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ΕΡΓΑΣΤΗΡΙΟ

«ΗΛΕΚΤΡΟΝΙΚΕΣ ΔΙΑΤΑΞΕΙΣ ΦΑΣΜΑΤΟΣΚΟΠΙΑΣ»

ΕΡΓΑΣΤΗΡΙΑΚΗ ΑΣΚΗΣΗ 5:

“ΕΓΚΑΤΑΣΤΑΣΗ – ΛΕΙΤΟΥΡΓΙΑ – ΔΙΑΤΑΞΗΣ
ΘΕΡΜΟΒΑΡΥΤΙΚΗΣ / ΘΕΡΜΙΚΗΣ ΑΝΑΛΥΣΗΣ
ΥΛΙΚΩΝ”

Α.ΚΑΝΑΠΙΤΣΑΣ

Καθηγητής

Στην παρούσα εργαστηριακή άσκηση θα μελετηθεί ο τρόπος εγκατάστασης, η υλοποίηση της κατάλληλης συνδεσμολογίας και η λειτουργία της διάταξης Θερμοσταθμικής Ανάλυσης (ταυτόχρονης Θερμοβαρυτικής και Θερμικής Ανάλυσης Υλικών(TGA/DTA) **STA 503 BAEHR**.

Παρατίθενται παρακάτω οδηγίες και τεχνικά χαρακτηριστικά απο το εγχειρίδιο χρήσης της διάταξης.

**Manual for the operation of a
BÄHR - STA / TGA**

**STA503
Simultaneous Thermal Analysis**

**TGA503
Thermal Balances**



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1.0 Introduction

We are delighted that you have decided to purchase a BÄHR STA /TGA respectively.

So that you have pleasure with your STA and TGA respectively right from the beginning, we provide you with this manual which should enable you to quickly set up the measuring instrument, to put it into operation and to carry out an initial sample measuring.

Before putting into operation such a high precision measuring instrument, it is a matter of course to take into account some points.

But don't worry, you will see that it will be very easy for you to start a sample measurement very soon after you had followed these instructions.

Therefore it is the best to look into this manual entirely and then following the various chapters, step by step:

- ☞ **Unpack the equipment**
- ☞ **Check the consignment of completeness**
- ☞ **Mount the equipment**
- ☞ **Carry out all necessary connections and cabling**
- ☞ **Install the measuring system**
- ☞ **Install WinTA software**

Please use this manual always in connection with the manual **General Software Description WinTA** which is also included in the delivery and which contains the precise description of the software modules. Before carrying out a sample measurement, please read the chapter **Data Record**.

Now you should be able to

- ☞ **Carry out a sample measurement**

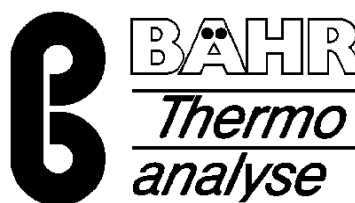
Should you have any further questions, suggestions of problems, please do not hesitate to contact our hotline.

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2.0 General Description

A Simultaneous Thermal Analysis serves the measurement of a change in mass and temperature (ΔG and ΔT) of a sample as a function of temperature (T).

For this purpose a sample and an inert sample are subjected to a defined temperature program (isothermal or dynamic). The resulting changes in mass as well as the heat turnover from physical conversion respectively chemical reaction and the prevailing temperature are recorded at a given time.

The Thermal Balances focus on the measurement of the change in mass (ΔG) and the temperature (T) of a sample.

The complete system may be operated under air, vacuum, statically respectively dynamically inert and many reactive gas atmospheres.

Measured and calculated parameters:

- ☞☞ Content determination
- ☞☞ Glass conversion temperature
- ☞☞ Decomposition temperature
- ☞☞ Temperature stability
- ☞☞ Oxidation stability
- ☞☞ Transformation temperature
- ☞☞ Enthalpy
- ☞☞ Phase diagrams

The measured and calculated values can be graphically represented in various axis coordinations in the software module **Evaluation**. Furthermore there are tabular representations available as well as a variety of functions for the evaluation. For this please look into the manual **General Software Description WinTA** which is also included in the delivery. Here you will find a detailed description of how to **Carry out a measurement**.

Areas of operation of STA503

Polymers
Clay
Glass, ceramics
Building materials, cement, plaster
Metals and their alloys
Organic and inorganic chemistry

3.0 Technical Specifications

3.1. Balance

Sample weight:	1 g (excluding sample crucible)
Tara range:	1 g (electric)
Measurement range:	250 mg or 1g
Resolution:	1 µg
Noise:	2 µg

3.2. Furnace

Temperature range:	Room temperature RT - 1500 °C
Heating rate:	0,01 - 100 K/min
Cooling time:	1500 °C from 100 °C in 15 min

3.3. Sensor

Crucible:	max. outer diameter 6 mm
Resolution:	? T 0,01 °C; T 0,1 °C

3.4. Atmosphere

Vacuum 10^{-5} mbar
Air
Inert gas
Various reactive gases

Please check the **completeness of the consignment** with regard to the ordered configuration.

- ☞☞ Measuring instrument
- ☞☞ Measuring system and crucible
- ☞☞ Transformer
- ☞☞ Computer, if ordered
- ☞☞ Software
- ☞☞ Peripheral equipment, if ordered (e.g. printer)
- ☞☞ Cable links (see **Mounting and Connecting of the components**)
- ☞☞ Manuals (**General Software Description WinTA, Instructions for First Use, Description of Database**)
- ☞☞ Dongle card, if the computer is not contained in the ordered configuration
- ☞☞ Reference material

Dongle card 6113

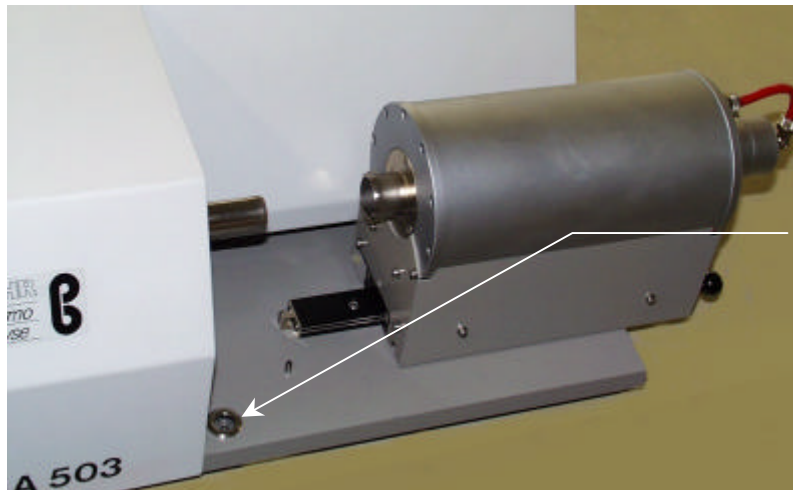


If the ordered configuration does not contain the computer and if the control board has design Interface 6113, then the Dongle card has to be installed in the computer (free ISA slot). The card needs the free basis address HEX 300 with address space from HEX300 to HEX 320.

