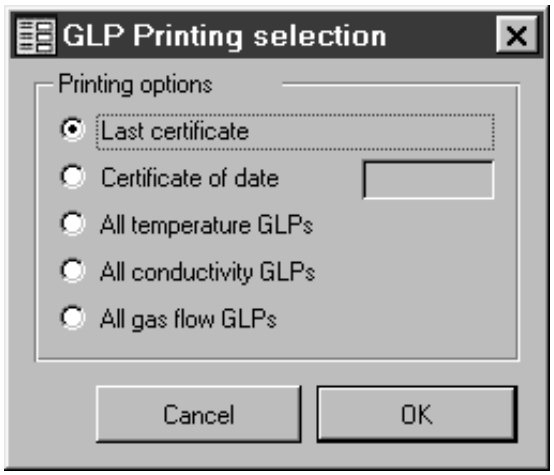
1 Open dialog window with overview

- Open the Results window *(see "Opening and closing the Results window", page 28)*.
- In the **Results window**, click on the menu item **View ▸ GLP** and select the required test results (**Temperature**, **Conductivity**, **Gas flow**).

2 Select data

- In the **Results window**, click on the menu item **File ▸ Print...**. If the database contains GLP test results which have been attained with different instruments, the dialog window **Select unit** appears.

- Select the **Serial number** of the required instrument and confirm with **[OK]**.
  The following dialog window appears:



- Select the required printing option and confirm with **[OK]**.

Parameter description

Printing options

Selection	**Last certificate** | **Certificate of date** | **All temperature GLPs** | **All conductivity GLPs** | **All gas flow GLPs**

Last certificate
Printout of the last GLP certificate of the selected overview window. The printout contains the **Serial number** of the instrument, the **Date**, the **Tester** and the **Test result**.

Certificate of date
Printout of the GLP certificate for the entered date. The printout contains the **Serial number** of the instrument, the **Date**, the **Tester** and the **Test result**.

All temperature GLPs
Printout of the detailed results for all GLP tests for temperature measurement.

All conductivity GLPs
Printout of the detailed results for all GLP tests for conductivity measurement.

All gas flow GLPs
Printout of the detailed results for all GLP tests for gas flow measurement.

5 Handling and maintenance

5.1 General information

5.1.1 Care

The 873 Biodiesel Rancimat requires appropriate care. Excess contamination of the instrument may result in functional disruptions and a reduction in the service life of the sturdy mechanics and electronics.

Spillages of chemicals and solvents should be cleaned up immediately. Above all, the plug connections on the rear of the instrument (in particular the mains connection socket) should be protected from contamination.

Caution

Although this is extensively prevented by design measures, the mains plug should be unplugged immediately if aggressive media has penetrated the inside of the instrument, so as to avoid serious damage to the instrument electronics. In such cases, the Metrohm Service must be informed.

5.1.2 Maintenance by Metrohm Service

Maintenance of the 873 Biodiesel Rancimat is best carried out as part of an annual service, which is performed by technicians of the Metrohm company. If working frequently with caustic and corrosive chemicals, a shorter maintenance interval could be necessary.

The Metrohm service department offers every form of technical advice for maintenance and service of all Metrohm instruments.

5.2 Replacing the dust filter

The dust filter *(3-**20**)* is mounted in the opening marked with **Filter** on the rear of the instrument and serves for filtration of the air sucked in through the air pump. It must be checked at periodic intervals and replaced in the case of more intense contamination (order number: 6.2724.010).

5.3 Regenerating or replacing the molecular sieve

Note

Regenerate the molecular sieve regularly.

The intervals at which you have to regenerate the molecular sieve depends on the **air humidity** in the laboratory and also on the **frequency of use** of the instrument.

The molecular sieve filled in the drying flask *(3-**11**)* serves for adsorption of disruptive oxidizing gases as well as of the water from the sucked in air. You can regenerate the molecular sieve in the drying oven at approx. +140… +180 °C for 24 to 48 h. You can order a new molecular sieve under the order number 6.2811.000.

Caution

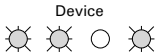
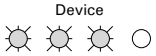
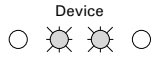
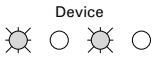
Do **not** fill the hot molecular sieve directly into the drying flask after regeneration, as otherwise the plastic filter on the filter tube will melt.

