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Manual

Pyrola® 2000

PYROL AB

Manual Pyrola® 2000

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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.

The Pyrola 2000 incorporates 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.

A further development of the Pyrola 2000 is the Pyrola 2000 MultiMatic, which has all the functionality of the Pyrola 2000, but can handle up to 14 pyrolysis probes automatically.

Since the Pyrola 2000 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 system

The Pyrola 2000 system is shown in Figure 1-1. It consists of:

- process unit (1)
- control unit (2)
- optic cable (3)
- control cable to the process unit (4)
- communication cable
- main power cable
- Computer with Pyrola 2000 software

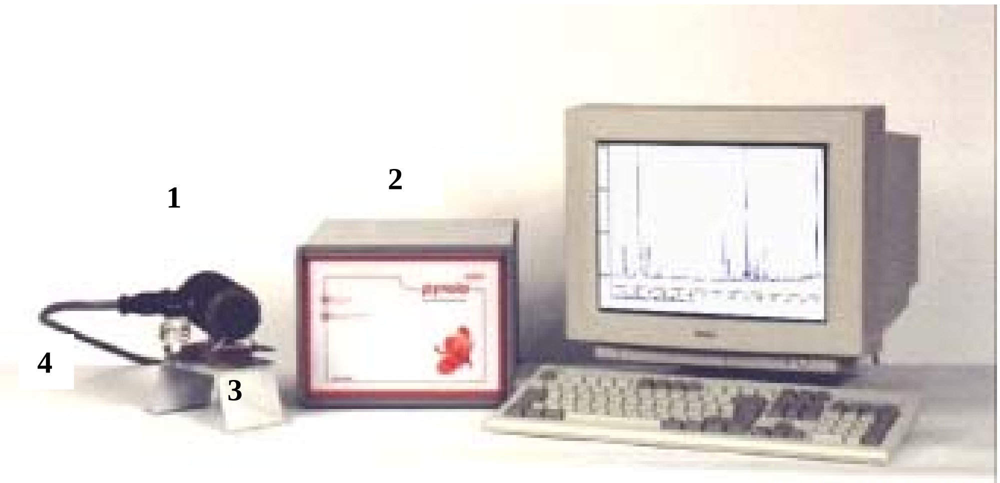


Figure 1-1. The Pyrola® 2000 pyrolyzer.

The process unit consists of the pyrolysis chamber and a probe. The samples that are to be pyrolyzed are placed on a platinum filament in the pyrolysis probe, 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).

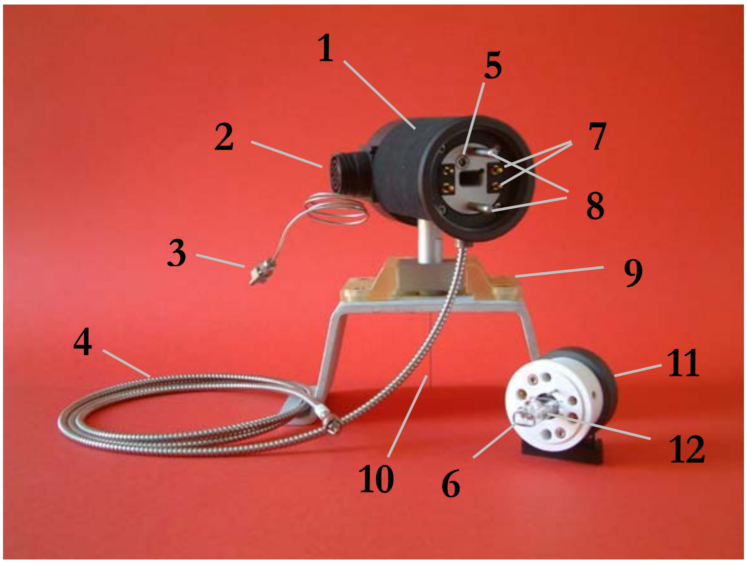
The control unit produces the current pulses and measures the temperature of the chamber (Tc), as well as the signal from the photo diode and the resistance of the Pt-filament. The control unit, in turn, is controlled by a PC with the Pyrola 2000 software.

1.2 The 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.

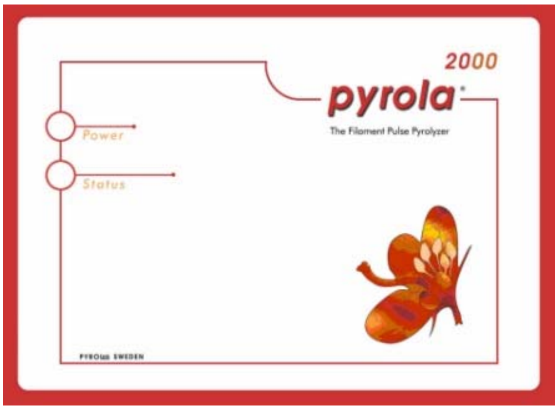


Figure 1-3. Front panel.

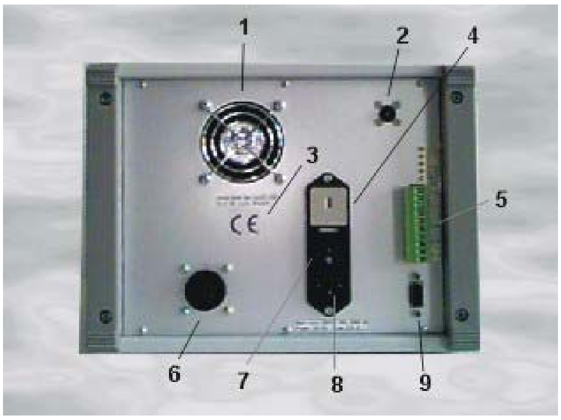


Figure 1-4. Rear panel.

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 •
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 • the platinum filament is broken • the probe is disconnected

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 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 is identical in appearance to the software for the Pyrola 2000 MultiMatic, but some options are not available for the Pyrola 2000 due to the automation of the MultiMatic.

title bar
menu bar
toolbar

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.

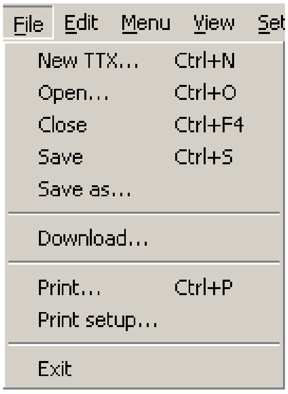
Button	Function
	Creates a new ttx-file (library file) and opens the 'Open file' dialog for the first ttp-file (result file)
	Opens a file
Cal.	Opens the parameter dialog for 'Calibration of filament'
Iso.	Opens the parameter dialog for 'Isothermal pyrolysis'
Seq.	Opens the parameter dialog for 'Sequential pyrolysis'
Frac.	Opens the parameter dialog for 'Fractionated pyrolysis'

Ramp	Opens the parameter dialog for 'Ramp I/t'
Cal.Data	Displays the calibration data

The functions for the buttons that are not highlighted, Proj. Probes, are only available for the automatic Pyrola 2000 MultiMatic.

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 unit
Print TTP
Change printer settings
Exit the Pyrola 2000 software

Figure 1-6. The file menu.

1.4.1.1 File types

The Pyrola 2000 handles three file types, .ini, .ttp and .ttx.

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.

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.

If you save the parameter settings to your default file they will be available automatically at the next start-up, except for the graph. Therefore, always make sure that you have saved your results in a ttp-file before exiting the software.

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

Under 'Menu' you can open the parameter setting dialog for the different types of pyrolysis -isothermal, sequential, fractionated or ramp. The parameter dialogs are similar for all four types and a detailed description of how to select your parameters can be found in section 4.2 of this manual.

The commands on this menu are also accessible via the toolbar. The toolbar is described in the beginning of this chapter.

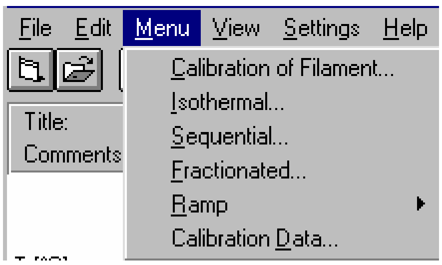


Figure 1-8. The 'menu' menu.

The 'Menu' menu also gives you accesses to the parameter setup for the calibration of a filament, which is described in the Appendix I, and to the calibration data.

1.4.4 View menu

In the 'View' menu you can select which TTP you want to be displayed in the result diagram.

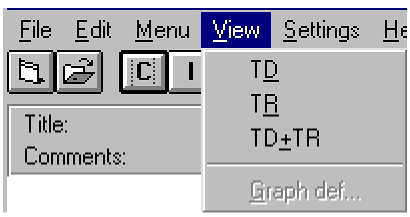


Figure 1-9. The view menu.

The available options are:

TD shows the TTP measured by the photo diode

TR shows the TTP measured by the resistance of the filament

TD+TR show the values from the photo diode and the resistance measurement

The command 'Graph def…' is available only for a library file (ttx-file) or a sequential or a fractionated pyrolysis. It can be used to display the pyrolysis conditions for a selected measurement. Double clicking on the diagram area of a TTP can also access this command. For a detailed description of this command please refer to section 4.4.3.

1.4.5 Settings menu

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

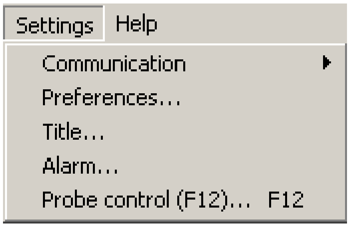


Figure 1-10. The settings menu.

<u>Communication</u>
Select the communication port to which the Pyrola 2000 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'.

<u>Preferences</u>
The preferences command opens the preferences dialog, see Figure 1-11. 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.

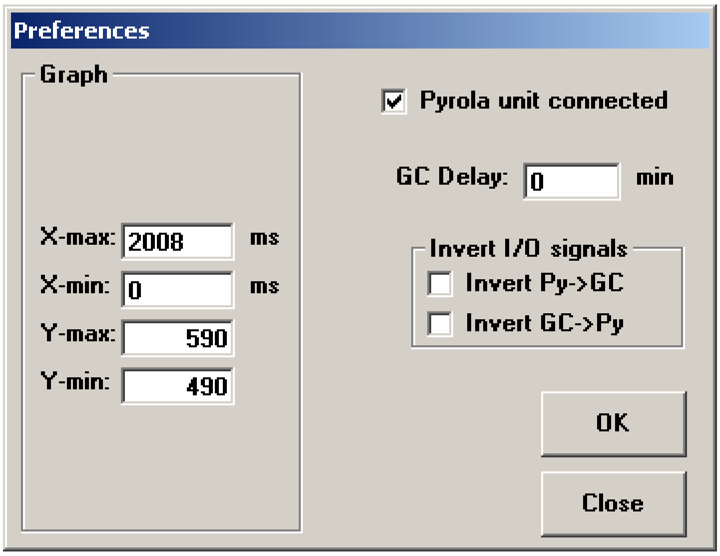


Figure 1-11. The preferences dialog.

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.

<u>Title</u>
In the 'Title' dialog you can enter information about your sample, such as sample title, comments and name of operator..

<u>Alarm</u>
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.

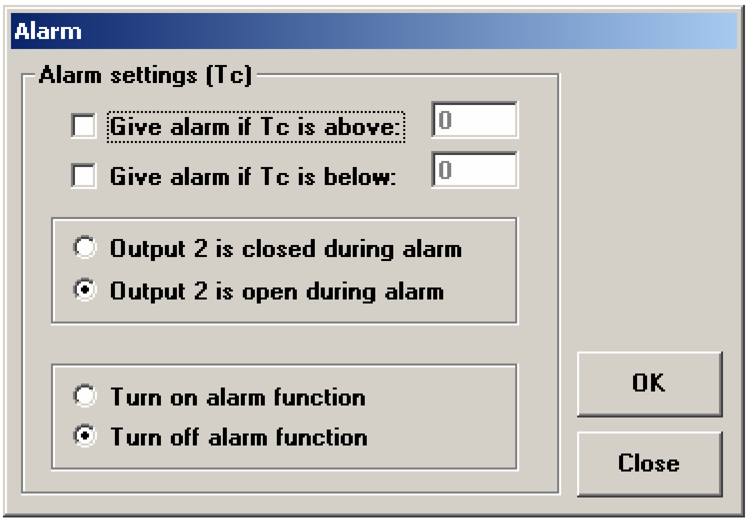


Figure 1-12. The alarm dialog.

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.

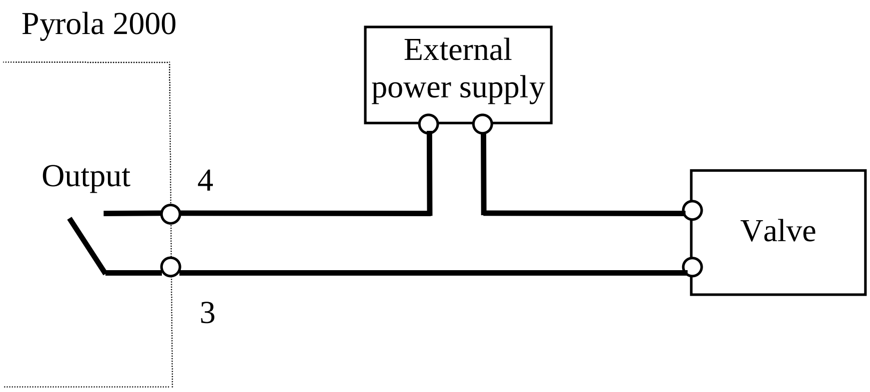


Figure 1-13. Principle of using output 2 for closing the He gas.

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

1.4.6 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 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.

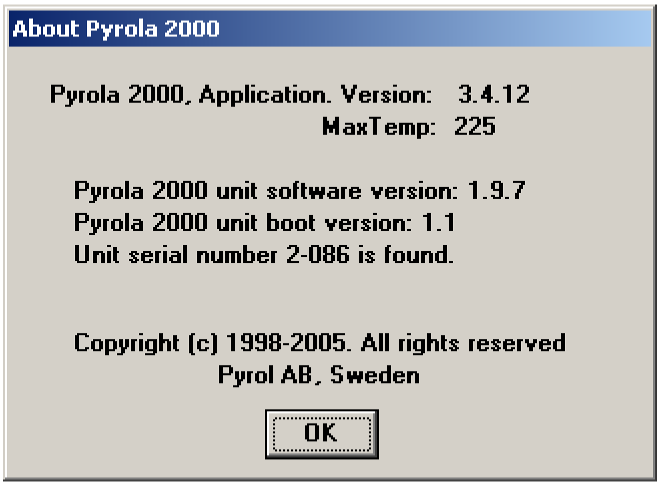


Figure 1-15. Example of output from 'About' command in the help menu.

Chapter 2

2 Installation

This chapter describes the installation of a Pyrola 2000 unit on a GC.

2.1 Connecting the process unit to the GC

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

The process unit of the Pyrola 2000 system is connected to the injector of your GC via an injection needle (10). It is equipped with a three-way valve that diverts the carrier gas either directly to the GC or through a quick connector (3) to the process unit. The three-way valve is shown in Figure 2-1, and its operation is shown in Figure 2-2.

Figure 2-1. Three-way valve.

Prior to installation please check the following:

- The process unit is equipped with a base (9) 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 (10) 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.

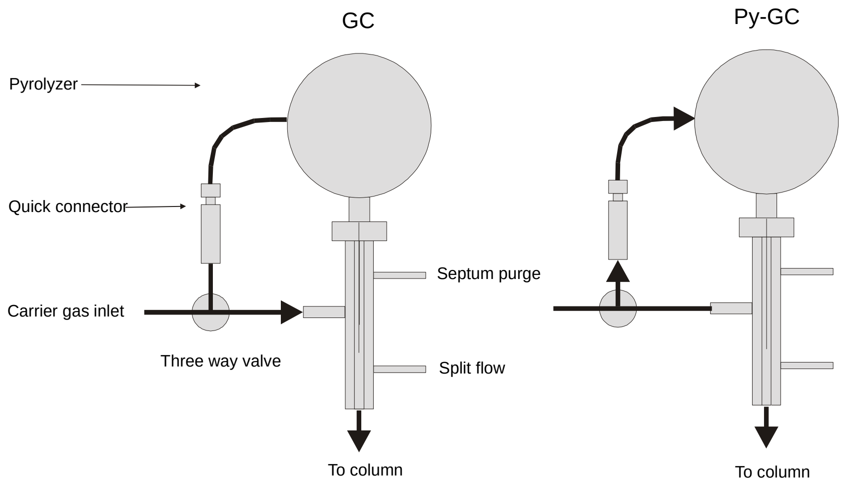


Figure 2-2. Three way valve in position GC and Py-GC, respectively.