4. Checking the location for the equipment and the necessary connections

4.1. Location

Locate the measuring instrument on a safe place without vibration, preferably on a balance table.



Point the equipment with the help of the level, so that it is perpendicular.

4.2. Power supply

230 Volt
50/60 Hz
protected with 16 Ampere

4.3. Water supply

Pressure: 3 bar
Max. 60 Liter/Hour
Pre-switched filter required, filter effect 50 μ
Connecting measurement for fabric hose:
6 mm inside diameter

4.4. Gas supply

(if required)

connecting measurement for fabric hose:
6 mm inside diameter

4.5. Fuses

6,3 A built-in fuse in plug of mains cable
(POWER SUPPLY)

4.6. Environment

Room-temperature of placement:
20 °C \pm 2 °C

5. Setting the various configurations

The equipment is delivered in various versions according to the ordered configurations.

For this reason we only represent the most important variants as an example:

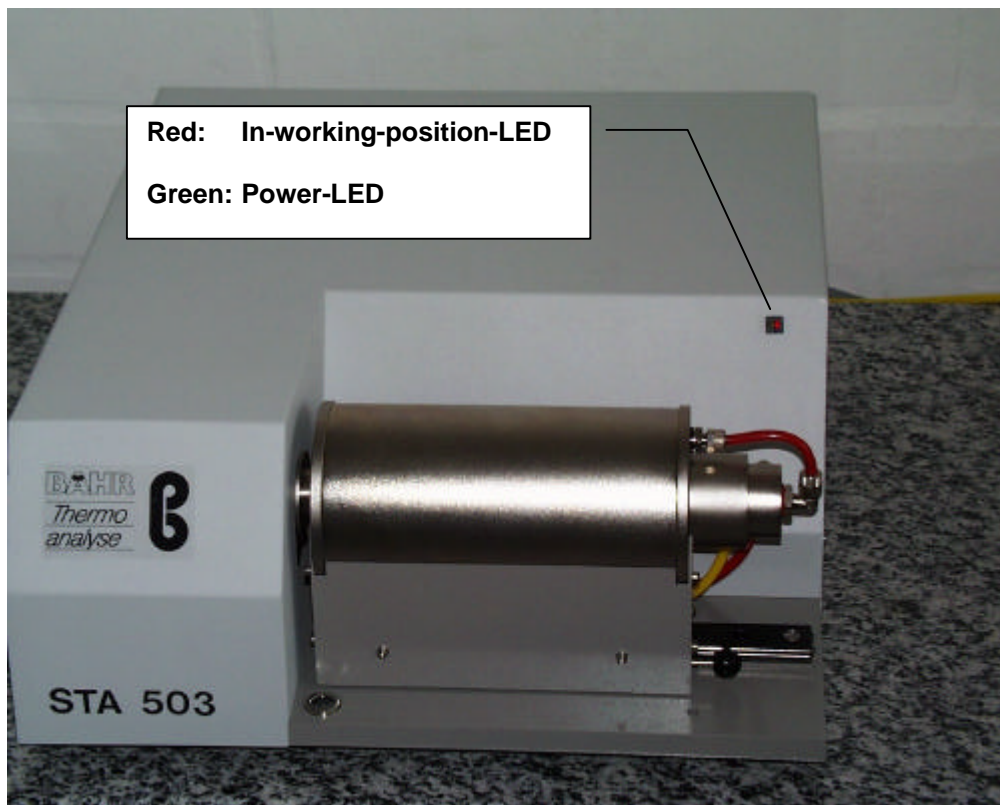
Control board:

Constructional form 1: Connected with the computer via a Dongle card 6113. The Dongle card has to be installed in the computer before.

Control LED's for Power (green) and In-working-position (red) at the front face.

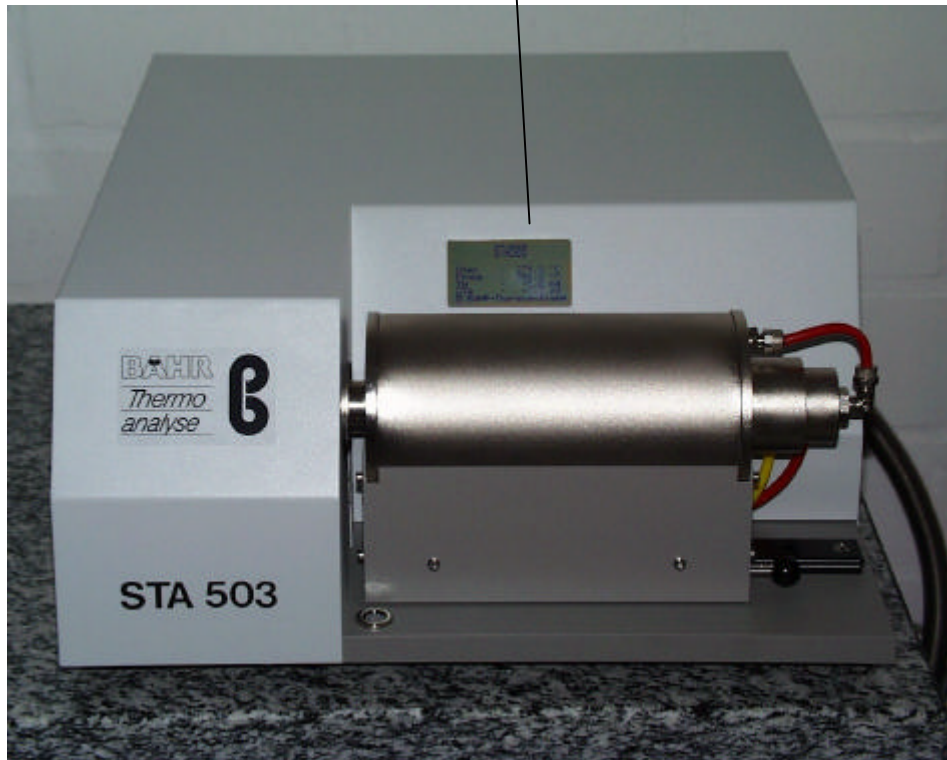
Important:

The computer and the control board may only be switched on and off combined (for instance by a socket board)!



