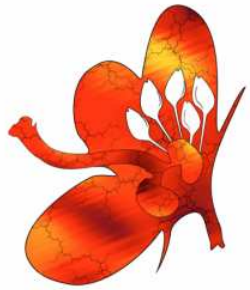

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Manual

Pyrola® 2000 MultiMatic

PYROL AB

Manual Pyrola® 2000 MultiMatic

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Chapter 1

1 Introduction

Pyrolysis, usually in combination with gas chromatography and/or mass-spectrometry (Py-GC/MS), is well known for the analysis of non-volatile samples because of simple sample handling and detailed qualitative and quantitative information.

The definition of analytical pyrolysis in the IUPAC Recommendations 1993 is: 'The characterization, in an inert atmosphere, of a material or a chemical process by a chemical degradation reaction(s) induced by thermal energy'. A more common definition is: 'Thermal degradation in an inert atmosphere'. Gas chromatography is used for the separation of the pyrolysis products, and mass spectrometry for the identification.

There is a rule of thumb which says that 'if the temperature is increased by ten degree Celsius for a chemical reaction, then the reaction rate is doubled'. By experience it is found that the same is valid for thermal degradation reactions. The conclusion is that in order to get reliable and repeatable results from analytical pyrolysis the temperature of the pyrolysis should be kept constant and be repeatable between samples. A measurement of the temperature time profile (TTP) is therefore essential to ensure the quality of the results. By knowing the TTP also much more information can be gained about the sample that is being analyzed. The more you know of your pyrolysis conditions the easier it is to understand the pyrolysis results.

The ideal TTP of a sample is the combination of a short temperature rise time (TRT), a constant temperature during a known pyrolysis time, and a short cooling time. This is essential when making an isothermal pyrolysis. To get an ideal TTP of the sample, a pyrolyzer is needed which can heat the sample fast (milliseconds) and keep the temperature constant until the sample is totally pyrolyzed or cooled quickly. The shorter the heating time of the pyrolyzer, and the smaller the sample size, the more probable that relatively volatile samples can be pyrolyzed before they are volatilized.

As a complement to isothermal pyrolysis, Pyrol AB has developed Four innovative pyrolysis techniques: thermal desorption, sequential pyrolysis, fractionated pyrolysis and pyrotomy. Before each pyrolysis, **thermal desorption** can take place in the heated process unit, to take care of the volatile substances in a complex sample. In **sequential pyrolysis** the sample is heated repeatedly to the same temperature, set sufficiently low as to maintain an ample amount of sample for the subsequent pyrolysis steps. The results can be used for determining the thermal degradation rate of the sample, and thus be used for qualitative analyses, for example to distinguish between a homo-polymer and a co-polymer.

In **fractionated pyrolysis** the sample is heated repeatedly, but to different temperatures. Then it is possible to separate substances with different degradation rates. For example if a sample consists of a substance that is degraded easily at 400° C, and another substance

that is degraded at 700° C. Then an initial pyrolysis at 400° C will give information of the former substance, while the subsequent pyrolysis at 700° C will give information of the latter. Thus fractionated pyrolysis is especially suited for the analysis of complex unknown samples.

In **pyrotomy** the sample is exposed to several extremely short thermal pulses (ms), made possible by the very fast temperature rise time (TRT) and the fast cooling of the sample by the Pyrola. Then only the part of the sample that is in direct contact with the platinum filament will be heated in each pulse, giving a separate pyrolysis of each 'layer' of the sample. Then if the sample consists of a laminate, the pyrograms will give information of each 'layer' separately, instead of having all of them mixed in a single pyrogram.

When the Pyrola 2000 was introduced, it incorporated new possibilities based on the experience from the Pyrola 85 and the Pyrola 9. It can heat a platinum filament up to 1400 °C in less than 8 milliseconds. The TTP is displayed and it is possible to make sequential and fractionated pyrolysis automatically. It can ramp the pyrolysis temperature (I/t), and it has the unique possibility to make a pyrotomy analysis.

The **Pyrola 2000 MultiMatic** has all the functionality of the Pyrola 2000, but it can handle up to 14 probes automatically. Being the first automated Pyrola, it opens up new frontiers in analytical pyrolysis.

Since the Pyrola 2000 MultiMatic is being subject to constant improvements, the manual might not exactly refer to your software. Please contact Pyrol AB for support, info@pyrolab.com.

1.1 Description of the Pyrola® 2000 MultiMatic system

The Pyrola 2000 MultiMatic system consists of a process unit (Figure 1-1) and a control unit (Figure 1-3). The process unit consists of the pyrolysis chamber and a carousel that can accommodate up to 14 pyrolysis probes. The control unit manages the probes and the carousel, produces the current pulses and measures the temperature of the chamber (Tc), as well as the signal from the photo diode and the resistance of the Pt-filament. The control unit, in turn, is controlled by a PC with the Pyrola 2000 MultiMatic software.

The samples that are to be pyrolyzed are placed on platinum filaments in the pyrolysis probes, either with or without a cavity. Two constant current pulses heat the filament, which are defined by their durations (t1, t2) and current amplitudes (I1, I2).

1.2 The process unit

The process unit of the Pyrola 2000 MultiMatic is shown Figure 1-1. It consists of

- pyrolysis chamber
- carousel with 6+1 or 14+1 probes

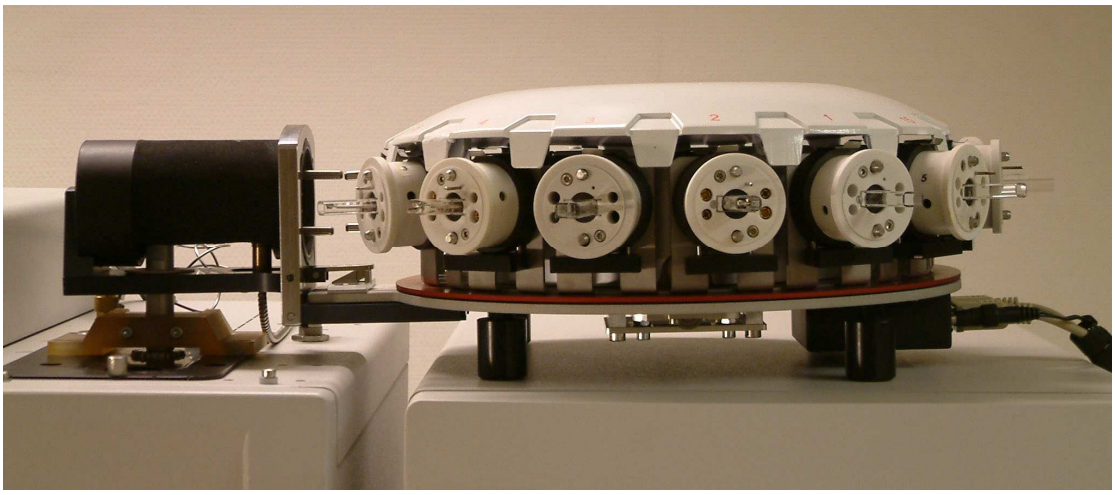
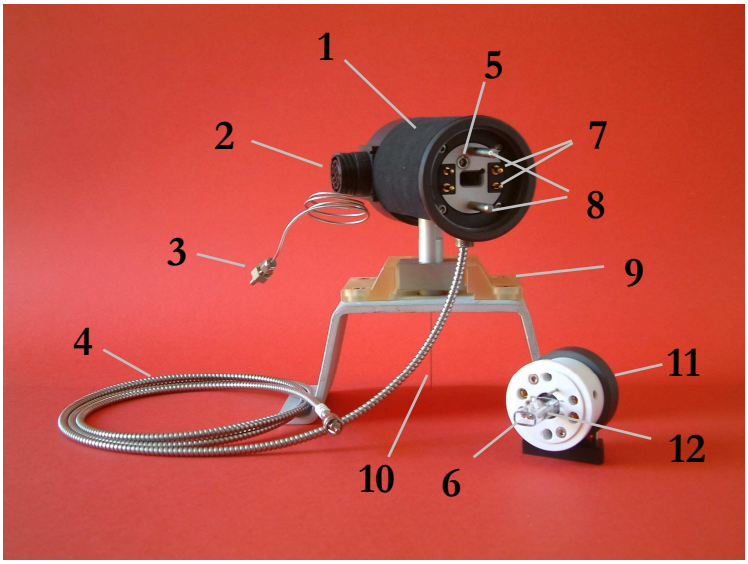


Figure 1-1. The Pyrola 2000 MultiMatic process unit.

One probe and the pyrolysis chamber are shown in detail in Figure 1-2.

The temperature range of the pyrolysis chamber is 50 °C to 225 °C. The most common pyrolysis temperature is 175 °C. The higher the temperature of the process unit the more of the high boiling pyrolysis products will be transported to the column.

The glass cell (6) protects the GC from non-volatile pyrolysis products and lets the emitted light pass through to the optic cable.



1 Pyrolysis chamber

2 Contact for the cable from the control unit

3 Gas inlet to pyrolysis chamber from quick connector.

4 Fiber optic cable

5 Gas outlet from chamber

6 Glass cell, the light from the filament can pass to photodiode and high boiling products can condense.

7 Electrical contacts for pyrolysis probe

8 Alignment pins for pyrolysis probe

9 Removable base for mounting unit on GC

10 Gas outlet from pyrolysis chamber to GC injector

11 Pyrolysis probe

12 Platinum holders for filament

Figure 1-2. Pyrolysis chamber and probe.

1.3 The control unit

1.3.1 Front panel

The two light emitter diodes (LED) on the front panel shown in Figure 1-3, 'Power' and 'Status', indicate the current status of the pyrolyzer. 'Power' shows a green light when the unit is switched on; the 'Status' LED can show different signals as described below.

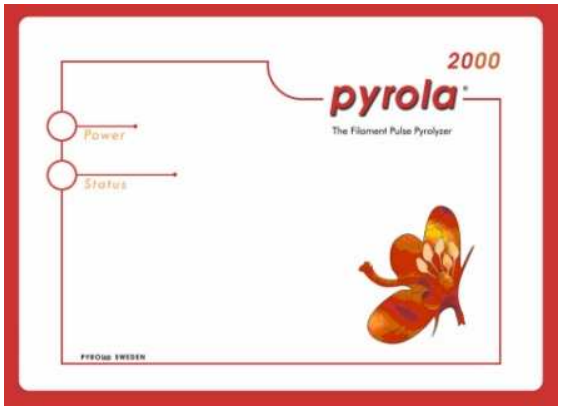


Figure 1-3. Front panel.

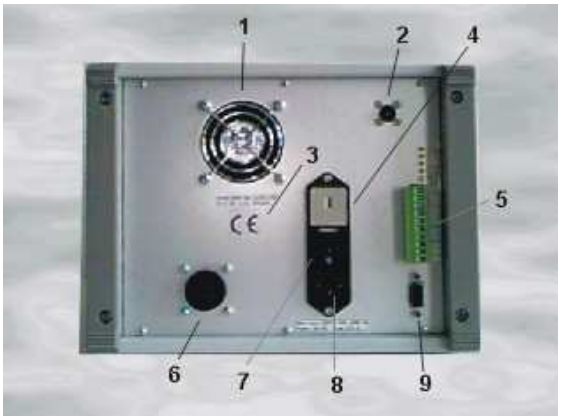


Figure 1-4. Rear panel.

Signal	Status	Information
continuous green	Ready	• the unit is ready and a pyrolysis can be run
flashing green	Waiting	• the chamber temperature has not yet reached the pre-set value • the 24 V DC has not reached the minimum level • the platinum filament is broken • the probe is disconnected
orange	Running	• the unit is active, a pyrolysis is currently being done
red	failure	• process unit is not connected to the control unit; check the cable

1.3.2 Rear panel

Please refer to Figure 1-4.

1. Fan
2. Connector for optic cable
3. Serial number and description
4. Power selector (115 or 230 VAC)
5. LED and connector for remote start and automation (see also Appendix II)
6. Connector for the process unit cable
7. Main power switch
8. Connector for main power cable
9. Connector for the communication cable, serial cable (9-pin) to PC.

1.4 Description of the software

This section contains a short description of the available software menus and functions. It is intended as a short reference only, and more detailed information about the operation of the software and how to select the parameters can be found in chapter 4, 'Operation'.

Notice: Before attempting to run the equipment, read chapter 4, 'Operation'.

The Pyrola 2000 MultiMatic software is developed for Microsoft Windows. It requires about 1 MB of empty disk space for the installation of the software files; some additional space should be available for the storage of your data files.

The software for the Pyrola 2000 MultiMatic is identical in appearance to the software for the manual Pyrola 2000, but with some other options available due to the automation of the MultiMatic. The analyses are organized in pyrolysis projects that controls which probes that are used, the pyrolysis methods and the pyrolysis conditions.

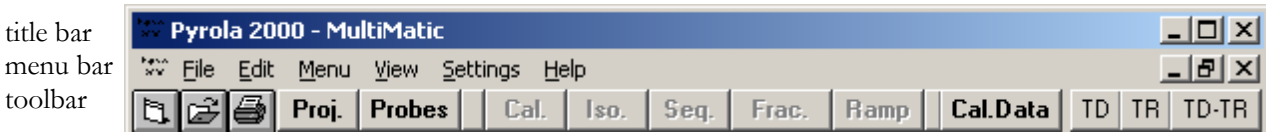


Figure 1-5. Main application window with title bar, menu bar and toolbar.

In Figure 1-5 the appearance of the main application window is shown. The **title bar** shows the application name as well as the name of the currently opened file. The **menu bar** contains the available menus and commands for the operation of the software; these will be described more in detail in the following sections.

The **toolbar** provides shortcuts to some of the commands that are available in the menu bar. A description of the command buttons is given in the table below.

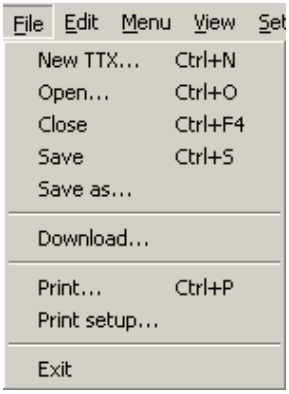
Button	Function
	Creates a new ttx-file (library file) and opens the 'Open file' dialog for the first ttp-file (result file)
	Opens a file
	Opens the print menu
Proj.	Opens the project window for creating or changing pyrolysis projects.
Probes	Opens the probe window for calibrating the pyrolysis probes.
Cal.Data	Displays calibration data.
TD	Shows the pyrolysis temperature time profile (TTP) measured with the

	photodiode, TD.
TR	Shows the TTP measured with the filament resistance, TR..
TD-TR	Shows the TTP measured with both the photodiode, TD, and by the filament resistance, TR.

The buttons that are not highlighted, Cal. Iso. Seq. Frac. Ramp, are only available for the manual Pyrola 2000. On the MultiMatic, these functions are instead controlled by the pyrolysis projects, see section 4.2, 'Projects'.

1.4.1 File menu

The File menu includes commands for the handling of files such as Open, Save, Print as well as the Exit command.



Create a new ttx-file
Open a file (ttp-, ttx- or ini-file)
Close the TTP window
Save the current parameter settings (in the file pyrini.ini)
Save a custom file (ttp-, ttx- or ini-file)
Load new program code (bin-file) to the Pyrola 2000 MultiMatic unit
Print TTP
Change printer settings
Exit the Pyrola 2000 MultiMatic software

Figure 1-6. The file menu.

1.4.1.1 File types

The Pyrola 2000 MultiMatic handles four file types, .ini, .ttp, .ttx and .mdb.

The **ini-file** (initialization file) pyrini.ini is delivered with the software and contains the instrument calibration as well as the last used parameter setup. This file is default and will be opened at every start-up of the software. The default parameter settings can be changed according to the user preferences and saved with the 'Save' command.

The **ttp-file** (result file) contains the parameter settings, current calibration and the result from a pyrolysis. It can be saved with a selected name using the 'Save as…' command; stored files can be retrieved with 'Open' and used as a parameter setup for a new pyrolysis. The **ttx-file** (library file) can be used to combine several previously stored result files and save them in one single file.

Finally, the calibration data for the probes are stored in a **mdb-file**.

1.4.1.2 Exit command

When the exit command is selected the Pyrola 2000 software prompts you to save the last used parameter settings to the default ini-file. Select 'Yes' to save the settings, or 'No' to quit the program without saving.

1.4.2 Edit menu

The 'Insert' command on the Edit menu is used to combine several result files (ttp) in one library file (ttx).

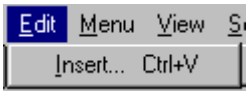


Figure 1-7. The edit menu.

To add a file click on the 'Insert' command and select your file from the appropriate drive and directory. The insert dialog is similar to the common Windows open dialog.

1.4.3 Menu

In the 'menu' menu only the 'Calibration Data' is available for the MultiMatic, since the pyrolysis methods are defined in the pyrolysis projects, see section 4.2 'Projects'. The 'Calibration Data' displays calibration data for the active probe file.

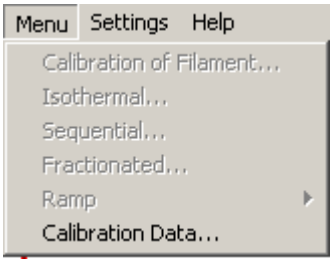


Figure 1-8. The 'menu' menu.

1.4.4 Settings menu

The commands on the 'Settings' menu are used to change general settings that are used in all measurement types.

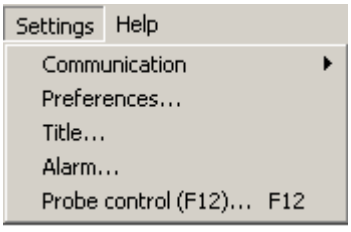


Figure 1-9. The settings menu.

Communication
Select the communication port to which the Pyrola 2000 MultiMatic has been connected

on your computer. After changing the port you will have to exit and re-start the software to make the changes valid. See also chapter 3, 'Start up'.

Preferences

The preferences command opens the preferences dialog, see Figure 1-10. The available options are:

Graph

The max and min values of the x- and y-axis in the graph can be chosen. These values can also be changed in the graph by clicking on the two axes.

Pyrola unit connected

Indicates whether the program has established communication with a Pyrola unit. If there are problems to connect the Pyrola, even if the cables are installed and right com port is chosen, check if the box is empty.

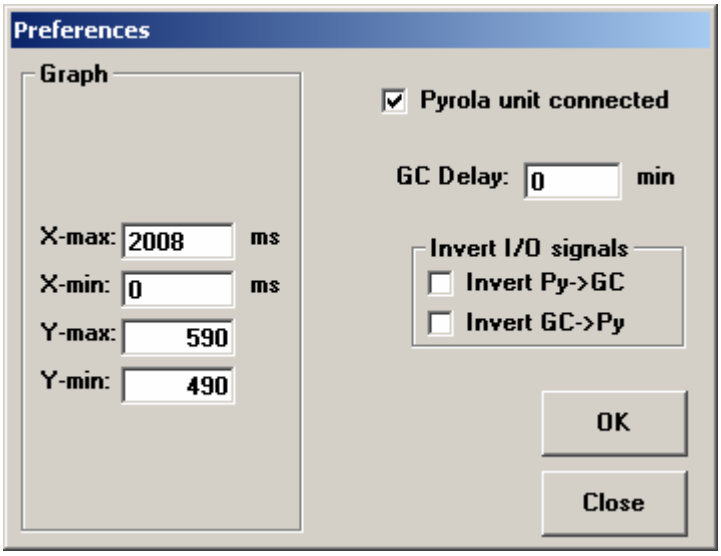


Figure 1-10. The preferences dialog.