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 (9) on the GC. Carefully align it with the GC injector.

4. Insert the injection needle (10) of the process unit through the septum of the GC injector and connect the quick connector (3).

5. Connect the fiber optic cable (4) to the process unit.

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

6. Connect the control cable, (4) in Figure 1-1, to the contact on the probe, (2) in Figure 1-2.

7. Check that the pyrolysis probe (11) is hand-tight. If the probe is tightened too much it may be damaged when the process unit is heated.

8. Wait 5 minutes to allow the probe to attain chamber temperature.

9. Check the split flow or head pressure of the system.

10. 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 injection needle by inserting the cleaning needle. Re-mount the chamber and check the gas flow again.

2.2 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.

2.2.1 Status indicators on the control unit

<u>Front panel</u>

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
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 • the platinum filament is broken • the probe is disconnected

<u>Rear panel</u>

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.

2.2.2 Automation

Sequential and fractionated pyrolysis can be automated if a suitable cable is installed between the GC and the Pyrola 2000 control unit. A detailed description of the cable can be found in Appendix II. The connection on the GC is dependent on the brand and model of your GC and is outside the scope of this manual. Please contact the manufacturer of your GC.

2.3 Software installation

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

2.4 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.

2.5 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 control unit.

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

2. Double click the Pyrola 2000 icon to start the software. The software will check for a Pyrola 2000 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.

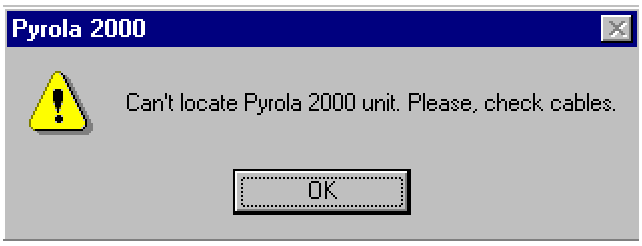


Figure 3-1

Figure 3-2

Figure 3-3

Chapter

4

4 Operation

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

4.1 Calibration

This section describes the calibration of the pyrolysis probe. Before any pyrolysis can be performed the probe need to be calibrated. 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.

4.1.1 Background

The Pyrola 2000 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 at the desired temperature.

The filament has to be calibrated at least at two temperatures, by finding the parameter settings for an 'ideal' TTP (time temperature profile) 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 to have a calibration temperature close to each pyrolysis temperature that will be used.

4.1.2 Calibration procedure

A step-by-step instruction on how 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. Press the 

button in the main application toolbar, Figure 4-1, to open the calibration of filament window. Alternatively, select 'Calibration of filament' on the 'Menu' menu. The calibration of filament window is shown in Figure 4-2.

Figure 4-1. Main application toolbar.

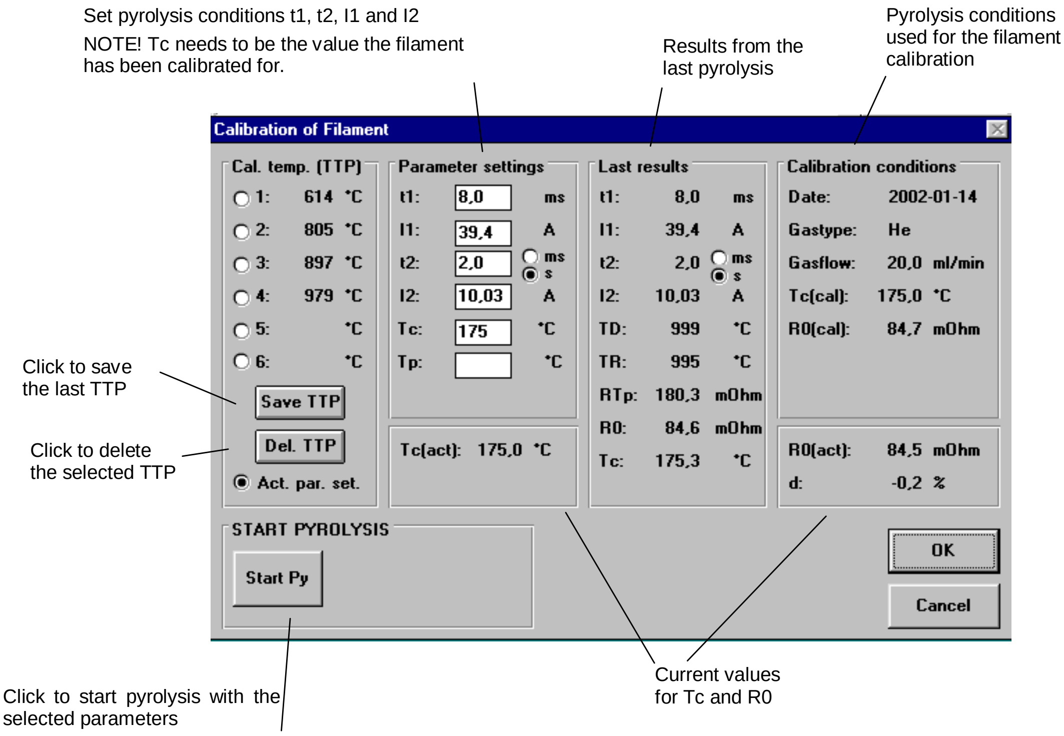


Figure 4-2. The calibration of filament window with explanations of the fields..

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. The most common chamber temperature is 175° C, but it may be set between 50° and 225° C.

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

4. 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.

5. 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.

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

7. The TTP diagram is shown, see Figure 4-3. 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. Data of the current pyrolysis is shown to the right of the TTP diagram. The same information is also shown in the Calibration of filament window under 'Last results'.

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.

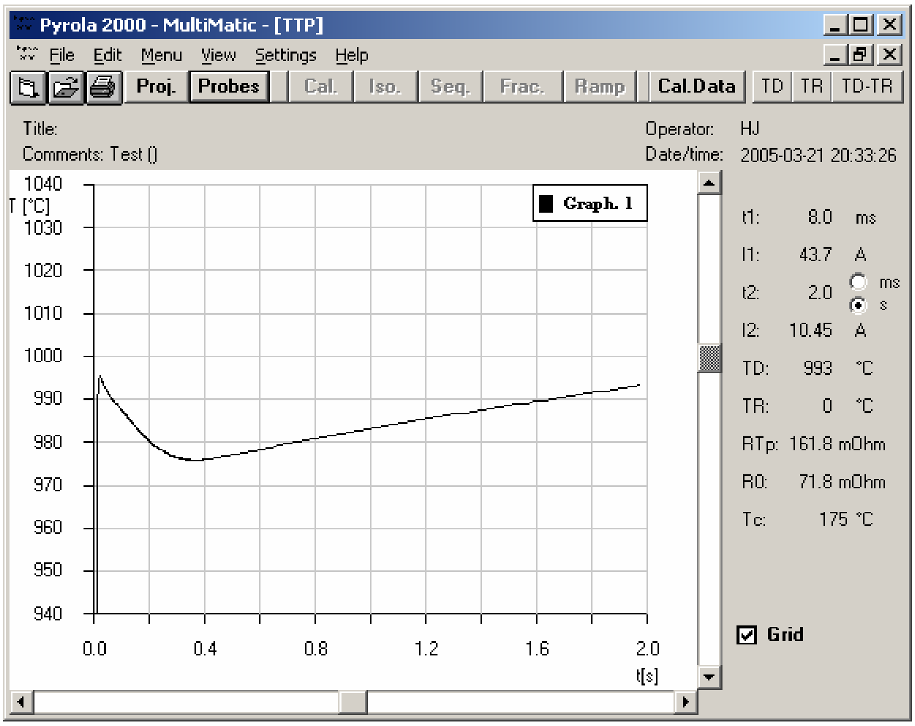


Figure 4-3. TTP diagram.

8. Save the calibrated temperature time profile in Cal. Temp. TTP by pressing the button 'Save TTP'. The information under 'Last results' is stored in the .ini-file you are currently using. Note that the graph itself is not saved, but may be saved as a ttp-file.

9. 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.

10. 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.

11. If needed, it is possible to remove a calibration temperature by selecting it and pressing 'Del. TTP'.

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

The resistance R0(cal) is measured at chamber temperature in 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 Calibration data

The current calibration data can be shown by pressing the Cal.Data button in the main application toolbar, Figure 4-1. Alternatively, select Calibration data in the 'Menu' menu. It displays data for all calibration points, as shown in Figure 4-4.

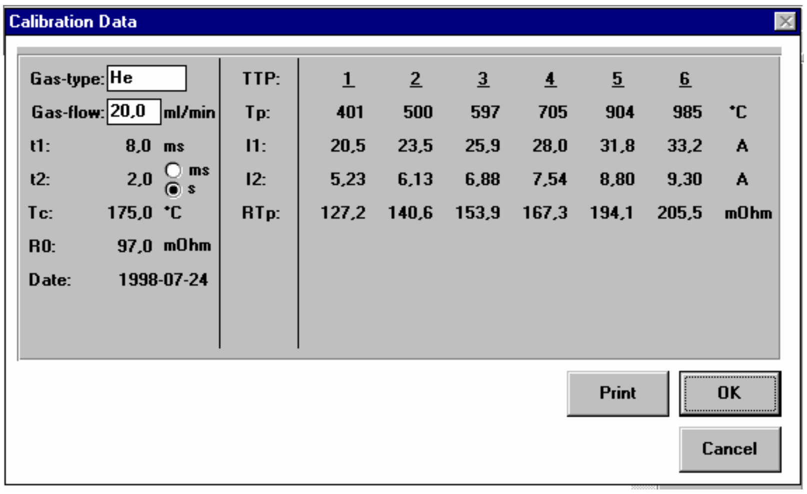


Figure 4-4. The calibration data window.

4.2 Pyrolysis menus

This chapter describes the different pyrolysis methods available, and the parameter settings for each method. The parameter dialog for all methods are similar to the one shown in Figure 4-2, therefore only the specific settings will be explained for each method. Each method is reached by pressing the appropriate button in the main application toolbar, or by choosing from the 'Menu' menu, see Figure 1-8. How to run a pyrolysis with the chosen parameters are described in section 4.3 below.

Please note that the probe must be calibrated prior to any pyrolysis, as described in section 4.1. However, once a probe is calibrated it is not necessary to make any recalibration unless the filament is replaced, or the chamber temperature or carrier gas (type or flow) is changed. If the actual resistance of the filament R0(act) has changed substantially from the R0(cal) used in the calibration, it is also necessary to make a new calibration.

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 calibration should be supplemented with the new pyrolysis temperature.

4.2.1 Isothermal pyrolysis

In isothermal pyrolysis, see Figure 4-5, the pyrolysis temperature Tp can be set to any value between the chamber temperature, Tc, and 1400° C. The parameters I1 and I2 are then calculated automatically. Additionally, it is possible to change the pyrolysis time t2, but note that when Tp is changed it will be set back to the value used in the calibration. The parameters t1 and Tc must be the same values as in the current probe file.

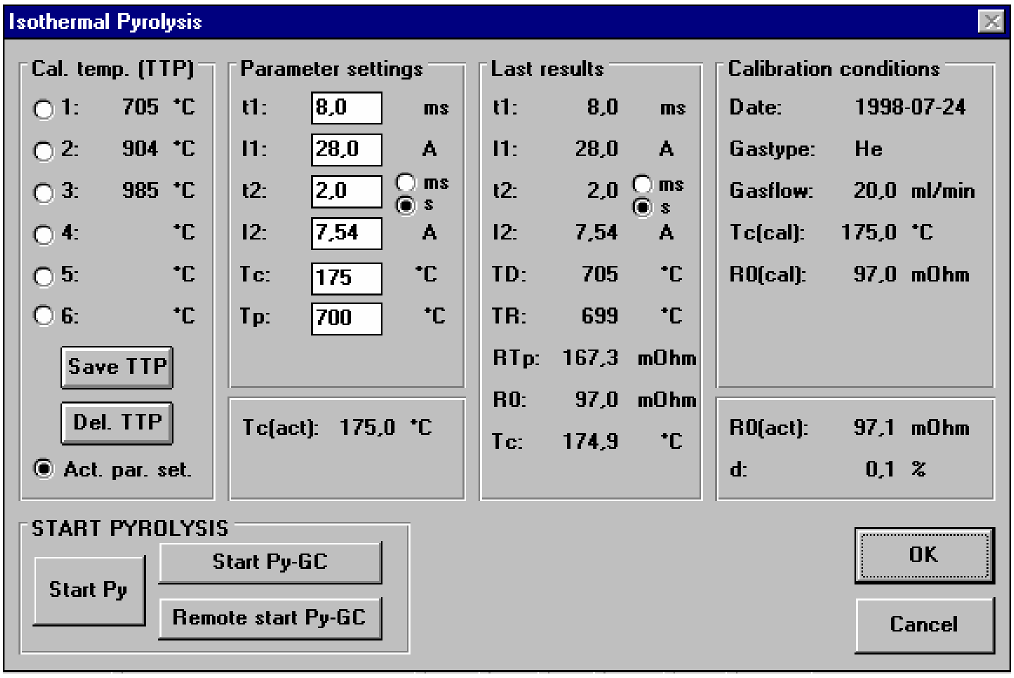


Figure 4-5. Isothermal pyrolysis.

The table below shows the available range for the parameter settings in an isothermal pyrolysis.

Parameter	Range	Unit
t1:	0-100.0	ms
I1:	0-50.0	A
t2:alt.1:	0-1000	ms
t2alt.2:	1.0-600.0	s
I2:	0-15.0	A
Tc:	50-225 (200)	°C
Tp:	Tc-1500	°C

a warning is displayed if Tp is greater than 1400°C, the user must then ensure that the filament can survive such high temperature, melting point for Pt is 1769°C

4.2.2 Sequential pyrolysis

With sequential pyrolysis, see Figure 4-6, you can automatically repeat the same TTP a number of times (up to 9) to be able to study the thermal degradation of the sample via the formation rates of the pyrolysis products.

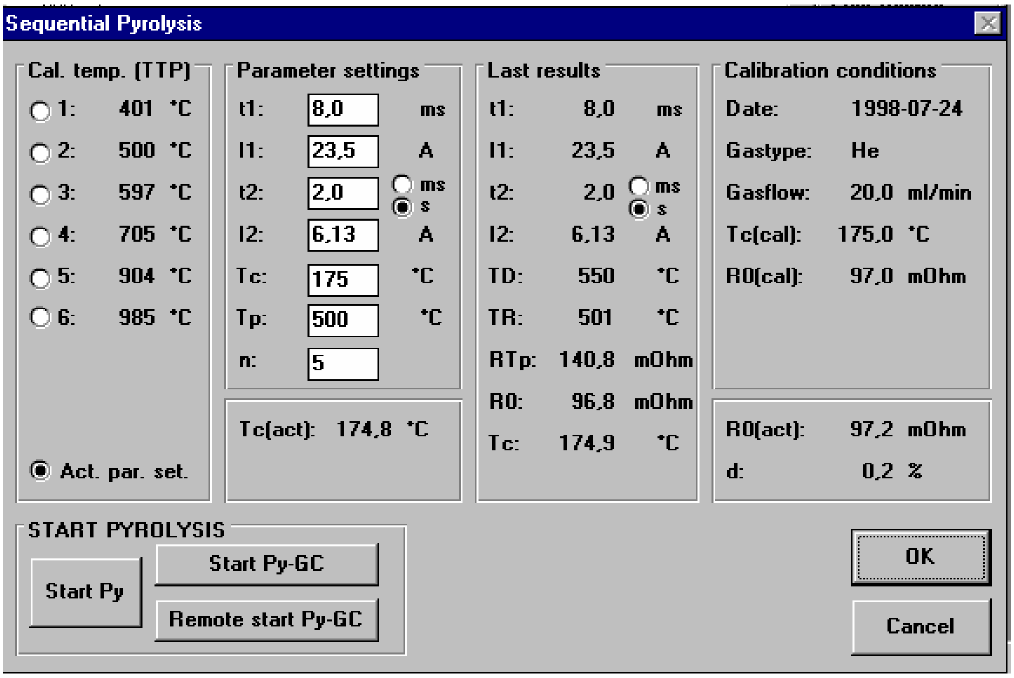


Figure 4-6. Sequential pyrolysis.