Constructional form 2: Connected with the computer via a serial interface. This constructional form has a built-in micro controller and an own display that shows the actual operating condition as well as the current measurements.

LCD-Display with current operating condition, current measurement values etc.



5.1. Weighing system

The weighing system, the heart of the STA503, is positioned at the side of the furnace.

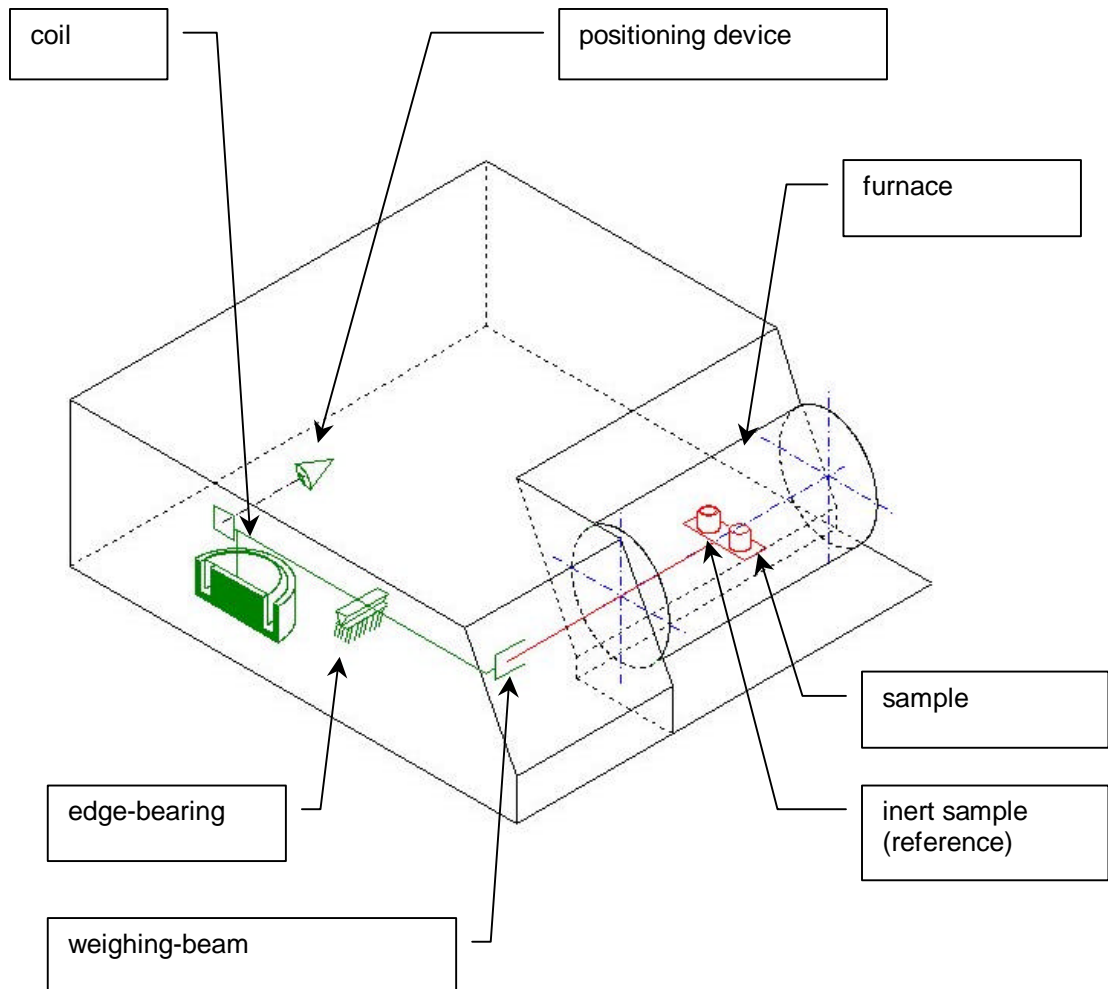
The sample weight is max.1 g, the measurement range covers 250 mg to 1 g respectively, depending on model specifications.

Due to a edge bearing the electromagnetic compensation balances achieve a sensibility of 1µg and a resolution of 10^{-6} .

The very good long-term stability of this balance is maintained even at high temperatures.

The Tara and the position of the weighing beam are adjusted automatically. Due to the volume-compensating design and the vacuum-tight casing (10^{-6} mbar) as well as the small sample space, changes in gas weight are insignificant. The lateral sample space design position reduces disturbances such as heat buoyancy, thermal noise and noise by gas entry.

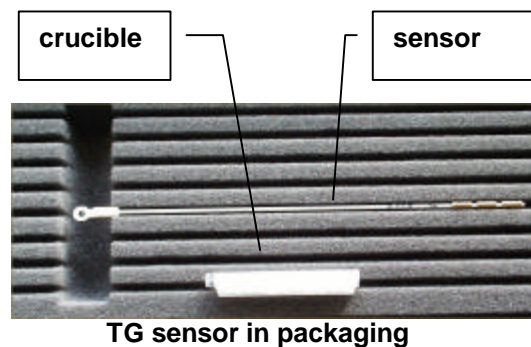
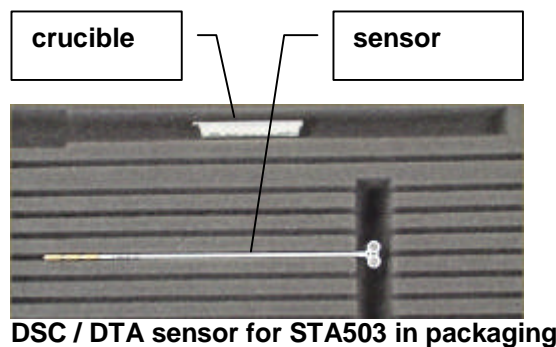
5.2. Principle construction of the weighing system:



There are still older constructional forms where the sample and the inert sample are positioned one behind the other. In this case the sample is located at the front (on the right) and the sample at the rear (on the left), seen from the front of the furnace.

5.3. Sensor

For the most part the sensor is dispatched in a special packaging.



Installed DSC / DTA sensor for STA503

DSC / DTA sensor

The flat metallic sensor measures the temperature difference between sample and inert material, e.g. Pt or NiCr, to which the thermocouples are connected from underneath. The upper side is even and thus guarantees an appropriate heat transmission to the crucible.

