METTLER TOLEDO STAR^e System

DMA/SDTA861^e

Operating Instructions

1 Preface and Safety Notes

Contents

1 Preface and Safety Notes

1.1 Preface

Thank you for choosing the DMA/SDTA861^e from METTLER TOLEDO. The DMA/SDTA861^e is a highly sensitive measuring instrument for dynamic mechanical analysis (DMA) and also offers the possibility of simultaneous differential thermal analysis. (SDTA). The DMA/SDTA861^e is part of the METTLER TOLEDO S**TA**R**^{e}** system.

In dynamical mechanical analysis (DMA) the dynamic mechanical behavior of a material sample is detected as a function of frequency and temperature. The sample mounted on the DMA/SDTA861^e is subjected to a predefined mechanical oscillation program, defined by frequency and amplitude, and a temperature program. Both the dynamic displacement of the sample and the dynamic force exerted on the sample during to the mechanical oscillation program are measured.

In simultaneous differential thermal analysis (SDTA) the difference between sample temperature and reference temperature is measured during the temperature program. Changes of chemical and physical material properties can be detected by readings of temperature changes.

For operation of the DMA/SDTA861^e and for display and evaluation of results you require version 7.0 or later of the S**TA**R**^{e}** software from METTLER TOLEDO.

The liquid nitrogen cooling system necessary for operating your DMA/SDTA861^e including liquid nitrogen Dewar and tubing is shipped with the instrument as standard accessory.

Address of manufacturer:

METTLER TOLEDO GmbH, Analytical

Sonnenbergstrasse 74

CH-8603 Schwerzenbach

Switzerland

1.2 Safety

This section contains information on the safe use of the DMA/SDTA861^{e}. All users of the DMA/SDTA861^{e} must read and observe the safety notes in this section.

1.2.1 Intended use

The DMA/SDTA861^{e} is intended for performing measurements in dynamic mechanical analysis.

All other use and operation beyond the limits of operation defined by METTLER TOLEDO without written consent from METTLER TOLEDO is considered as inappropriate.

♣ The limits of operation can be found in chapter 7, *Specifications*.

1.2.2 Safety notes

The DMA/SDTA861^{e} has been tested for the experiments and intentions documented in the appropriate operating instructions. However, this does not absolve you from the responsibility of performing your own tests of the products supplied by us regarding their suitability for the methods and purposes you intend to use them for. You should therefore observe following safety measures.

Measures for your protection

PC and DMA/SDTA861^{e}:

- Ensure that you plug the cable supplied into a receptacle outlet that is grounded! In the absence of grounding, a technical fault could be lethal!.

- Never work in an environment subject to explosion hazards! The housing of the instrument is not gas tight! (Explosion hazard due to spark formation, corrosion caused by the ingress of gases).

- The outer parts of the oven can become very hot which can ignite flammable gas mixtures.

DMA/SDTA861^e:

- Switch the instrument off and disconnect the power cable before you open the housing or change blown fuses! An electric shock could be lethal!

- The DMA/SDTA861^e is very heavy. At least four people should be available to lift it. Never try to lift the DMA/SDTA861^e alone – you could injure yourself.

- Never use gases, which may result in an explosive gas mixture! Explosive gas mixtures could produce an explosion!
- Never use combustible gases or explosive gas mixtures to purge the measuring cell! An explosion could occur!

- Never switch off the liquid nitrogen cooling when the temperature in the furnace is above 300 °C. The cooling of the module would no longer work and the surrounding of the cell could be heated unduly!

- Never touch the furnace halves or the clamping assembly during or immediately after a measurement. Always wear protective gloves or let the furnace and the clamping assembly cool down to ambient temperature before you open the furnace halves. Parts of the furnace and the clamping assembly can reach temperatures down to –150 °C or up to +500 °C. There is risk if burns.

- Make sure your fingers and other body parts are well clear of the clamping assembly and the furnace during the furnace closing process. There is risk of injury!

- Never remain in close proximity of the DMA module during a measurement without hearing protection. Always wear hearing protection when working near the DMA module. The DMA module can produce excessive noise that can impair your hearing. The noise level can exceed 85 dB(A) during certain measurements.

- Place the DMA module in a fume hood, when you measure substances which may produce toxic reaction gases !

- Check the set operating voltage before you switch on the instrument! The instrument may suffer damage if the operating voltage does not match the line voltage!
- Never switch off the instrument during power up! Otherwise problems occur during the next power up.
- Use only fuses of the type specified in the operating instructions!
- Never purge the measuring cell with a corrosive gas!
- Purge the measuring cell with an inert gas, when you are measuring samples which may produce corrosive reaction gases.

While mounting the sample in the clamping assembly:

- Never use excessive force to move the clamp. Exerting excessive force on the clamp could damage the force sensor, den displacement sensor or the drive motor.
- Do not drive the lower part of the adjustment aid mounted on the z-axis table too far up toward its upper part. The drive motor could suffer damage.

DMA/SDTA861^e and liquid nitrogen cooling:

- Always wear protective goggles and gloves when working with liquid nitrogen! It can cause severe burns on your skin.
- Make sure that you have been trained in the correct operation of the liquid nitrogen Dewar.
- Do not move the liquid nitrogen Dewar when measurements are in progress and if the electromagnetic valve is iced up! Frozen tubing could break and liquid nitrogen could flow out.
- Ventilate closed rooms as frequently as possible! High concentrations of nitrogen are dangerous and can cause suffocation.

- Always put the liquid nitrogen Dewar out of operation if it is not required for longer periods. There is always the danger that liquid nitrogen could escape uncontrollably.

- Lift and transport the liquid nitrogen Dewar using a forklift. Make sure the Dewar is supported from below.
- Never try to lift the liquid nitrogen Dewar with the handles or with hoisting loops wound round the outer part of the Dewar.

DMA/SDTA861^e and Gas ControllerTSO800GC/TSO800GC1

- Never use combustible gases or explosive gas mixtures to purge the measuring cell! An explosion could occur!
- Use the gas controller only with the specified gases. Explosive gas mixtures could produce an explosion!

Measures for operational safety

PC, DMA/SDTA861^e.

- Eliminate the following environmental factors:
  - strong vibrations,
  - strong draughts of air,
  - direct sunlight,
  - relative humidity at dewpoint
  - temperatures below 10 °C and above 31 °C
  - powerful electric or magnetic fields

FCC Rules and the Radio Interference Relation

This equipment has been tested and found to comply with the Limits for a Class A digital device, pursuant to both Part 15 of the FCC Rules and the radio interference regulations of the Canadian Department of Communications. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Cet appareil a été testé et s'est avéré conforme aux limites prévues pour les appareils numériques de classe A et à la partie 15 des règlements FCC et à la réglementation des radio - interférences du Canadian Department of Communications. Ces limites sont destinées à fournir une protection adéquate contre les interférences néfastes lorsque I' appareil est utilisé dans un environnement commercial. Cet appareil génère, utilise et peut radier une énergie à fréquence radioélectrique; II est en outre susceptible d'engendrer des interférences avec les communications radio, s'il n'est pas installé et utilisé conformément aux instructions du mode d'emploi. L'utilisation de cet appareil dans les zones résidentieIIes peut causer des interférences néfastes, auquel cas l'exploitant sera amené à prendre les dispositions utiles pour palier aux interférences à ses propres frais.