Wait until the molecular sieve has cooled down before filling.

5.4 Self test when switching on

An internal hardware self test executes after switching on the instrument. Each one of the 7 test steps lasts around one second and is displayed on the instrument number display *(2-**5**)* by a combination sample of the 4 display lamps. In the case of an error, the corresponding LEDs flash.

Display	Cause	Remedy
Device ○ ○ ○ ○	LED test. If not all LEDs light up during this test, an LED is defective.	Notify Metrohm Service.
Device ○ ☼ ☼ ☼	RAM test error.	Switch the instrument off and on again. If the error occurs again, please notify Metrohm Service.
Device ☼ ○ ☼ ☼	PROM checksum test error.	Switch the instrument off and on again. If the error occurs again, please notify Metrohm Service.

Display	Cause	Remedy
	BUSY test error.	Switch the instrument off and on again. If the error occurs again, please notify Metrohm Service.
	ADC test error.	Switch the instrument off and on again. If the error occurs again, please notify Metrohm Service.
	EEPROM test error.	Switch the instrument off and on again. If the error occurs again, please notify Metrohm Service.
	Defective mains voltage (deviation > 10 %).	Check power supply.

5.5 Quality Management and validation with Metrohm

Quality Management

Metrohm offers you comprehensive support in implementing quality management measures for instruments and software. Further information on this can be found in the brochure **«Quality Management with Metrohm»** available from your local Metrohm agent.

Validation

Please contact your local Metrohm agent for support in validating instruments and software. Here you can also obtain validation documentation to provide help for carrying out the **Installation Qualification** (IQ) and the **Operational Qualification** (OQ). IQ and OQ are also offered as a service by the Metrohm agents. In addition, various application bulletins are also available on the subject, which also contain **Standard Operating Procedures** (SOP) for testing analytical measuring instruments for reproducibility and correctness.

Maintenance

Electronic and mechanical functional groups in Metrohm instruments can and should be checked as part of regular maintenance by specialist personnel from Metrohm. Please ask your local Metrohm agent regarding the precise terms and conditions involved in concluding a corresponding maintenance agreement.

Note

You can find information on the subjects of quality management, validation and maintenance as well as an overview of the documents currently available at *www.metrohm.com/com/* under **Support**.

6 Troubleshooting

6.1 Problems

Problem	Cause	Remedy
The pump is louder than normal.	*The air flow is blocked somewhere before or after the pump.*	▪ Check dust filter *(3-**20**)* and, if necessary, replace. ▪ Check filter tube *(4-**4**)* on the drying flask cover for blockages and, if necessary, free with a gentle tap. ▪ Examine FEP tubings *(2-**6**)*, *(3-**13**)*, *(3-**14**)* and *(3-**17**)* for blockages and, if necessary, replace.
	Extra air is sucked in at somewhere else other than the dust filter. There is a leak somewhere in the system before the pump.	▪ Check FEP tubings *(3-**13**)*, *(3-**14**)* and *(3-**17**)* for cracks, kinks etc. and tighten. If necessary, replace. ▪ Place the drying flask cover *(3-**10**)* correctly on the drying flask and screw tight.
No air flow can be detected in the reaction vessel (it does not bubble), although the pump is running.	*The air supply is blocked.*	▪ Remove FEP tubing *(5-**1**)* from the thread adapter *(5-**3**)*. A slight air flow must be discerned here. If this is not the case, please contact Metrohm Service. ▪ Check FEP tubing *(2-**6**)* for blockages. If necessary, clean or replace. ▪ Check thread adapter *(5-**3**)* and air tube *(5-**5**)* on the reaction vessel cover for blockages. If necessary, clean or replace.
	The FEP tubing for the air supply is defective.	Check FEP tubing *(2-**6**)* for cracks, kinks etc. If necessary, replace.
	The FEP tubing for the air supply is not connected correctly.	Tighten FEP tubing *(2-**6**)* on both sides.
	*The air tube (5-**5**) does not immerse in the sample.*	▪ Push reaction vessel cover *(5-**8**)* down as far as the limit stop. ▪ Use more sample.