GC Delay

To be able to run the Py-GC system automatically, it is important that the GC is ready at the time for pyrolysis. For some GCs it is necessary to choose a delay time, long enough so the GC is ready before the next pyrolysis. For more information, see Appendix II.

Invert I/O signals

For an Agilent or HP GC, the 'Invert GC->PY' should be checked. For other brands, these boxes should be left unchecked.

Title

In the 'Title' dialog you can enter information about your sample, such as sample title, comments and name of operator. Normally not used, since this information is entered in the Projects menu, see section 4.2.

Alarm

You will reach the "Alarm" menu from the menu title "Settings". This is an extra output, which can be used for closing e.g. the He gas.

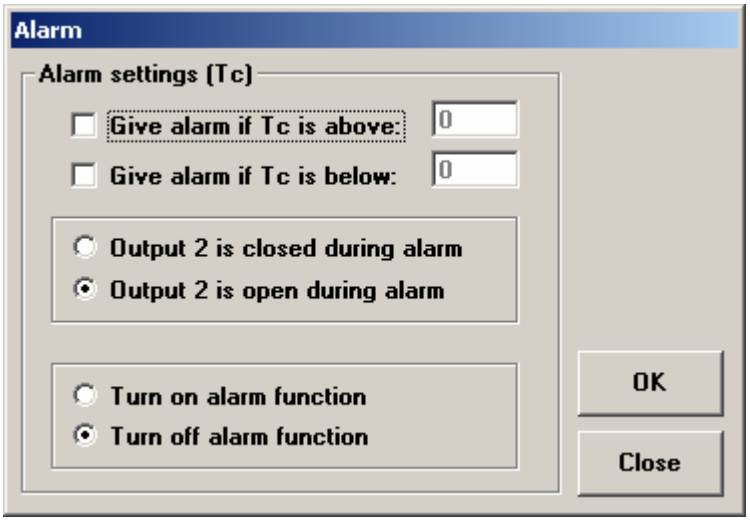


Figure 1-11. The alarm dialog.

The available options are:

1. Give alarm if Tc (chamber temperature) is above a certain temperature.
2. Give alarm if Tc (chamber temperature) is below a certain temperature. Both 1 and 2 may be checked at the same time.
3. Output 2 is closed during alarm.
4. Output 2 is open during alarm.
5. Turn on alarm function.
6. Turn off alarm function.

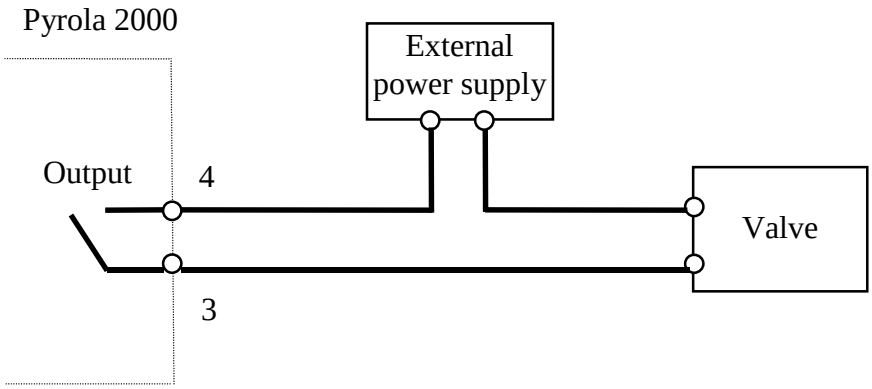


Figure 1-12. Principle of using output 2 for closing the He gas.

Output 2 is a relay output. If output 2 is set to be inactive during alarm, then the output is closed during normal operation.

Probe control

In 'Probe control' you can rotate the carousel and shift which probe is attached to the chamber. The 'change probe' window is shown in Figure 1-13.

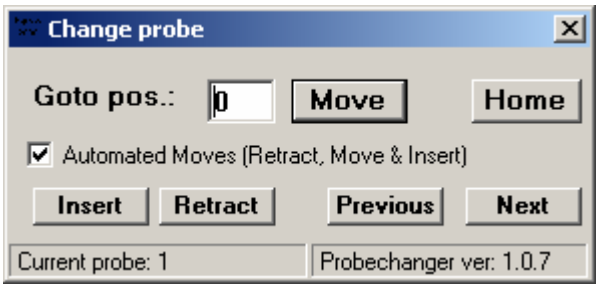


Figure 1-13. The 'Change probe' window.

1.4.5 Help menu

The 'Help' menu includes the commands 'Help topics', and 'About'.

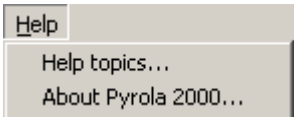


Figure 1-14. The help menu.

The 'Help topic' command opens the Pyrola 2000 MultiMatic online help.

The 'About' command displays information about the software and hardware versions. An example is given in Figure 1-15. Please have this information ready when contacting our support. Click 'OK' to close the 'About' window.

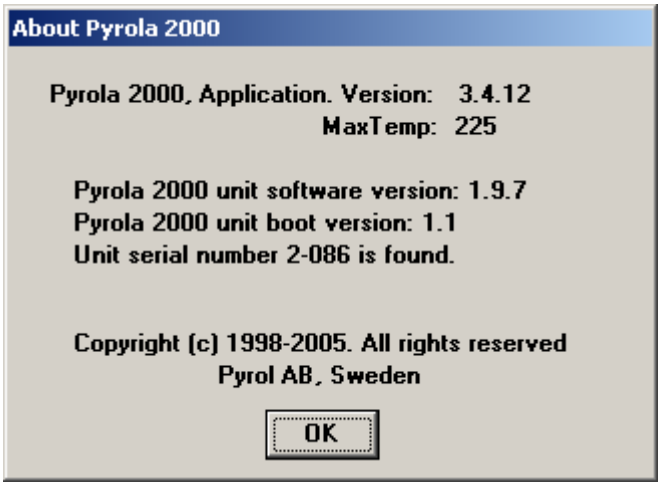


Figure 1-15. Example of output from 'About' command in the help menu.

Chapter 2

2 Installation

This chapter describes the installation of a Pyrola MultiMatic unit on a GC.

2.1 Definitions

A process unit connected to a GC is shown in Figure 2-1, together with definitions of constituent parts.

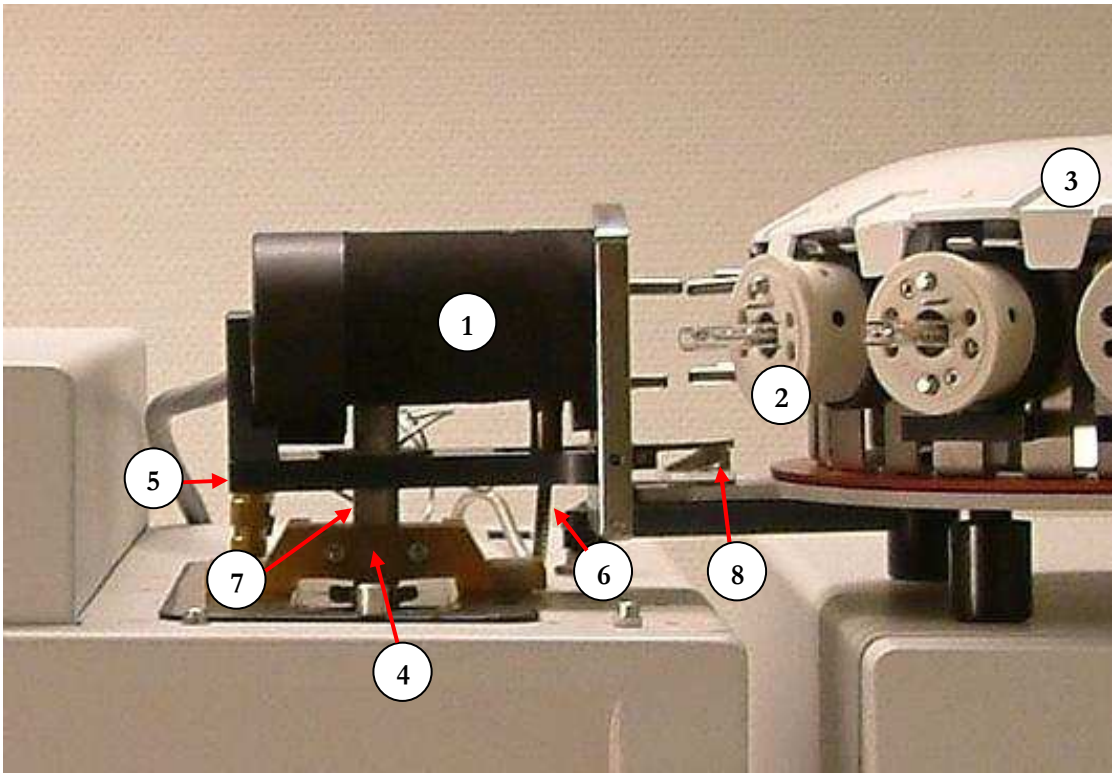


Figure 2-1. A process unit connected to a GC.

Item	Description	Item	Description
1	chamber	2	probe (item 1+2=process unit)
3	carousel	4	base
5	chamber foundation	6	optic cable
7	injection needle (inside neck)	8	carousel quick release

2.2 Connecting the process unit and the carousel to the GC

The figures in this section refer to Figure 2-1, unless stated otherwise.

The process unit of the Pyrola 2000 MultiMatic system is connected to the injector of your GC via an injection needle (7, hidden by the neck). It is equipped with a three-way valve that diverts the carrier gas either directly to the GC or through a quick connector to the process unit. The three-way valve is shown in Figure 2-2, and its operation is shown in Figure 2-3.

Figure 2-2. Three-way valve.

Prior to installation please check the following:

- The process unit is equipped with a base (4) which is used to keep it stable when mounted on the GC injector. Please check whether the base fits directly to your GC or whether you need spacers.
- If you have a new septum in the GC injector it is recommended that you make a "blind" injection or pre-drill the septum. Otherwise there is a risk that a fragment of the septum will plug the injection needle (7) of the process unit. One way is to put the cleaning needle in the injection needle before inserting it through the septum on the GC, and then remove the cleaning needle afterwards.

NOTE!
The glass liner in the GC injector should not contain any glass wool, as non-volatile pyrolysis products are easily condensed and trapped.

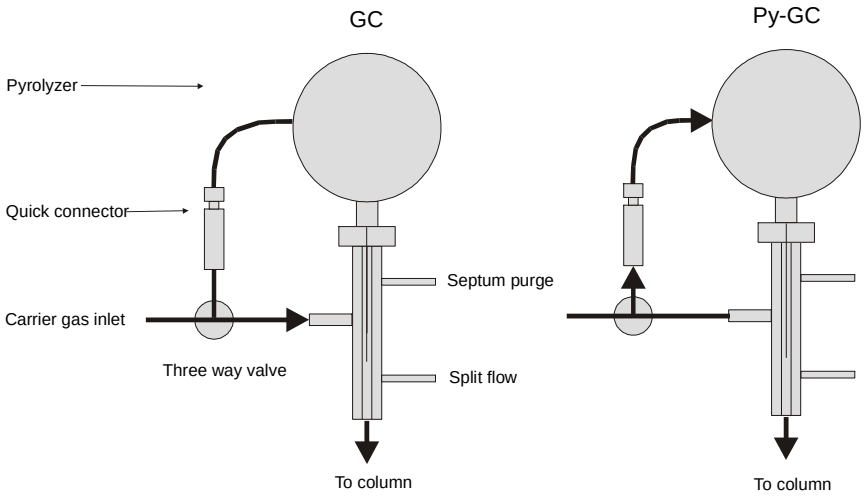


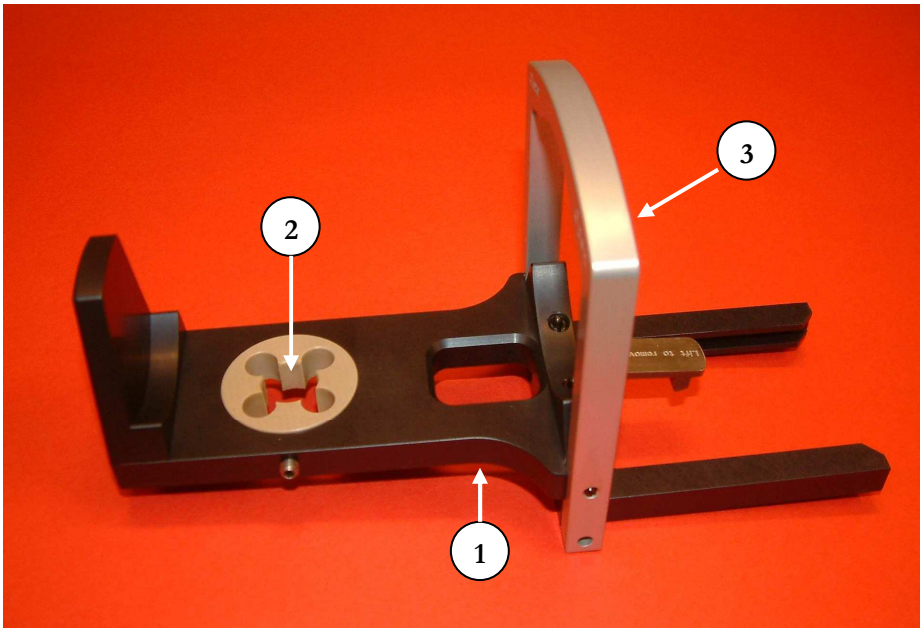
Figure 2-3. Three way valve in position GC and Py-GC, respectively.

The process unit can now be installed into the injector of the GC. The procedure can be somewhat different depending on which GC you are installing it on.

1. Install the three-way valve on a place where you can easily handle it. Cut the carrier gas tube close to the inlet of the GC-injector, and connect one end to the inlet of the three-way valve and the other to one of the outlets. The other outlet should be connected to the quick connector.

2. Install the quick connector and connect it to the three-way valve.

3. Mount the base (4) on the GC. Carefully align it with the GC injector.



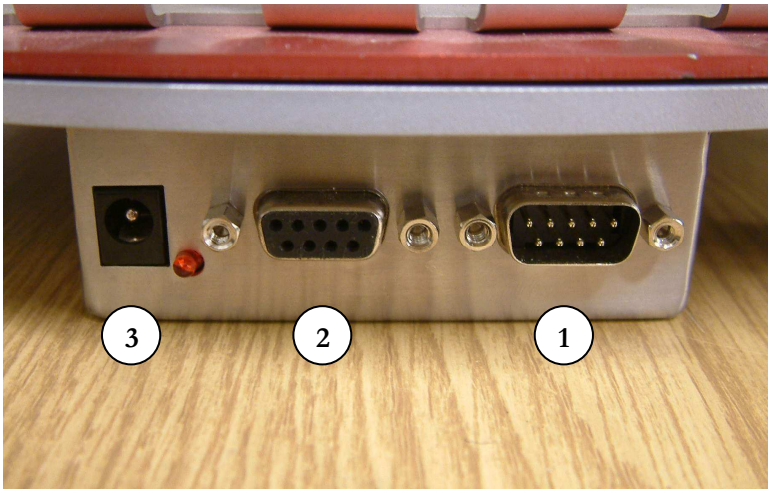
1. **Insert optic cable here.**
2. **Insert injection needle here.**
3. **Shackle for fixing the chamber to the foundation.**

Figure 2-4. The chamber foundation.

4. Insert the optic cable through the hole (1) in the chamber foundation, see Figure 2-4.

WARNING!
Handle the fiber optic cable with extreme care! It is sensitive to mechanical stresses. Especially, avoid any bending.

5. Fold down the shackle on the chamber foundation, (3) in Figure 2-4.
6. Hold the chamber over the foundation and attach the optic cable.
7. Put the chamber in place on the foundation, fold up the shackle to fix the chamber to the foundation.
8. Insert the injection needle through the GC injector.
9. Mount the chamber foundation, Figure 2-4, together with the MultiMatic carousel above the base. Align the hole in the foundation carefully with the hole in the base. Tighten all screws.
10. Connect the gas tube from the chamber with the quick connector.
11. Connect the control cable between the control unit and the chamber, contact (2) in Figure 1-2.
12. Connect the cables between the carousel and the control unit, (9) in Figure 1-4, the PC serial port and the power supply, see Figure 2-5.



1. **Attach the cable to the control unit here.**
2. **Attach the cable to the PC here.**
3. **Attach the 18-24 V power supply here.**

Figure 2-5. Connections on carousel, shown without top plate.

13. Start the Pyrola 2000 Multimatic software. Insert a probe into the chamber by using the Probe control window found under the 'Settings' menu, see Figure 1-13.
14. Wait 2 minutes to allow the probe to attain chamber temperature.
15. Check the split flow or head pressure of the system.
16. Turn the three-way valve to the pyrolysis position.

NOTE!
If there is no gas flow through the process unit the injection needle may be plugged. In this case remove the chamber and clean the needle with the cleaning wire. Re-mount the chamber and check the gas flow again.

2.3 Control unit

The numbers in this section refer to Figure 1-4 unless mentioned otherwise.

1. **Before you connect the process unit make sure that the power selector (4) has been set to the correct voltage. Failing to do so may cause severe damage to the process unit.** Set the power selector to 115 V or 230 V and make sure that the power supply meets the selected voltage within 10%.

2. Connect the control cable to connector (6) on the control unit.

3. Connect the fiber-optic cable from the process unit to connector (2).

4. Connect the power cable to connector (8).

5. Connect the communication cable to connector (9).

6. Connect the cable from the GC (5) controlling the remote start and automation of the system. The connection on the GC is dependent on the brand and model of your GC. For more information, see Appendix II.

2.3.1 Status indicators on the control unit

Front panel

The upper LED on the front panel is green when the control unit is switched on; the lower can show various signals as described in the table below.

Signal	Status	Information
continuous green	Ready	• the unit is ready and a pyrolysis can be run
flashing green	Waiting	• the chamber temperature has not yet reached the pre-set value • the 24 V DC has not reached the minimum level • the platinum filament is broken • the probe is disconnected
orange	Running	• the unit is active, a pyrolysis is currently being done
red	failure	• process unit is not connected to the control unit; check the cable

Rear panel

The LED indicators on the rear panel (Figure 1-4, no. 5) show the status of the signals for remote start and automation. They are described in detail in Appendix II.

2.4 Software installation

The Pyrola 2000 MultiMatic system is delivered with Windows software for parameter setup, instrument control and data handling and controls software for the control unit (see section 2.4 for the update procedure).

1. Insert the installation media.

2. On Windows 95, run DCOM95.exe to install a database handler, or run DCOM98.exe on a Windows 98 computer. On other systems no database handler needs to be installed.

3. Reboot if prompted to do so.

4. Install the main program by running setup.exe.
   Important! If you are using Windows Vista or Windows 7 do not install into c:\Program files, but to c:\Pyrol. This is because newer windows versions handle .ini files differently and the Pyrola software will not find the .ini files if installed in the c:\Program files directory.

5. Copy the two files PHOTO.INI and PYRINI.INI (resides in the root directory of the CD or on a separate media) to the Pyrola 2000 program directory.

2.5 Updating the control unit software (bin-file)

This section describes how to update the control unit with new software, in the form of a bin-file.

1. Go to 'File' and 'Download' in the Pyrola 2000 software.

2. Select the file 'pyrol.bin' in the dialog and click 'OK'. The default location of this file is the directory where you installed the Pyrola 2000 software. If you received the file separately select it from the appropriate drive and directory.

3. Click 'Download' in the window that is shown to start the download procedure. If you do not want to update at this point select 'Cancel'. The progress indicator shows the status during the download.