Both sensors have a deepening that ensures exact positioning of the crucibles.

TGA sensor

The TGA sensor has only one sensor. The measurement is carried out in the sample's vicinity.

5.4. Crucibles

The choice of the sample crucible depends on your sample.

We offer a range of crucibles made of different materials, such as aluminum, gold, silver, platinum, Al₂O₃, graphite, etc.

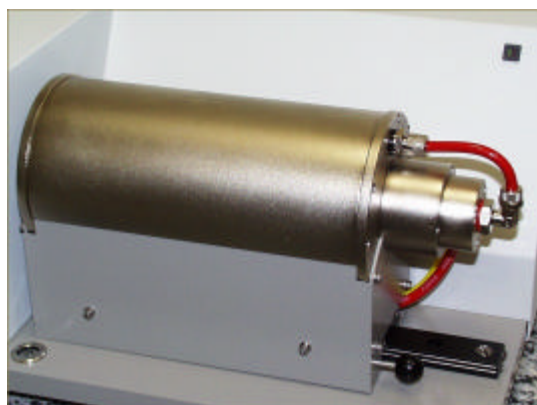
The volume of a standard crucible is approx. 0,8 to 0,11 cm³ with an outer diameter of 6 mm.

5.5. Transformer:



The transformer serves the transformation from 230V to low voltage.

5.6. Furnace



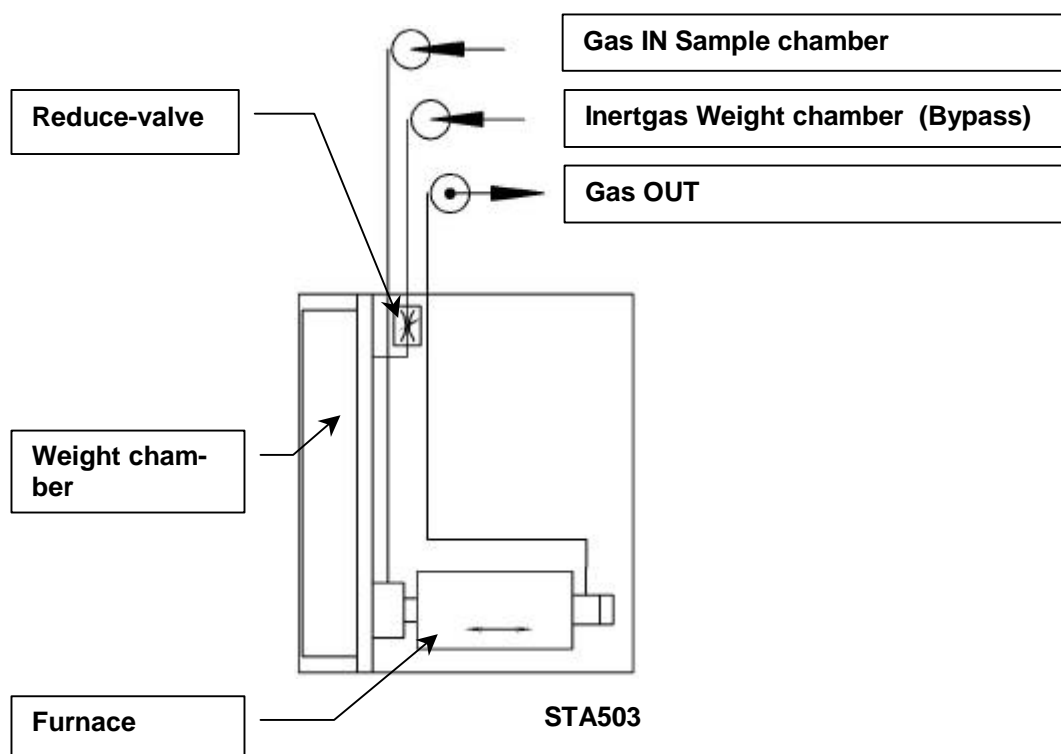
The bifilar-coiled furnace is determined for use in the temperature range from 20 °C - 1500 °C. The heating rate is at max. 100 K/min.

Due to the water-cooling the cooling time from 1500 °C to 100 °C is around 15 min.

The control thermocouple is directly positioned at the heating coil and thus guarantees exact temperature control. In the case of disturbance, e.g. break of the thermocouple or insufficient water flow, the furnace is shut down immediately.

5.7. Atmosphere

The STA503 may be used in vacuum (10^{-5} mbar) as well as in inert gas and a choice of reactive gases. The gas for the sample is led directly into the small sample chamber and there it can create a defined atmosphere without delay. A pre-switched gas box controls and regulates the gas flow. Changes of gas weight including vacuums and floods are set by control tracks from the software.