1.3 Typographic conventions

The following fonts and signs are used in these Operating Instructions to mark up certain items of the text:

Times New Roman

- Times New Roman font is used to mark text that appears on the screen of the computer (e.g. menu items, error messages, etc.).

ENTER

- Text in CAPITALS marks text that you have to enter via the keys on the PC keyboard or on the keypad of the DMA module

Rotate

- Text in **bold Arial Narrow** font marks up text that is displayed on the liquid crystal display (LCD) of the DMA module.

Italic

- *Italic font style* is used for cross references referring to titles and paragraphs within this document and in other documents.

♣

- The clover leaf ♣ signifies notes that contain additional information relating to the preceding or following text. This kind of text contains supplementary information and can help to understand the preceding or following text.

safety notes

- Safety notes are marked with safety triangles: The following warning triangles draw your attention to points concerning safety and danger. Ignoring this information could endanger the user and result in damage to the instrument and other malfunctions.

Risk of electric shock

Risk of explosion

Risk of fire

Risk of burns

Risk of injury

Caution

- Numbered paragraphs contain instruction steps in procedures. Example:

  (1) Loosen the Torx screws **6**, **7** of the clamp holder.

  (2) Pull the alignment insert carefully toward the right side out of the clamp holder, ❸.

- A check list with conditions that must be fulfilled to perform the following instruction procedure is included at the start of the procedure. Example:

  Start:
  - Alignment insert installed in large clamping assembly
  - Two halves of the furnace are fully open and swung to the rear

- Similarly, a check list with conditions that must be achieved at the end of the procedure is included at the end of the numbered steps. Example:

  End: Alignment insert removed from small clamping assembly.

furnace **10**

- Reference numbers in the text referring to figures or drawings are included in the text in bold font. The figure that is referred to can be several pages before or after the page containing reference number.

⇨*See Fig. 6.3*

- References to a figure to which the bold reference numbers in a procedure refer are located in the margin of the page at the start of the procedure and marked up by an arrow.

furnace arm, ❷

- Reference numbers in the text referring to pictures are included in the text as numerals in black circles. The picture that is referred to is usually on the same or opposite page as reference number.

1.4 Abbreviations

The following abbreviations are used throughout these operating instructions:

ALA	alignment aid
AAS	adjustment assembly
CA	clamping assembly
LCA	large clamping assembly
SCA	small clamping assembly
LCD	liquid crystal display on the DMA module

2 Design and Operating Principles

Contents

2 Design and Operating Principles

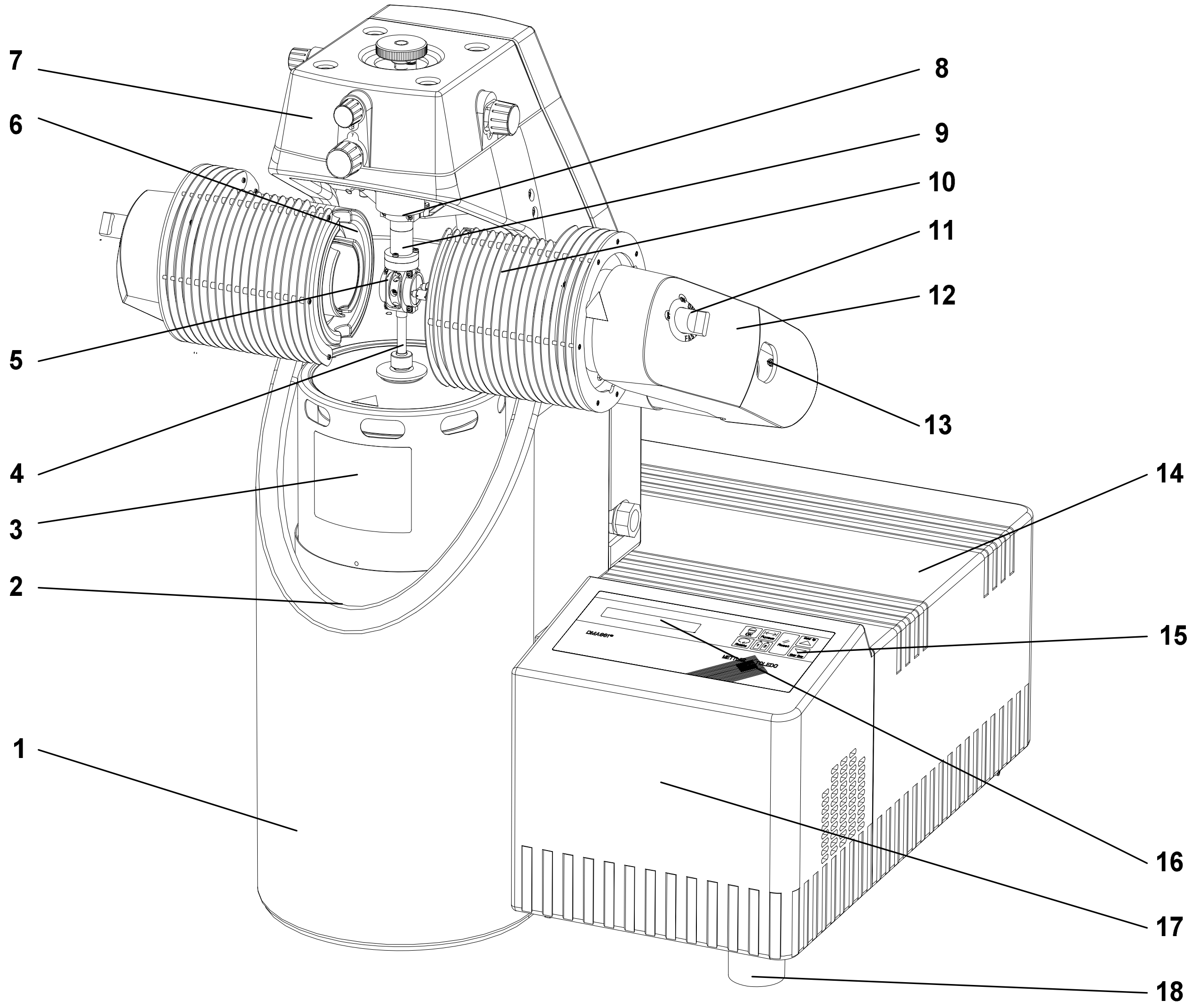
This chapter describes the design and operating principles of the DMA/SDTA861e.

2.1 Design

This section describes the design of the DMA/SDTA861e. It first presents an overview of the system and then describes the individual assemblies in more detail.

2.1.1 Overview

The following overview of the system shows the individual assemblies that make up the DMA/SDTA861e.



Parts

1	Stand	**10**	Furnace
2	Z-axis table	**11**	Fast cooling valve
3	Drive motor	**12**	Furnace drive
4	Drive shaft	**13**	
5	Clamping assembly	**14**	Module housing
6	Furnace insulation	**15**	Keypad
7	Four axes alignment device	**16**	Liquid crystal display
8	Force sensor	**17**	Front housing
9	Displacement sensor	**18**	Leveling screw

Fig. 2-1. Overview

2.1.2 Assemblies

The following sections describe the design of the individual assemblies of the DMA/SDTA861^e with the aid of the overview in Fig. 2-1.