4. A message is displayed after the download has been finished successfully. Confirm with 'OK'.

5. Exit the Pyrola 2000 software.

6. Shut down the control unit.

7. Restart the control unit.

8. Restart the Pyrola 2000 software.

The Pyrola 2000 system is now ready for use.

2.6 Updating the Pyrola 2000 software with new Pyrol2000.exe file

If you have received an update in form of a new Pyrol2000.exe file, proceed as follows:

1. Exit the Pyrola 2000 program.
2. Start Windows Explorer.
3. Locate the Pyrola 2000 directory. The default location is C:\program files\Pyrol 2000-Multimatic.
4. Rename the old Pyrola2000.exe file.
5. Copy the new Pyrola2000.exe file into the Pyrola 2000 directory.
6. Restart the Pyrola 2000 software.

Chapter 3

3 Start up

This chapter describes the start-up procedure of the control unit and software with possible errors and their remedies.

1. Start the Pyrola 2000 MultiMatic control unit.

Note!
The control unit needs to be switched on before you start the software

2. Double click the Pyrola 2000 MultiMatic icon to start the software. The software will check for a Pyrola 2000 MultiMatic unit on the selected communication port and display the serial number if a unit has been detected. The pyrolyzer can now be used.

If no control unit has been detected during the start-up of the software then two error messages will be displayed. Close these messages by clicking 'OK' and check the following, Figure 3-1.

- Make sure that the unit is connected to the power supply and switched on (upper LED on the control unit is green). If it is switched on after the software has been started you will have to exit and re-start the program.
- Check that the communication cable between the control unit and the computer has been connected properly, see Figure 3-2.
- Check which serial port the communication cable is connected to and go to 'Settings', 'Communication', see Figure 3-3. Change the port number if necessary. Then go to 'Settings', 'Preferences', check 'Pyrola unit connected' and click OK.

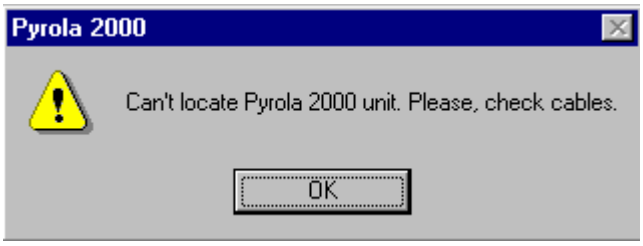


Figure 3-1

Figure 3-2

Figure 3-3

Chapter 4

4 Operation

This chapter describes the operation of the Pyrola 2000 MultiMatic unit and software.

4.1 Calibration

This section describes the calibration of the pyrolysis probes. There are 15 probe positions on the carousel. The probe in the home position is a passive probe used at the beginning and the end of a pyrolysis project, whereas up to 14 pyrolysis probes can be accommodated in the other positions. Before any pyrolysis can be performed the probes that are used need to be calibrated by creating a probe file. Additionally, a probe needs to be recalibrated whenever the filament has been replaced, when the chamber temperature or carrier gas (type or flow) is changed, or if the actual resistance of the filament R0(act) has changed substantially from the R0(cal) used in the calibration. Note that more than one probe file (i.e. calibration) can be saved for each physical probe, for example at different chamber temperatures.

4.1.1 Background

The Pyrola 2000 MultiMatic uses two different types of temperature measurement: light and resistance. The light emitted from the middle of the filament is measured with a photodiode and converted to a temperature value (TD). This measurement is correct without a calibration, provided the glass cell is clean, but it is only accurate at high temperatures.

Below 600° C the temperature is measured by means of the resistance of the filament, which changes with temperature. It is necessary to calibrate the filament with the photodiode because the measurement of TR (temperature measured by resistance) would not be correct otherwise. Please note that the type and flow of the carrier gas as well as the chamber temperature will also influence resistance of the filament, and the measurement of the temperature by the resistance will give incorrect results if not calibrated. Furthermore, a calibration makes it easier to find the conditions, I1 and I2 for an 'ideal' pyrolysis of your sample.

The filament has to be calibrated at least at two temperatures, by finding the parameter settings for an 'ideal' TTP at each temperature. It is necessary that one calibration temperature is between 975° C and 1000° C. The user can select the second calibration temperature; a value of about 900° C is suggested. Additionally, up to 4 calibrated temperatures may be added. It is recommended that the probe has a calibration temperature close to each pyrolysis temperature that will be used.

4.1.2 Creating a probe file

A step-by-step instruction on how to create a new probe file, i.e. to calibrate a probe, is presented below. A more detailed description of the calibration procedure is found in appendix I, 'Calibration – an example'. Before performing a calibration for the first time it is recommended that appendix I is read.

1. Insert the desired probe into the pyrolysis chamber by using the probe control window found under the 'Settings' menu, see Figure 1-13. Check that the three-way valve is in correct position, i.e. in such a way that the carrier gas is flowing through the pyrolysis chamber and probe.

2. Press the Probes button in the main application toolbar in order to open the Probes window, see Figure 4-1.

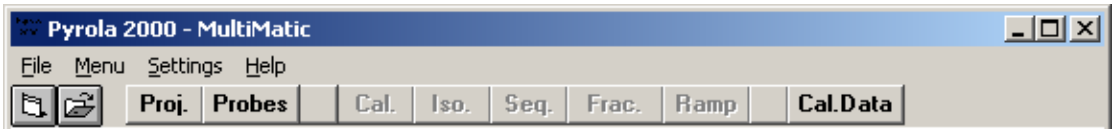


Figure 4-1. Main application toolbar.

3. Select New in the Probes window in order to open the Calibration window with a new probe file, see Figure 4-2.

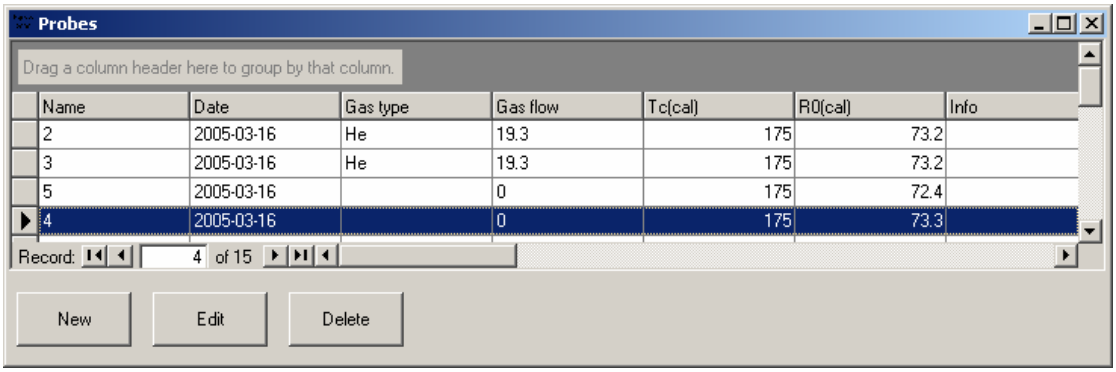


Figure 4-2. Probes window.

4. Rename the current probe file (default name 'Change Me!'), see Figure 4-3. Note that several probe files can be saved for each physical probe, for example at different chamber temperatures. It is therefore suggested that the name reflects both the number of the probe and the calibration conditions, for example 12-175 and 12-225 if probe 12 is calibrated at both 175° and 225° C.

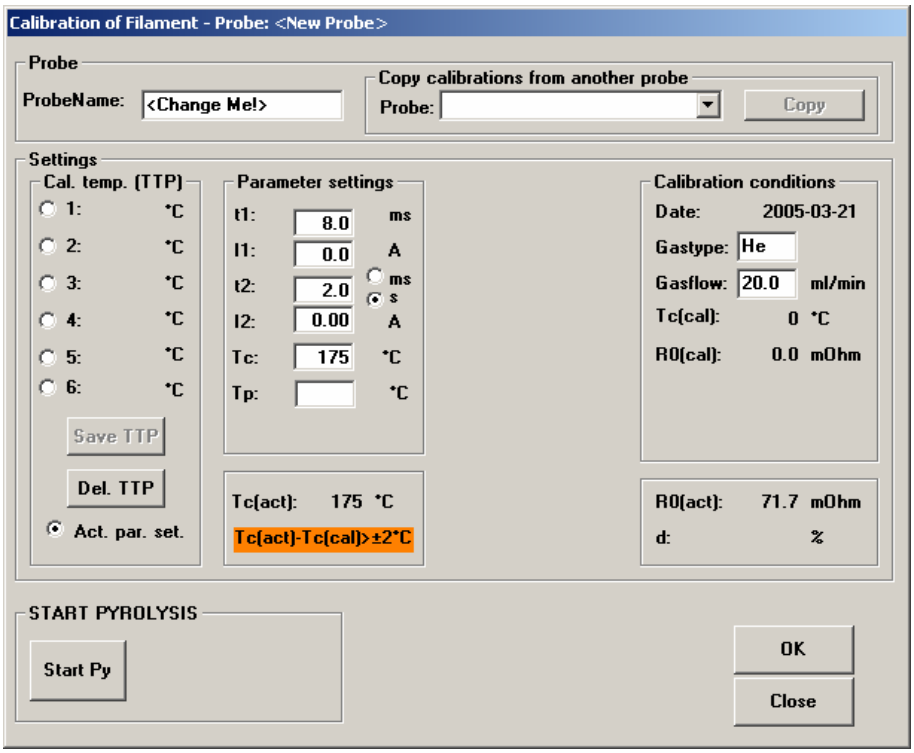


Figure 4-3. Calibration window.

5. Check that the chamber temperature, Tc, is correct. The most common chamber temperature is 175° C, but it may be set between 50° and 225° C.

6. Before proceeding, allow the probe to obtain the chamber temperature. Check that R0(act) has stabilized (usually takes 5-10 minutes).

7. Fill in the type of carrier gas and gas flow in the text box 'Calibrating conditions'. The text will not influence the calibration, but is good for documentation purposes. The current date, Tc(cal) and R0(cal) will be recorded automatically.

8. Choose the parameter settings t1, t2, I1 and I2. The default settings for t1 and t2 are 8 ms and 2 s, respectively. For more information of setting the parameters, see appendix I.

9. Start the pyrolysis by pressing 'Start Py'.

10. The TTP diagram is shown, see Figure 4-4. Check that the initial pyrolysis temperature Td is in the range from 975° to 1000° C, and that the final temperature is the same as the initial temperature, within ±5° C. If not, adjust I1 and I2 and repeat steps 8-9. If the calibration window is hidden behind the TTP diagram, you may show it by pressing the button 'Cal.' above the graph.

Note: the filament may break at high values of I1 and I2. The actual pyrolysis temperature depends on I1, I2, the chamber temperature Tc, the type carrier gas and flow, and the resistance of the filament.

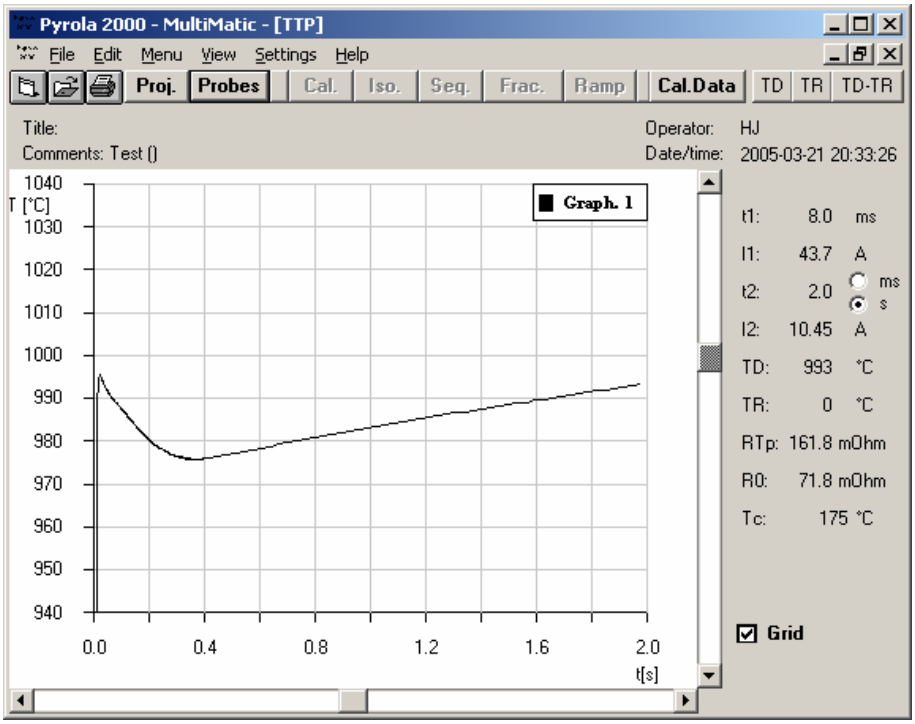


Figure 4-4. TTP diagram.

11. Save the obtained temperature time profile in Cal. Temp. TTP by pressing the button 'Save TTP'.

12. Repeat steps 7-10 at a different temperature. It is easy to get a second calibrated temperature at around 900° C by decreasing I1 with 1.5 A, and I2 with 0.5 A.

When two calibrated temperatures are saved, it is possible to obtain initial values of I1 and I2 automatically. Insert the desired temperature on the line 'Tp' in parameter settings, and press the button 'OK' or just press return. The values of I1 and I2 are calculated, and pressing 'Start Py' will start a new pyrolysis. Minor modifications of I1 and I1 may be needed in order to get a good time temperature profile (TTP). When the TTP is satisfactory, press 'Save TTP' in order to save the result.

13. Use the Tp function to make a calibration at each intended pyrolysis temperature. A total of six calibration temperatures may be stored. Note that the parameters t1 and t2 should be the same for all calibrated temperatures in a probe file.

As a rule of thumb, a decrease of the pyrolysis temperature by 80-100 °C is obtained by decreasing I1 1.5 A and I2 0.5 A, depending on the pyrolysis conditions.

14. Close the calibration window and save the probe file by pressing 'Close'.

The resistance R0(cal) is measured at chamber temperature in the the initial calibration at temperature 975-1000° C. The momentary resistance R0(act) is measured continuously, and is presented together with the deviation in % from R0(cal). In order to get a good temperature measurement TR it is necessary that the deviation between R0(cal) and R0(act) is small. Note that the temperature measured by resistance TR is calculated from

the present resistance of the filament and therefore needs calibration, whereas temperature measured by photodiode (TD) is a direct measurement.

4.1.3 Copying a probe file

It is possible to simplify the calibration procedure if a probe file exists for another probe calibrated under the same conditions. It is necessary that the resistance of the current probe, R0(act), is close to the calibrated resistance of the other probe, R0(cal). Then proceed as follows:

1. Insert the desired probe into the pyrolysis chamber by by using the probe control window found under the 'Settings' menu, see Figure 1-13. Check that the three-way valve is in correct position, i.e. in such a way that the carrier gas is flowing through the pyrolysis chamber and probe.

2. Press the Probes button in the main application toolbar in order to open the Probes window, see Figure 4-1. Information of the different calibration files is presented.

3. Select 'New' in the Probes window in order to open the Calibration window with a new probe file, see Figure 4-2.

4. Rename the current probe file (default name 'Change Me!'), see Figure 4-3. Since it is possible to save more than one probe file for each probe it is suggested that the name reflects both the number of the probe and the calibration conditions, for example 12-175 and 12-225 if probe 12 is calibrated at chamber temperatures of 175° and 225° C.

5. Before proceeding, allow the probe to obtain the chamber temperature. Check that R0(act) has stabilized (usually takes 5-10 minutes).

6. Select the probe file to copy data from in the upper right corner of the Calibration window. Press 'Copy' to copy the probe file.

It is possible to check the current calibration data by pressing 'Cal. Data' in the main application toolbar, see Figure 4-1.

7. Since the copied probe file has at least two calibration temperatures it is possible to get initial values of I1 and I2 automatically. Insert the desired temperature at the line 'Tp' in parameter settings and press the button 'OK', or press return. If the resistance of the current probe, R0(act), is close to the resistance of the copied probe, R0(cal), the calculated I1 and I2 will be close to the correct values. Start a pyrolysis by pressing 'Start Py'.

8. If the TTP curves are satisfactory at all calibrated temperatures, no modifications are needed. Save the probe file and exit the calibration window by pressing 'Close'.

9. If modifications are needed, change I1 and I2 appropriately and repeat the pyrolysis by pressing 'Start Py'. When a time temperature profile (TTP) is satisfactory, save the result by pressing 'Save TTP'.

10. Delete an inaccurate TTP, or a temperature that has not been tested, by selecting it and pressing 'Del. TTP'.

11. Repeat steps 8-9 until all calibrated temperatures are correct.

12. Save the probe file and exit the calibration window by pressing 'Close'.

4.1.4 Edit a probe file

To edit the existing probe file, proceed as follows:

NOTE!
When a probe file has previously been used in a project, it is recommended to copy the probe file and give it a new name instead of editing the file, in order to ensure trackability.

1. Insert the desired probe into the pyrolysis chamber by shifting the rotating table to the correct position by by using the probe control window found under the 'Settings' menu, see Figure 1-13. Check that the three-way valve is in correct position, i.e. in such a way that the carrier gas is flowing through the pyrolysis chamber and probe.

2. Press the Probes button in the main application toolbar in order to open the Probes window, see Figure 4-1. Information of the different probe files is presented.

3. Select the desired probe file and press 'Edit' in the Probes window. The Calibration window is opened with the selected probe file.

4. If R0(act) is close to the calibrated resistance R0(cal), proceed with steps 6-11 in the section 'Copying a probe' above.

5. Otherwise, delete the old calibrated temperatures by selecting them under Settings and pressing 'Del. TTP'. Then proceed with steps 7-13 in the section 'Creating a probe' above.

4.1.5 Deleting a probe file

How to delete a probe file is described in this section. Before deleting a file, consider the possibility to recalibrate the file using the procedure described in the section 'Edit a probe' above.

1. Press the Probes button in the main application toolbar in order to open the Probes window, see Figure 4-1. Information of the different probe files is presented.

2. Select the desired probe file and press 'Delete' in the Probes window.

3. If the selected probe file is used in any project the deletion is interrupted, and the corresponding projects are indicated. To proceed, first remove the probe file in the project, see section 4.2.3 'Changing a project'. Before editing the project, consider the possibility to recalibrate the probe file.

4. If the selected probe file is not used in any project, it may be deleted. Press ‘yes’ to confirm the deletion.

4.2 Projects

This section describes how to set up a project to run a set of analyses using the Pyrola 2000 MultiMatic. The project defines which of the up to 14 available probes that will be used, the pyrolysis conditions, and what kind of pyrolysis method that will be used, i.e isothermal, sequential or fractionated. The pyrolysis method and conditions may be set individually for each probe.

Before setting up a new project all probes that will be used must be calibrated, see section 4.1 'Calibration'. More information on calibration is given in Appendix I.

4.2.1 Creating a project

A step-by-step instruction on how to setup a new project is given below.

1. Press the 'Proj.' button in the main application toolbar to open the projects window, see Figure 4-1. All projects are shown together with some information, one at each line.