In the parameter settings you set the pyrolysis temperature Tp and the number of runs n you want the program to carry out. The software will automatically calculate the values for t1, I1 and t2, I2. The available ranges of the parameters are shown below.

Parameter	Range	Unit
t1:	0-100.0	ms
I1:	0-50.0	A
t2,alt.1:	0-1000	ms
t2,alt.2:	1.0-600.0	s
I2:	0-15.0	A
Tc:	50-225 (200)	°C
Tp:	Tc-1500	°C
n	1-9	

a warning is displayed if Tp is greater than 1400°C, the user must then ensure that the filament can survive such high temperature, melting point for Pt is 1769°C

In order to use the sequential pyrolysis the gas chromatograph must be connected to the Pyrola 2000 unit as is described in Appendix II. When you click **Start Py-GC** a pyrolysis and the gas chromatograph are started. **Remote start Py-GC** starts the pyrolyzer when the GC is ready, see Appendix II. When the first pyrolysis has been finished and the GC signals ready again the pyrolysis will be repeated with identical values until the selected number of runs has been reached.

The resulting TTPs are shown in a diagram, see Figure 4-7. Furthermore, a list with information of each pyrolysis can be displayed by double-clicking the diagram area of the visible curve, see section 4.4.3.

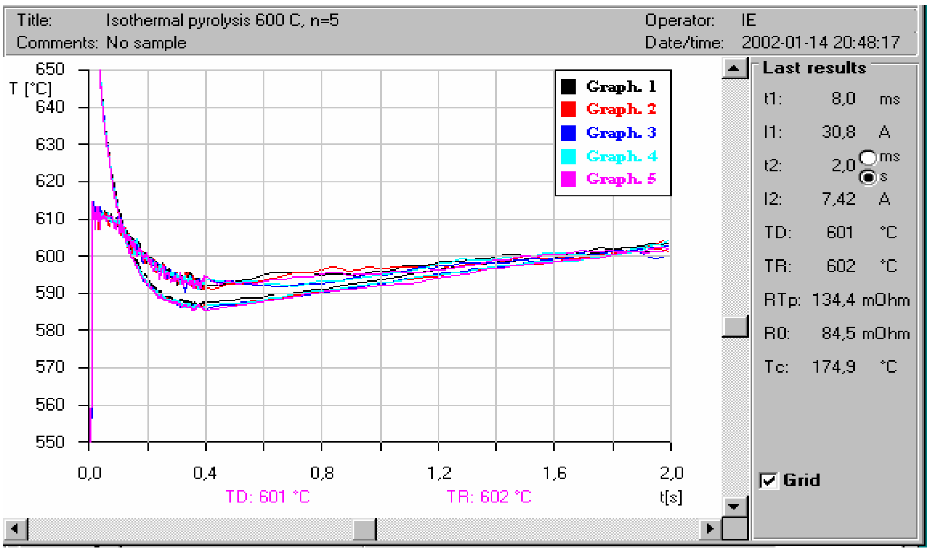


Figure 4-7. Five TTPs (TD and TR) from a sequential pyrolysis.

4.2.3 Fractionated pyrolysis

Complex materials can contain many substances with different degradation rates. If the same sample is pyrolyzed at several different temperatures a qualitative analysis becomes easier. ‘Fractionated pyrolysis’ allows you to analyze one sample at up to six temperature values.

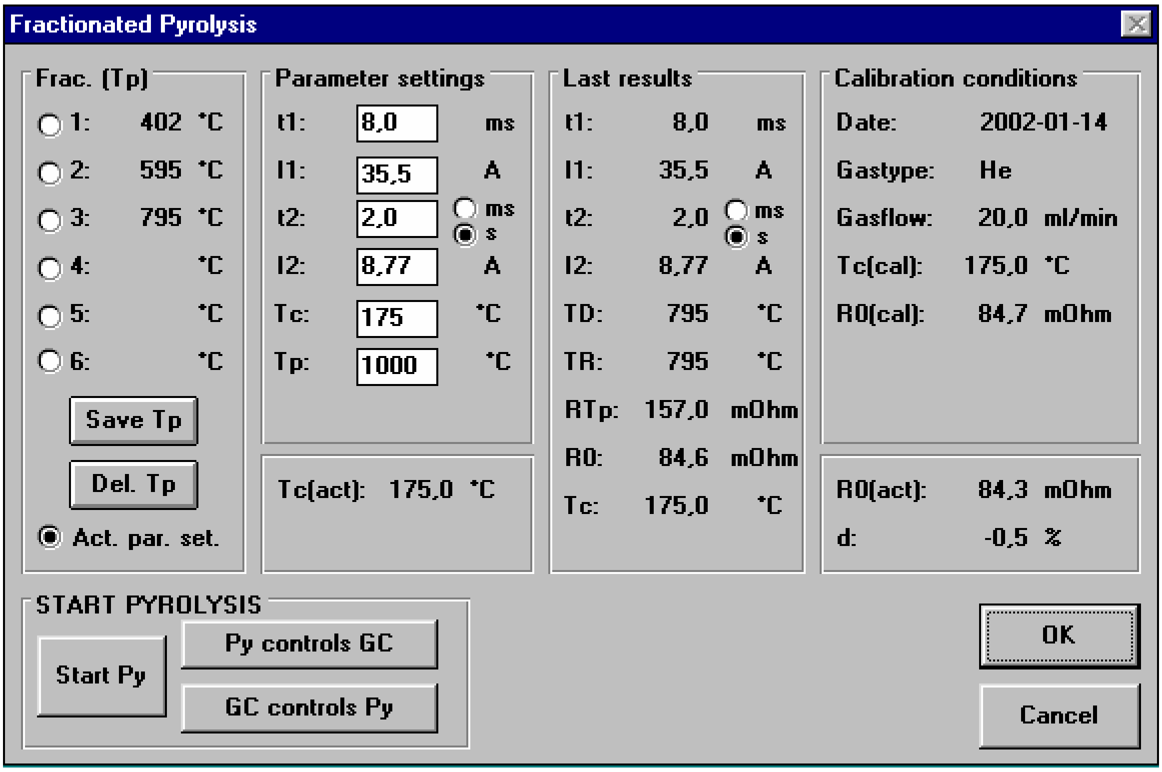


Figure 4-8. Fractionated pyrolysis.

The parameter dialog for ‘Fractionated pyrolysis’, see Figure 4-8, does not show the calibration TTP but the temperatures at which the pyrolysis should be carried out.

To select your temperature values enter the first value in the text box Tp in ‘Parameter settings’ and press ‘Enter’. The temperature will then be listed to the left. It is not necessary to enter the values in a particular order; they will be sorted by the program. Once a pyrolysis temperature has been set the software will automatically calculate the values for t1, I1 and t2, I2.

Click ‘Save Tp’ to store the temperature values. If you need to remove a value select it and click ‘Del Tp’.

The table below shows the parameter settings that are available for fractionated pyrolysis.

Parameter	Range	Unit
t1:	0-100.0	ms
I1:	0-50.0	A
t2,alt.1:	0-1000	ms
t2,alt.2:	1.0-600.0	s
I2:	0-15.0	A
Tc:	50-225 (200)	°C
Tp:	Tc-1500	°C

a warning is displayed if Tp is greater than 1400°C, the user must then ensure that the filament can survive such high temperature, melting point for Pt is 1769°C

When you click Start Py-GC a pyrolysis and the gas chromatograph are started. Remote start Py-GC starts the pyrolyzer when the GC is ready (see Appendix II). When the pyrolysis at the first temperature has been finished and the GC signals ready again the pyrolysis will be repeated with the next temperature until the last temperature value has been reached.

The result from a fractionated pyrolysis is shown in a diagram, see Figure 4-9. A list with information of each pyrolysis can be displayed by double-clicking the diagram area of the visible curve, see section 4.4.3.

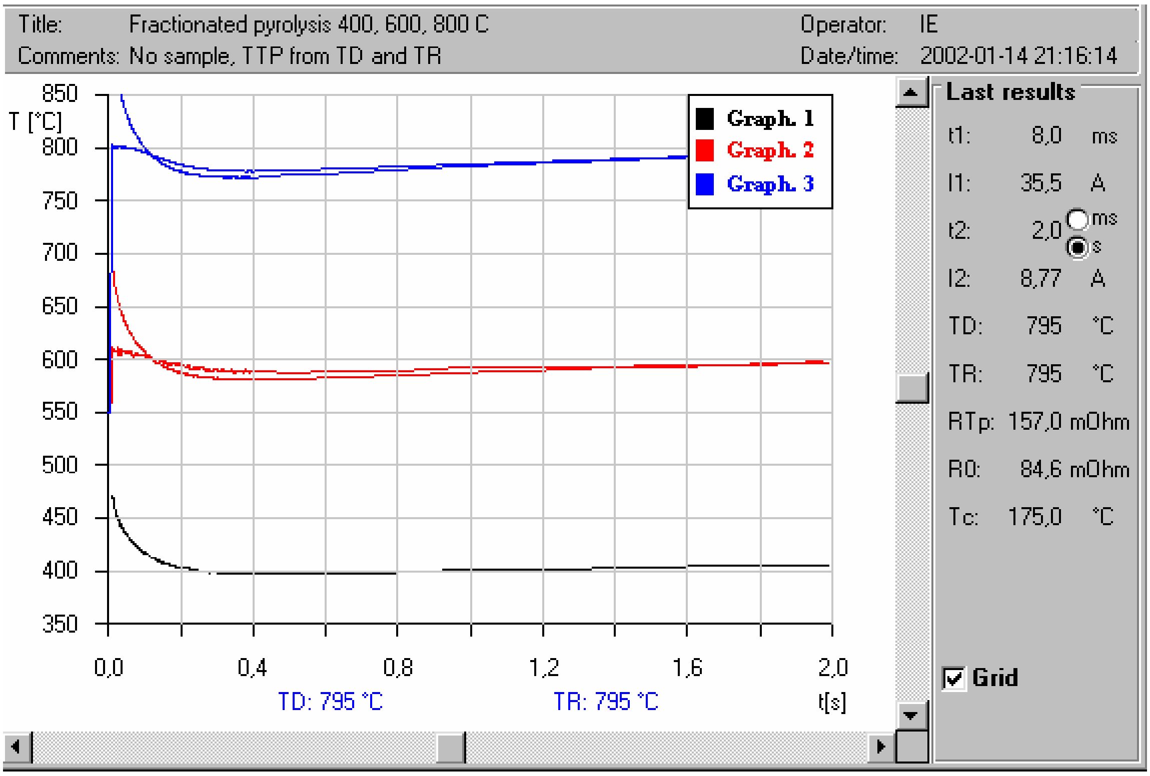


Figure 4-9. Three TTPs (TD and TR) from a fractionated pyrolysis.

4.2.4 Ramp I/t

'Ramp pyrolysis I/t' is used to heat a sample slowly up to a selected end temperature which can be useful if the sample is to be analyzed directly in a mass spectrometer. The parameter dialog for ramp pyrolysis I/t is shown in Figure 4-10.

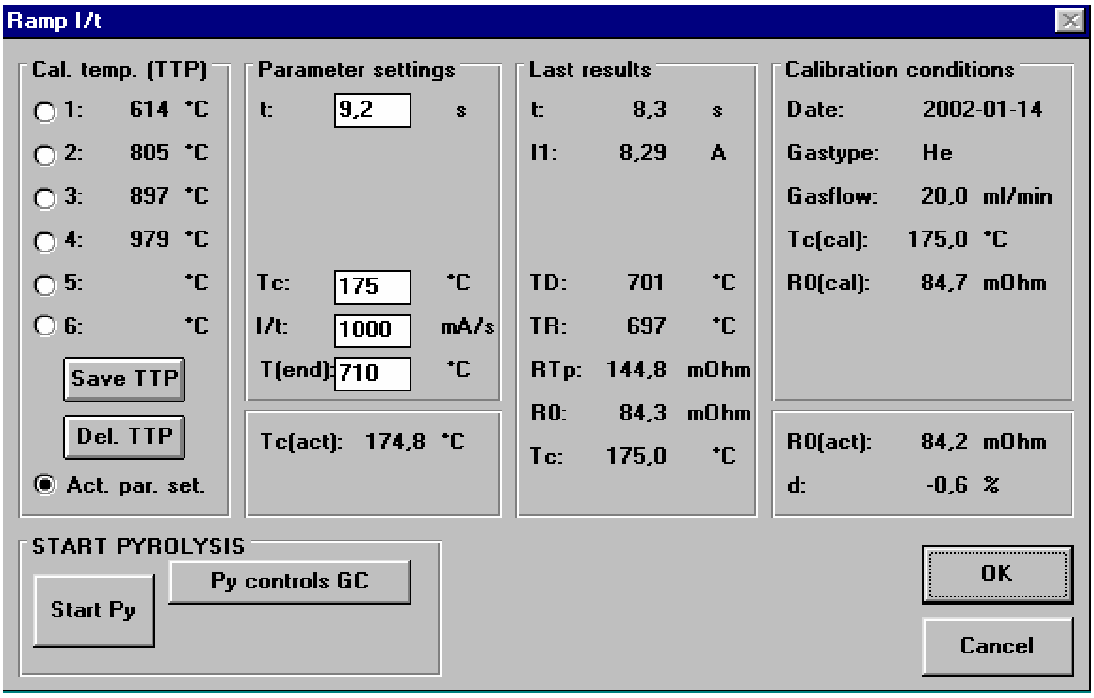


Figure 4-10. Ramp pyrolysis I/t.

For a ramp pyrolysis you select the heating rate I/t and the end temperature T(end). The Pyrola 2000 software will automatically calculate the current during the ramp when the probe is calibrated. The time t shows how long it will take the pyrolyzer to reach the end temperature at the selected rate. If the pyrolysis time is too long a warning is shown. The maximum value of t depends on the end temperature T(end). It is recommended to check the ramp pyrolysis first without sample.

The table below shows the parameter settings for ramp pyrolysis.

Parameter	Range	Unit
Tc:	50-225 (200)	°C
I/t:	10-1000	mA/s
T(end):	Tc-1500	°C

a warning is displayed if Tp is greater than 1400°C, the user must then ensure that the filament can survive such high temperature, melting point for Pt is 1769°C

An example of a ramp pyrolysis is shown in Figure 4-11.

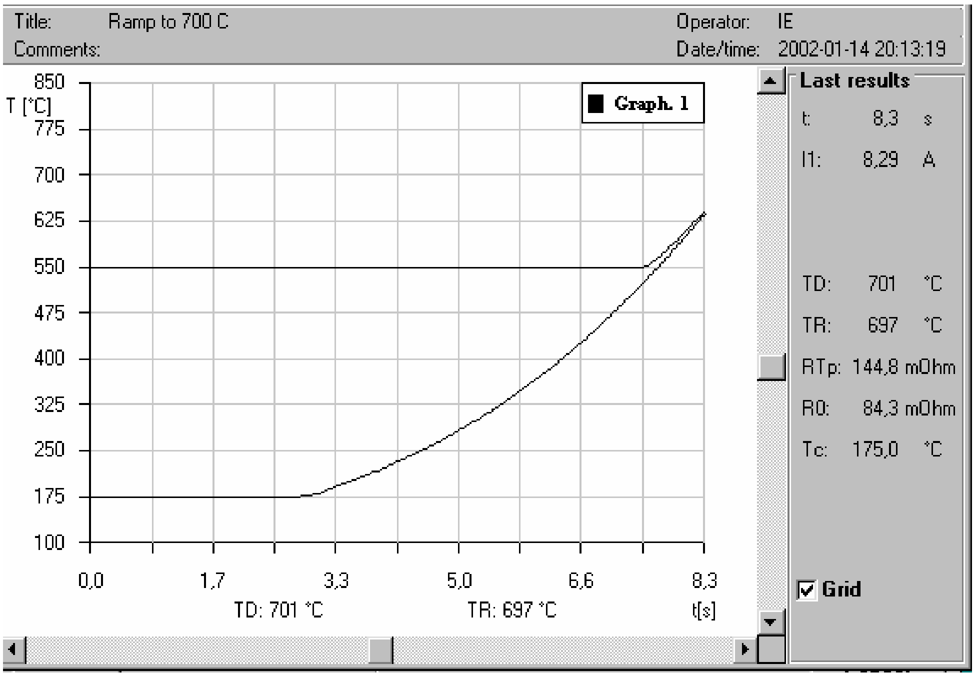


Figure 4-11. TTP results (TD and TR) from a ramp pyrolysis.