5.8. Extensions

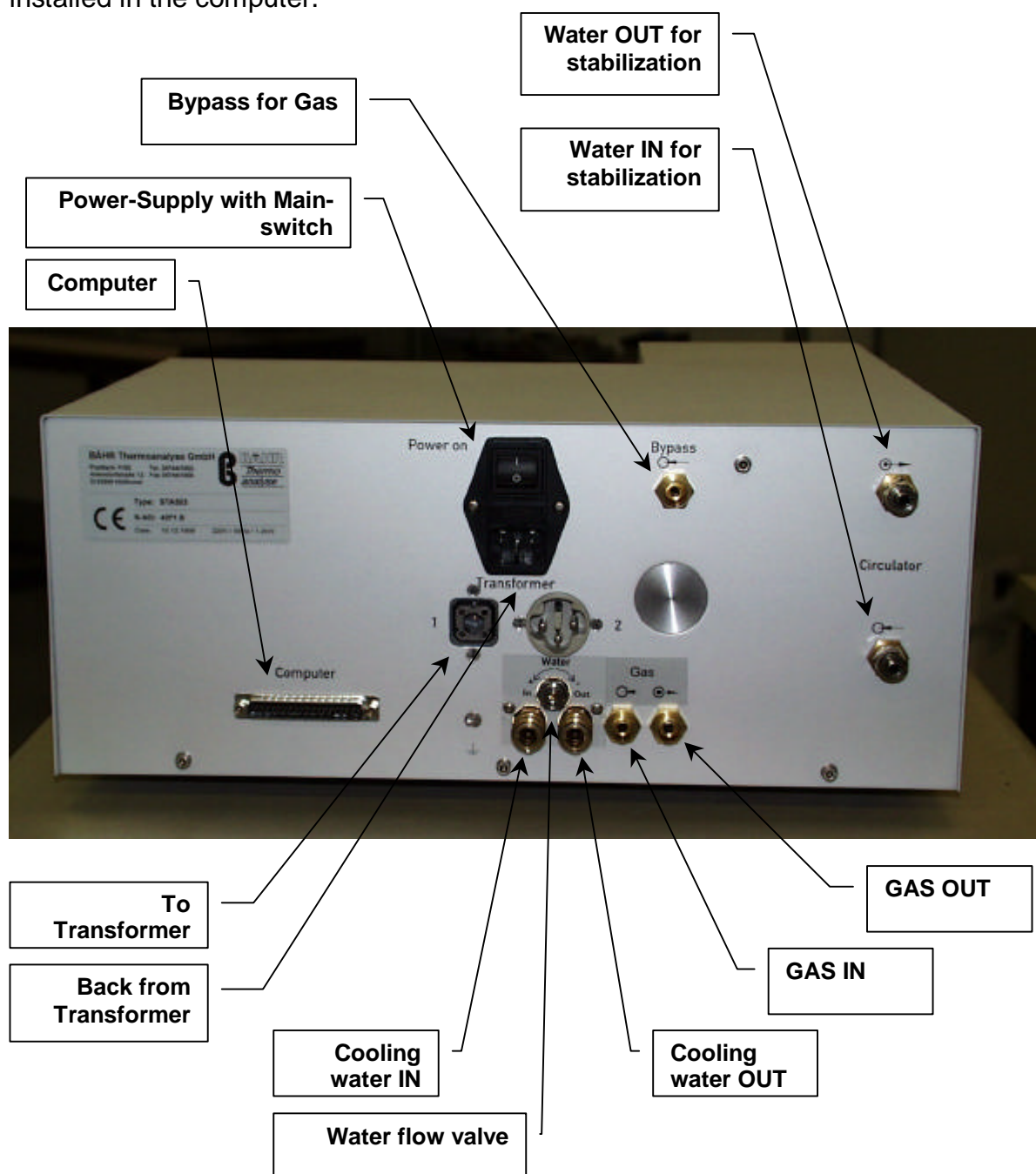
Despite the comprehensive basic equipment further extensions are possible without major efforts, e.g. adding a mass spectrometer, FTIR and sample changer. Furthermore the combination with other systems of thermal analysis such as Dilatometer, TMA or DSC is possible.

6. Mounting and connecting of the components

Here you also have to distinguish between the various constructional forms of the control board.

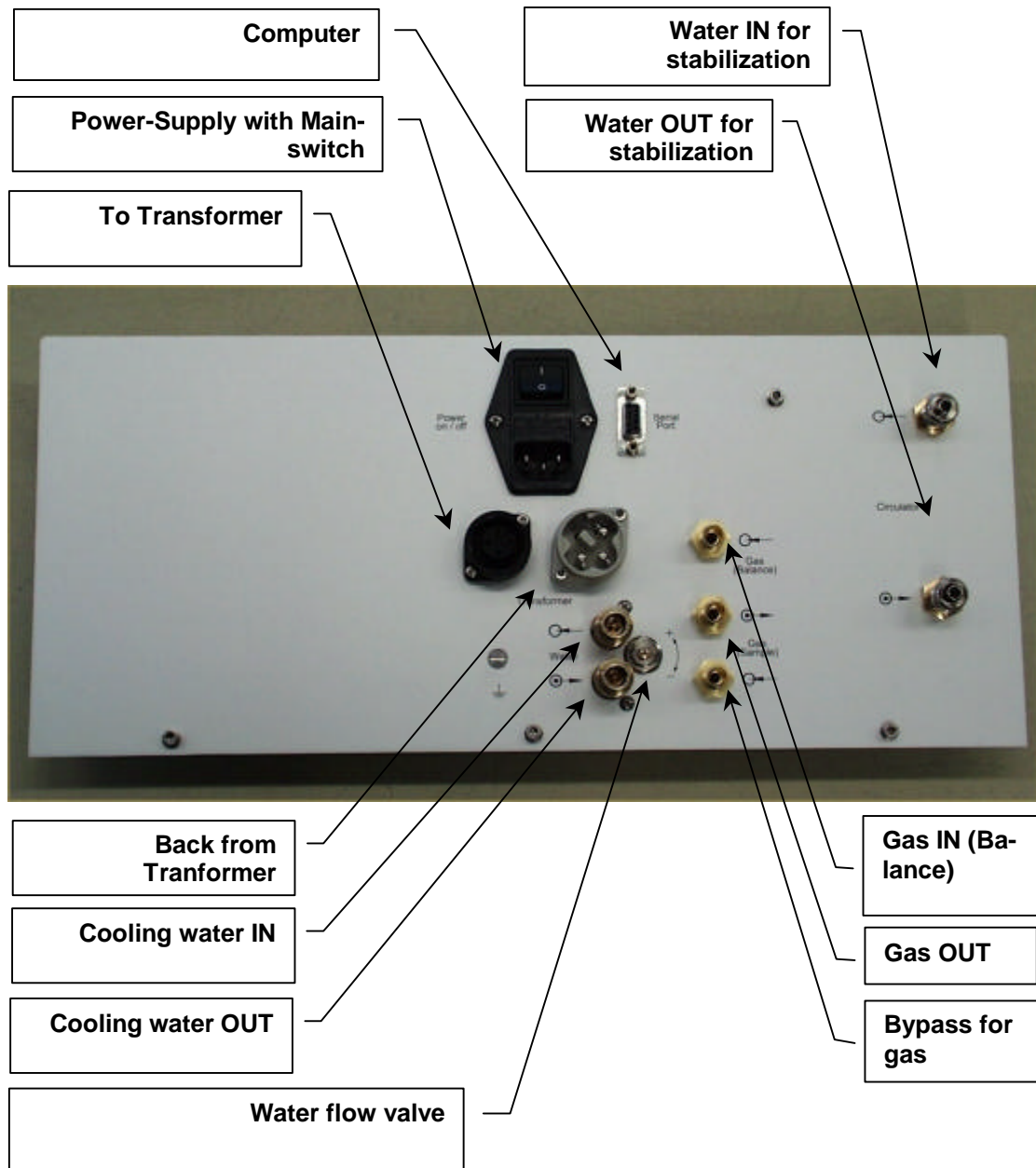
Control board, constructional form 1:

Connected with the computer via a Dongle card 6113. The Dongle card has to be installed in the computer.