2. Select 'New' in the projects window to add a line for the new project, see Figure 4-5.

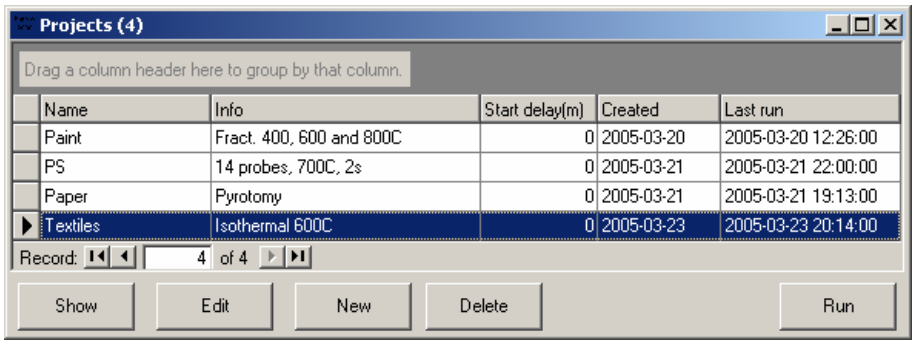


Figure 4-5. Projects window.

3. Fill in the name of the project and add some information in the info text box, for example the kind of samples or the chamber temperature. The name of the project as well as the information given in 'info' will be shown on all TTP-graphs on the comments line. Use TAB key and SHIFT+TAB to navigate between the text boxes.

4. Fill in the 'Start Delay (m)' text box to set the delay in minutes before the project starts when the button 'Run' is pressed. The delay 0 indicated that the pyrolysis starts as soon as the Pyrola 2000 MultiMatic, the GC and the possible MS are ready. In most applications no delay is needed. The text boxes 'Created' and 'Last run' will be set automatically.

5. Press RETURN.

A project has now been added at the bottom of the list. In order to specify which probes and what kind of analyses that should be performed, proceed as follows.

6. Select the project by navigating with the '<' and '>' buttons. The buttons '|<' and '>|' will move you to the beginning or the end of the list, respectively. Alternatively, single-click with the mouse on the desired project.

7. Press the 'Edit' button in order to open the edit project window. Alternatively, double-click on the desired project. The 'Edit project' window opens, see Figure 4-6.

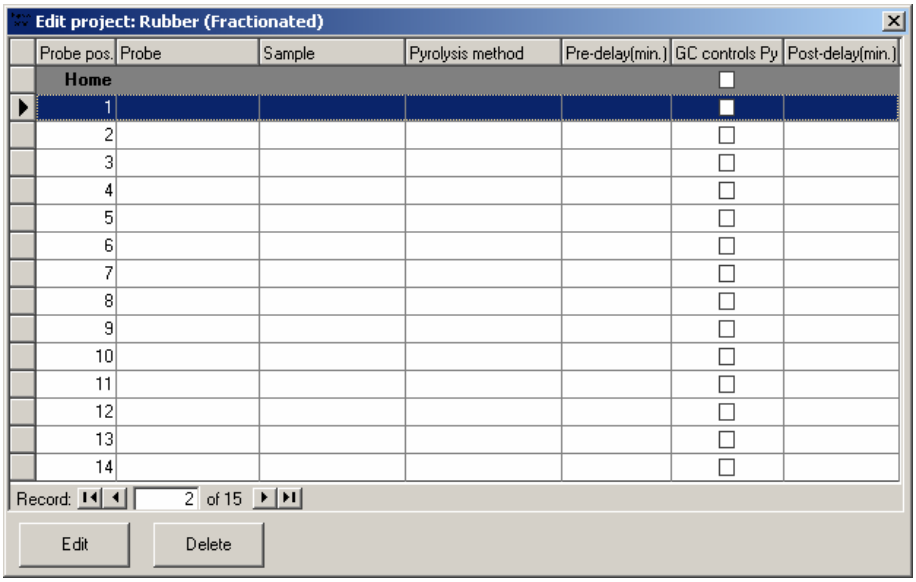
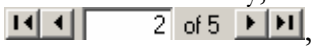


Figure 4-6. 'Edit project' window.

In the 'edit project' window a table is shown with all 14 probe positions, one at each line, where the numbers refer to the probe positions on the carousel. The columns Sample, Predelay, GC controls Py and Postdelay may be filled in directly, whereas the columns Probe and Pyrolysis Method are filled in by accessing a pop-up-menu, as described below.

8. Select the 'Sample' text box in the desired probe position by clicking with the mouse. Alternatively, select the appropriate line using the navigation bar 

, and then using the TAB and/or the SHIFT+TAB keys. It is also possible to navigate by using the arrow keys on your keyboard. Enter information about the sample. The text will appear in the title of the TTP graph of the analysis.

9. In the same way as above, enter the 'PreDelay', specifying the delay in minutes before the first pyrolysis is started for the current probe. A predelay of 5-10 minutes is recommended to assure that the probe achieves the chamber temperature before the pyrolysis starts.

10. The 'GC controls Py' and the 'PostDelay' are normally not used. The 'GC controls Py' is selected/deselected either by pressing SPACEBAR on your keyboard or by clicking in the box.

In order to specify the probe file and pyrolysis method and conditions for the current probe do in the following way:

11. Open the 'Pyrolysis and probe selector', Figure 4-7, by selecting the appropriate Probe text box and click on the 'Edit' button. Alternatively, double-click in the Probe text box.

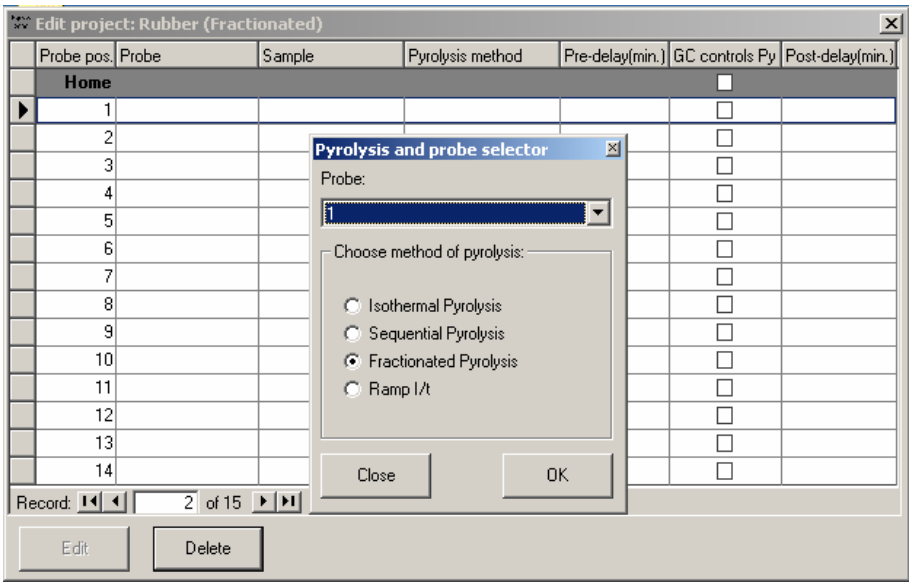


Figure 4-7. 'Pyrolysis and probe selector' window.

12. Select a probe file that is valid for the current probe on the 'Probe' line. Use the TAB and arrow keys to navigate, or use the mouse.

All probes need to be calibrated individually. Using a probe file for a different probe will give erroneous results.

13. Select the pyrolysis method, i.e. isothermal, sequential, fractionated or ramp I/t.

14. Press 'OK' to save the choice of probe file and pyrolysis method, then press 'Close' to close the window. If only 'Close' is pressed, the window will close without saving.

15. Depending on the choice of pyrolysis method, the appropriate setup window will be opened, see section 4.2.2 below. Enter the parameter settings for the pyrolysis as described below, and save the settings by pressing 'OK'. 'Close' will close the window without saving.

16. Repeat steps 8-15 for each probe that will be used in the project.

17. Close the 'Edit project' window and save the changes by clicking on the ☒ in the upper right corner of the window.

4.2.2 Setting parameters for the pyrolysis

The choice of pyrolysis method is done in the 'Pyrolysis and probe selector' as described in step 13 in 'Creating a project' above. The header of the current window indicates the probe position, pyrolysis method and current probe file. Depending on the choice of pyrolysis method, different sets of parameters shall be given. The process is described below.

4.2.2.1 Isothermal pyrolysis

The parameter that shall be set in isothermal pyrolysis is the pyrolysis temperature Tp, which can be set to any value between the chamber temperature, Tc, and 1400° C. Additionally, it is possible to change the pyrolysis time t2. The parameters t1 and Tc must be the same values as in the current probe file. The parameters I1 and I2 are calculated automatically. Note that if the desired pyrolysis temperature Tp differs significantly from the calibrated temperatures as shown in Cal.temp.(TTP), then the actual pyrolysis temperature may differ from the intended. In that case the probe file should be supplemented with the new pyrolysis temperature, see 'Edit a probe' above.

How to set the pyrolysis parameters is described below:

1. Enter the desired pyrolysis temperature on the line 'Tp' in parameter settings, and press the button 'OK', or just press return.

2. Press the button 'Close' to save the settings, close the window and return to the 'Edit project' window.

The other parameters must not be changed, or else the pyrolysis temperature will be wrong.

4.2.2.2 Sequential pyrolysis

The parameters that shall be set in the sequential pyrolysis are the pyrolysis temperature Tp, the pyrolysis time t2, and the number of pyrolyses n. The pyrolysis time t1 and the chamber temperature Tc must be the same as in the current probe file. Note that if the desired pyrolysis temperature Tp differs significantly from the calibrated temperatures as shown in Cal.temp.(TTP), then the actual pyrolysis temperature may differ from the intended. In that case the probe file should be supplemented with the new pyrolysis temperature, see 'Edit a probe' above. How to set the pyrolysis parameters is described below:

1. Enter the desired pyrolysis temperature on the line 'Tp' in parameter settings, and press the button 'OK', or just press return. Tp can be chosen in the range from Tc to 1400° C.

2. Insert the number of pyrolyses n (1-9), and press 'OK', or return.

3. Press the button 'Close' to save the settings, close the window and return to the 'Edit project' window.

4.2.2.3 Fractionated pyrolysis

The parameters that shall be set in the fractionated pyrolysis are the pyrolysis temperatures Tp (maximum 6 different temperatures are possible. For example to precede a pyrolysis with a thermal desorption to extract any volatile components of the sample, use a fractioned pyrolysis with a low temperature (typically 300° C) followed by an ordinary pyrolysis temperature (or several).

The pyrolysis time t1 and the chamber temperature Tc must be the same as in the current probe file. Note that if the desired pyrolysis temperatures Tp differ significantly from the calibrated temperatures, then the actual pyrolysis temperature may differ from the intended. In that case the probe file should be supplemented with new pyrolysis temperature(s), see 'Edit a probe' above. The 'fractionated pyrolysis' window is shown in Figure 4-8 below.

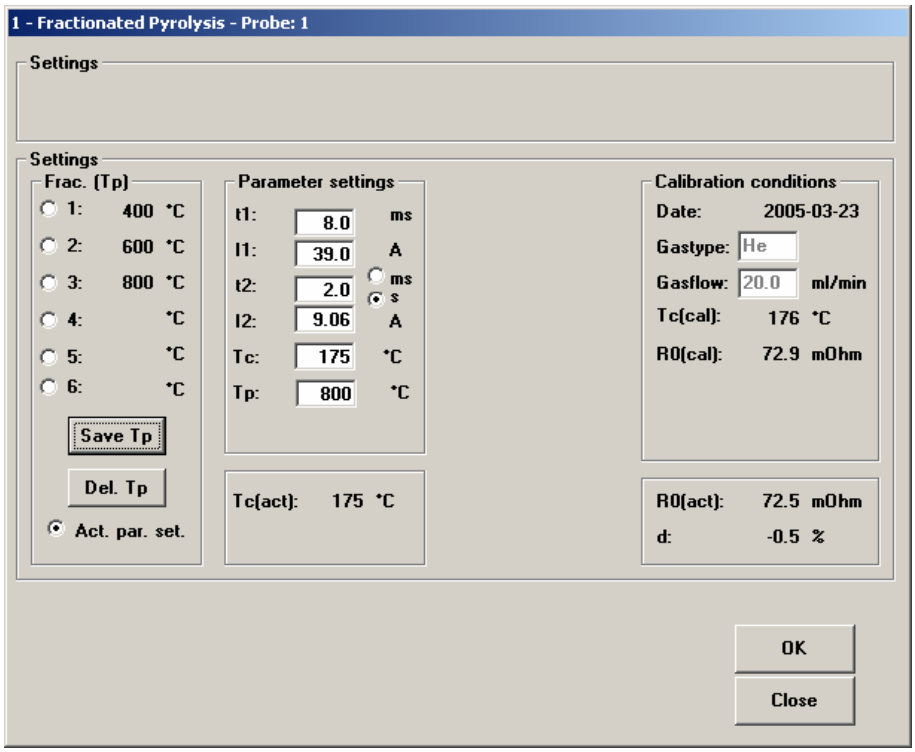


Figure 4-8. 'Fractionated pyrolysis' window.

How to set the pyrolysis parameters is described below:

1. Enter the desired pyrolysis temperature on the line 'Tp' in parameter settings, and press the button 'OK', or just press return. Tp can be chosen in the range from Tc to 1400° C.

2. Press the button 'Save Tp' in Settings Frac. (Tp) in order to save the current Tp.

3. Repeat steps 1-3 until all desired pyrolysis temperatures are entered. To delete an inserted Tp, select it and press 'Del Tp'.

4. Press the button 'Close' to save the settings, close the window and return to the 'Edit project' window.

4.2.2.4 Ramp I/t

Ramp I/t is used to heat a sample slowly up to a elected end temperature. The method can be useful if the sample is to be analyzed in a mass spectrometer. The 'ramp I/t' window is shown in Figure 4-9 below.

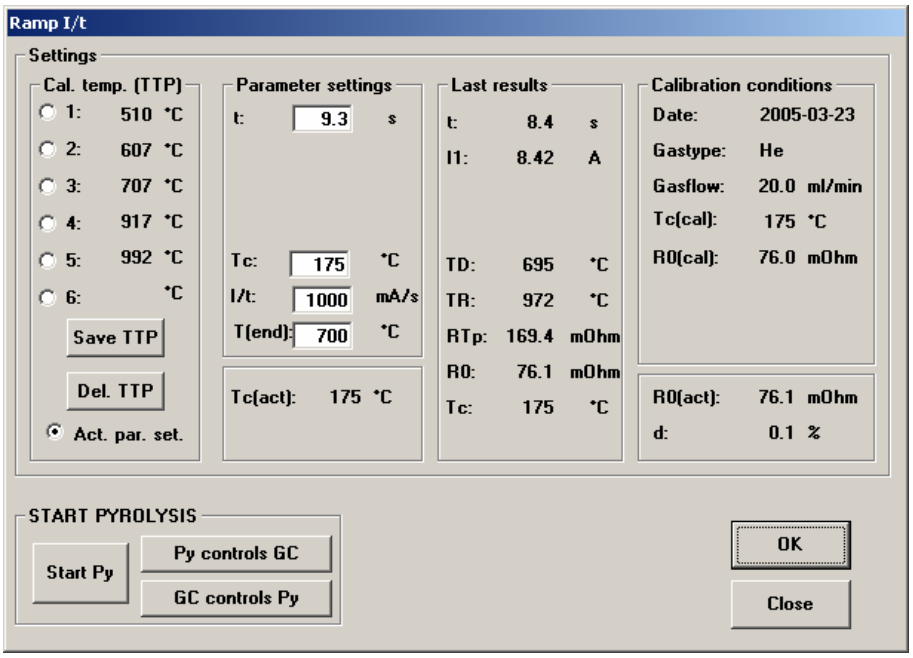


Figure 4-9. 'Ramp I/t' window.

The parameters that shall be set are the heating rate I/t, the end temperature T(end) and the time t to reach the end temperature.

How to set the pyrolysis parameters is described below:

1. Enter the desired end temperature on the line 'T(end)' in parameter settings, and press the button 'OK', or just press return. T(end) can be chosen in the range from Tc to 1400° C.

2. Enter the desired heating rate I/t, and press 'OK', or return. I/t can be chosen in the range 10-1000 mA/s.

3. Enter the desired time t to reach the end temperature, and press 'OK', or return. If the time is too long a warning is shown. The maximum value of t depends on the end temperature T(end).

4. Press the button 'Close' to save the settings, close the window and return to the 'Edit project' window.

4.2.3 Changing a project

To modify an existing project, proceed as outlined below:

1. Press the 'Proj.' button in the main application toolbar to open the projects window, see Figure 4-1. All projects are shown together with some information, one at each line.

2. Select the project that should be changed clicking with the mouse on the desired project. Alternatively, use the navigation bar 2 of 5.

3. Press the 'Edit' button in order to open the edit project window. Alternatively, double-click on the desired project.

4. To edit the text, single-click with the mouse in the appropriate text box. Alternatively use the navigation bar 

to select the line, and then use the TAB and/or the SHIFT+TAB keys to navigate between the text boxes. It is also possible to navigate by using the arrow keys on your keyboard.

5. The 'Sample', 'PreDelay', 'PostDelay' and 'GC control Py' boxes may be changed directly in the 'Edit project' window. Proceed as in steps 8-10 in 'Creating a project' above.

6. To change the 'Probe' or the 'Pyrolysis method' proceed as in steps 11-15 in section 4.2.1 'Creating a project' above.

7. Close the 'Edit project' window and save the changes by clicking on the  in the upper right corner of the window.

4.2.4 Showing a project

To show the information in the 'Edit project' window of an existing project proceed as outlined below:

1. Press the 'Proj.' button in the main application toolbar to open the projects window, see Figure 4-1.

2. Select the desired project by clicking with the mouse at the left edge of the window. Alternatively, use the navigation bar 

.

3. Press the 'Show' button in order to open the 'Show project' window. No changes are possible.

4. The 'Show project' window is closed by clicking on the  in the upper right corner of the window.

4.2.5 Deleting a project

The process of deleting an existing project is described below:

1. Press the 'Proj.' button in the main application toolbar to open the projects window, see Figure 4-1.

2. Select the desired project by clicking with the mouse at the left edge of the window. Alternatively, use the navigation bar

.

3. Press the 'Delete' button.

4. Confirm the delete project by pressing 'Yes' in the pop-up window.

4.2.6 Running a project

In order to run a pyrolysis project on the Pyrola 2000 MultiMatic proceed as follows:

1. Press the 'Proj.' button in the main application toolbar, see Figure 4-1, to open the projects window, Figure 4-10.

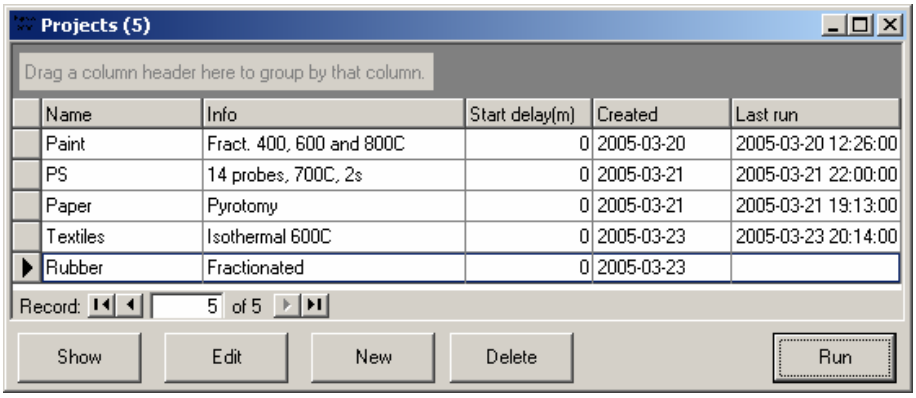


Figure 4-10. 'Projects' window.

2. Select the desired project by navigating with the '<' and '>' buttons. The buttons '|<' and '>|' will move you to the beginning or the end of the list, respectively. Alternatively, single-click with the mouse at the left edge to select the project.