4.3 Start pyrolysis

A pyrolysis, with the selected parameters, is started by clicking one of the start buttons in the parameter dialog. There are three different methods available which will be described below:

- Start Py 

- Py controls GC 

- GC controls Py 

Information on the cabling for remote pyrolysis can be found in Appendix II.

4.3.1 Start Py

A pyrolysis is started with the selected settings but no signal will be sent to the gas chromatograph. This method is only used for the filament calibration or for checking the settings without a sample.

Output 3 (see Appendix II) is activated during the pyrolysis.

4.3.2 Py controls GC

A pyrolysis will be started with the selected parameters and an output signal will be sent to the connected gas chromatograph.

If a sequential or fractionated pyrolysis is being run the subsequent pyrolysis will be started when the Pyrola 2000 control unit receives the ready signal from the gas chromatograph on input 1. This requires that the control unit and gas chromatograph have been set up for remote control.

Output 1 is activated for one second after the pyrolysis has been started and output 3 is activated during the whole pyrolysis.

4.3.3 GC controls Py

This command is only available for sequential and fractionated pyrolysis.

The remote start will load all necessary pyrolysis parameters to the Pyrola 2000 control unit and the pyrolysis will start as soon as the ready signal is received from the gas chromatograph on input 1.

If more than one pyrolysis is to be performed in a sequential pyrolysis or if more than one Tp is set in a fractionated pyrolysis, the next pyrolysis will be started when the Pyrola 2000 unit receives a ready signal from the GC on input 1.

Output 1 activated for one second after the start of the pyrolysis and output 3 is activated during the whole pyrolysis.

4.4 Graphs

A pyrolysis graph displays the time temperature profile (TTP) as measured by either the photodiode, the resistance of the filament or both (see Fig. 21). To display the different temperature values press the corresponding buttons:

TD to display the temperature measured by the photodiode

TR to display the temperature measured by resistance

TD+TR to display both temperature values

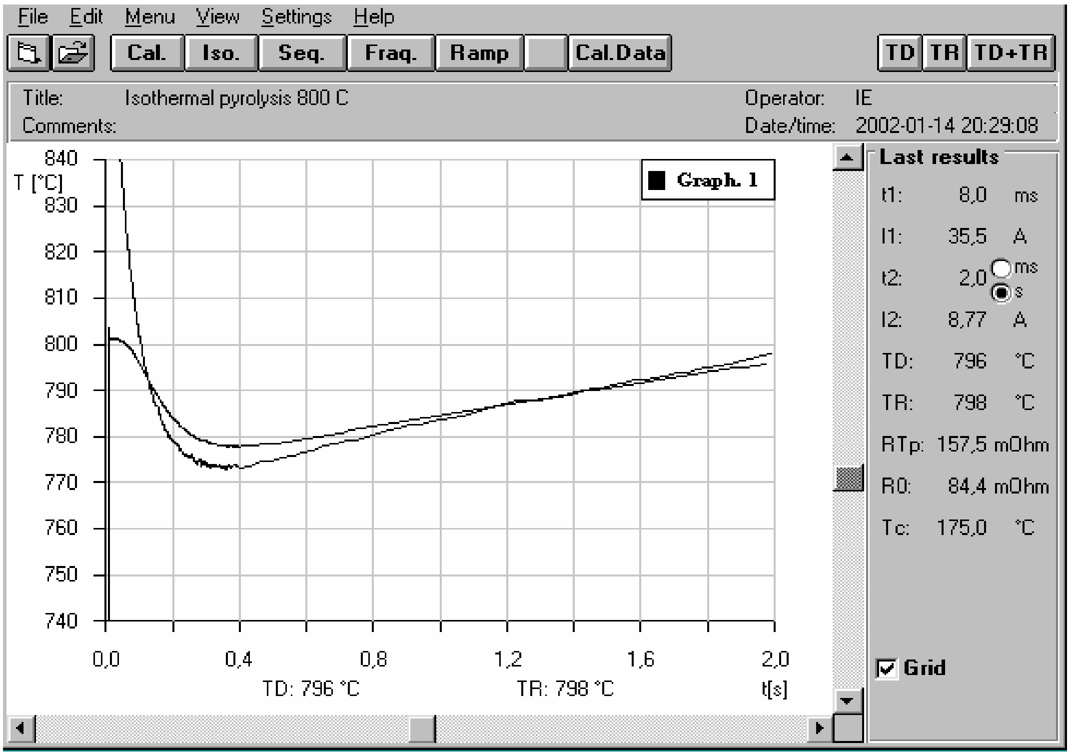


Figure 4-12. Pyrolysis result: TTP measured by photodiode, TD, and resistance, TR.

4.4.1 Title, Comments and Operator

An area above the diagram is available for displaying information about the pyrolysis. By double-clicking on the information area a dialog 'Pyrolysis title' is opened where it is possible to change the title, comments and operator. Enter the new information and press OK to exit the dialog. The new information will be displayed and stored with the graph. Alternatively the dialog can be accessed from the settings menu by choosing Title.

The information can be also be given before a pyrolysis. It is saved when you exit the program.

4.4.2 Scaling of the time and temperature axes

The time and temperature axes in the TTP diagram are adjusted automatically to the measured data, unless specified by the user.

The time axis will keep the same minimum and maximum values as long as the pyrolysis time is not changed.

The temperature axis will adjust the minimum and maximum values in such a way that the median value will be the end value for TD if TD is above 600 °C; otherwise the value for TR will be used. The selected unit length for this axis will remain constant until it is changed by the user.

Pre-defined graph settings

To specify predefined graph settings choose Preferences under the settings menu. The minimum and maximum value for the time and temperature axes can then be given.

Editing a graph

You can use the horizontal and vertical scroll bars to move the diagram. To edit one of the axes double click on the axis you want to change. The dialog window shown in Figure 4-13 will be displayed.

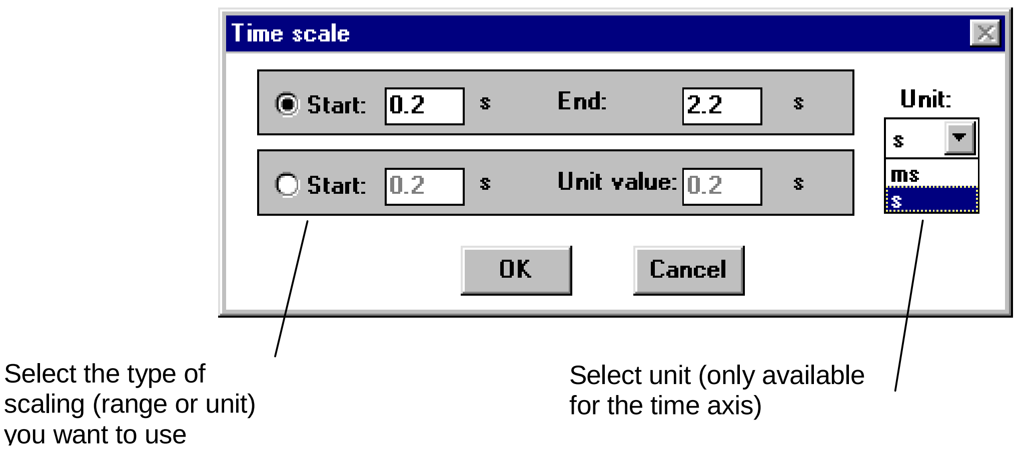


Figure 4-13. Scaling dialog.

If you select the first scaling option enter start and end value for the axis and click OK. For the time axis you also need to select the unit, ms or s.

For the second scaling option you need to enter a start value and the unit you want to use.

Please note that the number of increments that are displayed on one axis is has a constant value of ten. Therefore, if you select a unit of e.g. 10 s the length of the time axis will be 100 s. The maximum range of the temperature axis is 0 °C to 1500 °C.

4.4.3 Graph definitions

If pyrolysis result contains more than one curve a graph definition window -similar to a legend- can be displayed. The graph definition lists all pyrolysis results (ttp-files) with date, time, title and pyrolysis conditions. Examples from a sequential and fractionated pyrolysis are shown in Figure 4-14 and Figure 4-15 , respectively.

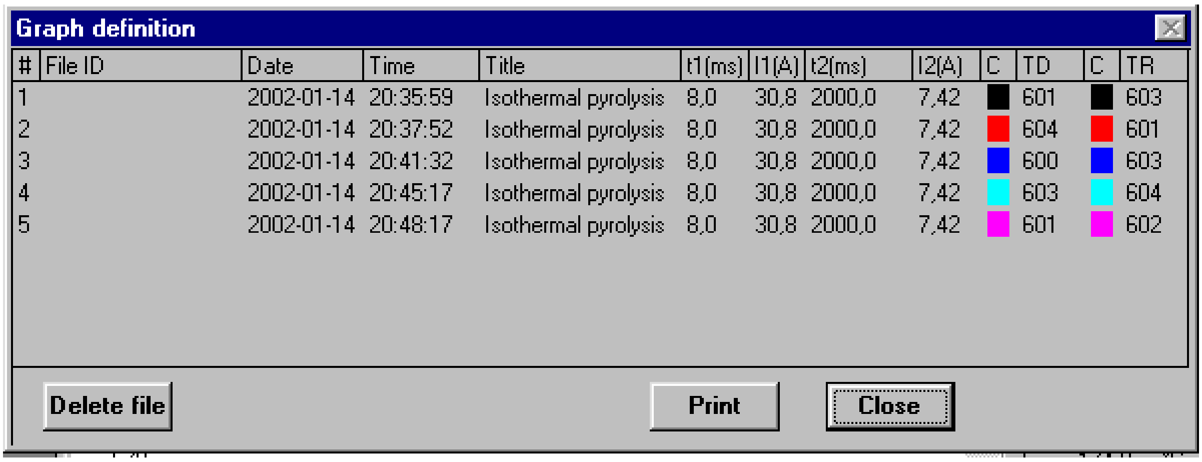


Figure 4-14. Graph definitions window from a sequential pyrolysis.

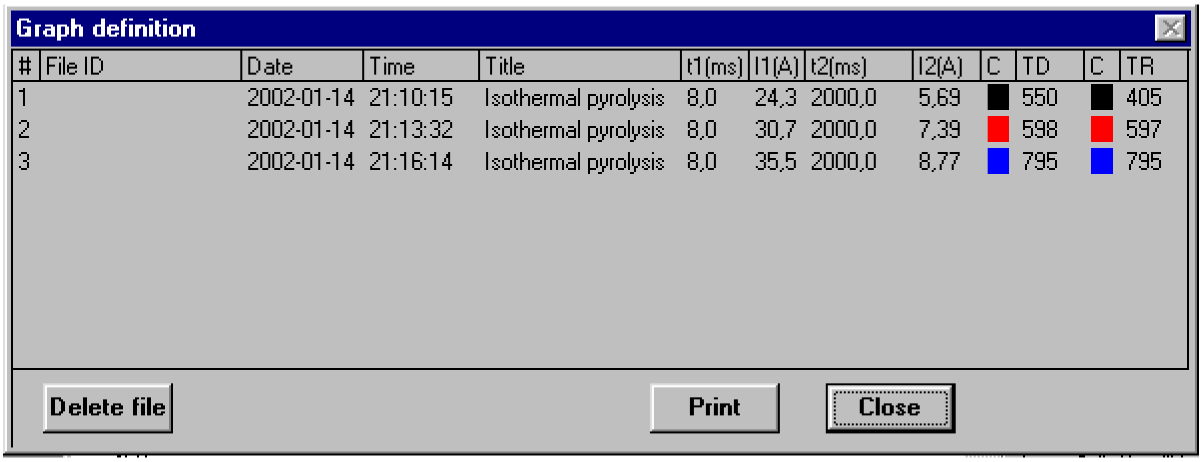


Figure 4-15. Graph definitions window from a fractionated pyrolysis. The large difference between TD and TR at 400 °C is due to the fact that the photo diode will not measure temperatures below 550 °C.

You can open the graph definitions window by double clicking on the diagram, or alternatively by choosing Graph def. in the view menu.

Use the print button to print the graph definition or use close to leave this dialog. If the graph definition of a library file is displayed you can use the delete button to remove selected file(s) from the file.

The table below summarizes the information in the graph definition window.

Column	Description
#	number of each TTP
File ID	name of the .ttx-file (if the file has been saved)
Date	date the .ttx-file was created
Time	time the .ttx-file was created
Title	experiment title as displayed in the graph
t1 (ms)	length of the first pulse (ms)
I1 (A)	current in the first pulse (A)
t2 (ms)	length of the second pulse (ms)
I2 (A)	current in the second pulse (A)
C	color used to display the TD curve
TD	temperature measured by the photodiode
C	color used to display the TR curve
TR	temperature measured by resistance

4.5 Printing

You can print the diagram, the graph definition and the calibration data. Graph definition and calibration data can be printed directly from their respective dialog windows by using the print button. A diagram is printed by choosing Print in the file menu.

The printer and printer settings may be changed by choosing Print setup in the file menu.

4.6 File formats and saving information

The Pyrola 2000 Software handles three file types, ini-files, ttp-files and ttx-files. I addition to these types data may be exported by generating a Tdtr.txt file. A short description of the files is given below.

1. ini-files; initialization files
   The ini-file is used to save pyrolysis parameters and calibration data. The software is delivered with a default ini-file, pyrini.ini, that contains the factory calibration for your unit.

2. ttp-files; result files
   The result files contain the pyrolysis results and the parameter setup used to get these results. Use 'Save as…' to save the result files under a selected name. Result files can be combined with each other in a ttx-file or exported for use in another program.

3. ttx-files; library files
   Library files consist of several user-selected result files and are used for comparison of the results.

4. Tdtr.txt files; export files
   This text file contains the temperature values from the last pyrolysis for TD and TR in the same order as they are measured. It is stored in the same directory as the Pyrola 2000 software and can be imported to other programs e.g. Excel.

To save any file other than the default pyrini.ini, always use the command 'Save as…'. Select drive, directory and file type, enter the filename and extension. The file extension of these files need to be exactly as spelled above, otherwise the Pyrola 2000 program will not recognize the files.

Note: The command 'Save' will only save changes to the current ini-file.

To open a file use the 'Open' command, select file type, drive and directory and select your file.

4.6.1 ini-files; Initialization files

The ini-file contains the parameter setup for all pyrolysis menus and the calibration data and is stored in the same directory as the Pyrola 2000 software. There is one default ini-file, pyrini.ini, which includes the factory settings.

Any changes that are made during the operation of the software can be stored to the file pyrini.ini with the 'Save' command. This file will automatically be loaded the next time you start the Pyrola 2000 program.

If needed, more than one ini-file may be saved, for example if the chamber temperature or type of carrier gas is changed (for example from He to H_2 or air). Then save the calibration data in a different ini-file with 'Save as…'. This way you can recall all calibration conditions according to the settings you want to use for a pyrolysis.

The ini-files can also be used to store different parameter settings. Change the parameter settings for the pyrolysis menus as required and save them in a new ini-file using the 'Save as…' command.

To use a specific ini-file you need to load it after the software has been started (during start-up the default file pyrini.ini will be loaded). Go to Open in the file menu; a warning as shown in Fig. 24 will be displayed.

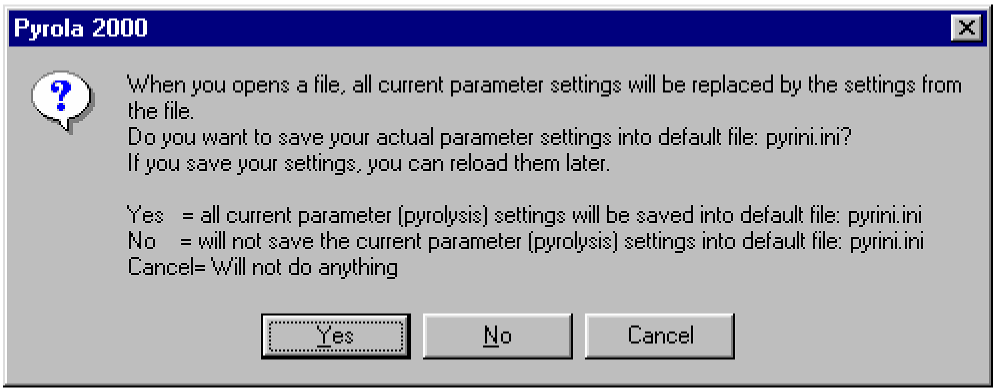


Figure 4-16. 'Open file' warning.