Stand, 1

measurement system

The stand is on the left-hand side at the front of the DMA/SDTA861^e and contains the measurement system assemblies. The stand determines the height, and characterizes the appearance of the DMA/SDTA861^e. There is a wide opening to the front of the DMA/SDTA861^e.

Drive motor, 3

The drive motor is mounted below the clamping assembly and projects upward out of the stand opening. This makes it easily accessible from the front side of the instrument.

Clamping assembly and clamp, 5

clamping assembly and clamps

The clamping assembly with the clamp is situated between the drive motor and the four axes alignment device. Different combinations of clamping assembly and clamps are used depending on measurement mode.

The **clamping assembly** consists of the following components:

- drive shaft
- clamp holder
- displacement sensor core

There is a small and a large clamping assembly. Depending on the sample size and the measurement mode, the small or the large clamping assembly is used. The clamp is mounted in the clamp holder of the clamping assembly. Both the small and the large clamping assemblies allow you to use different **clamps** for the various types of measurement:

- The small clamping assembly allows you to perform shear measurements, compression measurements and tension measurements with films and fibers. For each type of measurement there is a suitable clamp.
- The large clamping assembly allows you to perform 3-point bending and dual cantilever measurements, as well as compression and tension measurements on films, fibers and rods. For each type of measurement there is a suitable clamp.

Furnace, 10

furnace halves

heating and cooling assemblies

cooler

The furnace consists of two halves, each of which is fixed to an extendable furnace arm on the side of the DMA/SDTA861^e. The furnace is opened by moving the furnace arms outward. The two halves of the furnace are symmetrically arranged and include heating and cooling assemblies as well as an insulating cover. The cooling element is called the “cooler”.

When the furnace is closed, the two halves of the furnace completely enclose the clamp.

fast cooling device

The two **knobs of the fast cooling device, 11** are located on the outer sides of the furnace arms. The fast cooling valves are used to rapidly cool the clamping assembly and sample to low start temperatures before the actual measurement

Furnace drive, 12

The furnace drive is a mechanism that consists primarily of the two arms of the furnace support, a rack and three guide rods. The actual furnace is mounted on the furnace arm.

Furnace insulation, 6

The furnace insulation encloses the heating element. It consists of two symmetrical mirror-image halves.

Gas tubing

The gas tubing and the manifold under the module housing on the left side of the DMA module are part of the liquid nitrogen cooling system of the furnace. Two lengths of tubing connect the furnace halves with the manifold. A third length of tubing leads away from the manifold to the liquid nitrogen cooling Dewar.

Force sensor, 8, and displacement sensor, 9

The **displacement sensor** is mounted above the clamp assembly, and can be seen in the opening of the stand.

The **force sensor** is mounted between the displacement sensor and the four axes alignment device.

Four axes alignment device, 7

knobs

The four axes alignment device is mounted at the top of the stand. It has two adjustment knobs on the front side and one knob each on the left and right sides for adjustment of the x and y positions and the α and β angles. In operation, for example when the sample holder is changed, only x and y usually need to be re-adjusted. The DMA module is therefore delivered with the two adjustment knobs the α and β angles removed to prevent unintentional misalignment. The knobs are shipped with the instrument and can be mounted when required.

Z-axis table, 2

spindle

The z-axis table is mounted below the in the stand housing and cannot be seen from outside. It consists essentially of a platform on which the drive motor is mounted, a threaded roller spindle, a housing with guide rods and a stepper motor with electronic control system.

Module housing 14 und front housing, 17

The housing of the module consists of two parts: the front housing and the module housing. The module housing accommodates the electrical supplies for the furnace heating system and the measurement system, as well as electronic components for the control of the measurement system.

The keypad and the liquid crystal display are mounted on the front housing. Electronic components for current and voltage conversion and the drive motor are also accommodated here.

Keypad and liquid crystal display, 15, 16

The keypad consists of function keys that are labeled according to their function. The liquid crystal display (LCD) is situated on the left side of the keypad. It displays the measurement values, messages and the status of the measuring cell.

Electronic units and connections on the rear panel

Various **electronic connections** are mounted on the rear panel of the DMA module. Fig. 2-2 shows the rear panel of the DMA module with the furnace supply, electronics supply, module electronics and DMA measuring electronics units.

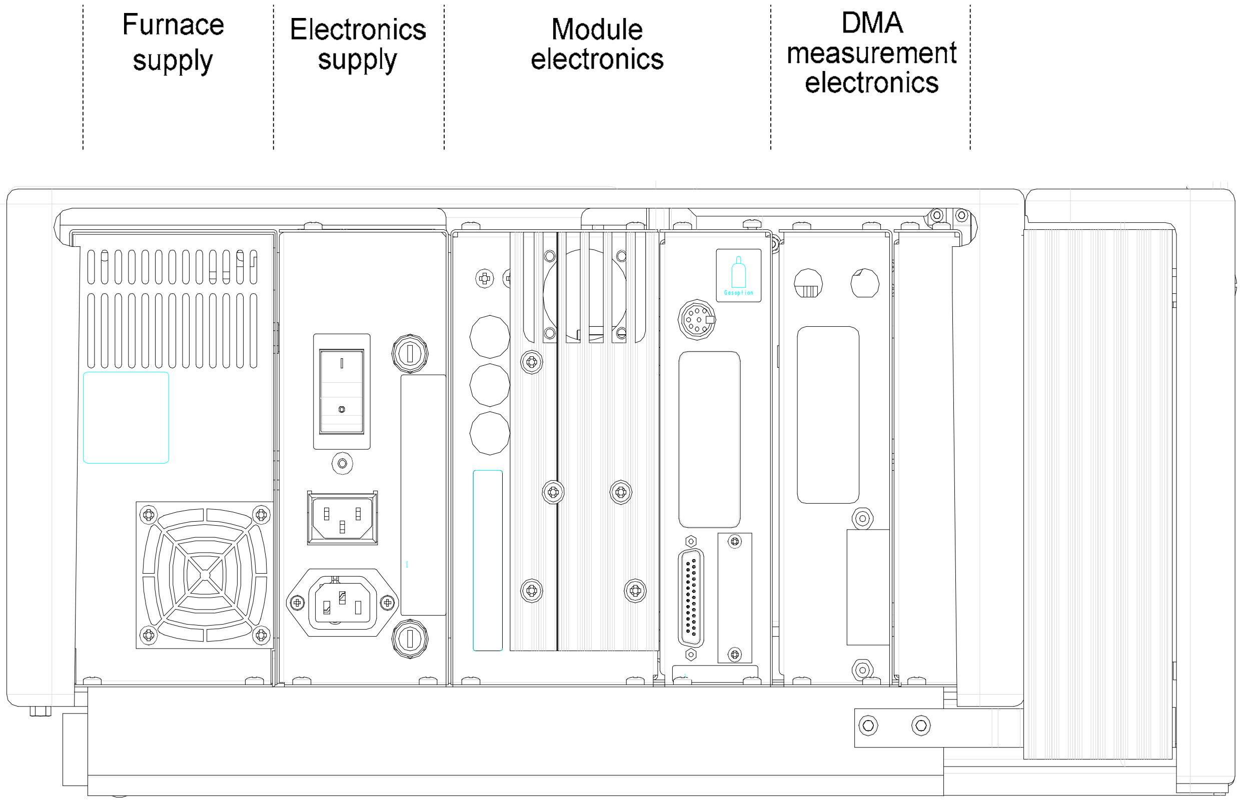


Fig. 2-2. Rear panel of the DMA module

The DMA/SDTA861e power supply system consists of two parts: the **electronics power supply** and the **furnace power supply**. The power supply system is located in the module housing.