3. Make sure that all probes that will be used are calibrated and prepared with the correct samples.

4. Make sure that the GC or GC/MS system is ready and prepared with a GC sample list and GC/MS-methods.

5. Make sure that the carrier gas is flowing through the pyrolysis chamber.

6. If necessary, make a folder using Windows Explorer where the TTP-files and the log-file can be saved. It is recommended that the files are saved in a folder different from pyrolysis program, marked with date and/or analysis.

7. Press 'Run' in the projects window.

8. Select the folder where the TTP-files and log-file will be saved, see item 6 above.

9. Enter the operator of the analyses. The information will be written in the TTP graphs as operator.

10. Start the project pressing by pressing 'OK'.

Sample Handling

Clean the Pt-filament before each pyrolysis by heating with a micro-torch, but be careful not to overheat the filament as the septum may be damaged. If necessary, clean a heavily contaminated filament with diluted HCl or by careful mechanical cleaning.

The sample is placed on the platinum filament of the pyrolysis probe. The filament may either be flat, or have a cavity made with a special tool. The cavity is used for e.g. paper or powder to keep the sample in position.

If the sample is soluble the required amount can easily be applied with a syringe or pipette. The solvent is evaporated outside the gas chromatograph either by the heat of the probe, a heating lamp or a soldering iron.

There are two different sample handlers (optional), one for e.g. paper and one for insoluble powder. The handlers are shown in Figure 4-11 together with a probe and the tool for making cavities in the filament.

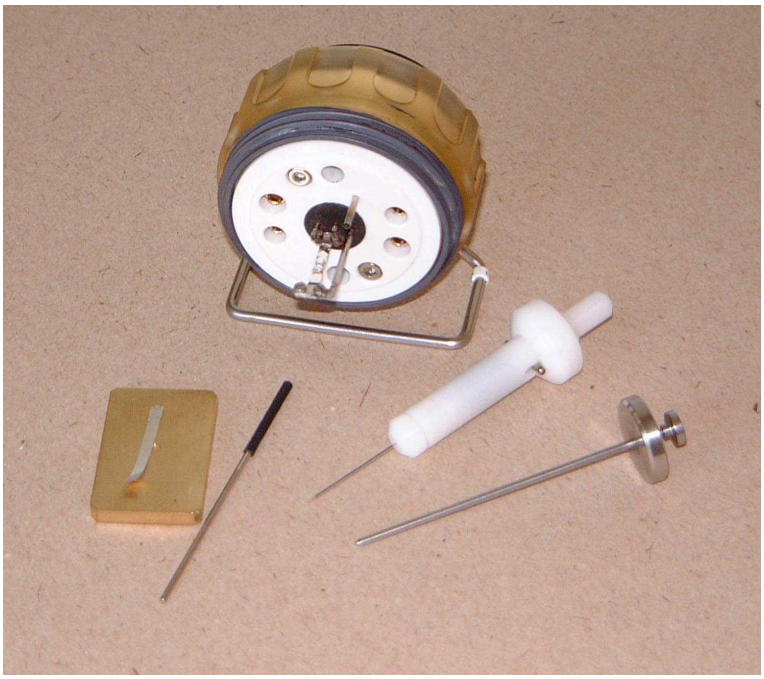


Figure 4-11. Sample handlers.

4.3 Maintenance

4.3.1 Cleaning the glass cell

The purpose of glass-cell in the process unit (see Figure 1-2, no. 7) is to be able to let the light from the heated Pt-filament pass to the fiber-optic cable. Furthermore, it protects the GC-column from non-volatile pyrolysis products. Therefore, the glass-cell might need to be cleaned after a certain time. An indication of this is e.g. when you find non-reproducible pyrolysis products with long retention times.

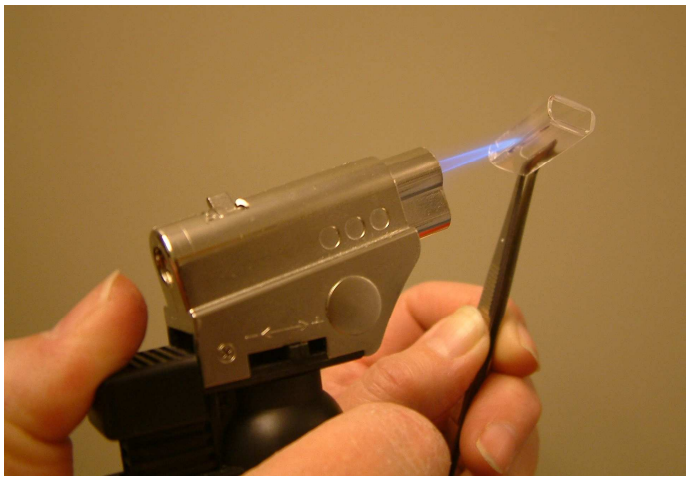
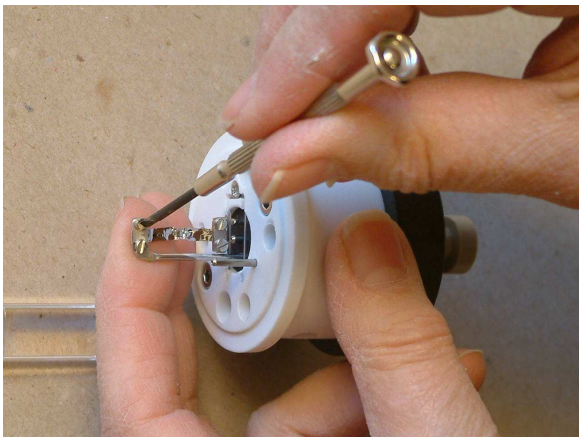


Figure 4-12. Cleaning the glass cell with a micro torch.

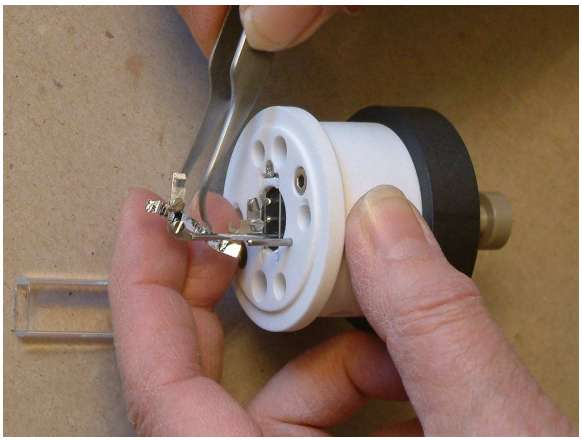
The easiest way to clean it is to put it in a Bunsen burner or to use a micro torch, see Figure 4-12. Notice, that air should be able to pass inside the cell, otherwise the products will be pyrolysed and generate carbon, which is difficult to remove.

WARNING!
Make sure to be adequately protected when cleaning the glass cell with heat. Also, handle the glass cell with care and avoid sudden temperature changes.

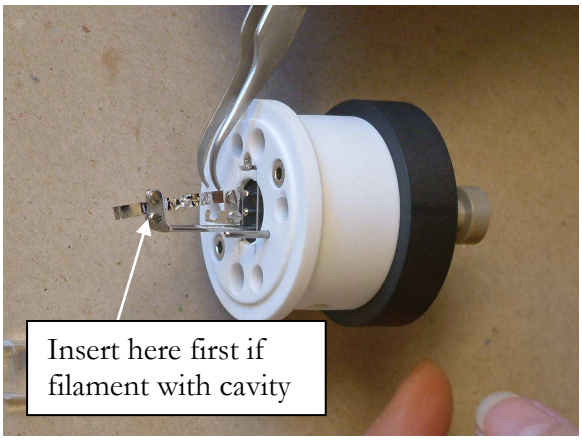
4.3.2 Replacing the filament



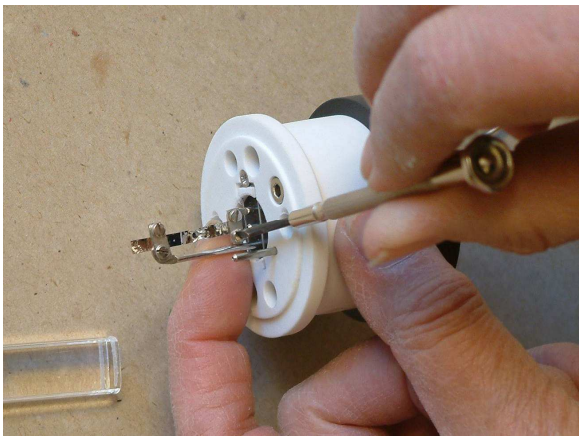
Loosen the screws holding the filament.
Do not remove the screws!



Open the lids and remove the old filament.



Insert the new filament. If a filament with a cavity is used, insert the filament furthest away from the probe first.



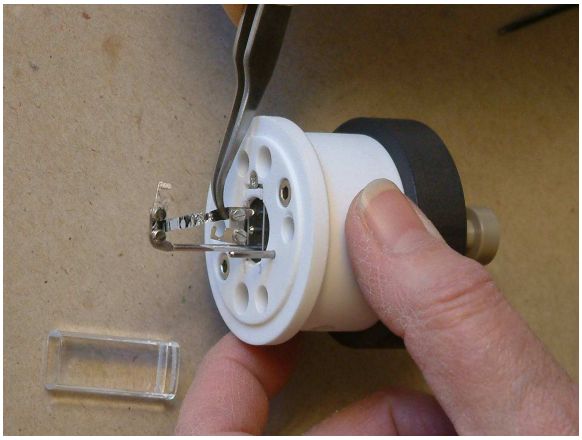
Adjust the position of the filament to be in line with the Pt-flags closest to the probe. Fasten the end closest to the probe first.

Stretch the filament and bend any excess filament down. Fasten the other end.

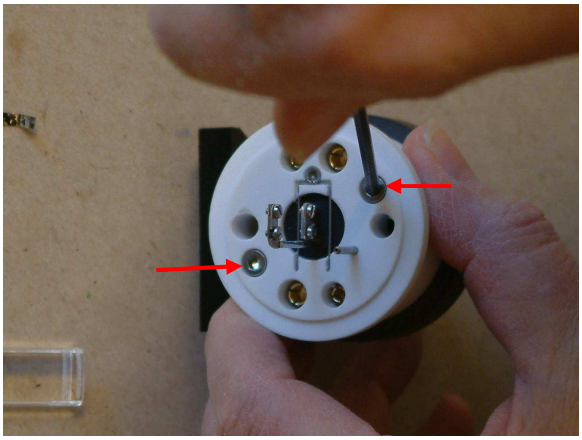
Clean the filament from finger prints by heating with a micro torch.

Calibrate the new filament.

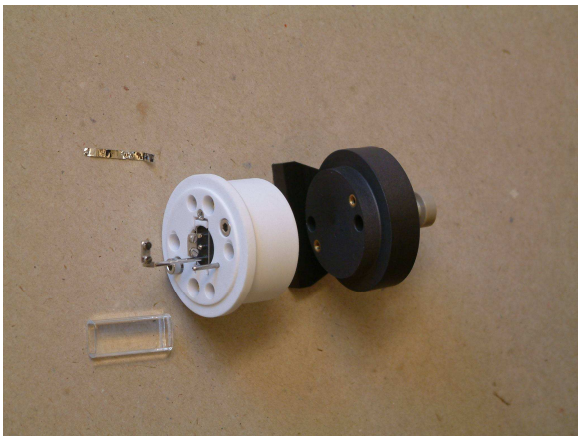
4.3.3 Replacing the septum



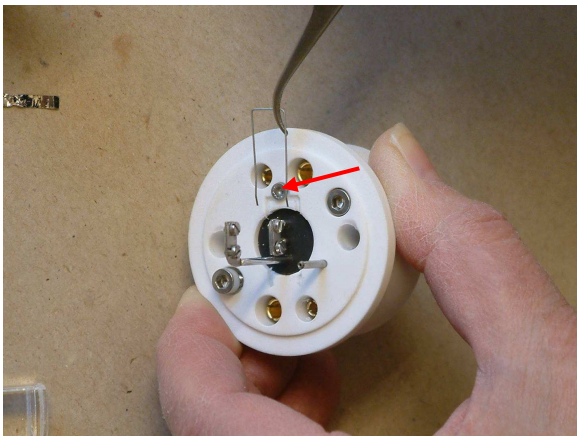
Remove the filament.



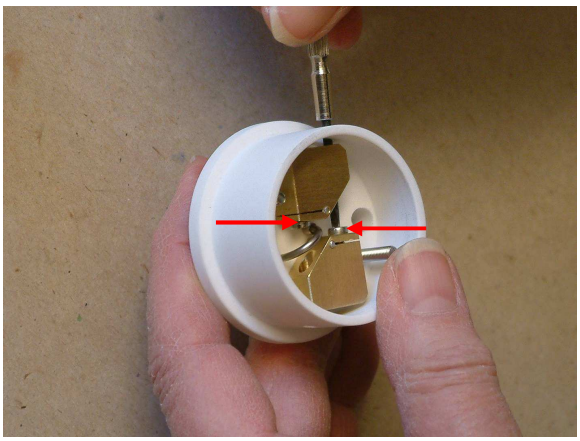
Remove the screws holding the probe together.



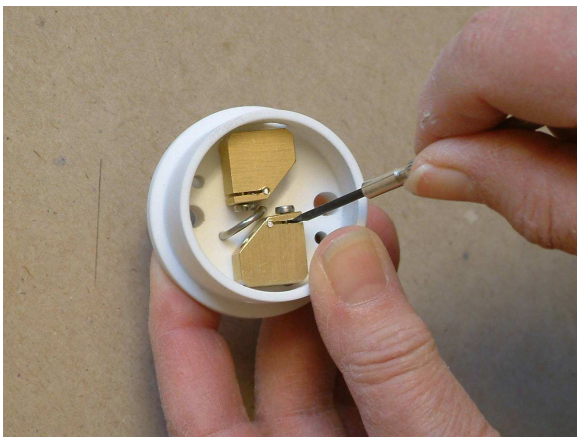
Separate the probe.



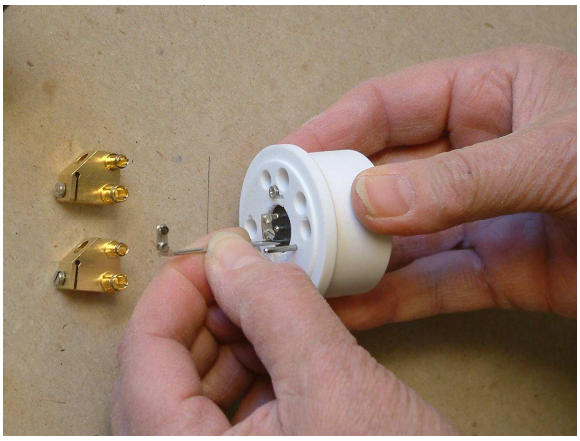
Loosen the screw holding the spring for the glass cell, and remove it carefully.



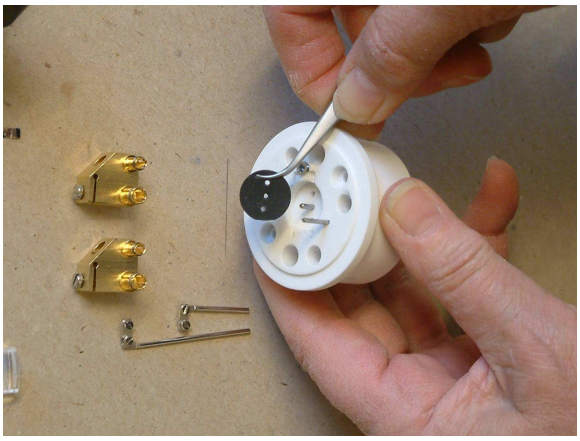
Loosen the screws holding the Pt-flags.



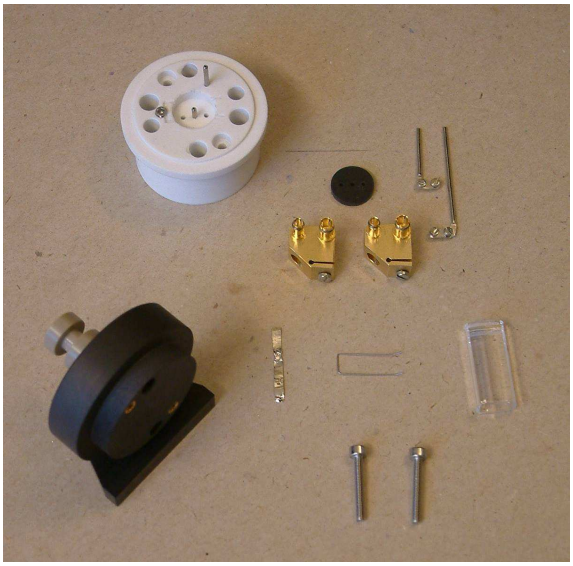
Widen the slit holding the Pt-flags carefully. Remove the flag holders with contacts.



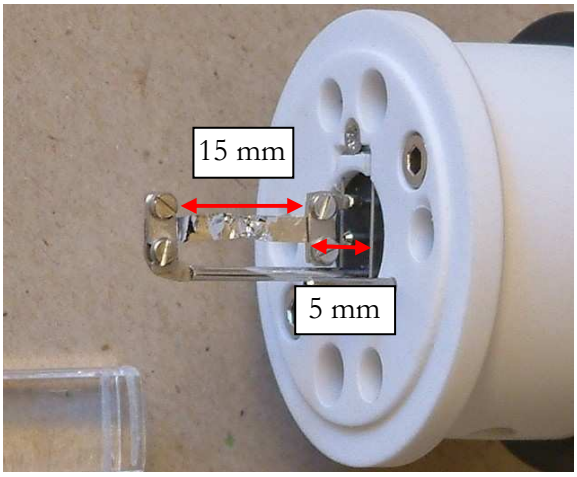
Remove the Pt-flags. Be careful not to damage the flags.



Remove the septum.



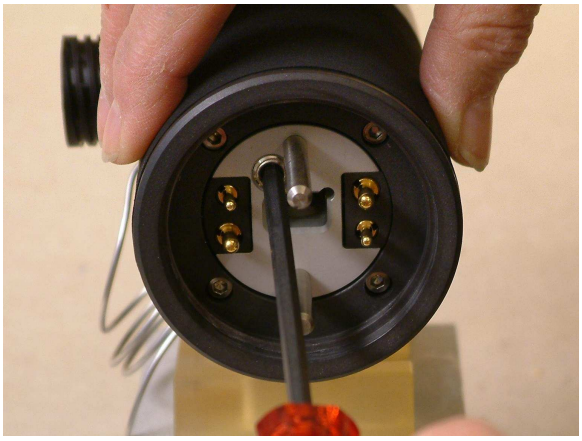
Insert the Pt-flags in the holes in the new septum before mounting it on the probe. Reassemble the probe.



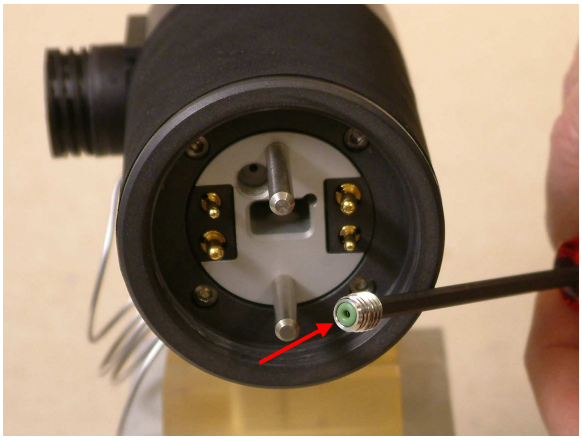
The distance between the outer edge of the inner Pt-flag and the septum shall be 5 mm, and the distance between the flags shall be 15 mm.