Problem	Cause	Remedy
No air flow can be detected in the measuring vessel (it does not bubble), although an air flow can be discerned in the reaction vessel.	*The connection is blocked.*	▪ Check tubing connector *(5-**4**)* on the reaction vessel cover for blockages and, if necessary, clean. ▪ Check Iso-Versinic® tubing *(5-**2**)* for blockages and, if necessary, clean. ▪ Check tubing adapter *(5-**11**)* and PTFE tube *(5-**17**)* on the measuring vessel cover for blockages and, if necessary, clean.
	The connection is leaking.	Check Iso-Versinic® tubing *(5-**2**)* for leakages and, if necessary, replace.
	*The reaction vessel cover (5-**8**) is not mounted correctly or is too loose.*	▪ If the reaction vessel cover is oblique or not completely mounted, press down as far as the limit stop. ▪ If the reaction vessel cover is loose on the reaction vessel despite correct assembly, the cover has to be replaced.
	The connection is wrongly connected.	▪ Ensure that the PTFE tube *(5-**17**)* is mounted to the air supply at the opening **In** *(5-**13**)* of the measuring vessel cover. ▪ Ensure that the Iso-Versinic® tubing *(5-**2**)* is mounted on the tubing adapter, which is mounted in the opening **In** *(5-**13**)*. ▪ Ensure that the reaction vessel is connected to the measuring vessel belonging to the corresponding measuring place.
The conductivity is displayed in the upper part of the Live window during startup.	*The conductivity of the water used is too high.*	Check the conductivity of the water used and, if necessary, use deionized water from another source.
	The conductivity measuring cell is contaminated.	Check cell constant of the measuring cell. The cell constant should be between 1.0 and 1.2. If this is not the case, the measuring cell must be cleaned *(see Chapter 4.6.4, page 80)*.

Problem	Cause	Remedy
The induction times are not reproducible for multiple determinations.	*The reaction vessels used are not clean.*	▪ Free the reaction vessel of particles (dust, cardboard, glass fragments etc.) with compressed air before weighing in the sample. ▪ Only use new, unused reaction vessels.
	The reaction vessels used are scratched on the inside.	Only use new, unused reaction vessels.
	*The reaction vessel cover (5-**8**) is not mounted correctly or is too loose.*	▪ If the reaction vessel cover is oblique or not completely mounted, press down as far as the limit stop. ▪ If the reaction vessel cover is loose on the reaction vessel despite correct assembly, the cover has to be replaced.
	The connection to the measuring vessel is not mounted correctly.	Ensure that no air can escape through leaks when transferring from the reaction vessel to the measuring vessel.
	The temperature in different channels of a heating block differs, as there are burn marks at one or more places in the opening of the heating block.	Check temperature in the different positions *(see Chapter 4.8.4.1, page 141)*. If necessary, carefully remove the impurities from the **cold** heating block.
	The temperature in different heating blocks differs.	Determine the temperature correction Delta T for each block separately *(see Chapter 4.4.2, page 50)*.
	The sample is not homogenous.	Homogenize sample.
The stability times are not reproducible for multiple determinations.	*The cell constant was not determined or does not correspond to the value entered.*	▪ Determine cell constant *(see Chapter 4.4.1, page 48)*. ▪ Ensure that the measuring cell is always used in the same channel, so that the cell constant determined once also corresponds to the measuring cell. ▪ Ensure that the measuring cell is not contaminated. If necessary, clean *(see Chapter 4.6.4, page 80)*.

Problem	Cause	Remedy
	The conductivity measuring cell is contaminated.	Check measuring cell *(see Chapter 4.8.4.2, page 146)* and, if necessary, clean.
	See also: The induction times are not reproducible for multiple determinations.	
The induction time is longer/shorter than expected.	*The temperature is not selected correctly.*	▪ Ensure that the correct method for the determination has been selected. ▪ Check that **Temperature** and **Delta T** have been specified correctly in the method *(see Chapter 4.5.2.1, page 60)*.
	See also: The induction times are not reproducible for multiple determinations.	
The stability time is longer/shorter than expected.	*Delta Kappa is not defined correctly.*	Ensure that the value of **Delta Kappa** defined in the method is correct *(see Chapter 4.5.2.2, page 64)*.
	See also: The stability times are not reproducible for multiple determinations.	
	See also: The induction time is longer/shorter than expected.	
The measurement curves are extremely noisy.	*The air supply for the measuring solution is directed to the conductivity measuring cell.*	Loosen tubing adapter *(5-**11**)* on the measuring vessel cover, turn the PTFE tube *(5-**17**)* in such a way that the air is no longer directed to the measuring cell and fix in this position.
	Gas bubbles adhere to the conductivity measuring cell during measurement.	▪ Ensure that the measuring cell is clean and free of fat. If necessary, clean thoroughly *(see Chapter 4.6.4, page 80)*. ▪ In some cases ultra pure water contains a large proportion of dissolved air which escapes during measurement. In this case, degas the ultra pure water before the measurement for 5 to 10 min in a vacuum.