Control cabinet constructional form 2:

serial interface connected to computer



7. Initial switching on of the equipment

Before initially switching on the equipment, please check all connections to the control board, as described in the previous chapter.

Important for constructional form1:

The computer and the control board always have to be switched on and off combined!

This can be managed for instance by using a socket board!

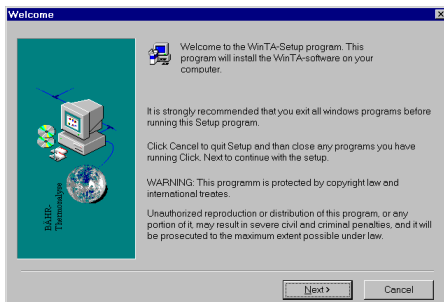
After the computer has run-up the WinTA software can now be installed with the enclosed WinTA CD.

8. Install WinTA software

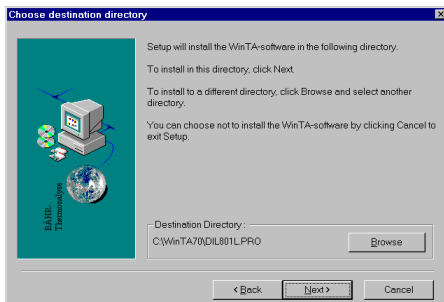
Please call the setup-program on the WinTA CD.

You will find Setup.exe in the directory <Equipment>\Disk1 on the CD, whereas <Equipment> has to be regarded as a variable for the type of equipment you want to install (here e.g. „STA503“).

Please follow the Setup-dialogue:



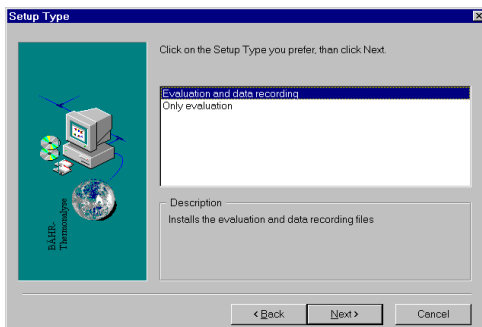
**Setup dialogue 1:
Welcoming**



**Setup dialogue 2:
Choice of install path**

Select the install path.

Standard: Taking over the suggested install path.

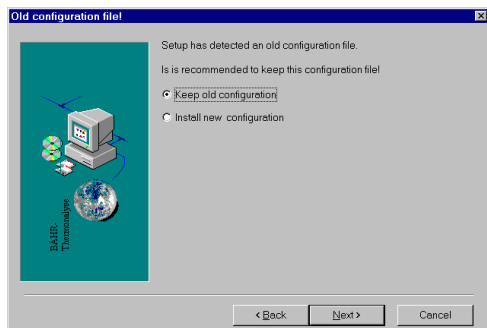


**Setup dialogue 3:
Optional installation
of data recording**

The software can be installed with or without data recording. Without data recording the software is, e.g. installed on evaluation computers or it is installed whilst updating the computer.

Standard: Installation with data recording

Please note: If you want to install the software on another computer you will need a second license and a Dongle card.

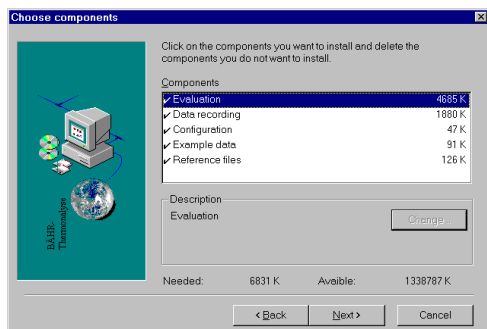


Setup dialogue 4:
Optional taking over of possible existing configuration

This message will only appear in case of an existing older version of the measuring instrument, that is to be installed on the target computer.

As a rule the existing configuration is to be taken over with an update. You will be separately informed about possible deviant rules.

Standard: Taking over the existing configuration

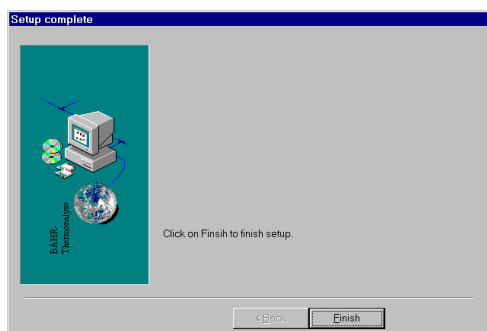


Setup dialogue 5:
Summary of the install settings

Here please check the selected install settings.

Additionally you will see the required and available storage space on the target drive.

After this the necessary files are copied and possibly required configuration settings are carried out.



Setup dialogue 6:
Conclusion of the setup dialogue

After conclusion of the setup dialogue you can start the program by clicking the corresponding program icon of the **WinTA** program group.

The software contains the following modules:

- ☞☞ Evaluation
- ☞☞ Data record
- ☞☞ Temperature program generator
- ☞☞ Data base
- ☞☞ Configuration

You will find more detailed information concerning the architecture and the operation of the WinTA software in the manual **General Software Description WinTA 6.0**, which is included in the delivery.

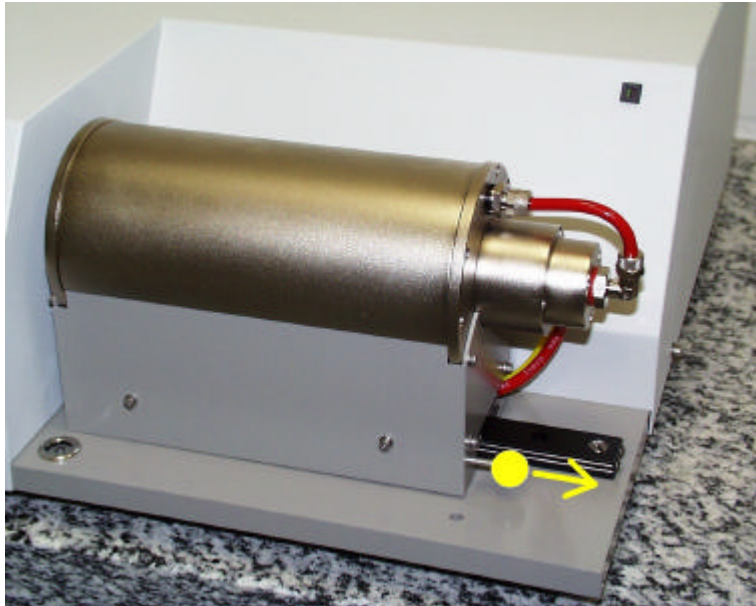
In addition you will find a separate manual which describes the database functions.