The electronics power supply is set to either 115 V or for 230 V AC (60 Hz or 50 Hz) during manufacture in the factory. The setting cannot be changed afterward.

The furnace power supply automatically recognizes the power outlet voltage and adapts itself accordingly.

The DMA/SDTA861e has the following fuses:

Fuse	for 115 V	for 230 V	Information
SI 1	3.15 AT	1.6 AT	Fuse for line output via power switch
SI 2	6.3 AT	3.15 AT	Fuse for line output via switched line socket

2.2 Operating principles

The DMA/SDTA861^e is an instrument designed for dynamic mechanical analysis: It measures the dynamic mechanical behavior of materials as a function of temperature, e.g. the complex modulus of elasticity and the complex shear modulus.

By the term **elasticity** we mean the way in which materials change their shape through the action of external forces. The **modulus of elasticity** of a material is the ratio of the mechanical stress to the relative deformation.

In **Dynamic Mechanical Analysis**, DMA, a sample is subjected to a sinusoidal mechanical deformation of frequency, f, and the corresponding forces measured. Conversely, the sample can be subjected to a defined force amplitude and the resulting deformation measured.

This section describes the operating principles of the DMA/SDTA861^e and explains the function of the individual assemblies.

2.2.1 Operation of main assemblies

The diagrams below include the main assemblies and show the design principles of the DMA/SDTA861e.

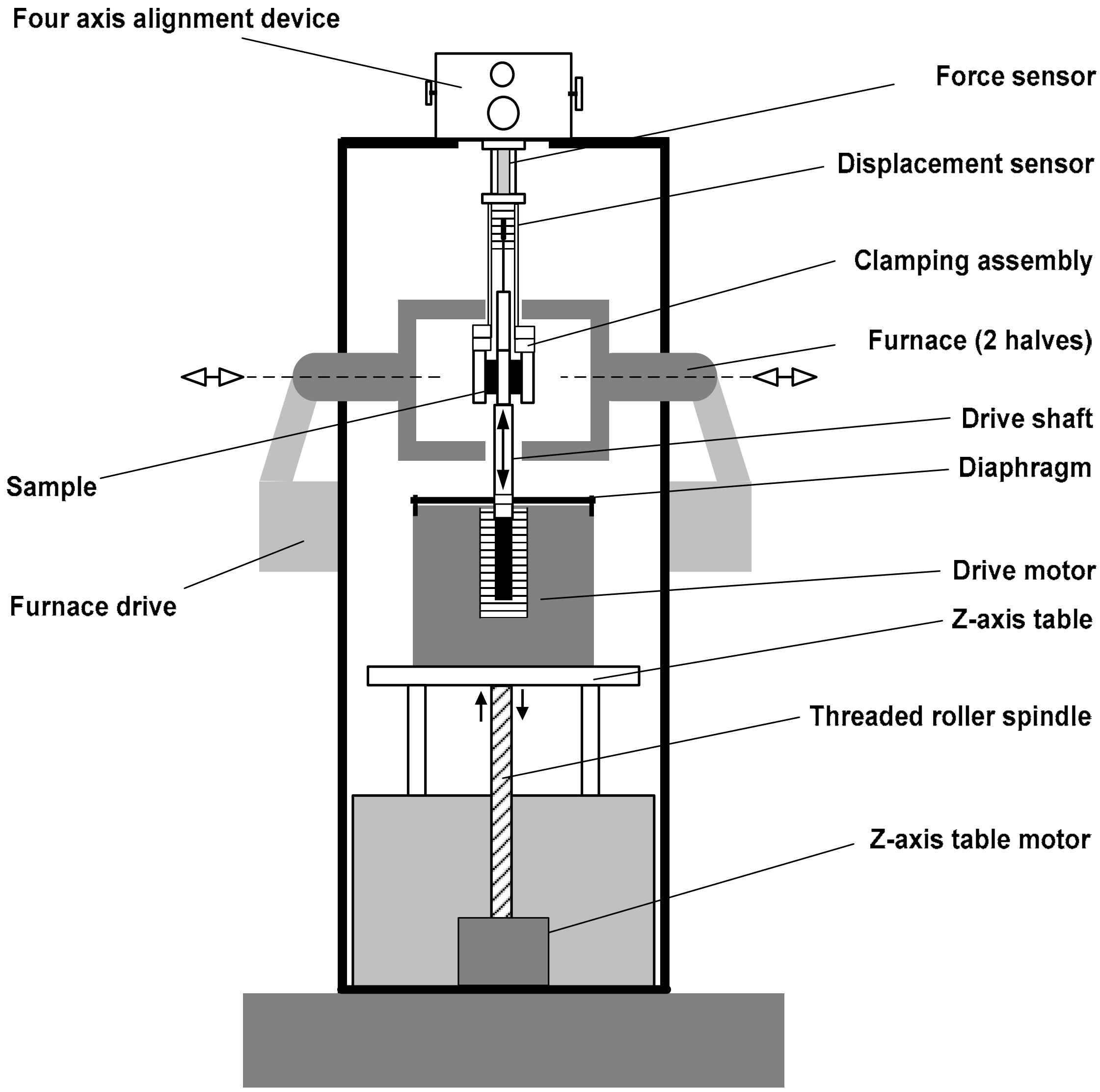


Fig. 2-3. Schematic showing design principles of the DMA/SDTA861e (with shear clamp installed)