Mount the Pt-filament as described in section 4.3.2.

4.3.4 Replacing the O-ring

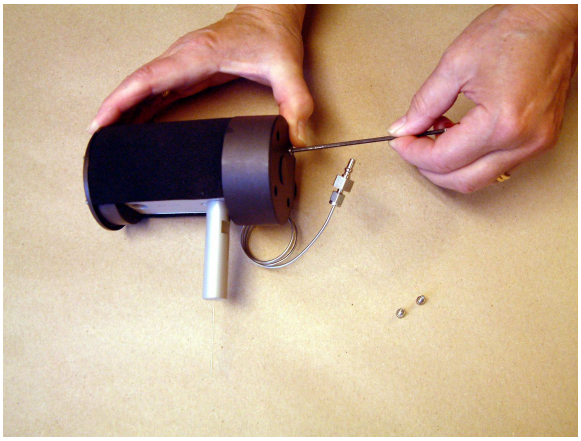


Use a 4 mm socket head cap screw driver to remove the gas outlet screw.

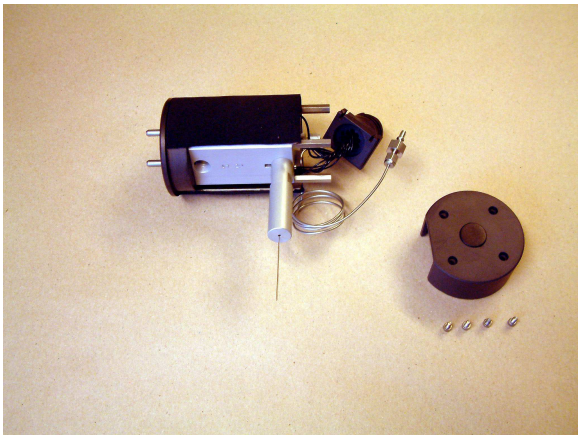


The o-ring is situated on the end of the gas outlet screw. Change it carefully and replace the screw.

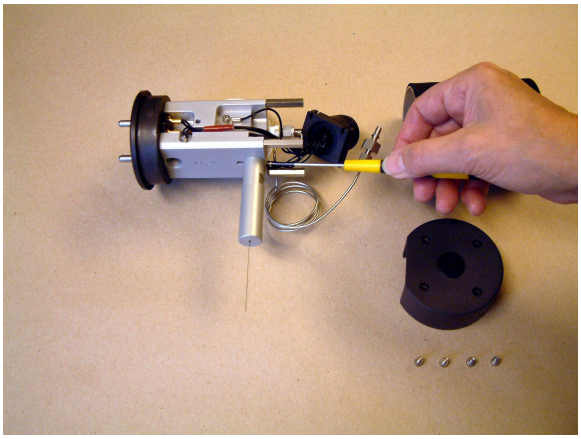
4.3.5 Changing the injection needle



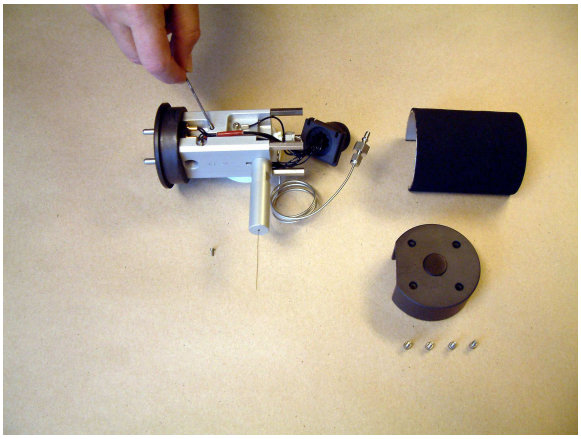
Unscrew the four screws on the coupling box.



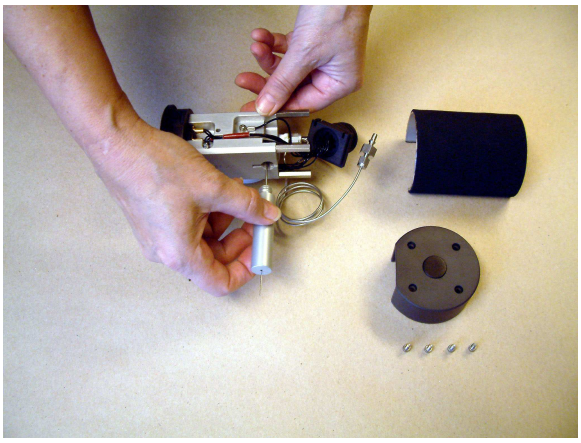
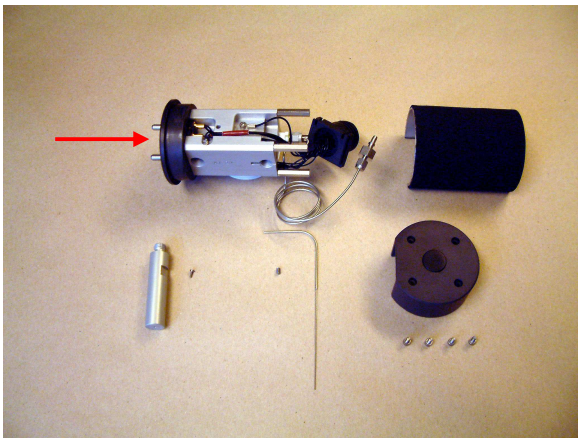
Remove the four screws, the coupling box and lift away the cover.



Unscrew the screw locking the neck.



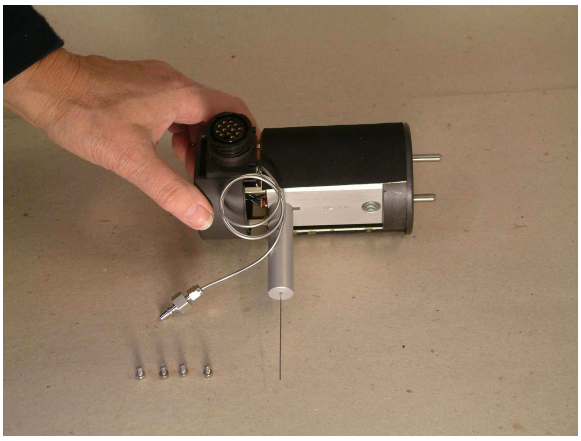
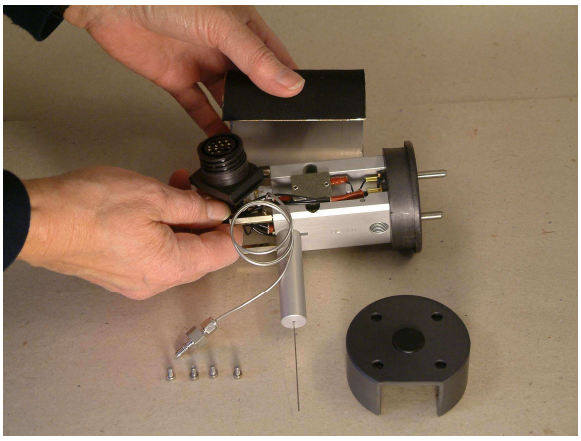
Unscrew the screw locking the needle.

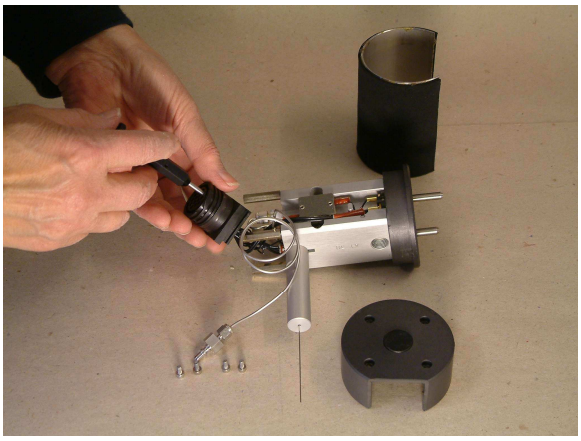
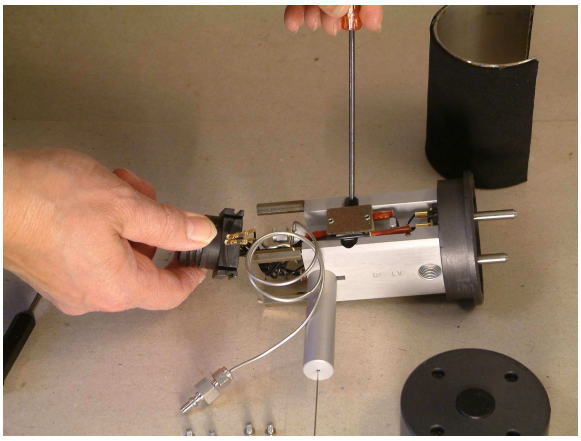
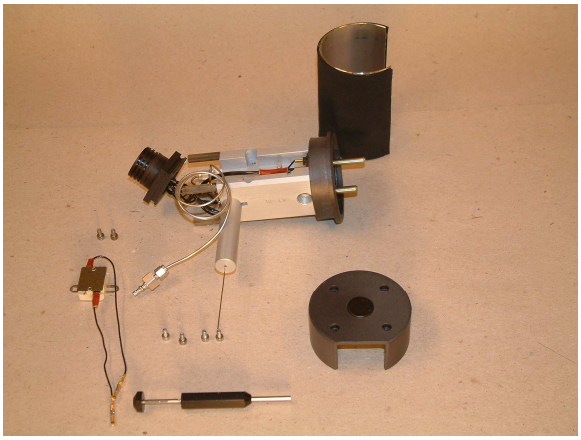
	
Unscrew the neck.	Push the needle back through the septum by pressing with a socket head cap screwdriver in the direction indicated by the arrow. Remove the needle.

Mount the neck on the new injection needle before it is put in position. Reassemble the chamber. Insert the cleaning needle into the injection needle before remount the chamber on the GC. Remove the cleaning needle.

4.3.6 Replacing the thermo fuse in the pyrolysis chamber

A special tool is needed for this operation, shown in the figure below.

	
Unscrew the four screws on the coupling box, and remove it.	Remove the cover.

	
Locate pin no. 11 and 14 in the contact, and use the special tool to detach them.	Remove the two screws for the thermo fuse and remove it.
	Mount the new thermo fuse. Attach the cable to the contact by carefully inserting the pins in position 11 and 14. Check that the pins fit tightly in the contact and cannot be removed by hand, only with the tool. Reassemble the chamber.

4.3.7 Replacing other electrical components in the chamber

To replace other electrical components in the chamber, proceed as described in section 4.3.6, 'Replacing the thermo fuse in the pyrolysis chamber' above'. The pin layout is shown in Appendix III.

Chapter 5

5 Warnings and precautions

Make sure that all personnel involved in handling the Pyrola 2000 MultiMatic equipment are informed of and adhering to the warnings and precautions in this chapter and throughout this manual.

- Accessible parts of the chamber may be warm! Make sure to be protected adequately when handling the pyrolysis chamber if the chamber temperature Tc exceeds 65°C.
- The maximum chamber temperature is 225 °C. Failing to observe these limits may result in severe damage to the unit.
- Always make sure you have carrier gas around your filament whenever the chamber is heated.
- Helium can conduct heat much more efficient than e.g. air or N2. Therefore a higher current amplitude I2 is necessary when Helium is used as a carrier gas. Make sure to decrease the amplitude if the carrier gas is switched to air or N2. A too high current may break the filament.
- There are dangerous voltages inside the Pyrola 2000 MultiMatic control unit. Do not open the unit. In case of failure, contact your distributor service center to get help.
- Never leave the chamber at temperatures above 200 °C for a longer period of time. Decrease the chamber temperature to 175 °C.
- Be sure that you have the same carrier gas as when you calibrated the filament.
- Start the control unit before the starting the program.
- Exit the program before turning off the control unit.
- Clean the glass cell if you found non-reproducible peaks with high boiling points and/or if the chamber temperature Tc is increased, see section 4.3.1.
- Clean the Pt-filament before each pyrolysis by heating with a micro-torch, but be careful not to overheat the filament as the septum may be damaged.
- If necessary, clean a heavily contaminated filament with diluted HCl or by careful mechanical cleaning.
- Handle the optic cable with great care. It can easily be damaged by bending or any other mechanical stress.

Chapter 6

6 Troubleshooting

NOTE!
No quarts wool in the injection liner! Non-volatile pyrolysis products are easily condensed and trapped.

6.1 Installation

No gas through the pyrolyzer:	The injection needle is plugged. Remove the process unit from the gas chromatograph and clean the needle with the cleaning wire. The gas line from the three way valve to the pyrolysis chamber is leaking. Check the valve. The quick fitting on the gas tube is not connected to the pyrolysis chamber.
”Cannot locate the Pyrola®2000”	See chapter 3 ‘Start up’.

6.2 Process unit

The chamber is not heated	The thermo fuse is blown. Replace the fuse. Tc is not activated Go to the pyrolysis menu, enter the chamber temperature value and press ‘Enter’
No gas through the pyrolyzer	The injection needle plugged. Remove the process unit from the gas chromatograph and clean the needle with the cleaning wire. The three way valve is in the wrong position. Check the valve and adjust if necessary The quick fitting on the gas tube is not connected to the pyrolysis chamber.

Gas leakage	The chamber has not reached its final temperature. Wait 5-10 minutes. The glass cell is damaged. Check the cell and replace if necessary The septum in GC injector is not OK. Check the septum and replace if necessary The septum in the pyrolysis probe is aged. Replace the septum The septum in the bottom of the glass cell inside the chamber is aged. Replace the septum (section see section 4.3.3) The o-ring behind the screw where the carrier gas passes to the probe needs to be changed Replace the o-ring (see section 0)
Ro not reproducible	Filament not fixed. Check the filament and adjust. Chamber temp. (Tc) not constant.
Roact.= 0 mohm or smaller than normal	There is contact between the two Pt-flags. The Longer Pt-flag is in contact with the gas tube to the GC-injector.
Roact.= 350 mohm	There is no pyrolysis probe. The Pt-filament is broken. There is a home probe in the pyrolysis chamber.
TD constant at 550° C.	Pyrolysis temperature below 550° C (The photodiode does not measure temperatures below 550° C) The optic cable is broken.
TD not reproducible	The optic cable not fixed. Make sure the cable is connected securely to both the process unit and control unit
TR not shown	There is no calibration temperature between 975-1000° C. Calibrate the probe, see section 4.1.
TR not reproducible	Ro not constant.

6.3 Service

In case of failure, contact your distributor service center:

Tel:

Fax:

e-mail:

Please use the box in which the Pyrola 2000 unit was delivered if it will be necessary to ship the unit for service or repair.

6.4 Warranty

There is one year of warranty from date of purchase unless stated otherwise in your contract on the Pyrola 2000 equipment.

NOTE!
The warranty does not cover disposable goods or damage caused by the user, expressly the platinum filament is not covered by any warranty.

NOTE!
The control unit and the carousel do not contain any parts that can be serviced by the user. If service is needed, please contact your distributor.

Opening the control unit and the carousel will void the warranty.

6.5 Technical description

Electrical specification:
Input voltage: 115 / 230 VAC
Frequency: 60 / 50 Hz
Power: 200 W

Fuses:
On the rear panel: 2 fuses, 6x32 mm,
115 VAC: 3.15 A slow
230 VAC: 1.6 A slow
Inside on the power board: 5x20 mm, 1 A slow

Mechanical specification control unit
Width: 260 mm (10.2")
Height: 205 mm (8.1")
Depth: 315 mm (12.4")
Weight: 6.7 kg (14.8 lb.)

Appendix

I

I Calibration – an example

The calibration of a filament should be carried out:

- if you have a new filament
- if you want to re-calibrate because:
  - the filament resistance R0 has changed too much
  - the chamber temperature Tc has been changed
  - the carrier gas and/or flow has changed

The Pyrola 2000 system uses two different types of temperature measurement: light and resistance. The light emitted from the middle of the filament is measured with a photodiode and converted to a temperature value. This measurement is correct without a calibration but it is only accurate at high temperatures>≈ 600° C.

Below 550° C the temperature is measured only by means of the resistance of the filament which changes with temperature. It is necessary to calibrate the filament because the measurement of TR (temperature measured by resistance) would not be correct otherwise. Please note that, the type and flow of the carrier gas as well as the chamber temperature also influence the measurement of the temperature TR to a greater or lesser extent. Furthermore, a calibration makes it easier to find the conditions (i.e. currents I_1 and I_2) for an ideal pyrolysis of your sample.

In this appendix an example of the calibration procedure is given.

1. Press 'Probes' in the toolbar to open the Probes window. Press 'New' to open a new Calibration of Filament window, see Figure I-1.

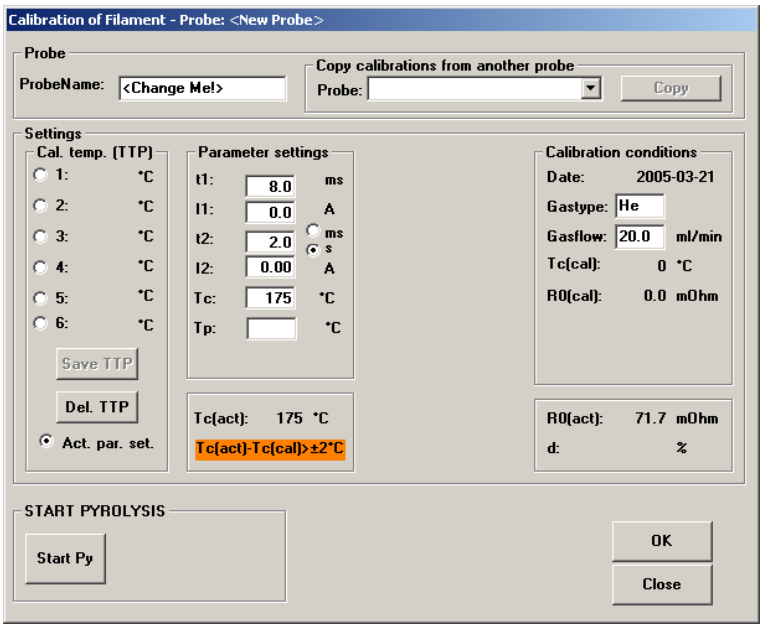


Figure I-1. 'Calibration of filament' window.

2. Choose the chamber temperature Tc in 'Parameter settings', in this case Tc=175°C, and press 'Enter' to send the temperature to the control unit.

> **NOTE!**
> The red field below the chamber temperature Tc(act) indicates that the Tc(act) is different from the temperature when the filament was calibrated, Tc(cal).
> **Here Tc(cal) is 0 °C since we have not yet calibrated the filament.**

3. Rename the probe file (the default name is <Change Me!>). Note that each probe can have several probe files, for example with different chamber temperatures. It is suggested that the name reflects both the number of the probe and the calibration conditions, for example 1-175 and 1-225 if probe 1 has been calibrated at Tc=175°C and Tc= 225°C, respectively.

4. Enter the type of carrier gas you are using and the gas flow. In this example He is used as carrier gas with a total flow of 20 ml/min. Confirm with 'OK'.

   The stored values do not influence the calibration but serve as a reminder that you need to re-calibrate if you change them, especially if the carrier gas is changed from He to N_2 or air.

5. Under parameter settings, set t1 = 8 ms (t1=TRT, temperature rise time) and t2 = 2 s (pyrolysis time). These settings are often applicable, but other values may be used depending on the application.