Problem	Cause	Remedy
	During the measurement, sample evaporates in the reaction vessel and condenses in the measuring vessel. This results in contamination of the conductivity measuring cell, which in turn promotes adherence of gas bubbles.	▪ Keep the measuring time as short as possible, by about 4 to 6 h. The induction time can be reduced by about half by increasing the temperature by 10 °C. ▪ Reduce the temperature so that less sample evaporates or no sample at all. However, this can substantially extend the measuring time. The induction time approximately doubles by reducing the temperature by 10 °C.
The curve shows a step which means that the induction time is no longer determined correctly.	*Side reactions occur at the start or during the measurement, which cause the conductivity in the measuring cell to rise.*	▪ In the method, use the option **Evaluation delay** or **Evaluation suppression** *(see Chapter 4.5.2.2, page 64)*. ▪ Do not use **Endpoint(s)** as a stop criterion in the method, but instead a defined **Conductivity** (e.g. 200 µS/cm) *(see Chapter 4.5.2.1, page 60)*. The curves can then also be evaluated manually after finishing the determination.
The curve shows a step at the start of the measurement, which has not occurred in previous measurements.	*The reaction vessel cover (5-**8**) and/or the tubing (5-**2**) still contain residues which are transported into the measuring vessel through the warm air flow when a new measurement is started.*	▪ Thoroughly clean the reaction vessel cover and the Iso-Versinic® tubing *(see Chapter 4.6.4, page 80)*. ▪ Replace the reaction vessel cover and the Iso-Versinic® tubing from time to time.
The induction time is not evaluated automatically, although a significant break point can be noticed in the curve.	*The option **Evaluate induction time** is deactivated in the method.*	Activate the evaluation of the induction time in the method *(see Chapter 4.5.2.2, page 64)*.
	*The option **Evaluation delay** or **Evaluation suppression** is defined in the method, which prevents the evaluation of the curve in the corresponding time period.*	Deactivate the corresponding option in the method *(see Chapter 4.5.2.2, page 64)*.
	Automatic detection of the induction time is not yet possible.	Keep the determination running until the induction time is automatically found.

Problem	Cause	Remedy
	The curve progression is too flat with the result that automatic detection of the induction time is not possible.	Do not use **Endpoint(s)** as a stop criterion in the method, but instead a defined **Conductivity** e.g. 200 µS/cm *(see Chapter 4.5.2.1, page 60)*. Evaluate the curves afterwards manually using tangents *(see "Setting tangents manually", page 112)*.
The measurement aborts without an endpoint being found.	*The measurement has been stopped manually.*	Keep the measurement running until the endpoint is automatically found.
	A time or conductivity is defined as a stop criterion in the method, which was reached before the endpoint.	Deactivate time or conductivity as a stop criterion in the method *(see Chapter 4.5.2.1, page 60)* or increase the corresponding value for the time or conductivity.
	*The heating is defective.*	Switch the instrument off and on again. If the error occurs again, please contact Metrohm Service.
	The maximum temperature (220 °C) has been exceeded by 10 °C.	Switch the instrument off and on again. If the error occurs again, please contact Metrohm Service.
	*Error during self test.*	Switch the instrument off and on again. If the error occurs again, please contact Metrohm Service.