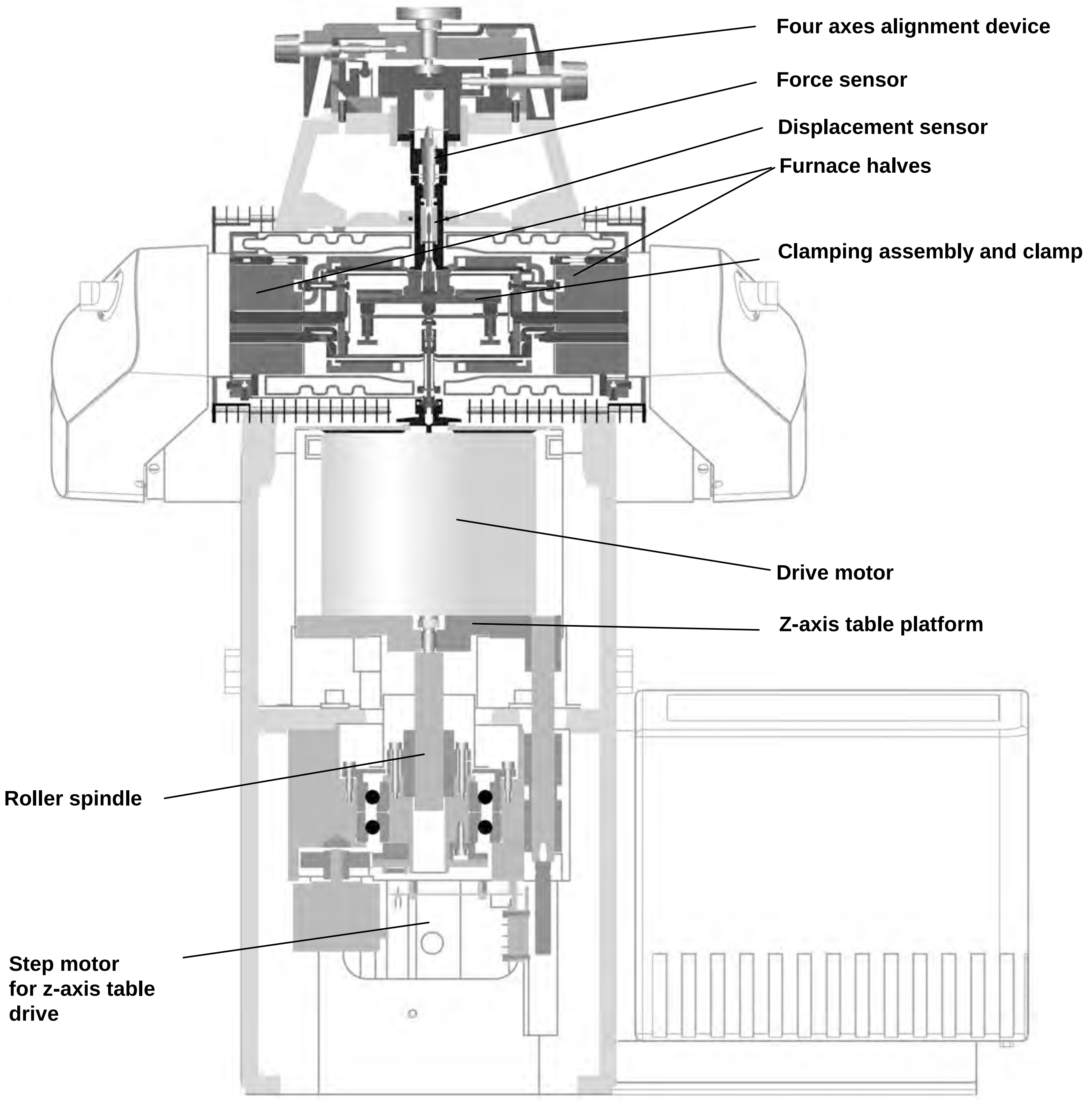


Fig. 2-4. Main assemblies of the DMA/SDTA861e
(with bending clamp installed)

Force and displacement measurement

The sample is mounted in a clamping assembly. The type of clamping assembly used depends on the type measurement to be performed.

mechanical oscillation

The drive motor generates a mechanical oscillation. The current flowing through the drive motor coil sets the diaphragm oscillating through induction. The drive motor current determines the oscillation form of the diaphragm, which in turn determines the dynamic force exerted on the sample and its amplitude.

The drive shaft transfers the mechanical oscillation to the sample.

The clamp assembly consists of a movable part and a fixed part. The drive motor sets the movable part and the displacement sensor core connected to it into oscillation. The displacement sensor core oscillates within the magnetic coil of the displacement sensor and generates the displacement signal by magnetic induction.

LVDT principle

The displacement measurement is based on the LVDT principle (linear variable differential transformer). A special temperature resistant LVDT measures the displacement over an extremely wide range with nanometer resolution.

The displacement sensor is located close to the sample so that only the deformation of the sample is measured. This eliminates any effects due to possible deformation of the stand and improves the accuracy with which the delay time (i.e. phase shift) between the force and displacement is determined. The reproducibility of the displacement measurement is improved by measuring the temperature of the LVDT sensor. Deviations from the reference temperature are then compensated.

The fixed part of the clamp assembly is rigidly connected to the end of the displacement sensor and transfers the dynamic force generated by the drive motor to the force sensor via the body of the displacement sensor.

force and displacement control

Force is measured directly with a piezoelectric crystal. The force measured is that which is actually applied to the sample. Force measurement by the force sensor allows the instrument to be operated under either force or displacement control. A mixed operating mode is also possible. Forces form a few mN to 40 N can be measured.

stiffness range

The stiffness range is given by the force and displacement ranges. More than six decades in stiffness are available.

Heading and cooling during a temperature program

The sample can be subjected to a temperature program at the same time as it undergoes oscillations.

heating and cooling assemblies

The sample is situated in the furnace unit, which consists of two halves. There are heating and cooling assemblies in both halves of the furnace unit. A temperature control loop controls the sample temperature by heating and cooling according to the temperature program set.

The heating assembly consists of lithographically deposited heater tracks that are wrapped around a ceramic tube in meander form. This neutralizes any electromechanical forces that might otherwise affect the sample.

cooler

The cooling assembly, the cooler, is fed with liquid nitrogen from the liquid nitrogen Dewar. The cooler is a heat exchanger in which liquid nitrogen evaporates. The cooler temperature is also displayed on the LCD.

During the actual measurement, the cooling effect is transferred to the sample via the furnace atmosphere so that the measurement results are not affected by the inflow of cold gas.

fast cooling

For rapid cooling to low start temperatures before the actual measurement, the fast cooling feature can be used. Cold liquid nitrogen vapor can be blown directly into the furnace chamber to achieve high cooling rates that allow you to start a new measurement more quickly.

2.2.2 Measurement modes

A number of different measurement modes are used:

- Shear for materials with a very large range shear modulus from about 1 kPa to 2 GPa. This allows viscous liquids and even solids, e.g. polymers in the glassy state, to be measured.
- Three-point bending for stiff materials with a modulus of elasticity of up to 1000 GPa.
- Single and dual cantilever bending for materials that deform too strongly with three-point bending.
- Tension for thin bars, films and fibers.
- Compression for materials with a modulus of elasticity of up to about 1 GPa.

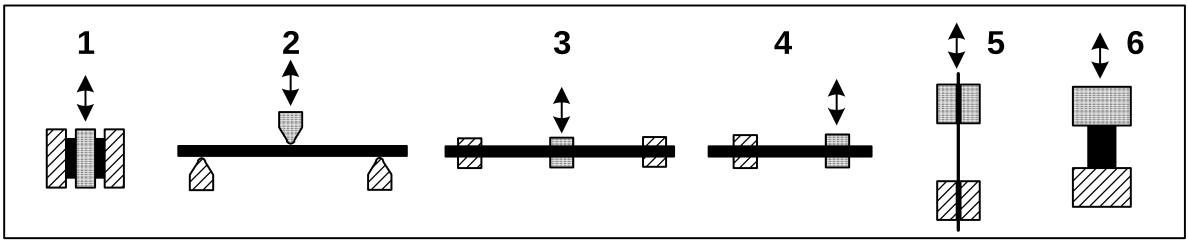


Fig. 2-5. Schematics of the measurement modes

1 shear; **2** three-point bending; **3** dual cantilever (similar to bending but the sample is fixed); **4** single cantilever; **5** tension for thin bars, films and fibers; **6** compression. The clamping assembly is colored black and the sample red. The hatched areas show the parts of the clamping assemblies that remain fixed in position.

2.2.3 The quantities measured

Fig. 2-6 shows a typical measurement of force and displacement versus time.