Furthermore you can call the online-help for nearly every program item which is available either via the **Menu item Help** or (in dialogue boxes) by clicking the corresponding **Button Help**.

Please note that the software can only be started if the Dongle card 6113 (control board constructional form 1) has either been installed in the computer or if the control board (constructional form 2) has been connected to the computer by the serial cable.

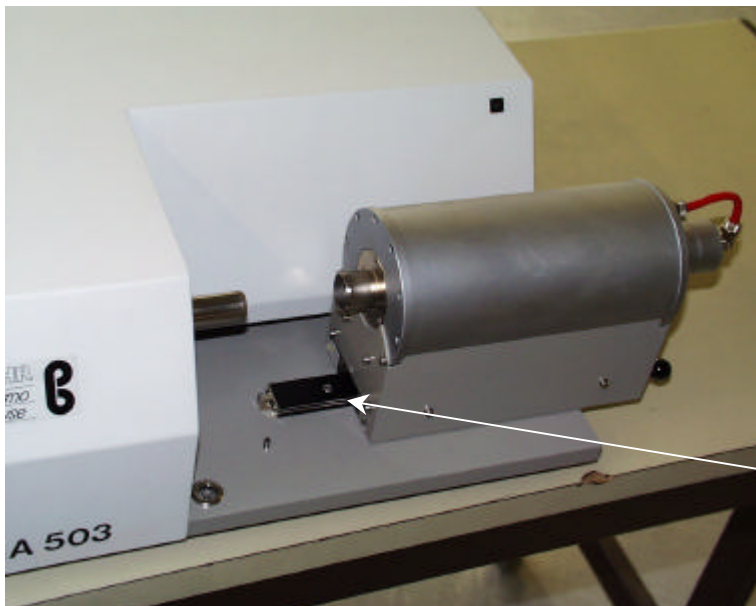
9. Operation

9.1. Installation of the measuring system

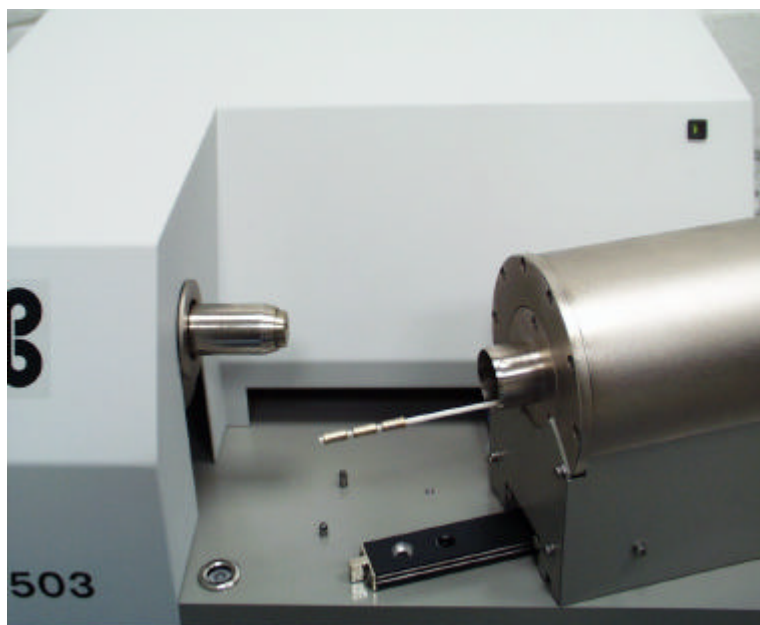


Firstly the furnace has to be opened in order to install the measuring system.

The furnace is fixed on a crosshead.
Draw the furnace with the knob to the right towards the direction of the arrow.



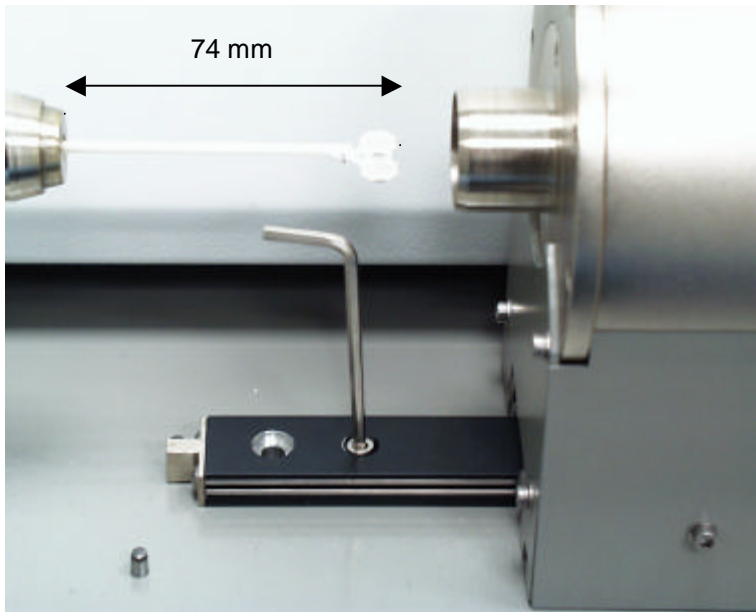
Afterward the furnace has to be turned to the front.
For this please undo the screw which is marked with the arrow and remove the screw.
Now turn the furnace to the front.



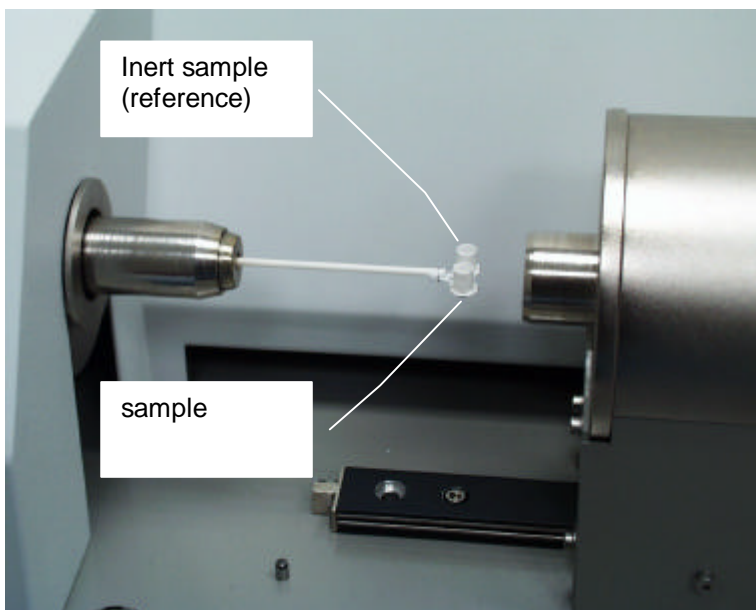
Carefully push the sensor with the crucibles at the head into the opening of the furnace. Then turn the furnace back into the initial position. Do not yet fix the furnace with the screw.