7 Technical specifications

7.1 General data

Number of instruments that can be connected	1…4
Number of samples per instrument	1…8 (4 each per heating block)
Sample amount	Several grams / several milliliters

7.2 Temperature regulation and measurement

Range setpoint temperature	50…220 °C
Temperature correction (Delta T)	0…±9.9 °C (can be entered manually or determined automatically using the external temperature sensor)
Temperature measuring range	0…250 °C
Resolution	0.1 °C
Max. deviation of the heating block temperature from the set value (50… 220 °C)	±0.3 °C
Reproducibility of the set temperature	typ. < 0.2 °C
Max. temperature difference between different measuring positions per block	typ. < 0.3 °C
Temperature fluctuations	typ. < 0.1 °C (at attained operating temperature, inserted and identically filled reaction vessels and 20 L/h air throughput)

Shutdown temperature	260 ± 11 °C (if this temperature is exceeded, the heating is shut down, and switched on again at 210 ± 11 °C)
Heating up time for the instrument	approx. 45 min (from 20 °C to 120 °C) approx. 60 min (from 20 °C to 220 °C)
Instrument external temperature	< 50 °C (at operating temperature 220 °C)

7.3 Conductivity measurement

Sensor	Conductivity measuring cell, integrated in the measuring vessel cover 6.0913.130
Structure	Conductivity measuring cell with 2 steel electrodes
Measuring principle	AC 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 entered manually or determined automatically)
Measuring range	0…400 µS/cm
Resolution	0.1 µS/cm
Maximum error	±1 % ± 0.5 µS/cm

7.4 Gas flow regulation

Pump	Membrane pump
Adjustable range	7…25 L/h
Max. deviation from the set range	±10 %

7.5 GLP test set

External temperature sensor

Type	Pt100 casing element ⌀1.9 mm, in 4-conductor technology; DIN IEC 751 Class B
Calibration accuracy	0…90 °C: ±0.02 °C 90…220 °C: ±0.03 °C
Max. deviation of the measured temperature from the true value (50…220 °C)	±0.3 °C
Test resistor	10 kΩ

7.6 RS-232 interface

Plug	Sub-D plug, 9-pin (male)
Basic settings	9600 Baud, 8 Bit, 1 stop bit, no parity, XON/XOFF

7.7 Mains connection

Voltage	2.873.0014: 220…240 V ± 10 % 2.873.0015: 100…120 V ± 10 %
Frequency	50…60 Hz
Power consumption	450 W
Fuse	Diameter 5 mm, length 20 mm 100…120 V: 2.0 ATH (slow-acting) 220…240 V: 4.0 ATH (slow-acting)

7.8 Safety specification

Design and testing According to EN/IEC/UL 61010-1, CSA-C22.2 No. 61010-1, EN/IEC 61010-2-010, protection class I, degree of protection IP20

Safety instructions The documentation contains safety instructions which have to be followed by the user in order to ensure safe operation of the instrument.

7.9 Electromagnetic compatibility (EMC)

Emission Standards fulfilled:
- EN/IEC 61326-1
- EN/IEC 61000-6-3
- EN 55011 / CISPR 11
- EN/IEC 61000-3-2
- EN/IEC 61000-3-3

Immunity Standards fulfilled:
- EN/IEC 61326-1
- EN/IEC 61000-6-1
- EN/IEC 61000-4-2
- EN/IEC 61000-4-3
- EN/IEC 61000-4-4
- EN/IEC 61000-4-5
- EN/IEC 61000-4-6
- EN/IEC 61000-4-11

7.10 Ambient temperature

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

7.11 Housing

Cover material	Polyurethane hard foam (PUR) with flame retardation for fire class UL94VO, CFC-free
Base material	Steel, painted
Width	405 mm
Height	268 mm (without accessories) 353 mm (with accessories)
Depth	466 mm
Weight	20.2 kg (without accessories) 27.6 kg (with accessories)

8 Conformity and warranty

8.1 Declaration of Conformity

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

873 Biodiesel Rancimat

Instrument for automatic determination of the oxidation stability of biodiesel and its blends.

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

Electromagnetic compatibility

Emission:	EN/IEC 61326-1: 2006, EN/IEC 61000-6-3: 2004, EN 55011 / CISPR 11: 2007, EN/IEC 61000-3-2: 2006, EN/IEC 61000-3-3: 2005
Immunity:	EN/IEC 61326-1: 2006, EN/IEC 61000-6-1: 2007, EN/IEC 61000-4-2: 2001, EN/IEC 61000-4-3: 2006, EN/IEC 61000-4-4: 2004, EN/IEC 61000-4-5: 2006, EN/IEC 61000-4-6: 2007, EN/IEC 61000-4-11: 2004