6. The aim of the first run is to find an ideal TTP (time temperature profile) with a temperature between 975 and 1000°C. 'Ideal' in this context means having the correct temperature and the same temperature in the end of the pyrolysis as in

the beginning. The amplitudes of the currents I1 and I2 need to be set or adjusted. Choose I1=41.0 A and I2=10 A as start values if you are using He as a carrier gas and a filament with a low resistance, R0 ≈ 70 mΩ.

NOTE!
Always check that you have carrier gas in the pyrolysis chamber before you start the pyrolysis. The filament may otherwise be damaged.

7. Click 'Start Py' to start a pyrolysis with the selected parameter settings.
   A diagram is displayed, see Figure I-2, which shows the temperature time profile (TTP) measured by the photo diode; the diode temperature TD.
   To the right of the diagram the final temperature TD = 926 °C is indicated; TR is zero because the filament has not been calibrated yet. This TTP is not 'ideal'.
   There is an under-shoot of ≈130°C, and the end temperature, TD, is ≈70 °C too low compared to the intended value between 975 and 1000°C. Both I1 and I2 need to be adjusted and increased.

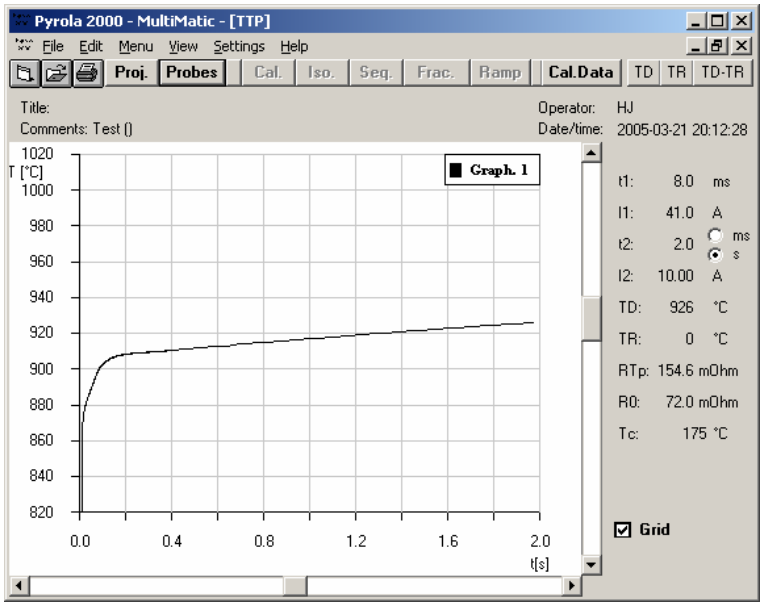


Figure I-2. TTP after the first pyrolysis.

8. Increase I1 to 44 A and I2 to 10.50 A and click 'Start Py' again, see Figure I-3.
   A change of I1 and I2 with 1.5 and 0.5 A, respectively, will change the temperature ≈ 80-100°C. Now the TTP reaches 1010°C initially, but the final temperature is 1001°C. Thus the TTP is still not 'ideal' since it does not reach the same temperature at the end of the pyrolysis as in the beginning, and the temperature is too high. Further adjustments are needed.

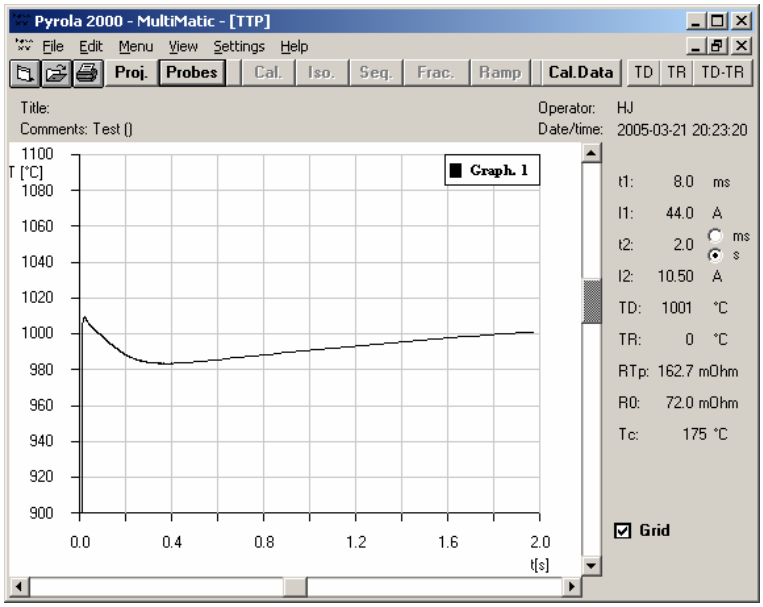


Figure I-3. TTP after the second pyrolysis.

9. Decreasing both I1 and I2 gives an acceptable TTP, see Figure I-4.

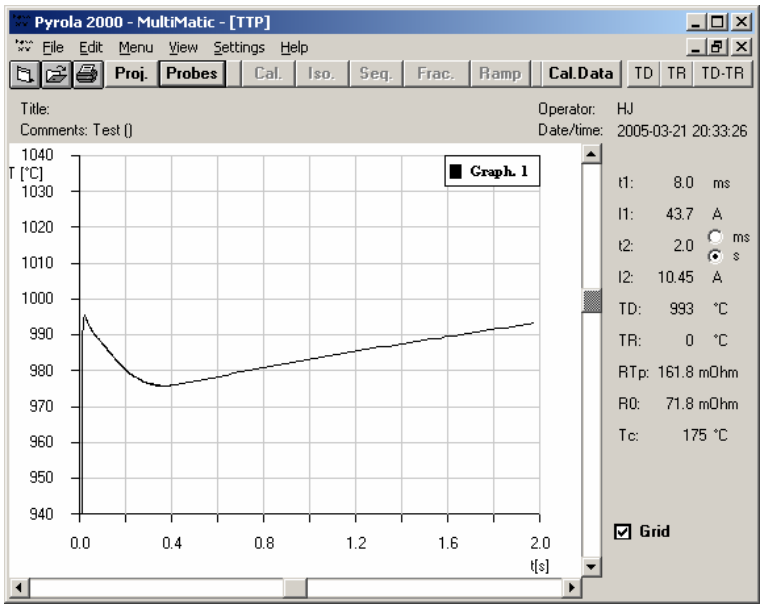


Figure I-4. The TTP after the third pyrolysis gives an acceptable result. Note the changed scaling on the y-axis.

10. For reliable pyrolysis results the TTP has to be repeatable as well as "ideal". Therefore re-run the pyrolysis with the same parameters and compare the results. The result of the repeated test for this example is shown in Figure I-5. The results are very close compared to those in Figure I-4 and thus the repeatability is good.

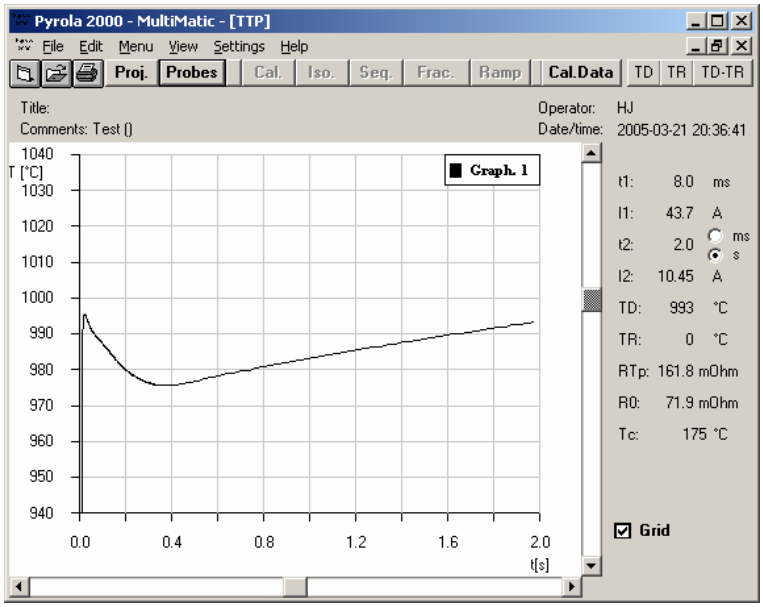


Figure I-5. Repeating the pyrolysis gives the same result, compare with Figure I-4.

NOTE!
Remember that when you have pyrolyzed e.g. at 1000°C the filament takes some time to cool. This cooling can be followed by a decrease in R0.

11. Since the results are acceptable, go back and click 'Save TTP' in the 'Calibration of Filament' window. When the first calibration temperature (975-1000°C) has been saved both TD and TR are displayed in the TTP diagrams.

12. A second pyrolysis temperature can be easily found by decreasing I1 with 1.5 A and I2 by 0.5 A. The result is shown in Figure I-6.

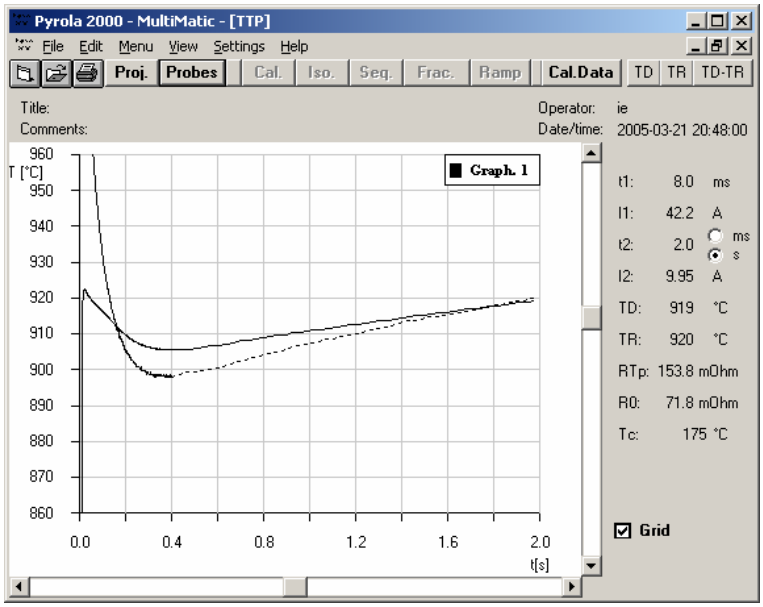


Figure I-6. A second pyrolysis temperature is found by decreasing I1 and I2 with 1.5 and 0.5 A, respectively.

13. . Click 'Save TTP' in the 'Calibration of Filament' window.

14. After ideal TTP's have been found and calibrated at two temperatures, the program calculates initial values of I1 and I2 at other pyrolysis temperatures. Continue the calibration:

    - Set Tp to 700°C and press 'Enter' to confirm. The program will calculate and suggest I1 and I2.

    - Click 'Start Py' and check the beginning of the TTP, then increase or decrease I1 or I2 accordingly to get an 'ideal' TTP.

    - When an ideal TTP is obtained click 'Save TTP'. Every new calibration temperature is displayed under 'Cal. temp. (TTP)', see Figure I-7.

    - If you want to pyrolyze at other temperatures, repeat this step with the appropriate temperatures you want to use.

    - Close the 'Calibration of filament' window by pressing 'Close'.

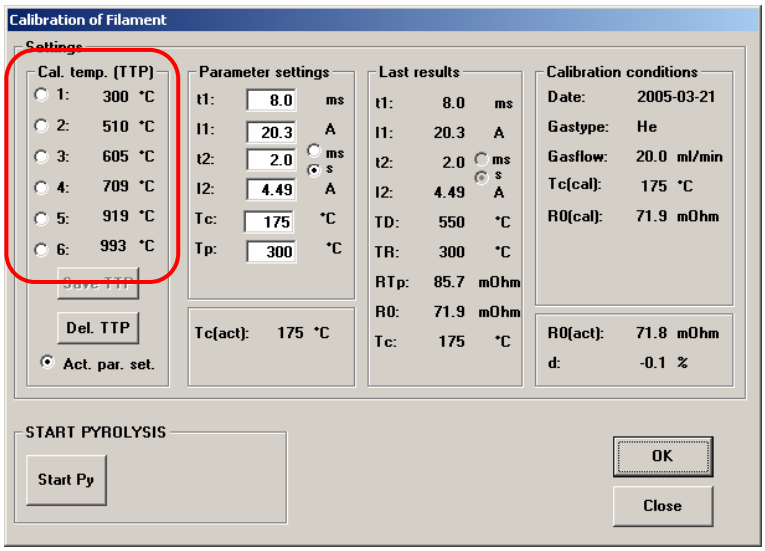


Figure I-7. 'Calibration of filament' window after calibrating at 6 different temperatures.

15. The calibration conditions and all stored TTP's can be found in the calibration data dialog. Go to 'Menu' and 'Calibration data' or click Cal.Data in the toolbar to open the dialog shown in Figure I-8. The calibration data for the current probe file can also be printed or saved as a result file (ttp-file).

NOTE!
When TD is lower than 600° C, TR will be set as the calibration temperature. The temperature values measured by the photo diode are not accurate and reproducible enough at lower temperatures. The lowest TD = 550° C.

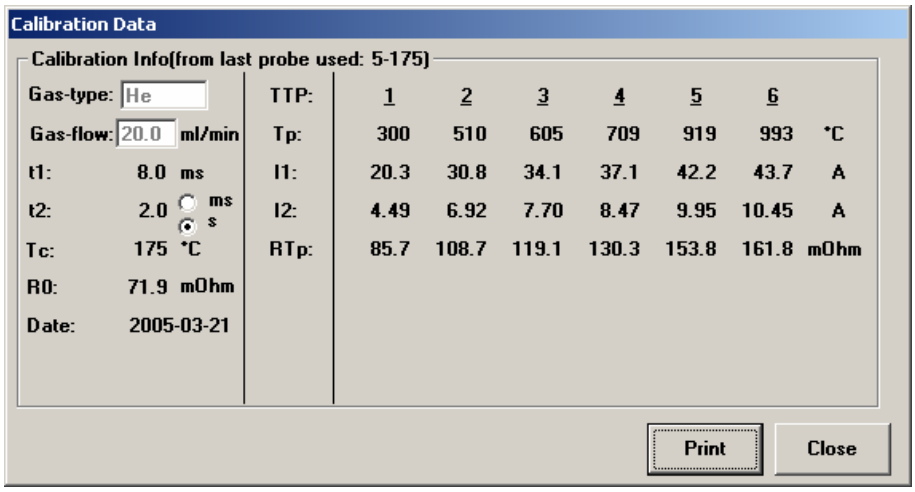


Figure I-8. 'Calibration data' window.

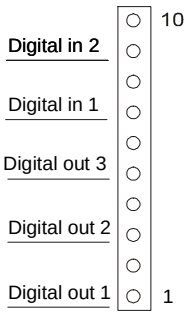
Appendix

II

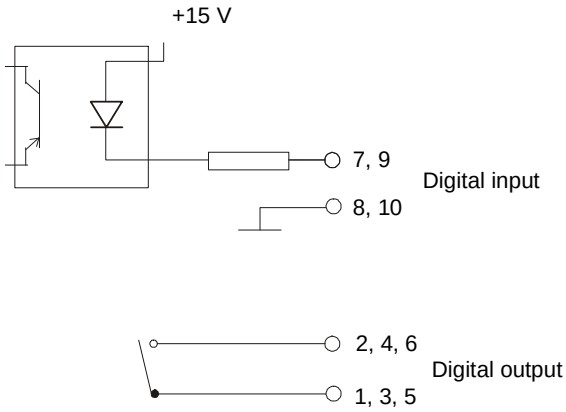
II Connection to a gas chromatograph

When a pyrolysis is started a signal is sent from the control unit to the gas chromatograph which starts the gas chromatograph cycle. After the GC has finished it's analysis a signal is returned to the control unit. If the Pyrola 2000 MultiMatic has been set to run another pyrolysis, it will start and send a new signal to the GC until the whole pyrolysis program has been executed.

Pyrola 2000 Rear panel

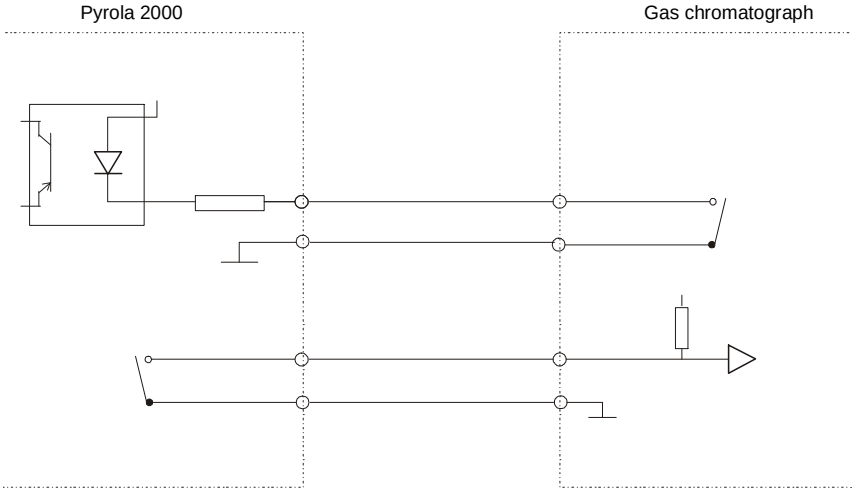


Electrical drawing

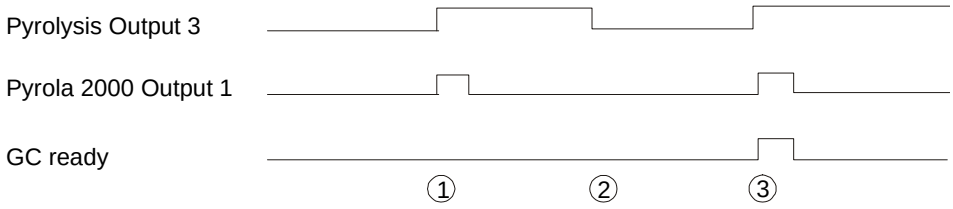


Installation of the cable between Pyrola 2000 and gas chromatograph

Connect a cable between digital out 1 of the Pyrola 2000 and 'GC start in'.
Connect a cable between 'GC Ready output' and digital in 1 of the Pyrola 2000.



Service



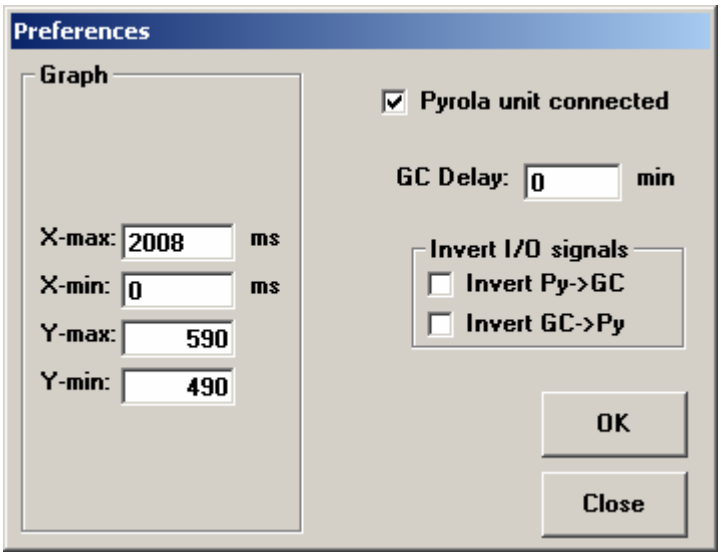
1. A pyrolysis starts and P2000´s output 1 is activated (closed) for 1 second. GC reads this and starts it´s cycle. Output 3 is activated (closed) during the whole pyrolysis cycle.