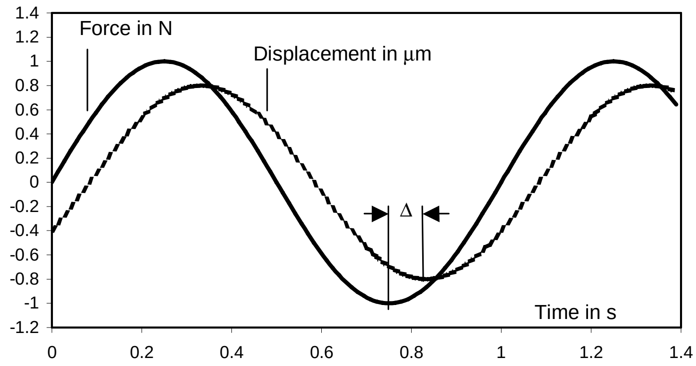


Fig. 2-6. Force and displacement at a frequency (f) of 1 Hz. The phase shift, δ, can be calculated from the time delay, Δ, using the equation $\delta = 2\pi f\Delta$.

The raw data, i.e. the measured force and displacement amplitudes, F_a and L_a, and their phase shifts, δ, are used to calculate the desired material properties:

- Complex modulus (M*): modulus of elasticity, Young's modulus (E) or the shear modulus, G*
- Storage modulus, M', proportional to the energy stored elastically and reversibly
- Loss modulus, M", proportional to the energy transformed into heat and irreversibly lost
- Loss factor, tan δ. With completely elastic materials no phase shift, δ, occurs; completely viscous materials show a 90° phase shift. The loss factor of viscoelastic materials is between 0 and infinity (δ = 90°). The term tan δ corresponds to the ratio of M" to M'.

The moduli are calculated according to the following formulas:

$|M^*| = \frac{F_a}{L_a} g$ where the quotient $\frac{F_a}{L_a}$ is defined as the stiffness S and g is known as the geometry factor calculated from the sample dimensions.

$$M' = |M^*| \cos\delta$$

$$M'' = |M^*| \sin\delta$$

$$\tan\delta = \frac{M''}{M'}$$

E and G are related by Poisson's ratio, μ:

E = 2 (1+ μ) G

For most isotropic materials, μ lies between 0.2 and 0.5, and E is 2.4 to maximum 3 times greater than G. In the rubbery-elastic region of unfilled materials, E ≈ 3 G and in the glassy state E = 2.7 G.

With anisotropic materials, e.g. unidirectional fiber reinforced plastics, E can be more than one hundred times larger than G.

If a material is heated, the storage modulus decreases stepwise by several orders of magnitude. The step corresponds to a peak in the loss modulus. If the transitions are frequency-dependent, they are in fact relaxation transitions, which with increasing frequency shift to higher temperatures.

stiffness

The stiffness, $S = \frac{F_a}{L_a}$, is gained directly from the raw force and displacement data. This property does however not take into account the geometry of a sample: A thick sample is stiffer than a thin one, as the figure below illustrates. The stiffness, S, is therefore not a material property.

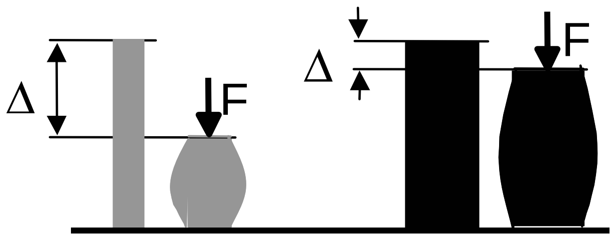


Fig. 2-7. Stiffness of thin and thick samples

2.2.4 Measurement details

- Usually with DMA measurements, the measurement is performed at constant **displacement amplitude**, and a **maximum** force is set that should not be exceeded even with stiff samples. (Measurements under force control are also possible, however.)
- An unsuitable choice of the displacement or force amplitude can affect the measurement accuracy. Amplitudes greater than 1 µm and 10 mN are optimal, as long as the displacement amplitude does not exceed 1% of the corresponding sample dimension. With larger amplitudes, the modulus can change (non-linearity of the sample).
- **Heating rates** of ≤ 3 K/min are usually used because of the low thermal conductivity of plastics and the relatively large samples – except for trial measurements. The same applies to cooling measurements.
- To determine the **frequency dependence**, measurements are performed with several frequencies. The frequencies can be either mixed (simultaneous multi-frequency mode) or applied individually one after the other (sequential frequency series).

Besides measurements with a dynamic temperature program, the DMA/SDTA861e can also perform isothermal measurements with increments of increasing or decreasing

- frequency,
- displacement amplitude and
- force amplitude.

2.2.5 The presentation of DMA curves

Since modulus values tend to change by several orders of magnitude, a linear presentation cannot adequately display the measurement data (Fig. 3). For example, a step of 1 GPa to 10 MPa cannot be distinguished from a step of 1 GPa to 1 MPa. In the logarithmic display, however, such differences can be easily seen (Fig. 4).

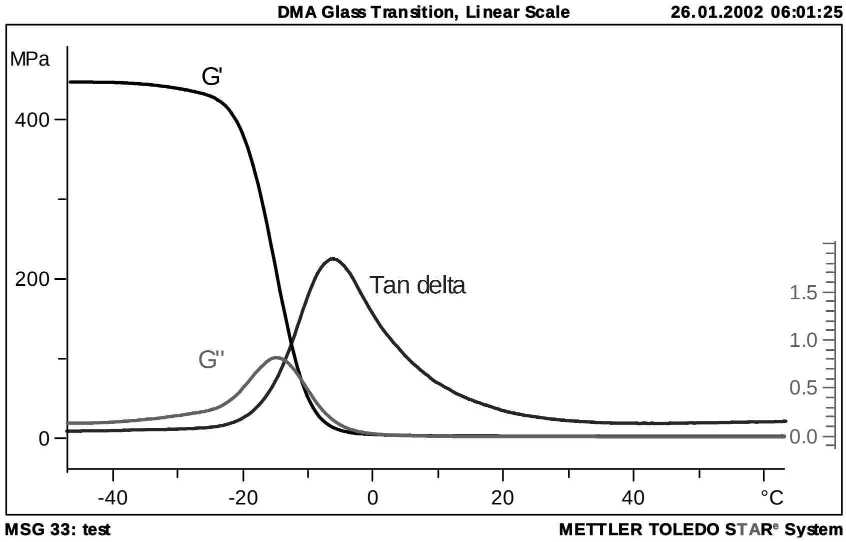


Fig. 2-8. Linear presentation

The linear presentation of the modulus overemphasizes the region with high values. The point of inflection of the storage modulus corresponds approximately to the maximum of the loss modulus. The latter is often referred to as the glass transition temperature, T_g, at the frequency concerned. Because tan δ = G'' / G', the maximum of tan δ is at higher temperature. At the point of intersection of G' and G'', tan δ = 1.

Sample: SBR, 1 Hz, 2 K/min.