Select 'Yes' to save the settings from the new file to the default file or 'No' to keep the default file unchanged. Then select the ini-file you want to use and click OK.

4.6.2 ttp-files; Result files

The result file contain the TTP of the pyrolysis and the pyrolysis conditions. For sequential and fractionated pyrolysis the ttp file contains information of each separate pyrolysis. For these pyrolysis types the graph definition, see section 4.4.3, is also included.

As ttp-files also include the parameter setup for the pyrolysis menus they can be used as parameter files instead of using different ini-files. If you open a previously saved ttp-file the software will show the warning in Figure 4-16. Select 'Yes' to save the settings from the ttp-file to the default ini-file or 'No' to keep the default file unchanged. Then select the ini-file you want to use and click OK.

When a result file has been opened you can select whether to use the parameter settings in the ini-file, or in the result file, when you open any of the pyrolysis menus.

To save a result file use the command 'Save as..' on the file menu, select drive and directory, enter the filename with extension and click OK.

4.6.3 ttx-files; Library files

Library files are used to compare up to nine different result files that have been stored previously.

To create a new library file choose 'New TTX' on the file menu, and choose a result file in the open dialog that is shown. Once the library file has been created you can add result files up to a total number of nine graphs with the 'Insert' command on the edit menu. Please note that the size of the library file is limited by the number of result files and the number of graphs, i.e. if a result file contains more than one graph the number of files in the library file is reduced accordingly.

To display the graph definition (list of files) for a library file, double click on the diagram area or use Graph definitions on the view menu. The graph definitions window displays a list of all result files included in the library file with file information such as date, name and pyrolysis conditions. To remove a result file from the list select the file and click 'Delete'.

To save a library file use 'Save as…' on the file menu. Select drive and directory, enter the filename with extension and click OK.

If you wish to save a ttx-file, use the 'Save as…' menu item. Save the file with the file extension '*.ttp'. To load this ttx-file later, use the 'Open…' menu item and choose ttx-files under 'List Files of Type'.

After a library file has been opened you can select whether to use the parameter settings in the ini-file or in the result file when you open one of the pyrolysis menus.

4.6.4 Tdtr.txt; Export files

This file contains the temperature values for TD and TR in the same order as they are measured for the last pyrolysis that has been carried out, and is stored in the same directory as the Pyrola 2000 software. It is saved when the command 'Save' is used and when you exit the software.

The file uses ASCII format and can be imported to other Windows programs, e.g. Excel.

4.7 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 4-17 together with a probe and the tool for making cavities in the filament.

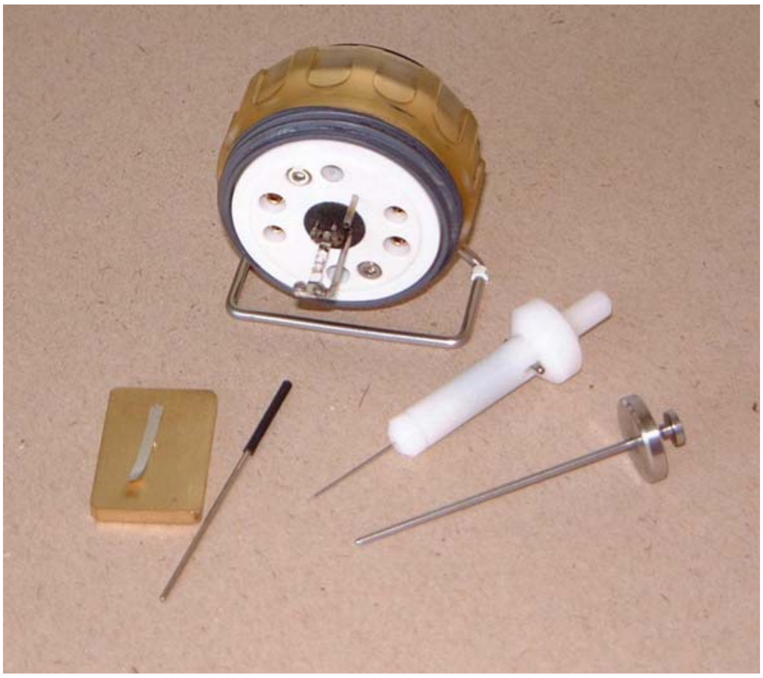


Figure 4-17. Sample handlers.

4.8 Maintenance

4.8.1 Cleaning the glass cell

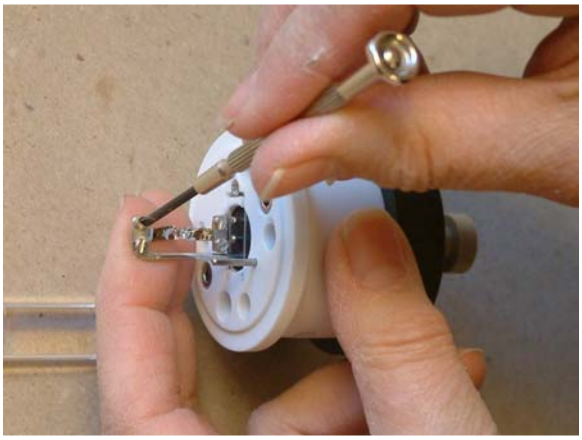
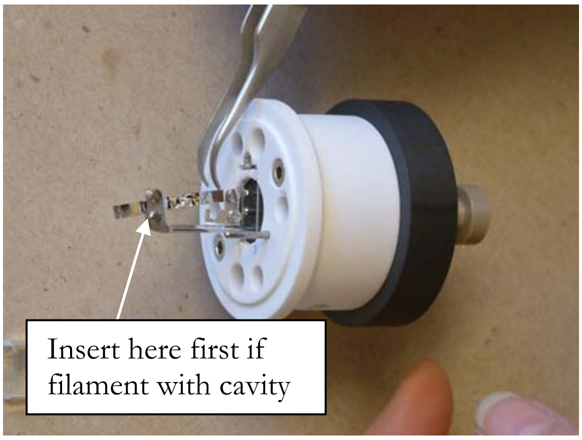
The purpose of glass-cell in the process unit (see Figure 1-2, no. 7) is to be able to let the light from the heated Pt-filament pass to the fiber-optic cable. Furthermore, it protects the GC-column from non-volatile pyrolysis products. Therefore, the glass-cell might need to be cleaned after a certain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

Figure 4-18. Cleaning the glass cell with a micro torch.

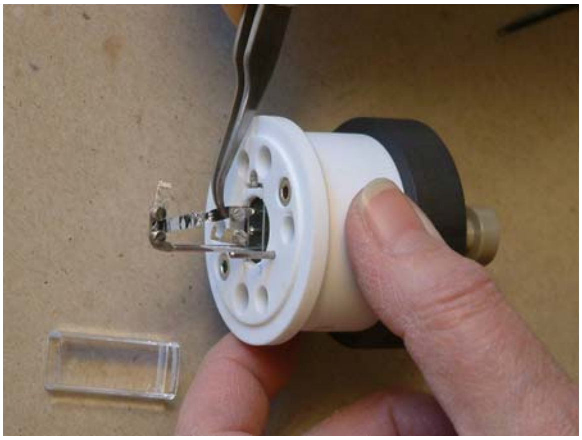
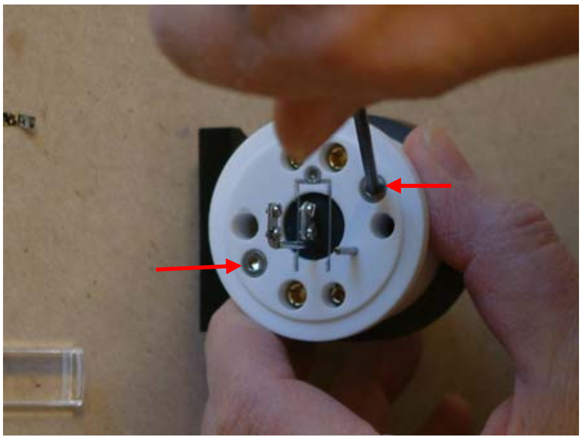
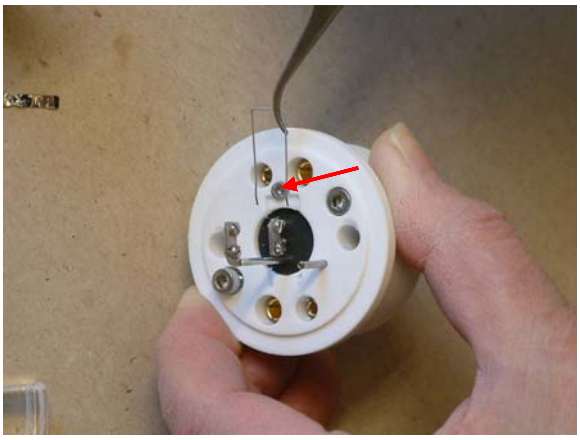
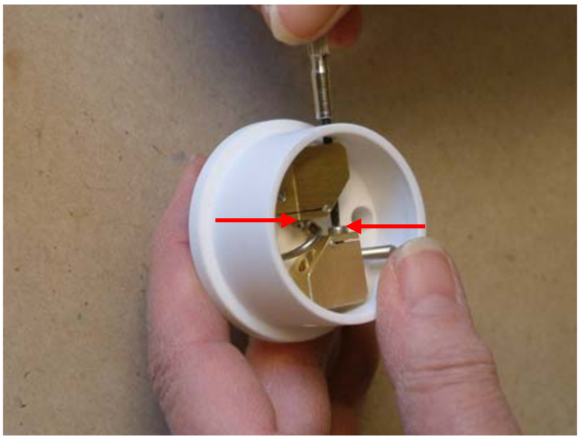
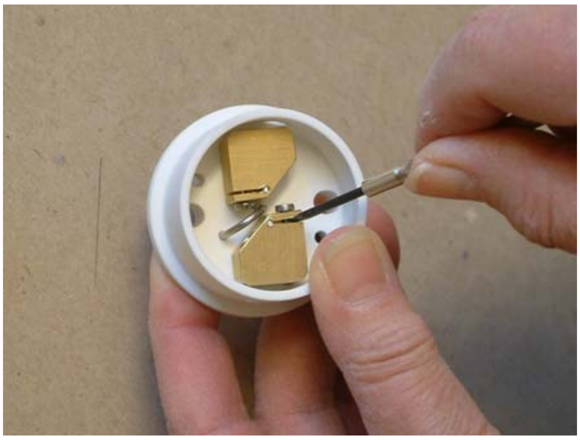
The easiest way to clean it is to put it in a Bunsen burner or to use a micro torch, see Figure 4-18. Notice, that air should be able to pass inside the cell, otherwise the products will be pyrolyzed and generate carbon, which is difficult to remove.

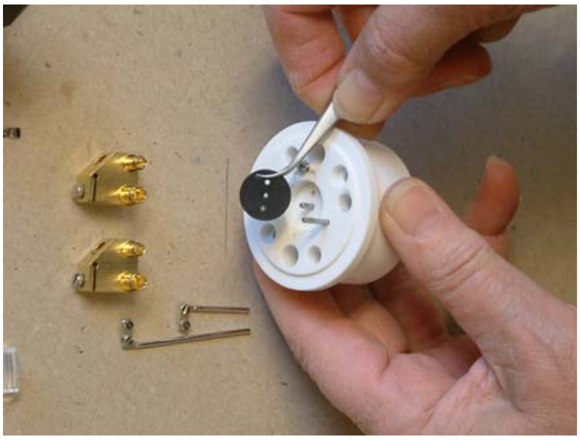
WARNING!
Make sure to be adequately protected when cleaning the glass cell with heat. Also, handle the glass cell with care and avoid sudden temperature changes.

4.8.2 Replacing the filament

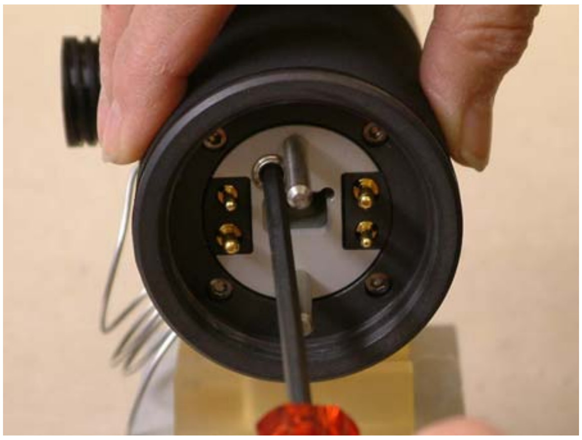
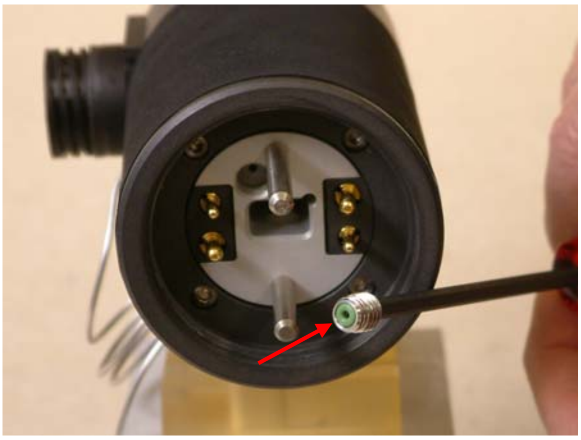
 Loosen the screws holding the filament. **Do not remove the screws!**	 Open the lids and remove the old filament.
 Insert the new filament. If a filament with a cavity is used, insert the filament furthest away from the probe first.	 Adjust the position of the filament to be in line with the Pt-flags 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.