2. Pyrolysis is ready. P2000 waits for GC ready on digital input 1.

3. GC is ready and signals to P2000 which starts another cycle if relevant.

Software settings:

The GC settings in the Pyrola 2000 program are found under the Settings menu, Preferences, shown in the figure below.



GC Delay
For some GCs it is necessary to choose a delay time, long enough so the GC is ready before the next pyrolysis.

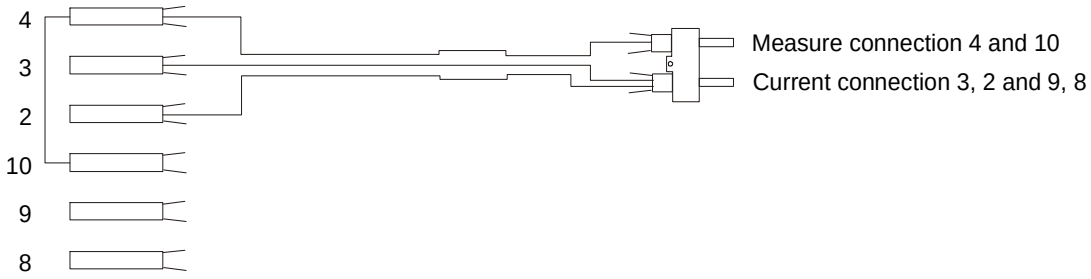
Invert I/O signals
For an Agilent or HP GC, the 'Invert GC->PY' should be checked. For other brands, these boxes should be left unchecked.

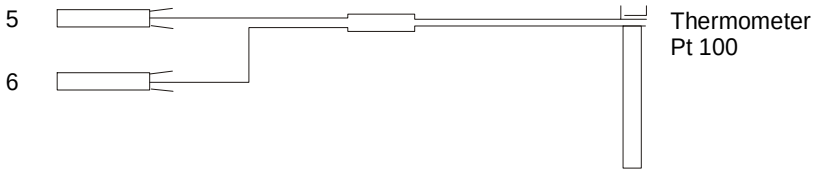
Appendix

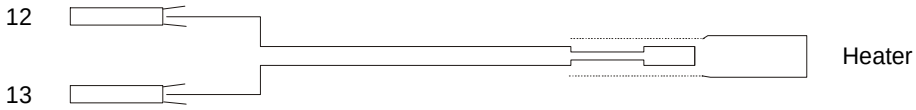
III

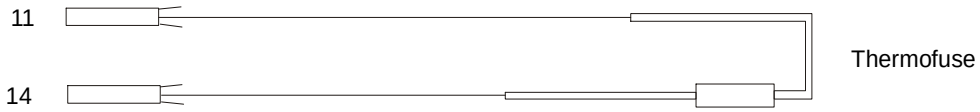
III Pin layout

The position of the pins in the contact of the process unit is indicated below:







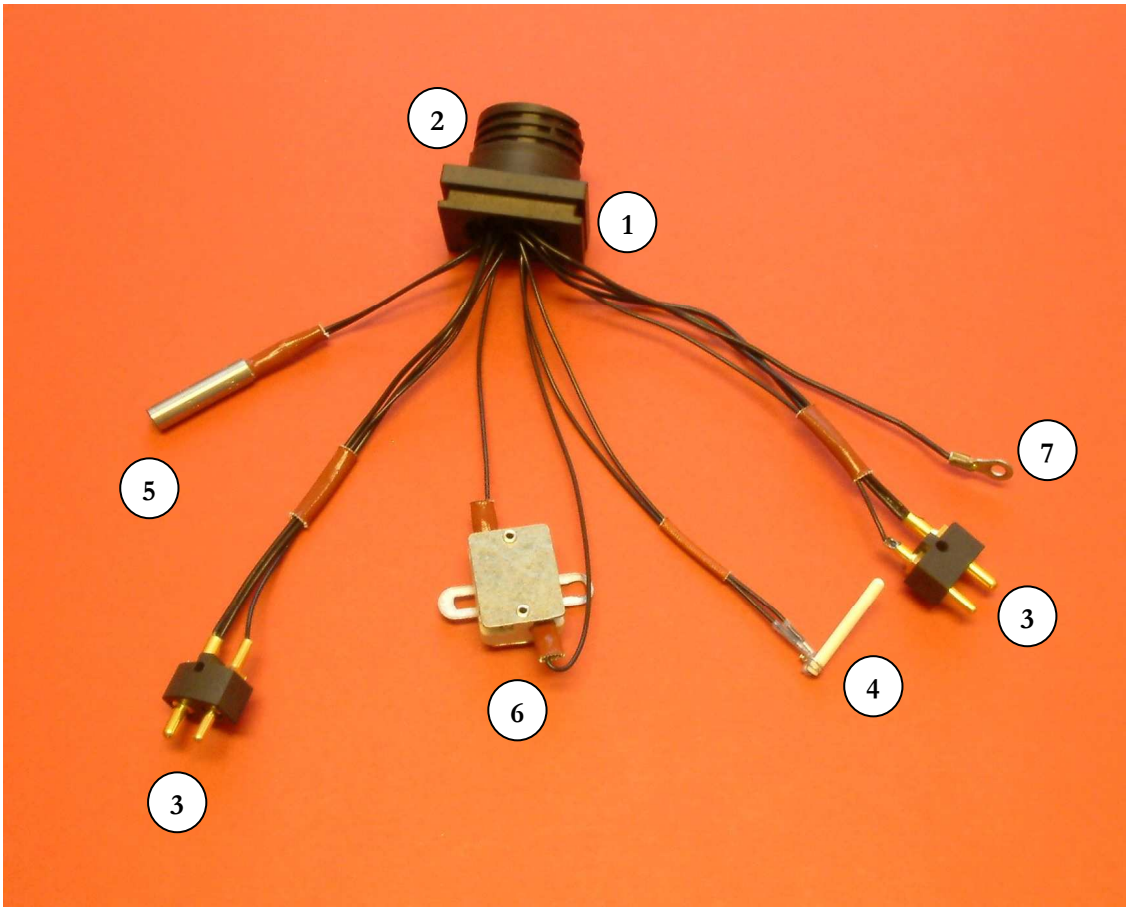


Appendix

IV

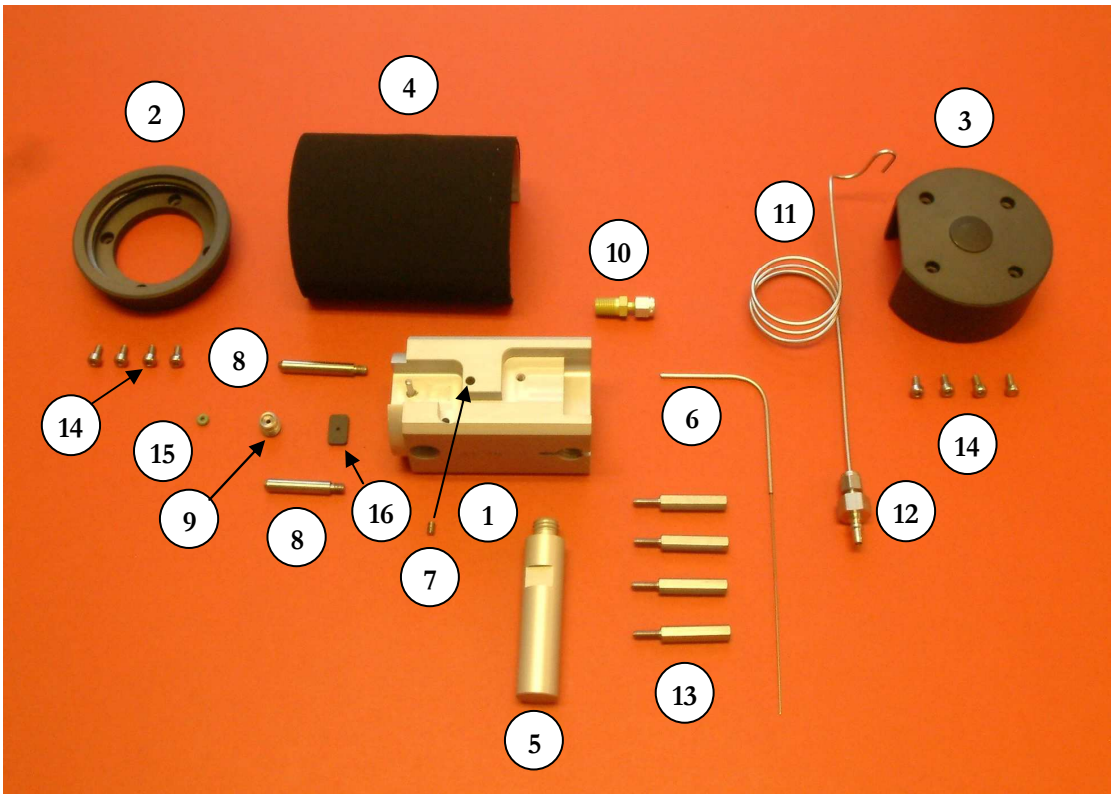
IV Parts and order numbers

In this appendix parts and order numbers are presented.



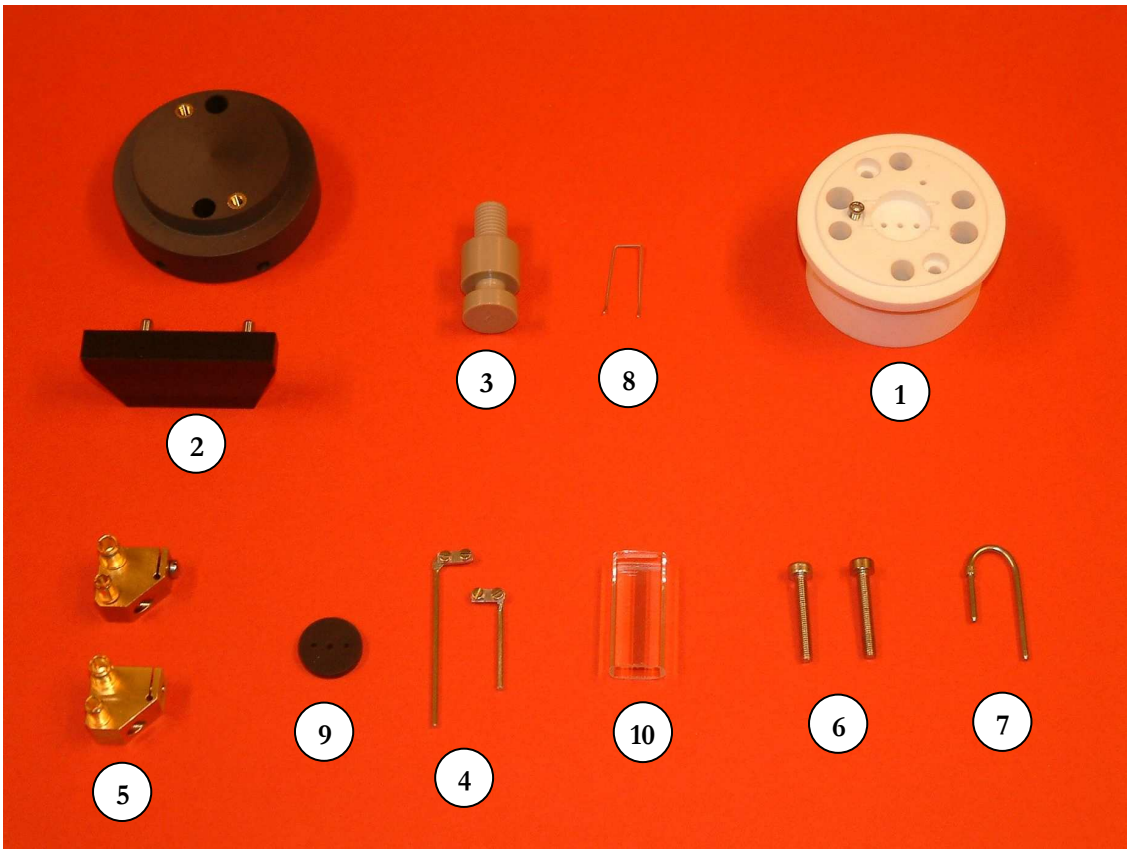
Cabling

Item	Article number	Article
1	2200-02	Connector
2	2200-04	Connector support
3	2200-11	Current and measure cables+holder
4	2200-12	Thermometer, Pt-100
5	2200-14	Heater
6	2200-16	Bimetal thermofuse
7	2200-18	Ground cable



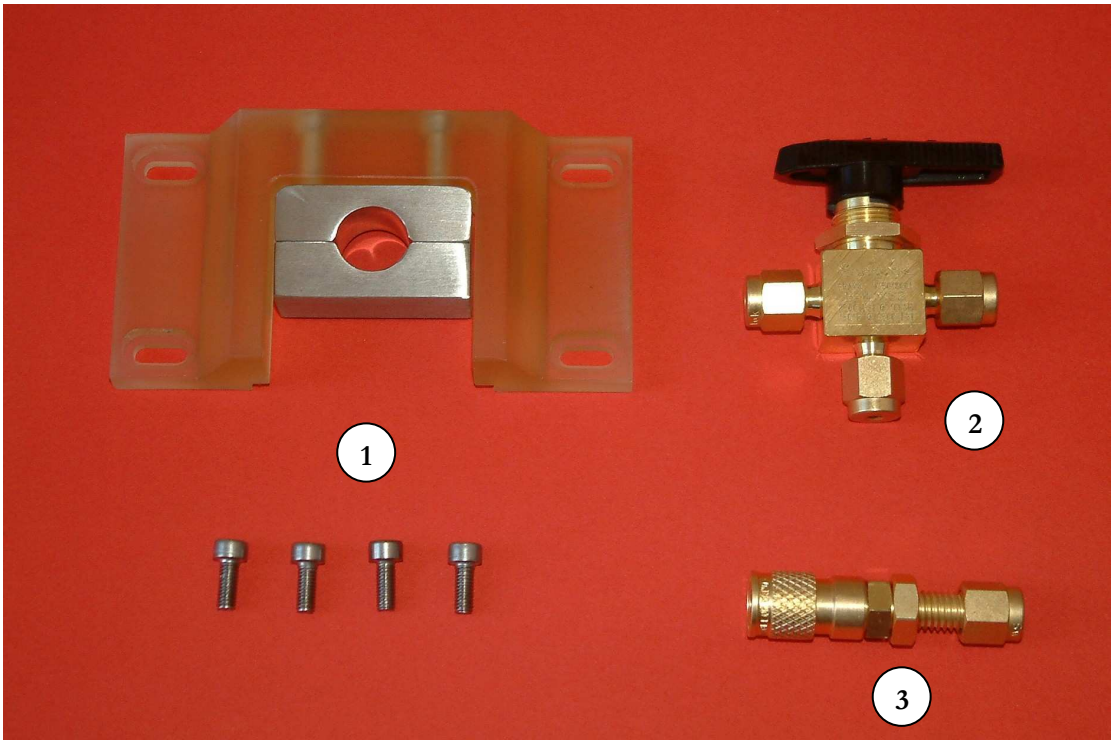
Chamber

Item	Article number	Article
1	2000-02	Block
2	2000-04	Front gable
3	2000-06	Coupling box
4	2000-08	Cover
5	2000-10	Neck, M6
5	2000-11	Neck, M10
6	2000-12	Injection needle for GC
7	2000-14	Screw for fixing injection needle
8	2000-18	Alignment pins, long and short
9	2000-20	Guide for gas tube in probes
10	2000-22	Gas Connector
12	2000-24	Quick fitting, male
10+11+12	2000-23	Gas tube complete
13	2000-26	Distances
14	2000-28	Screws
15	2000-32-05	O-ring
16	2000-34-05	Septum, rectangular



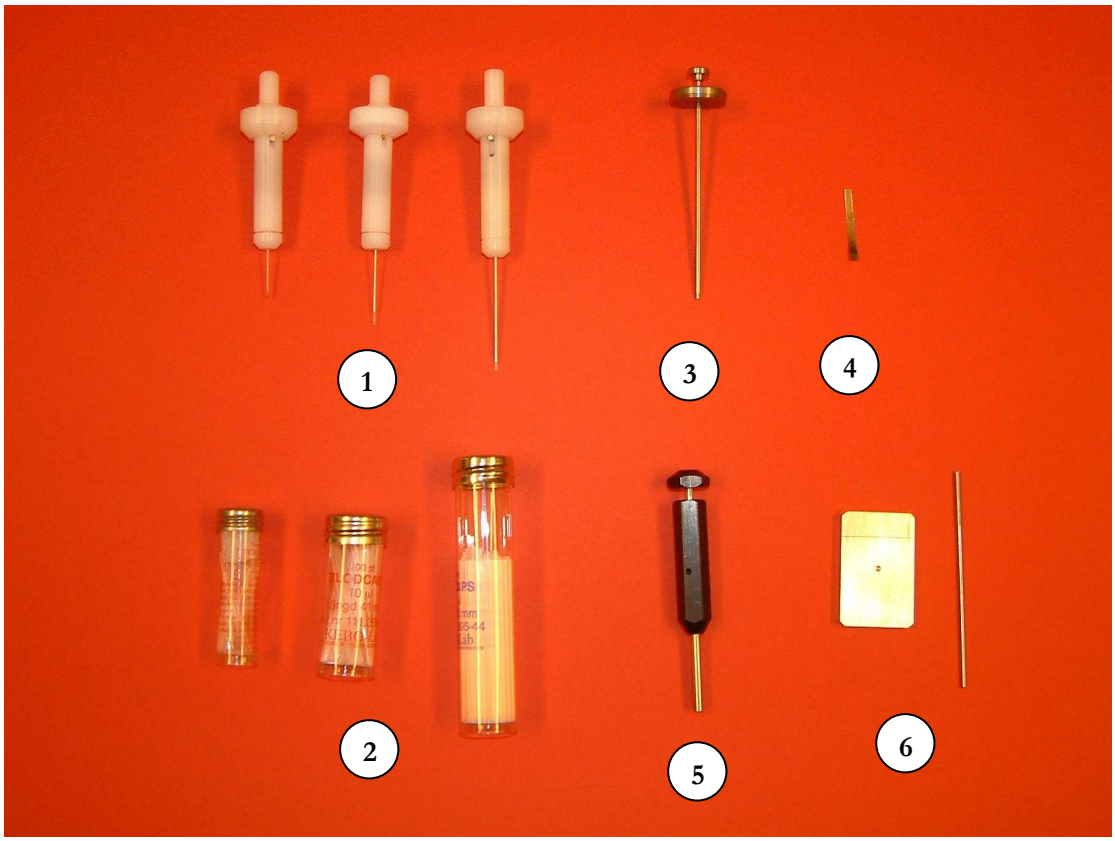
Probe, MultiMatic

Item	Article number	Article
1	8300-04	Adapter
2	8300-06	Back part, including foot
3	8300-05	Probe holder
4	8300-08	Pt-holder, complete
5	8300-12 8300-14 8300-16	Holders Screws Contacts, females
6	8300-18	Screws
7	8300-20	Gas tube
8	8300-28-15	Springs for glass cells, 15 pcs
9	8300-24-05	Septum, 3 holes, 5 pcs
10	8300-29-05	Glass cell 28 mm, with notch, 5 pcd



Parts for installation

Item	Article number	Article
1	1100-02	Base + 4 screws
2	1100-08	Three-way valve
3	1100-10	Quick connector



Sample handlers

Item	Article number	Article
1	1100-14	Sample handler, powder 3 µl, 10 µl, 44.7 µl
2	1100-16	Micropipettes, 100 pcs 3 µl, 10 µl, 44.7 µl
3	1100-18	Sample handler, solids
4	3000-26-10	Pt-filaments, 10 pcs
5	1100-06	contact tool
6	1100-12	filament cavity tool