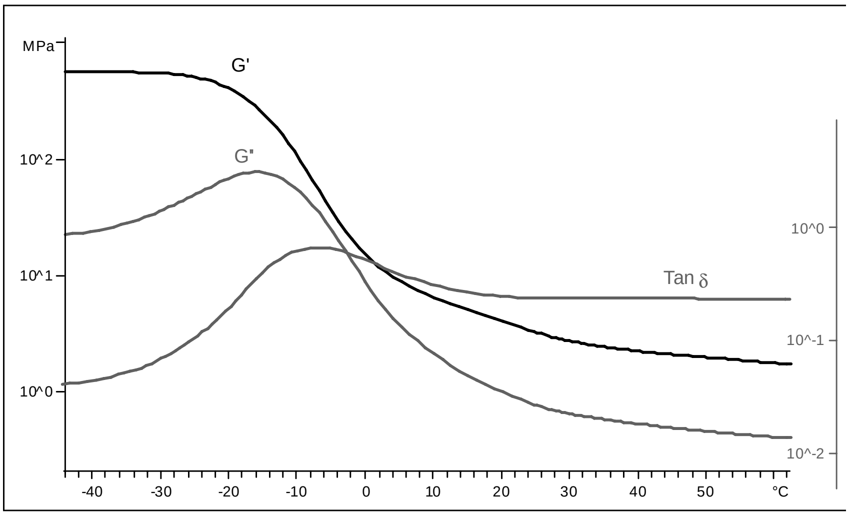


Fig. 2-9. Logarithmic presentation

Same measurement as in Fig. 2-8, but displayed in the usual logarithmic presentation. Compared with the linear presentation, the low-value region of the modulus now appears scale-expanded. In this presentation, T_g corresponds to the onset of the decrease of G‘. The loss factor in the rubbery-elastic state is clearly larger than in the glassy state. The ordinate of the loss factor is displayed on the right of the diagram.

2.2.6 Operation of the individual assemblies

This section describes the function of the individual assemblies of the DMA/SDTA861e and their functional interdependence.

Abbreviations:

For better orientation, the margins of the pages in this chapter include the following signs to indicate the device to which the text in the corresponding section refers:

SCA Small clamping assembly, used for the text relating to the small clamping assembly

LCA Large clamping assembly, used for the text relating to the large clamping assembly

Clamping assembly and clamps

The clamping assembly and the clamp are used to mount the sample.

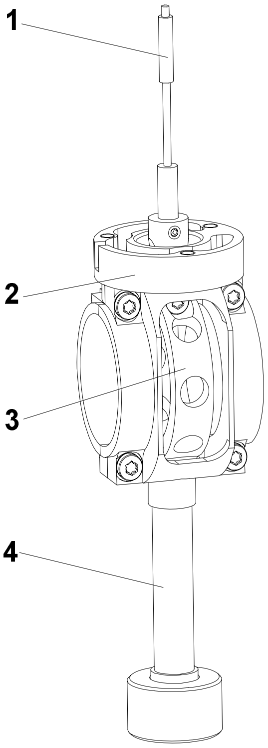
The table below summarizes the various combinations of clamping assemblies and clamps that can be used:

Clamping assembly	Clamp
Small clamping assembly	• Standard shear clamp for highly viscous materials • Special shear clamp for low-viscosity liquids • Tension clamp (films, fibers, rods and bars)
Large clamping assembly	• Bending clamp for dual/single canti-lever and 3-point bending • Tension clamp (films, fibers, rods and bars) • Compression clamp
Adjustment assembly	None *

* No clamp is used with the adjustment assembly

Small clamping assembly (SCA)

Fig. 2-10 shows the design of the small clamping assembly:



1 Displacement sensor core
2 Clamp holder, fixed part
3 Clamp holder, movable part
4 Drive shaft

Fig. 2-10. The small clamping assembly

The small clamping assembly consists of two parts: a movable part and a fixed part.

The movable part is connected to the drive motor and sets the sample into oscillation. It consists of a drive shaft **4**, the movable part of the clamp **3** and the displacement sensor core **1.**

The other part, the fixed part of the clamp holder **2**, is rigidly connected to the displacement sensor and fixes the sample in the clamp. The force induced in the sample is transferred via this part to the force sensor.

In the small clamp assembly you can use the following clamps:

- Shear clamp for highly viscous fluids
- Shear clamp for low-viscosity fluids
- Tension clamp (for films, fibers, rods and bars)

♣ The shear assembly for highly viscous materials is available in two versions. The assembly with the rough surfaces is delivered as standard. For special applications, a version with smooth surfaces is available (ME 51140093).

All clamps consist of three disks, which, depending on the type of clamp, represent the sample support, the sample fitting or the end disk. The middle disk is enclosed by the movable part of the clamp holder. The two outer disks are mounted in the fixed part of the clamp holder.

The guide pins serve to fix the sample when installing the assembly in the clamp holder.

The following sections describe the function of the assemblies.

Shear clamps

Two shear clamps are available for the small clamping assembly: the standard shear clamp for highly viscous materials and the special shear clamp for low viscosity liquids.

The shear clamps allow you to perform shear measurements and determine the dynamic shear modulus.

The advantage of this measurement mode is that both liquids and viscous-to-hard materials can be measured.

standard shear clamp for highly viscous materials

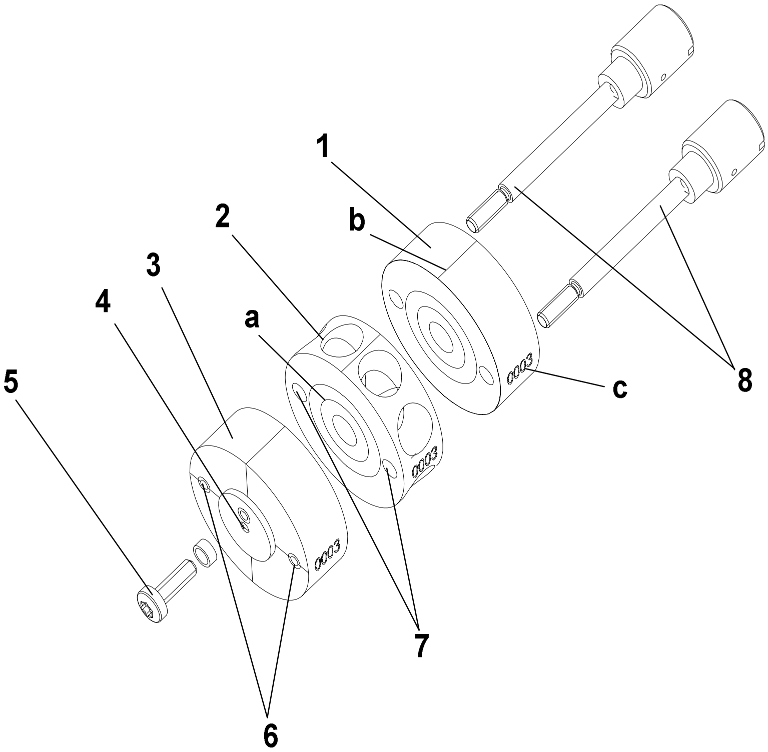
The standard shear clamp for highly viscous materials guarantees a homogeneous temperature environment. It is possible to attach a thermocouple that measures the sample temperature so accurately that simultaneous calorimetric effects of the sample can also be measured (SDTA). This shear clamp is therefore suitable both for elastomers and for thermoplastics and thermosets.