Safety specifications

EN/IEC 61010-1: 2001, UL 61010-1: 2004, CSA-C22.2 No. 61010-1: 2004, EN/IEC 61010-2-010: 2003, protection class I

CE

This instrument meets the requirements of the CE mark as contained in the EU directives 2006/95/EC (LVD), 2004/108/EC (EMC). It fulfils the following specifications:

EN 61326-1	Electrical equipment for measurement, control and laboratory use – EMC requirements
EN 61010-1	Safety requirements for electrical equipment for measurement, control and laboratory use

Manufacturer

Metrohm Ltd., CH-9101 Herisau/Switzerland

Metrohm Ltd. is holder of the SQS certificate ISO 9001:2000 Quality management system for development, production and sales of instruments and accessories for ion analysis.

Herisau, 23 January, 2009

D. Strohm

Vice President, Head of R&D

A. Dellenbach

Head of Quality Management

8.2 Quality Management Principles

Metrohm Ltd. holds the ISO 9001:2000 Certificate, registration number 10872-02, issued by SQS (Swiss Association for Quality and Management Systems). Internal and external audits are carried out periodically to assure that the standards defined by Metrohm's QM Manual are maintained.

The steps involved in the design, manufacture and servicing of instruments are fully documented and the resulting reports are archived for ten years. The development of software for PCs and instruments is also duly documented and the documents and source codes are archived. Both remain the possession of Metrohm. A non-disclosure agreement may be asked to be provided by those requiring access to them.

The implementation of the ISO 9001:2000 quality management system is described in Metrohm's QM Manual, which comprises detailed instructions on the following fields of activity:

Instrument development

The organization of the instrument design, its planning and the intermediate controls are fully documented and traceable. Laboratory testing accompanies all phases of instrument development.

Software development

Software development occurs in terms of the software life cycle. Tests are performed to detect programming errors and to assess the program's functionality in a laboratory environment.

Components

All components used in the Metrohm instruments have to satisfy the quality standards that are defined and implemented for our products. Suppliers of components are audited by Metrohm as the need arises.

Manufacture

The measures put into practice in the production of our instruments guarantee a constant quality standard. Production planning and manufacturing procedures, maintenance of production means and testing of components, intermediate and finished products are prescribed.

Customer support and service

Customer support involves all phases of instrument acquisition and use by the customer, i.e. consulting to define the adequate equipment for the analytical problem at hand, delivery of the equipment, user manuals, training, after-sales service and processing of customer complaints. The Metrohm service organization is equipped to support customers in implementing standards such as GLP, GMP, ISO 900X, in performing Operational Qualification and Performance Verification of the system components or in carrying out the System Validation for the quantitative determination of a substance in a given matrix.

8.3 Warranty (guarantee)

Metrohm guarantees that the deliveries and services it provides are free from material, design or manufacturing errors. The warranty period is 36 months from the day of delivery; for day and night operation it is 18 months. The warranty remains valid on condition that the service is provided by an authorized Metrohm service organization.

Glass breakage is excluded from the warranty for electrodes and other glassware. The warranty for the accuracy corresponds to the technical specifications given in this manual. For components from third parties that make up a considerable part of our instrument, the manufacturer's warranty provisions apply. Warranty claims cannot be pursued if the Customer has not complied with the obligations to make payment on time.

During the warranty period Metrohm undertakes, at its own choice, to either repair at its own premises, free of charge, any instruments that can be shown to be faulty or to replace them. Transport costs are to the Customer's account.

Faults arising from circumstances that are not the responsibility of Metrohm, such as improper storage or improper use, etc. are expressly excluded from the warranty.

9 Accessories

Note

Subject to change without notice.

9.1 Scope of delivery

2.873.0014 and 2.873.0015 873 Biodiesel Rancimat

Qty.	Order no.	Description
1	1.873.0014 (230 V) 1.873.0015 (115 V)	**873 Biodiesel Rancimat**
8	6.0913.130	**Measuring vessel cover for stability measuring instruments** With built-in conductometric measuring cell. Shaft material: PP Measuring range: 0 - 400 Measuring unit: µS*cm-1 Cell constant (1/cm): 1
24	6.1428.030	**Glass measuring vessel for stability measurements** Conductivity measuring vessel made of clear glass for 873 Biodiesel Rancimat, 743 Rancimat and 763 PVC Thermomat. Material: Clear glass Height (mm): 91 Outer diameter (mm): 46