4.8.3 Replacing the septum

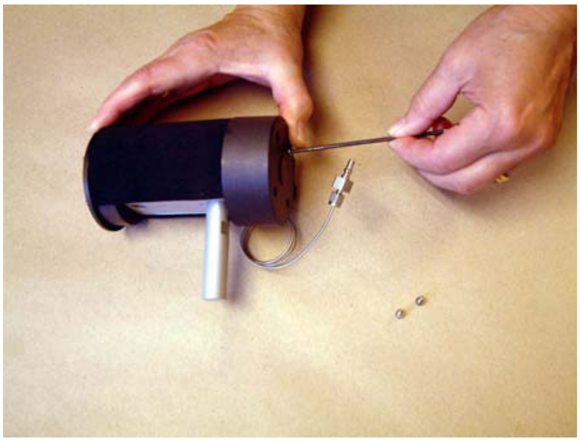
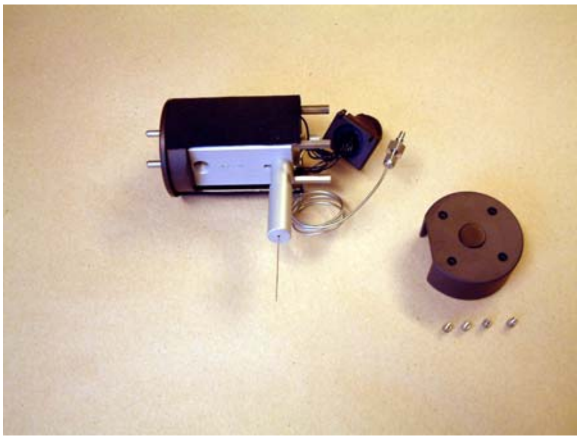
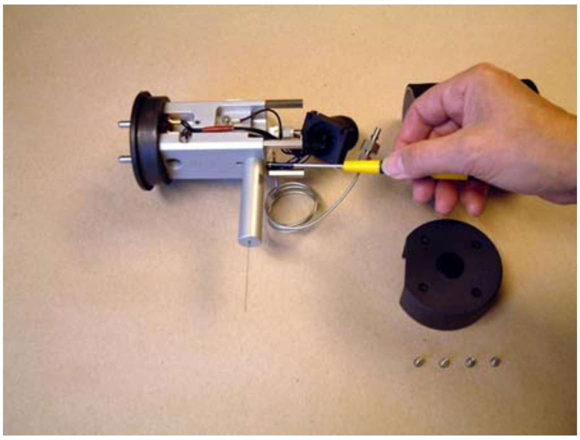
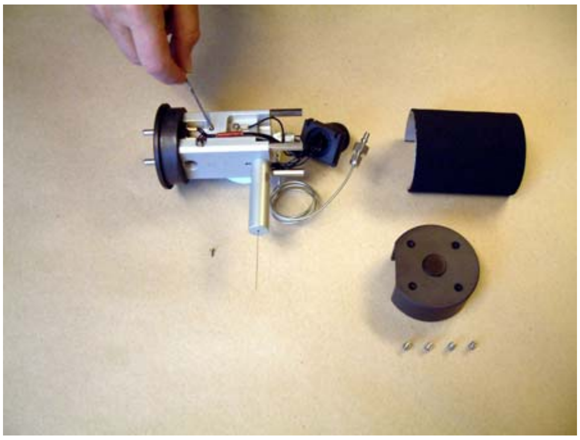
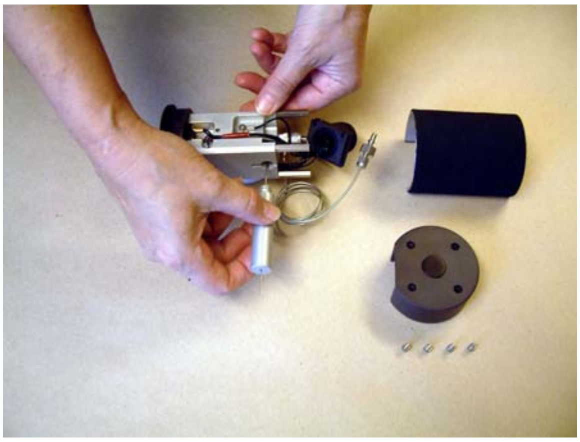
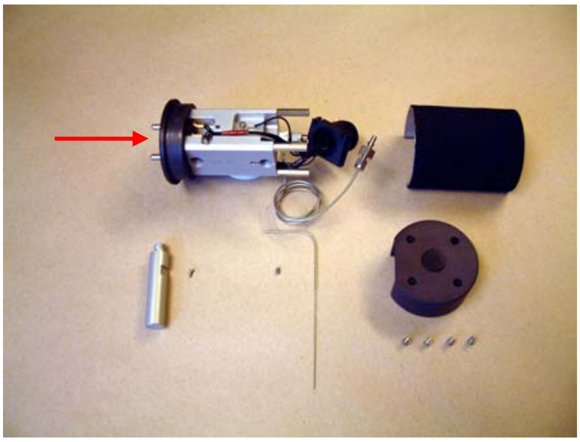
 Remove the filament.	 Remove the screws holding the probe together.
 Separate the probe.	 Loosen the screw holding the spring for the glass cell, and remove it carefully.
 Loosen the screws holding the Pt-flags.	 Widen the slit holding the Pt-flags carefully. Remove the flag holders with contacts.

 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. Mount the Pt-filament as described in section 4.8.2.

4.8.4 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.

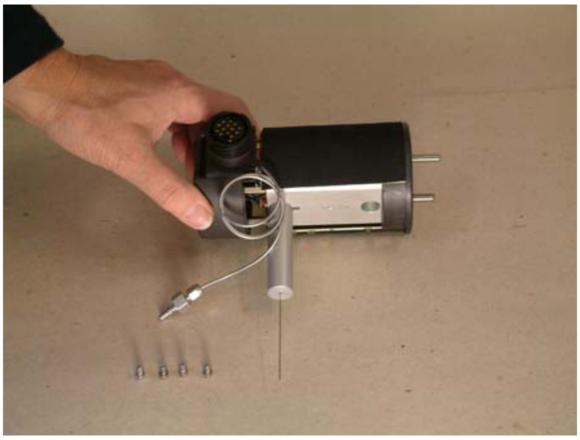
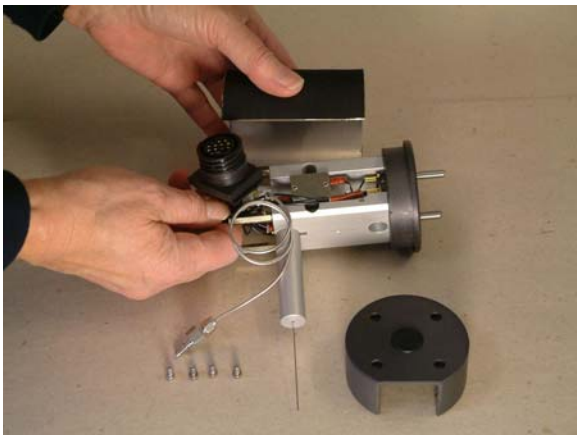
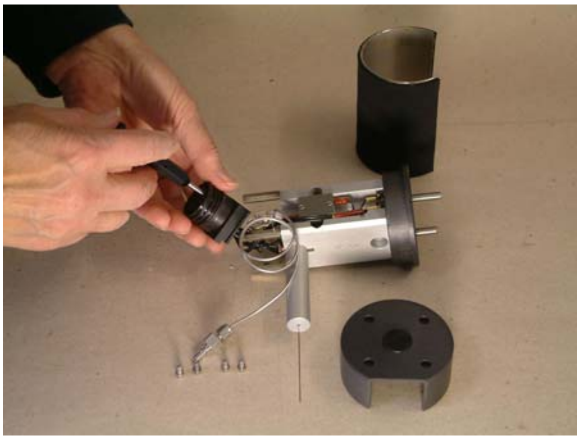
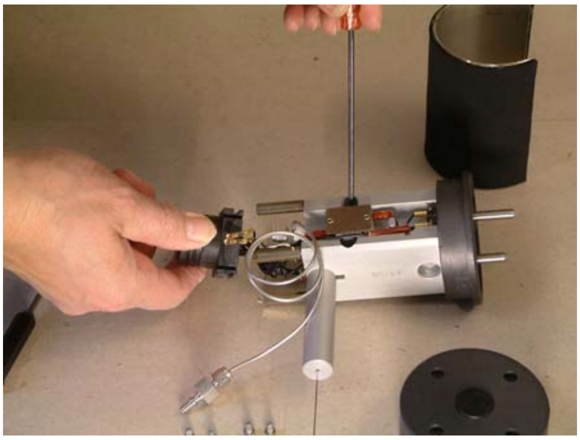
4.8.5 Changing the injection needle

 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.

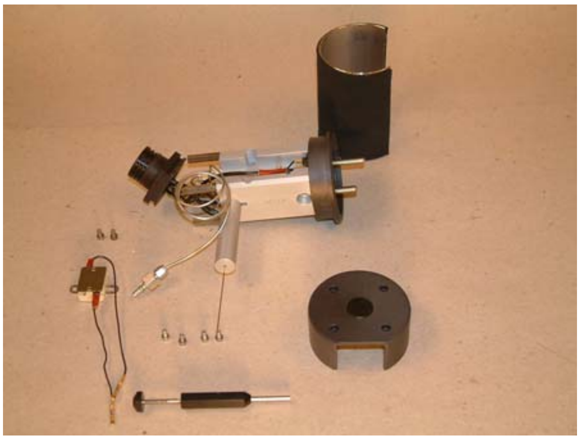
Mount the neck on the new injection needle before it is put in position. Reassemble the chamber. Insert the cleaning needle into the injection needle before remount the chamber on the GC. Remove the cleaning needle.

4.8.6 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.

The instruction continues on next page.



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.

4.8.7 Replacing other electrical components in the chamber

To replace other electrical components in the chamber, proceed as described in section 4.8.6, ‘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 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 hot! 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 N_2. 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 N_2. A too high current may break the filament.
- There are dangerous voltages inside the Pyrola 2000 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 4.8.1.
- 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 4.8.3) The o-ring behind the screw where the carrier gas passes to the probe needs to be changed Replace the o-ring (see section 4.8.4)
R0 not reproducible	Filament not fixed. Check the filament and adjust. Chamber temp. (Tc) not constant.
R0(act)= 0 mΩ 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.
R0(act)= 350 mΩ	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	R0 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.

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 'Cal' in the toolbar to open the Calibration of Filament window, see Figure I-1.

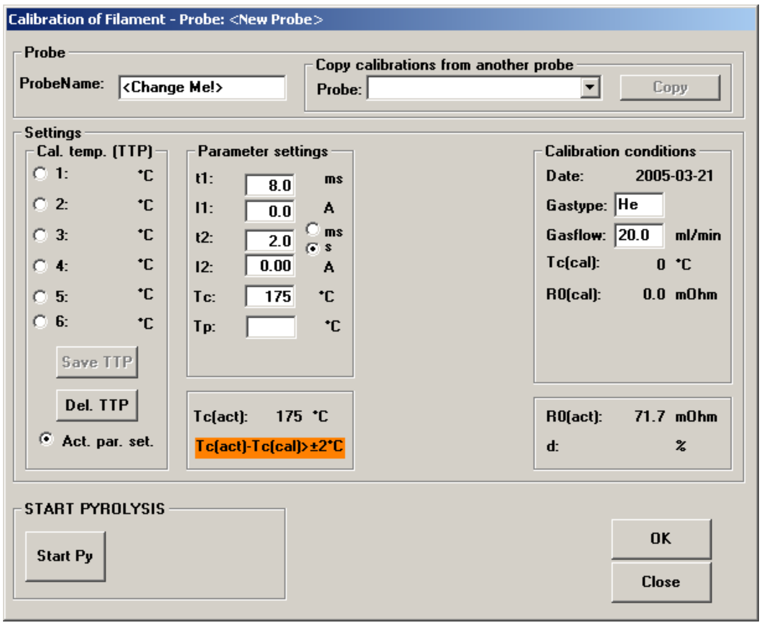


Figure I-1. 'Calibration of filament' window.

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. 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.

4. 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.

5. 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.

6. 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.

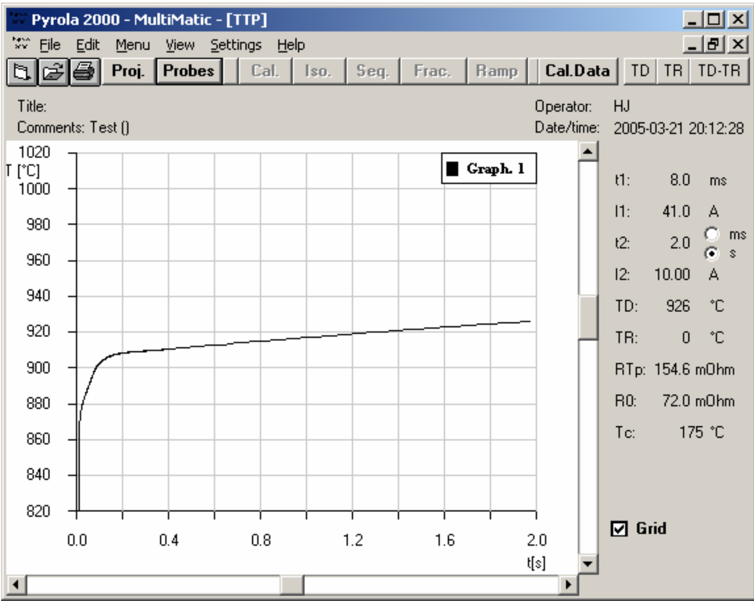


Figure I-2. TTP after the first pyrolysis.

7. 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.

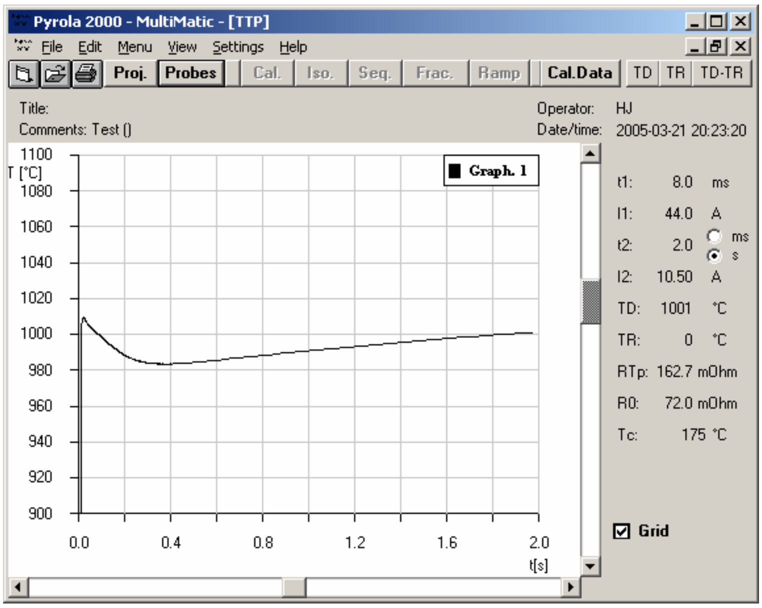


Figure I-3. TTP after the second pyrolysis.

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

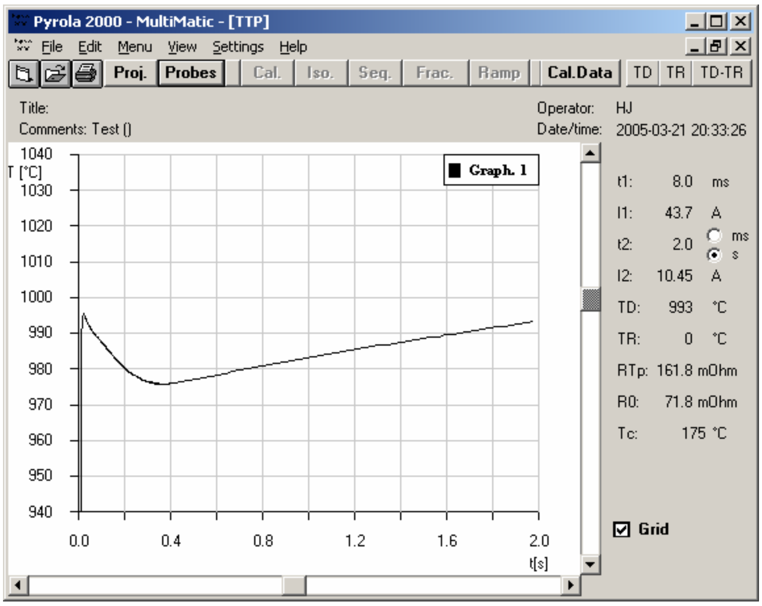


Figure I-4. The TTP after the third pyrolysis gives an acceptable result. Note the changed scaling on the y-axis.

9. 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.

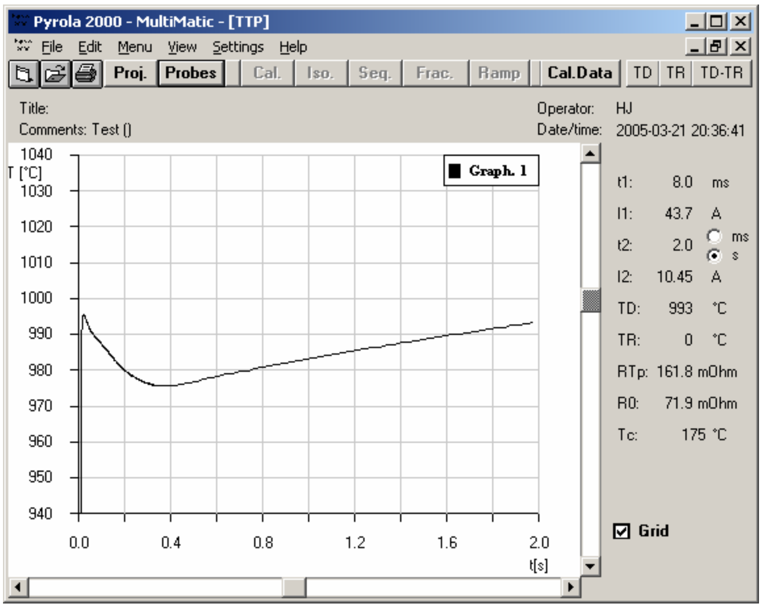


Figure I-5. Repeating the pyrolysis gives the same result, compare with Figure I-4.

> **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.

10. 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.

11. 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.