Now carefully push the sensor at the other side with the metal sleeve at the head into the measuring head.



Turn back the furnace until to the stop.
Then fix the furnace with the screw.



Put the crucibles on the sensor:
On the right: Inert-sample
On the left: Sample
(seen from the furnace)

In case of an older configuration, do not put the crucibles side by side but one behind the other.

Here applies:
At the front (to furnace):
Inert sample
At the back (to measuring head):
Sample

9.2. Correction of reference line by carrying out a dry run

The measurement results of a STA/TGA are incorrect due to tolerances of the component parts or physical influences. For the precise determination of the course of the test respectively the derived values, correcting values have to be created which are used to correct the measuring result.

For this you have to run an adjustment test. An adjustment test is a dry run which is carried out under the same conditions like the actual test. The test is carried out with empty crucibles.

Please carry out the dry run before the actual test. Use the same crucibles as in the test.

Please note the following points:

- same temperature program as in the actual test
- same starting conditions
- same crucibles
- same atmosphere

Important:

If you change the temperature program you have to carry out a new adjustment test. Even if you do not change the temperature program you should also regularly carry out a new adjustment test since the material properties (e.g. a different crucible material, a different crucible form, etc.) could have been changed.

In the manual **General Software Description WinTA 6.0** which is included in the delivery, you will find a detailed description of how to carry out an **Adjustment test** and you will also find the uncorrected respectively corrected evaluation of measurement data.

9.3. Calibration

The calibration is carried out by calibration substances as listed in the next chapter. The function $K(T)$ is determined by the calibration, and this function enables a scaling of the transitional enthalpies to the weighted-in quantity. On carrying out a calibration, please take care that the calibration conditions are to the largest extent the same as in future measuring conditions.

This means:

- ?? Same temperature program
- ?? Same crucible material
- ?? Same sample geometry
- ?? Same kind of transition

The way of how to carry out a calibration and a peak area integration for the enthalpy determination is described in detail in the manual **General Software Description WinTA 6.0** which is included in the delivery.

9.4. Calibration material

The following table contains the melting points of various materials together with their thermal capacity under standardized conditions. Please note that the melting point also depends on the purity of the material (better and 99.9%) and the environmental atmosphere (normal pressure = 1013.25 mbar).

Material	Peak [°C]	Delta H [J/g]	Calibration T = Temperature calibration H = Enthalpy calibration
Naphthalene	80.3	147.0	T
Benzoic acid	122.4	147.4	T
In	156.6	28.5	T, H
Sn	231.9	59.5	T, H
Bi	271.3	53.1	T, H
Pb	327.5	23.1	T, H
KNO3	334.0	95.5	T, H
Zn	419.6	112.0	
Ag2SO4	426.4	51.0	T, H

9.5. Effect of the heating rate

Almost all Onset temperatures listed in the literature refer to conditions of equilibrium, i.e. the heating rate = 0.

An increasing heating rate shifts the signal towards higher temperatures. If the equilibrium is to be determined, you initially have to measure with different heating rates. After that the temperatures are graphically represented and the equilibrium temperature for = 0 is extrapolated.

Furthermore you have to take into account the fact that an increasing heating rate will cause both an increasing base width of the DTA signal and an increasing peak height.

Thus an increasing base width is synonymous with a decreasing resolution power.

10. Carrying out a sample measurement

You will find a detailed description of how to carry out a measurement in the manual **General Software Description WinTA** in the chapter **Data Record**, which is also included in the delivery.

Please read this chapter carefully before starting a sample measurement. The best is to use a well-known material so that you can check the result with the help of the example diagrams.

11. Evaluation of the sample measurement

As a current software upgrade you will find the file card **Log** in the module **Data Record**. Here the parameters of the last carried out measurement are indicated. Additionally a protocol file containing all carried out measurements is carried along and can either be printed by a mouse click or can be copied on a disc for troubleshooting purposes.

In this context the parameter **Abortion Reason** is mainly important and will inform you about the proper carrying out of the test (Abortion Reason = 0) or possible errors. In any case the program will indicate you the current reason for the completion via the dialogue box . So the file card **Log** can be mainly considered for the use of the subsequent troubleshooting.

The different abortion reasons can be displayed via the button ? on the file card and can also be printed.

The actual evaluation of the measurement is carried out in the software module **Evaluation**. Here you can see the both the „crude data“ such as time, temperature or change in lengths or the calculated values such as the differentiated DSC. Additionally there are various functions (such as tangents, text entries, etc.) available.

For this please read the **General Software Description WinTA** which was referred to repeatedly.

The software contains the module **Database** für the data management.

13. Troubleshooting

Whenever you should need help, our hotline is pleased to help you.

Before calling us please have the following information ready:

- ☞☞ Type of equipment
- ☞☞ Order picking number
- ☞☞ Serial number
- ☞☞ Software version and operating system

- ☞☞ Precise description of the case respectively of the problem (e.g.: is it possible to exactly reproduce the problem?)

- ☞☞ In case of software problems in the module Data Record: Abortion reason of the test (as described under *Evaluation of sample measurement*)

- ☞☞ **Attention: It is strictly necessary to pull out the power plug before inspecting fuses or cabling!**

- ☞☞ In case of hardware problems you firstly should inspect the 6.3A fuse which is installed in the plug (POWER SUPPLY) as well as the required connections (as described under *Mounting and connecting of the components*).

- ☞☞ For the troubleshooting we may probably require some information e.g. test data, the configuration file or the protocol file containing important parameters of the carried out test. Corresponding menu items are available for this. For instance, it is possible to copy test files on a disc or in a certain directory from the main menu via the menu item **Disk**. In the **Configuration** module you will find the item **Save Configuration Files** under **Options**. You can also save the current protocol file on a disc on the file card **LOG** (after starting **Data Record**). As speedily as possible we can get the required data via e-mail. Furthermore is also possible to exchange data via our FTP server or via disc.