Qty.	Order no.	Description	
1	6.1429.050	**Reaction vessel long for stability measurements** Reaction vessel for stability measurements involving volatile oils, mineral oils and biodiesel blends with 743 Rancimat and 873 Biodiesel Rancimat. In combination with 6.2418.130. Set of 100 pieces. Material: Clear glass Outer diameter (mm): 24 Length (mm): 250	
8	6.1451.010	**Foam barrier** Foam barrier in reaction vessels for stability measurements. Material: PP Outer diameter (mm): 20 Inner diameter (mm): 5	
4	6.1454.040	**O-ring** O-ring for air tube for stability measurements. Set of 6 pieces. Material: Nitrile rubber Height (mm): 1.5 Inner diameter (mm): 3.5	
1	6.1602.145	**Drying flask cover** Screw cap with GL45 thread for 6.1608.050 drying bottle.	

Qty.	Order no.	Description	
1	6.1608.050	**Drying bottle / 100 mL / GL 45**	
	Material:	Clear glass	
	Height (mm):	100	
	Outer diameter (mm):	56	
	Volume (mL):	100	
1	6.1805.010	**FEP tubing / M6 / 13 cm**	
	With light and kink protection.		
	Material:	FEP	
	Inner diameter (mm):	2	
	Length (mm):	130	
10	6.1805.080	**FEP tubing / M6 / 25 cm**	
	With light and kink protection.		
	Material:	FEP	
	Inner diameter (mm):	2	
	Length (mm):	250	

Qty.	Order no.	Description
1	6.1808.020	**Tubing adapter olive / M6 inner** 1 M6 inner thread and 1 tubing nozzle. Part of the screw connections for Exchange Units and stability measuring instruments. Material: PCTFE Length (mm): 30
16	6.1808.050	**Tubing adapter olive / M8 outer** 1 M8 outer thread and 1 tubing olive. E.g. for thermostat jacket of exchange units and stability measuring instruments. Material: PVDF Length (mm): 31.5
8	6.1808.090	**Thread adapter M8 outer / M6 inner** Outer thread M8, inner thread M6, e.g. for mounting 6.2418.XXX air tubes in 6.1429.XXX reaction vessels of stability measuring instruments. Material: PVDF Length (mm): 23

Qty.	Order no.	Description
8	6.1819.080	**Cannula for gas feeding**

For stability measuring instruments.

Material:	PTFE
Outer diameter (mm):	4
Inner diameter (mm):	3
Length (mm):	85

1	6.1821.040	**Filter tube**

Filter tube for 6.1608.050 drying bottle. For Rancimats and Karl Fischer ovens.

Length (mm):	112

24	6.1839.000	**Iso-Versinic tubing 0.22 m**

Connection tubing for stability measuring instruments for determining the oxidation stability of biodiesel.

Material:	FPM
Outer diameter (mm):	9
Inner diameter (mm):	6
Length (mm):	220

1	6.2041.190	**Holder for reaction vessels**

Reaction vessel holder for stability instruments; for 8 reaction vessels.

Material:	Aluminum
Width (mm):	170
Height (mm):	110
Depth (mm):	110

Qty.	Order no.	Description
1	6.2134.100	**RS-232 cable 5 m, 9-pin - 9-pin**

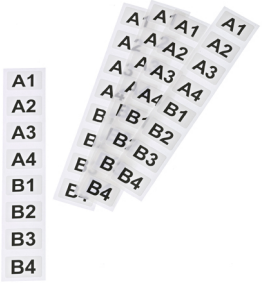
Connecting Metrohm RS-232 interface - Titrinos, 756 KF Coulometer, 762 or 771 IC Interfaces, 790 Personal IC, 743 Rancimat, 763 PVC Thermomat, 873 Biodiesel Rancimat.

Length (m):	5

1	6.2250.000	**Labels laminated**

Labels for measuring vessel cover for stability measuring instruments. Labeling: A1, A2, A3, A4, B1, B2, B3, B4. 5 sets of 8 items.