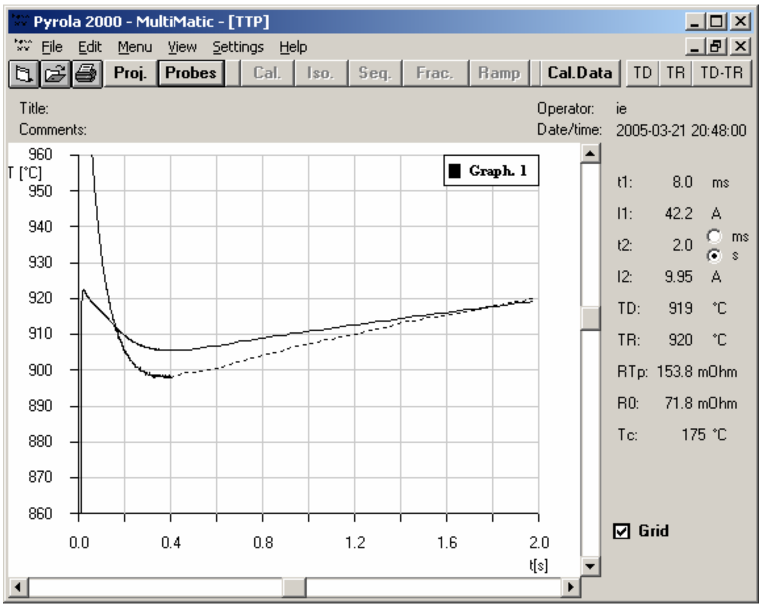


Figure I-6. A second pyrolysis temperature is found by decreasing I1 and I2 with 1.5 and 0.5 A, respectively.

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

13. 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'.

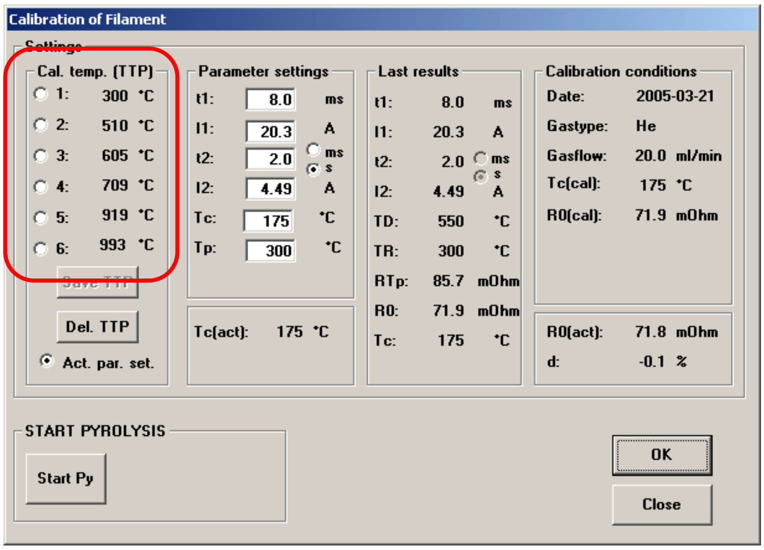


Figure I-7. 'Calibration of filament' window after calibrating at 6 different temperatures.

14. 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.

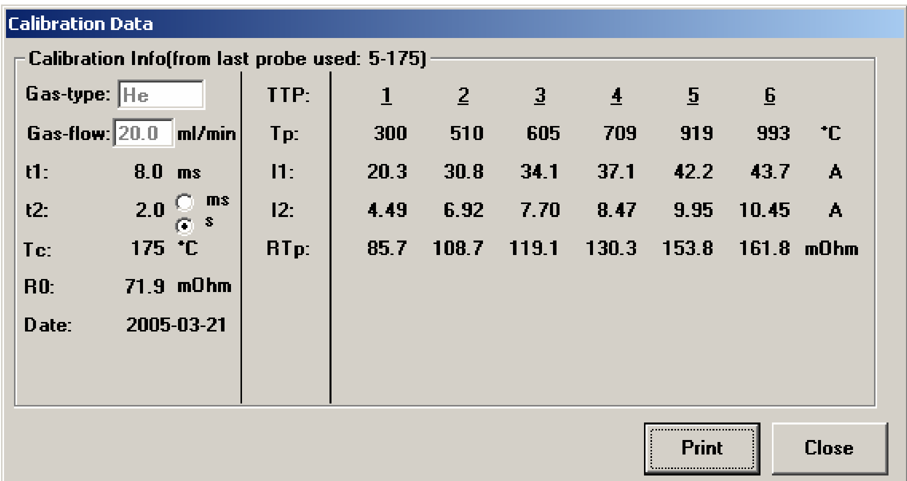


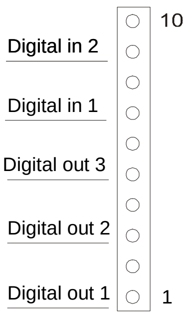
Figure I-8. 'Calibration data' window.

Appendix
II

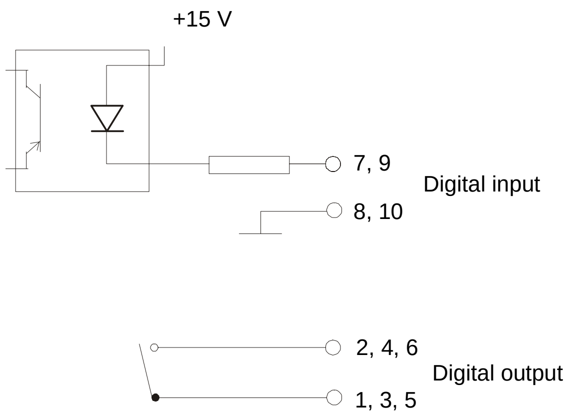
II Connection to a gas chromatograph

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 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

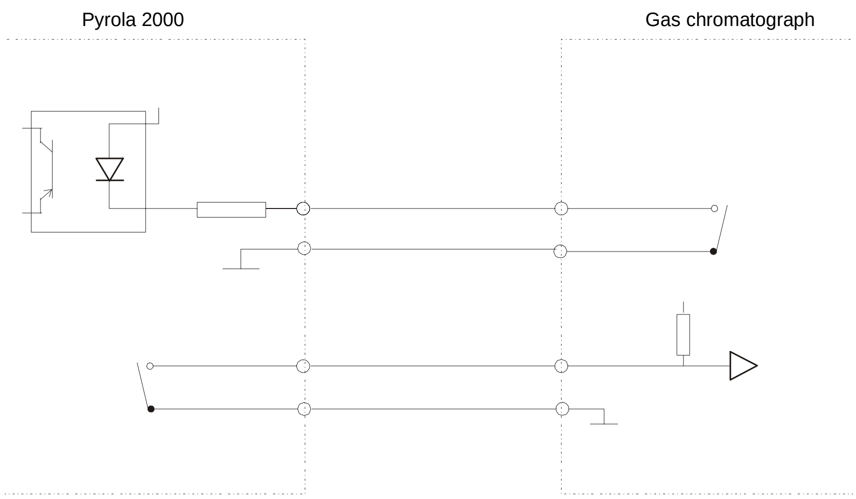


Electrical drawing

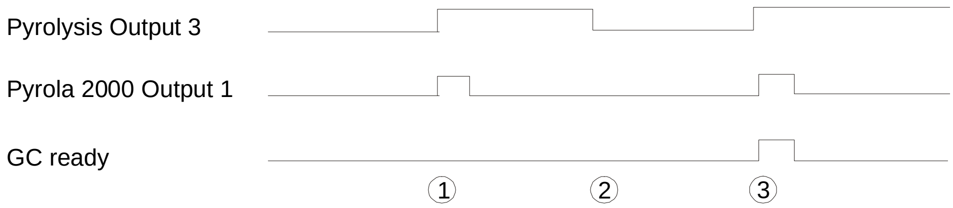


<u>Installation of the cable between Pyrola 2000 and gas chromatograph</u>

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



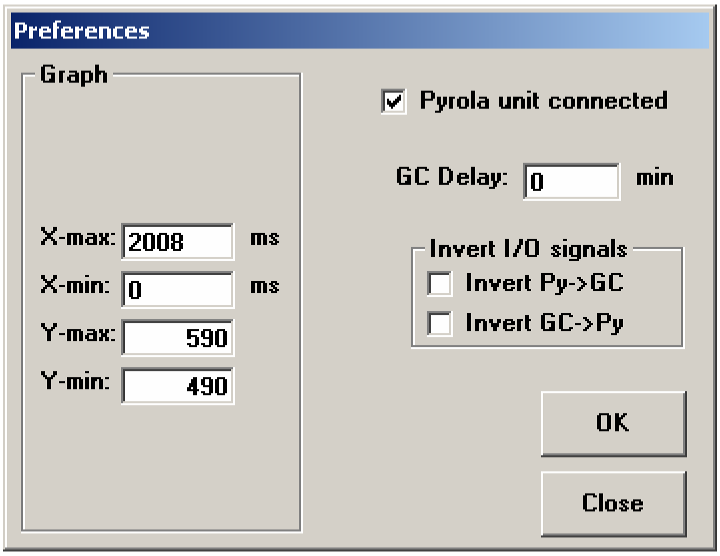
1. A pyrolysis starts and Pyrola 2000 ´s output 1 is activated (closed) for 1 second. GC reads this and starts it´s cycle. Output 3 is activated (closed) during the whole pyrolysis cycle.

2. Pyrolysis is ready. Pyrola 2000 waits for GC ready on digital input 1.

3. GC is ready and signals to Pyrola 2000 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.



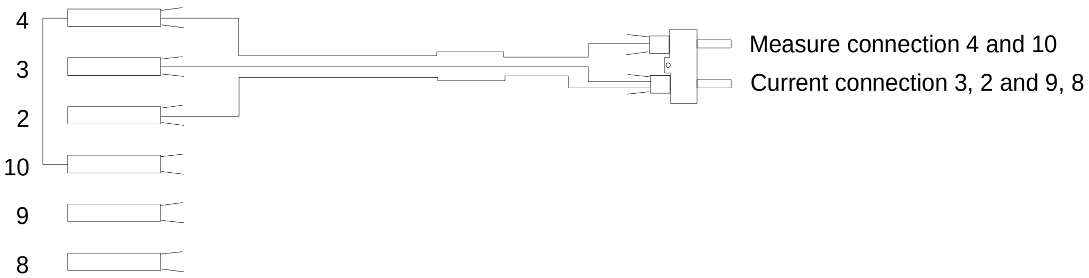
GC Delay
For some GCs it is necessary to choose a delay time, long enough so the GC is ready before the next pyrolysis.

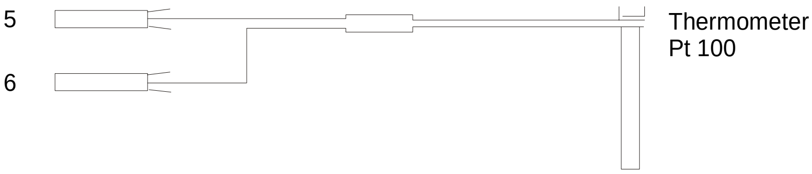
Invert I/O signals
For an Agilent or HP GC, the 'Invert GC->PY' should be checked. For other brands, these boxes should be left unchecked.

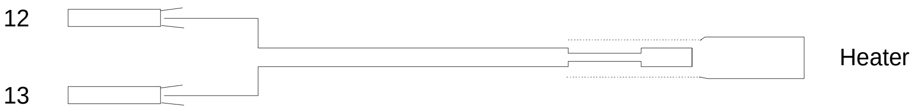
Appendix III

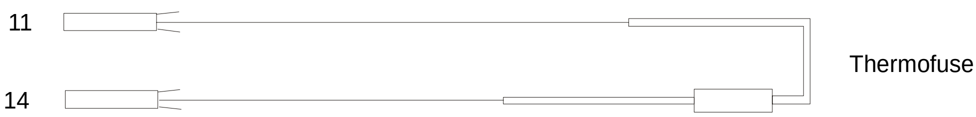
III Pin layout

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





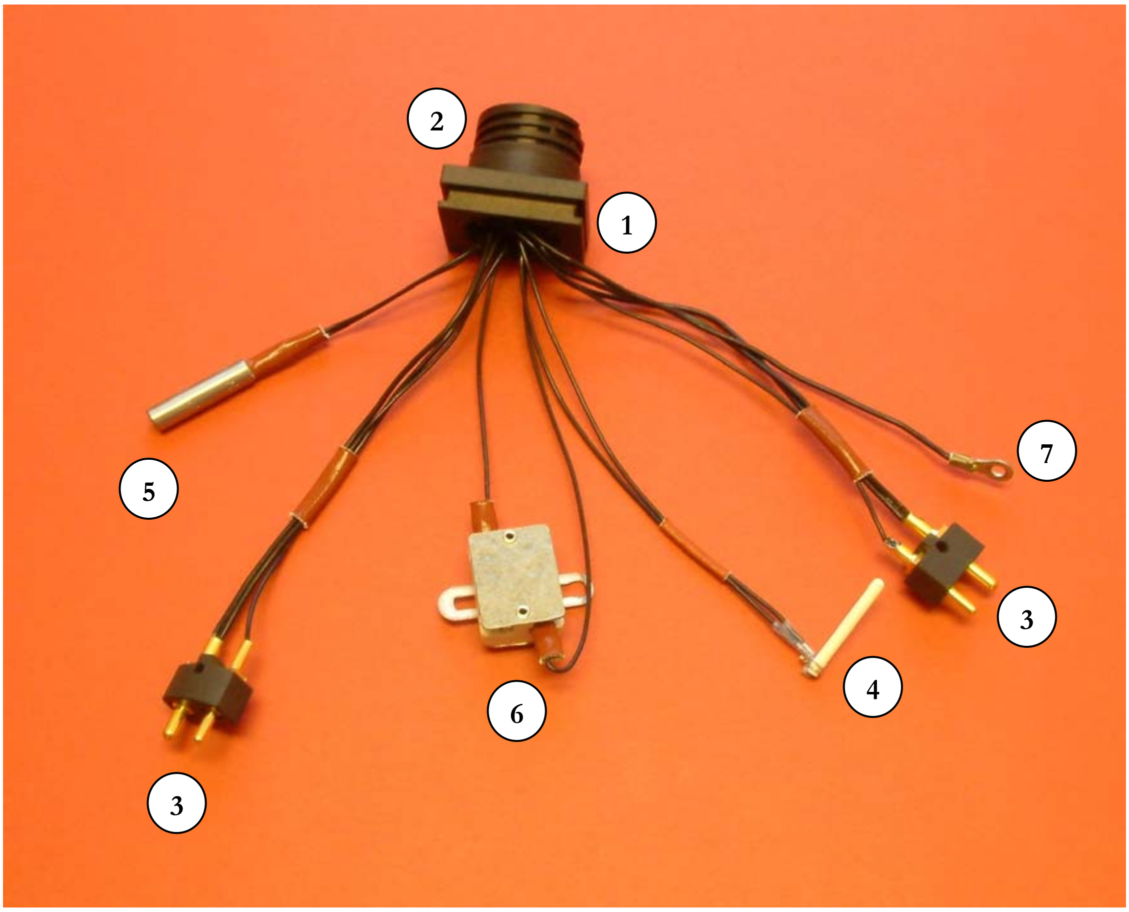




Appendix
IV

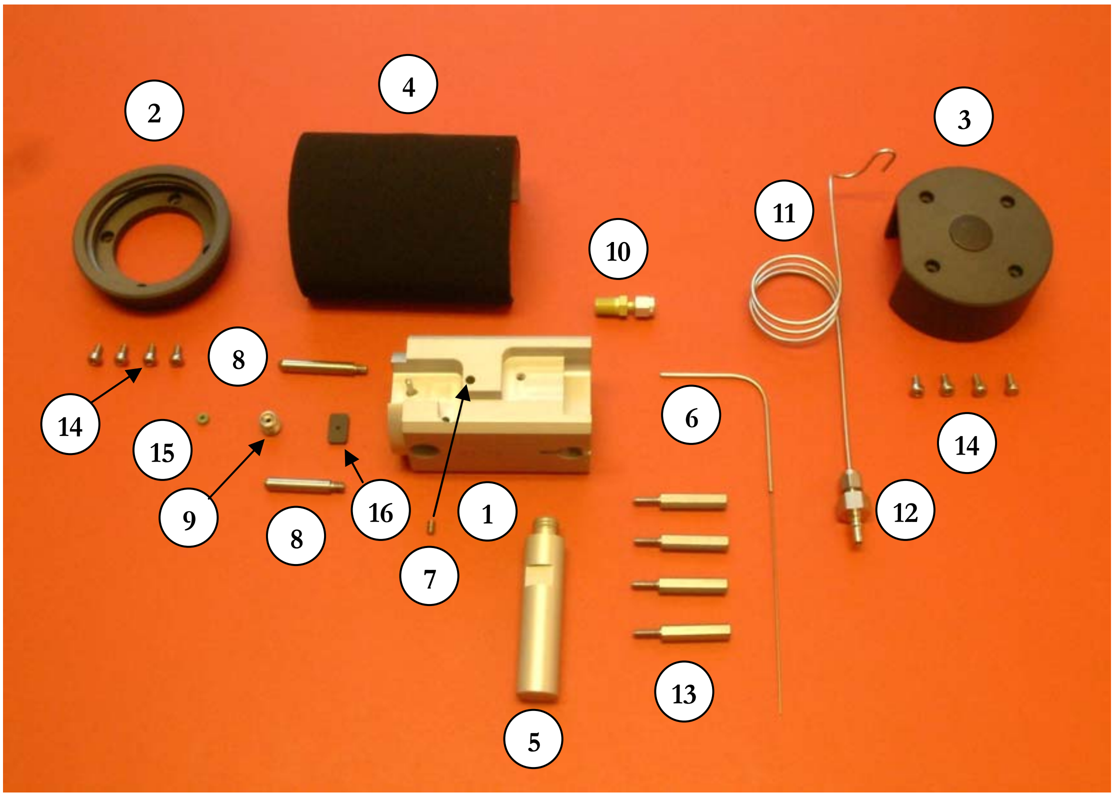
IV Parts and order numbers

In this appendix parts and order numbers are presented.