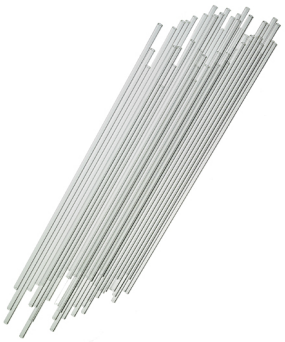
Material:	PE
Material 2:	PP
Width (mm):	25
Height (mm):	15



1	6.2418.130	**Air tube long for biodiesel measurements**

Set of 100 pieces. In combination with 6.1429.050.

Material:	Glass
Outer diameter (mm):	4
Inner diameter (mm):	2.4
Length (mm):	248



1	6.2628.000	**Holder for reaction vessel**

Reaction vessel holder for stability instruments; for 1 reaction vessel for weighing in the sample.

Material:	Aluminum
Width (mm):	50
Height (mm):	123
Depth (mm):	50
Hole diameter (mm):	26.5

Qty.	Order no.	Description	
1	6.2724.010	**Dust filter** Dust filter for Rancimats and Karl Fischer ovens. Outer diameter (mm): 44 Length (mm): 53	
1	6.2753.107	**Reaction vessel cover for stability measurements** For 6.1429.040 or 6.1429.050 reaction vessel. Set of 100 pieces. Material: TEEE Height (mm): 14.7 Height description: Without connections Outer diameter (mm): 28.6	
1	6.2811.000	**Molecular sieve** Molecular sieve. Bottle containing 250 g. Pore size: 0.3 nm. Without moisture indicator. For Rancimats and Karl Fischer instruments.	
1	6.6060.010	**873 Biodiesel Rancimat software 1.1** Software for instrument control and data handling.	
1	6.2122.0x0	**Mains cable with C13 line socket IEC-60320-C13** Cable plug according to customer requirements. Switzerland: Type SEV 12 6.2122.020 Germany, …: Type CEE(7), VII 6.2122.040	

Qty.	Order no.	Description
	USA, …:	Type NEMA/ASA 6.2122.070
1	**8.873.8003EN**	**Manual 873 Biodiesel Rancimat**

9.2 Optional accessories

Order no.	Description
2.145.0320	**Port extension USB - 4xDB9**

For connection (DB9) of 4 additional instruments to the USB interface of the computer (WIN98, 2000 or XP).

6.1111.020 **Pt100 Temperature sensor long**

Pt100 temperature sensor for 6.5616.010 GLP test set for stability measuring instruments.

Shaft material:	Stainless steel (AISI 304)
Measuring range:	-200 ... 300
Measuring unit:	°C
Shaft diameter top (mm):	2
Shaft diameter bottom (mm):	2
Shaft length to head (mm):	300
Min. immersion depth (mm):	20
Temperature sensor:	Pt100

6.2059.000 **Turning ring**

Turning ring for the swiveling setup of stability measuring instruments.

Height (mm):	32
Outer diameter (mm):	250

6.2125.110 **RS-232 cable 3 m, 25-pin - 9-pin**

Connecting Metrohm RS-232 interface - PC. Used with pH Meters, Titrinos, KF Coulometers, VA Trace Analyzer.

Length (m):	3

Order no.	Description

6.2301.060 KCl ion standard 250 mL

KCl ion standard (250 mL, c(ion) = 0.1000 +/-0.0005 mol/L), can also be used as standard solution for conductometry.

Volume (mL): 250

6.2324.000 Conductivity standard 100 µS/cm, 250 mL

Conductivity standard for calibration of conductivity measuring cells with cell constant = 0.1/cm.

6.2757.000 Air collection tube for stability instruments

Including 8 stoppers.

Outer diameter (mm): 30

Length (mm): 367

6.2821.090 Aspiration filter

Pore size 20 µm, set of 5 pieces. For 6.1834.000 aspiration tubing and 6.1821.040 and 6.1821.050 filter tubes.

Material: PE

Outer diameter (mm): 9.5

Length (mm): 35.5

Order no.	Description	
6.5616.010	**GLP Test Set**	
	GLP test set for the exact adjustment and validation of temperature on 873 Biodiesel Rancimat. Consists of:	
	6.1111.020: Pt100 temperature sensor long	
	6.1253.000: Measuring insert for temperature sensor	
	6.2042.040: Clamp for temperature sensor	
	6.2109.030: Plug with resistor	
	6.2621.120: Hexagon key 1.5 mm	
6.9988.733	**Validation Documentation for 873 (English)**	

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