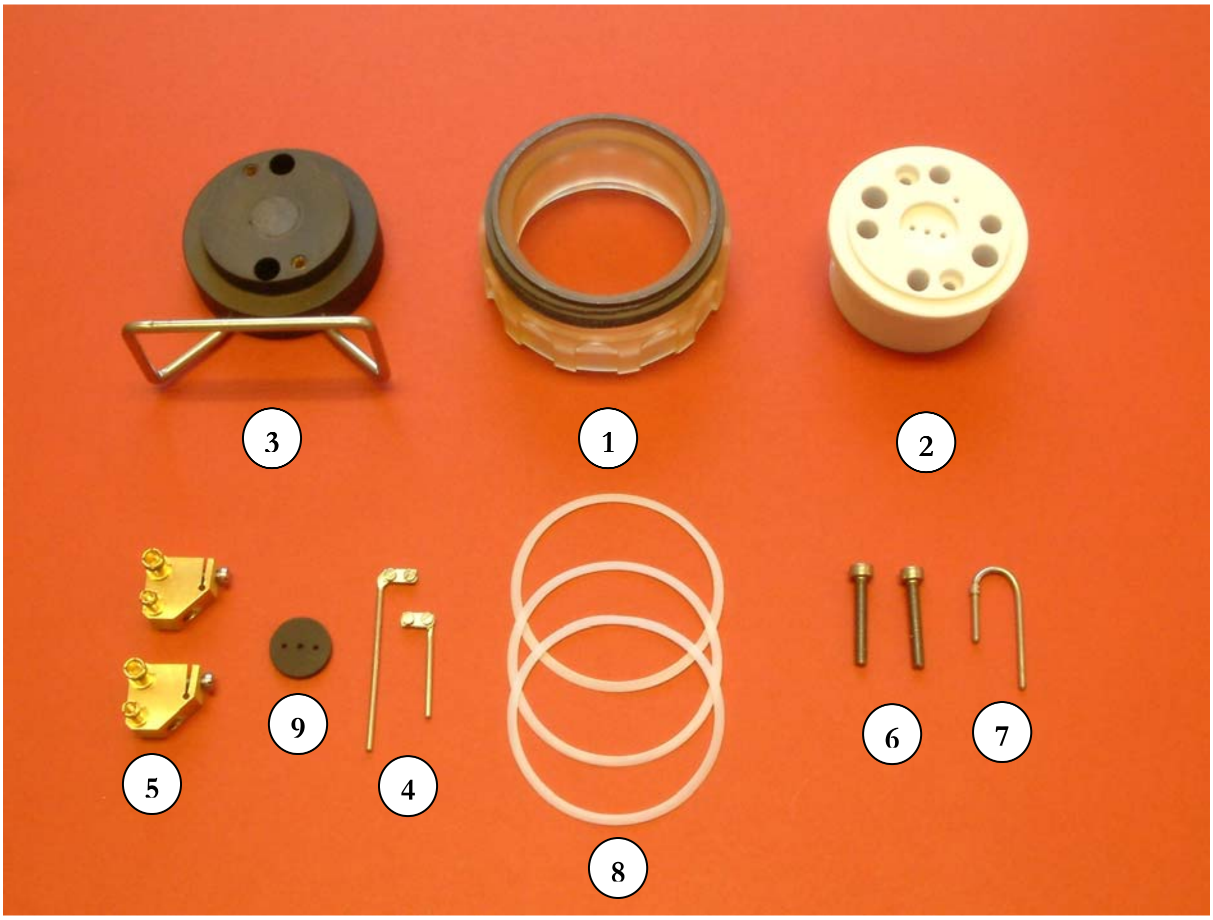
Cabling

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



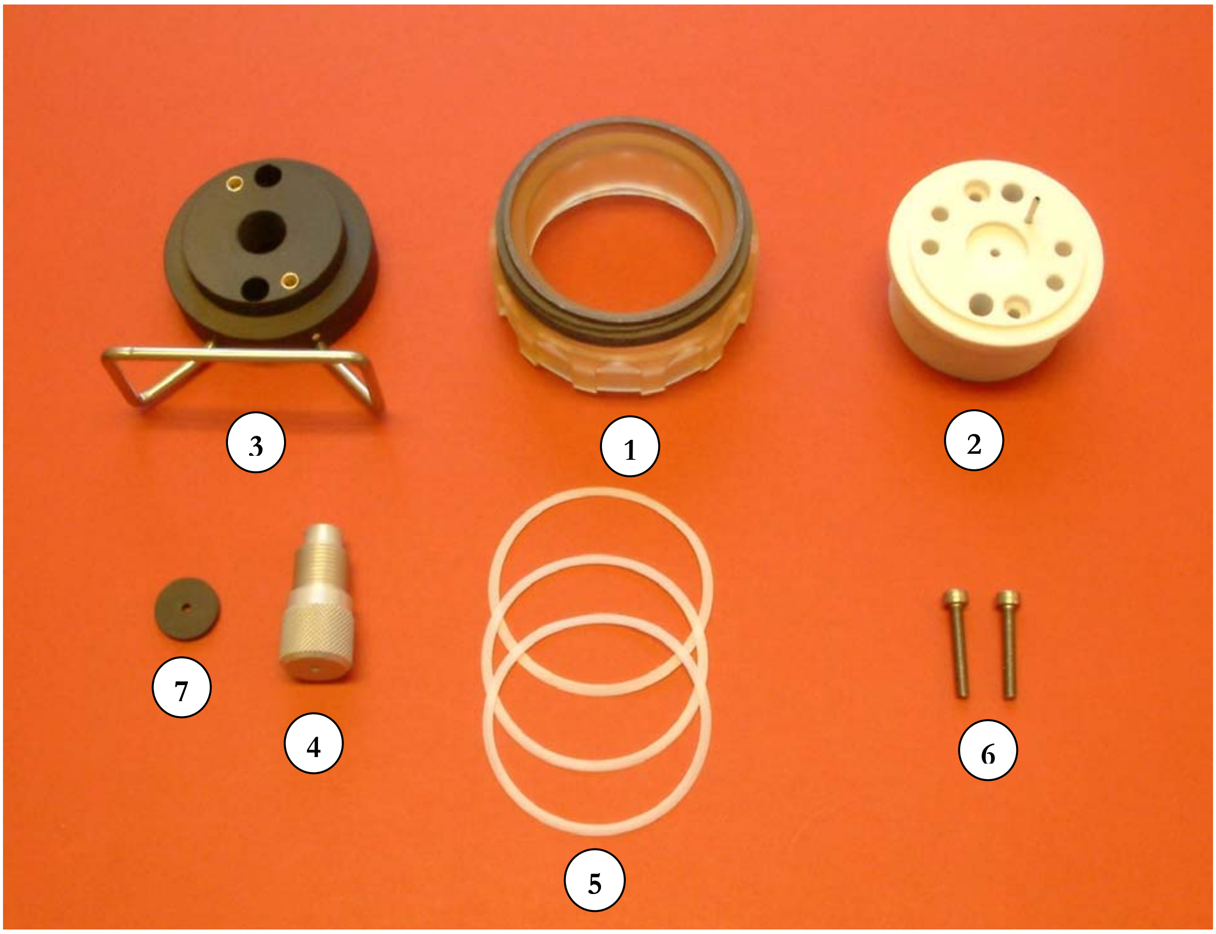
Chamber

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



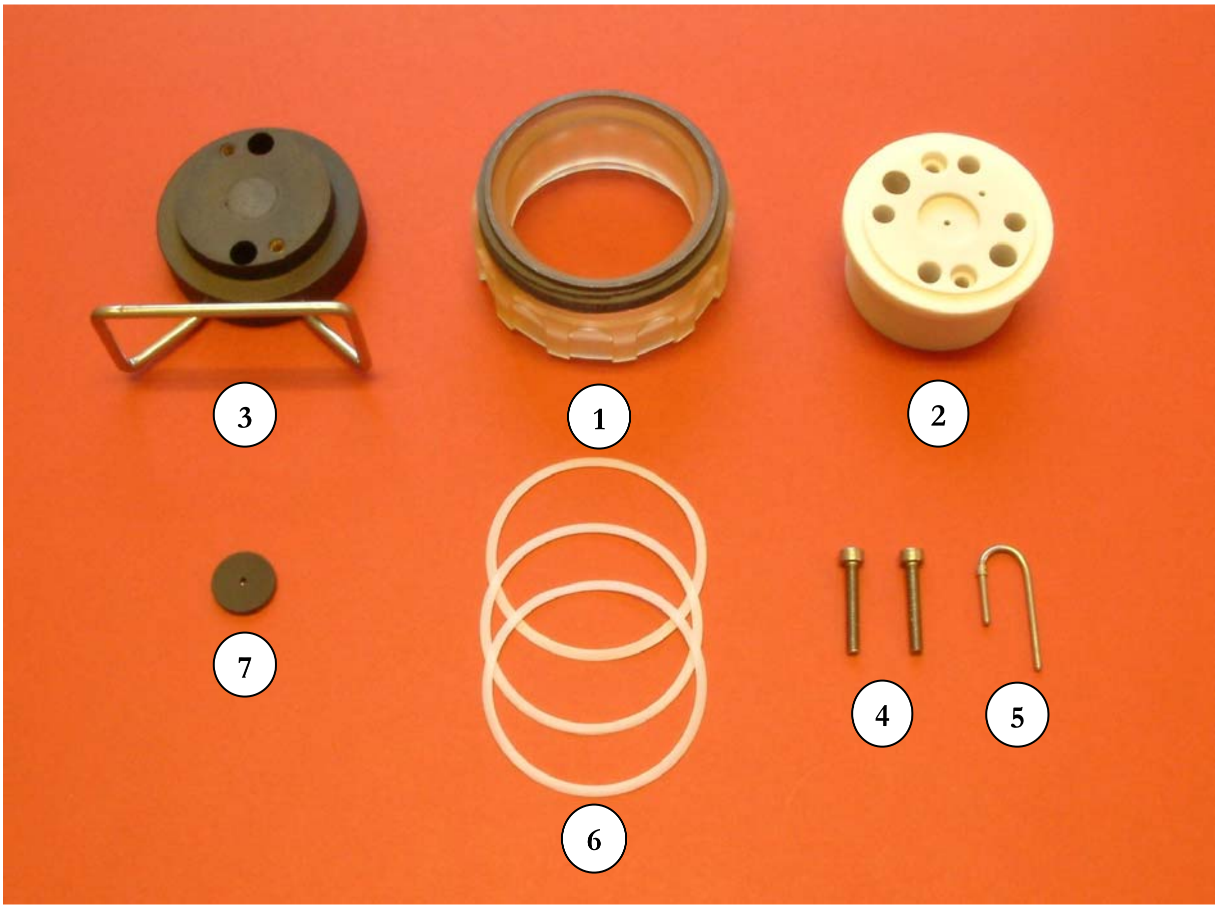
Probe

Item	Article number	Article
1	3000-02	Ring screw
2	3000-04	Adapter
3	3000-06	Back part
4	3000-08	Pt-holder, complete
5	3000-12 3000-14 3000-16	Holders Screws Contacts females
6	3000-18	Screws
7	3000-20	Gas tube
8	3000-22-05	Glide washer
9	3000-24-05	Septum, Py-prob



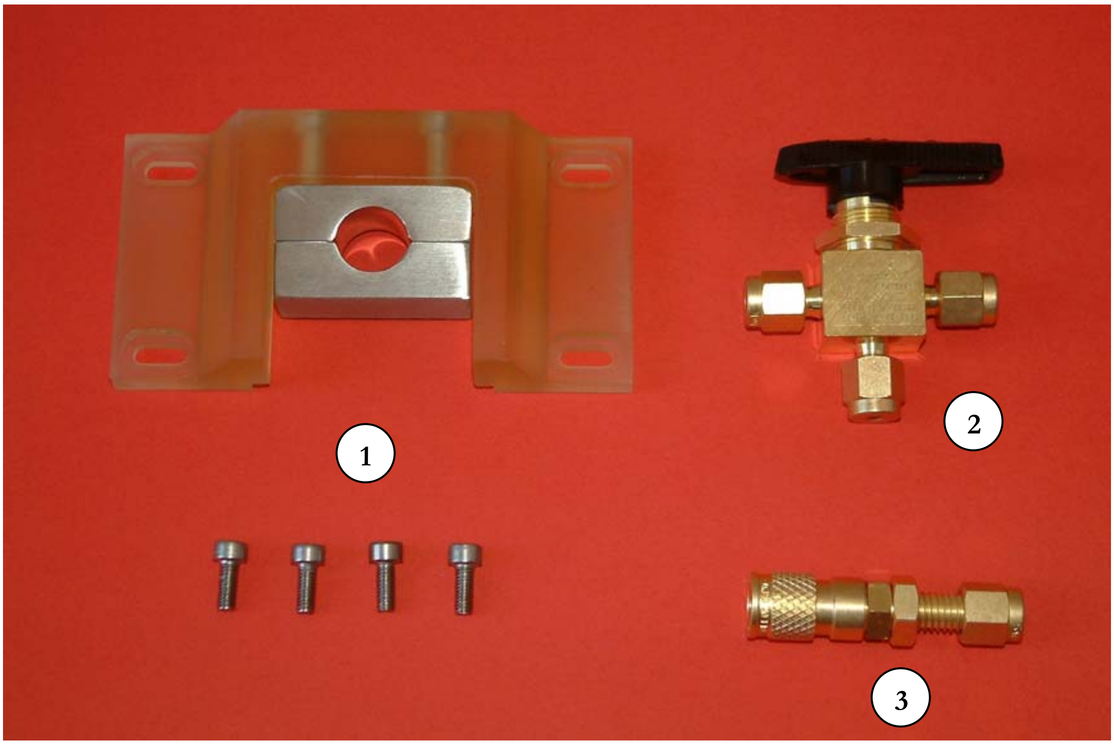
Calibration Probe

Item	Article number	Article
1	4000-02	Ring screw
2	4000-04	Adapter
3	4000-08	Back part
4	4000-10	Injection screw
5	4000-12-05	Glide washer
6	4000-14	Screws
7	4000-16-05	Septum, Cal.-probe



Tighten Probe

Item	Article number	Article
1	4100-02	Ring screw
2	4100-04	Adapter
3	4100-08	Back part
4	4100-14	Screws
5	4100-06	Gas tube
6	4100-12-05	Glide washer
7	4100-16-05	Septum, Tighten probe



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	1100-04	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