SDTA

special shear assembly for low viscosity liquids

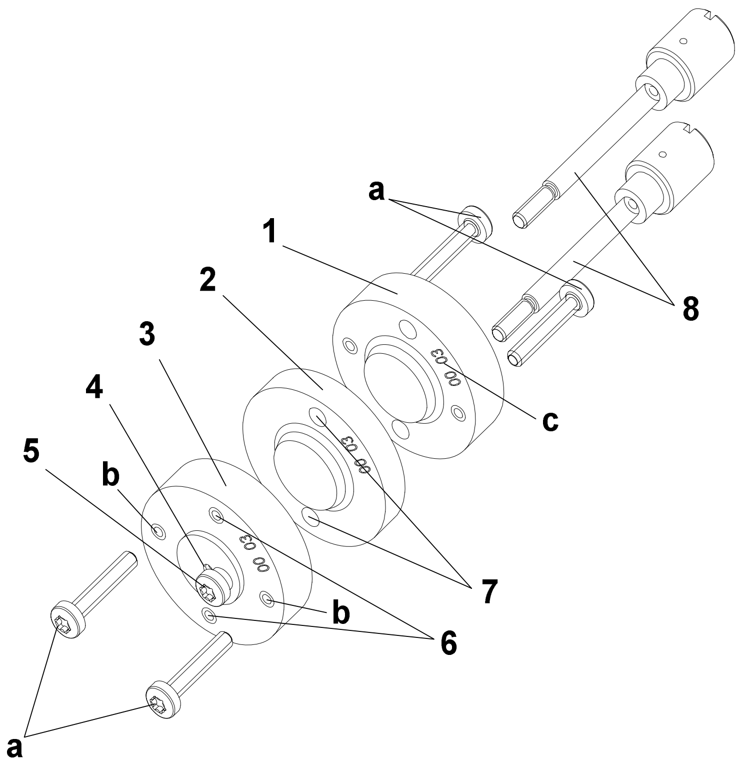
The special shear clamp for low viscosity liquids allows materials of low viscosity to be measured (e.g. low viscosity silicone oils).

Fig. 2-11 and Fig. 2-12 show the design of the two shear clamps.



1,2 Sample supports
3 End disk
4 Hole for SDTA thermocouple
5 Torx screw for SDTA thermocouple
6 Tapped holes
7 Guide holes
8 Guide pins
a Laser markings
b Markings
c Clamp identification number

Fig. 2-11. Standard shear clamp for highly viscous materials



1, 2 Sample supports
3 End disk
4 Hole for SDTA thermocouple
5 Torx screw for SDTA thermocouple
6 Tapped holes
7 Guide holes
8 Guide pins
a Distance screws
b Tapped holes for distance screws
c Clamp identification number

Fig. 2-12. Special shear clamp for low-viscosity liquids

The shear clamp consists basically of three disk-like parts, namely two sample supports **1** and **2**, and the end disk **3**. Separate samples of material under investigation are placed on the two sample supports **1** and **2** in both the spaces between the disks. The laser markings on the surfaces are a guide to position the samples properly. Both the sample support and the end disk **3** are pressed together in the clamp holder of the small clamping assembly.

The sample support **2** in the middle is connected directly to the drive motor via the movable part of the clamp holder and the drive shaft and transfers the shear forces to the sample.

The end disk **3** has tapped holes for the guide pins **8**. The purpose of the guide pins is to clamp the sample between the disks and to hold them in place when mounting the clamp in the clamp holder. The guide pins are removed for the measurement.

Two pairs of guide pins **8** of different length are supplied with the standard shear clamp. Either pair of the guide pins can be used, depending on the thickness of the sample.

The SDTA thermocouple is inserted in the hole **b** and fixed by a Torx screw **a**.

special shear clamp for low viscosity liquids

With the special shear clamp for low viscosity liquids, the sample supports and the end disk are somewhat different. The sample support surfaces are raised with respect to the faces. The distance between the disks (sample supports and end disk) is set by means of four distance screws.

- ♣ There are markings on the side of the sample supports and the end disk of the standard shear clamp for highly viscous materials. These markings must be aligned when the shear clamp is properly assembled. If your sample has an internal structure, you can use the markings on the longitudinal side of the clamp to take into account the orientation of the structure when mounting the clamp.
- ♣ The sample supports and end disk are inscribed with a number in order to identify them as part of a particular shear clamp.

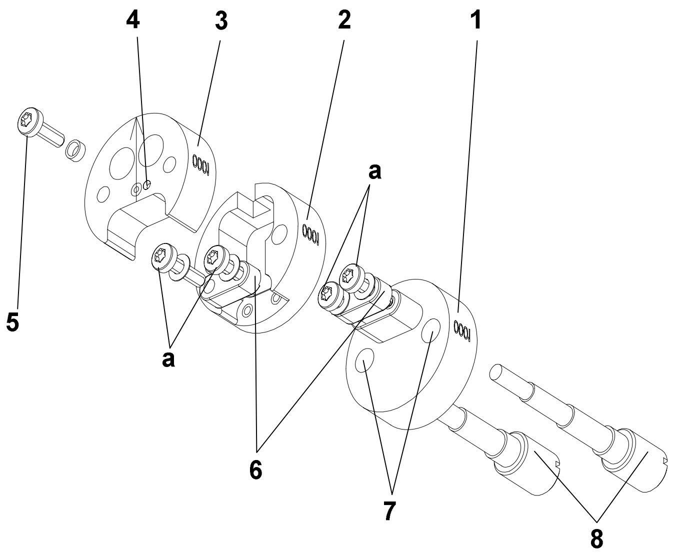
Tension clamp (small clamping assembly)

The tension clamp allows you to perform measurements in tension and to determine the modulus of elasticity (Young's Modulus). In this mode, the sample is subjected to oscillating tensile forces. One end of the sample is fixed and the oscillation is applied to the other end. A preload force must be applied to the sample so that buckling does not occur during the oscillation. When the offset control is set to Auto, a static tensile load is maintained.

With the tension clamp, the sample can be prepared and mounted in the clamp separately outside the DMA module, for example while a different sample mounted in another clamp is being measured. Once you have mounted the sample in the clamp, the clamp can be quickly installed and the next experiment started.

This tension clamp is most suitable for measuring films, fibers and thin rods or bars. The advantage is that sample clamping has practically no influence on the deformation.

Fig. 2-13 shows the design of the tension clamp for the small clamping assembly.



1,2 Sample supports

3 End disks

4 Hole for f SDTA thermocouple

5 Torx screw for SDTA thermocouple

6 Clamping plates

7 Guide holes

8 Guide pins

a Torx screws for clamping plates

Fig. 2-13. Tension clamp for the small clamping assembly

The tension clamp for the small clamping assembly consists basically of three disk-like parts, the sample supports **1** and **2** and the end disk **3**. The sample for tensile measurement is clamped between the sample supports **1** and **2**. The two sample supports and the end disk **3** are mounted in the clamp holder of the small clamping assembly.

The middle sample support **2** is connected directly to the drive motor via the moving part of the clamp holder and the drive shaft, and applies the tensile forces to the sample.

In the middle sample support **2** and in the end disk **3** there are threaded holes for the guide pins **8**. The guide pins serve to hold the sample supports, end disk and sample in place when the clamp is installed in the clamp holder. The guide pins are then removed for the actual measurement.

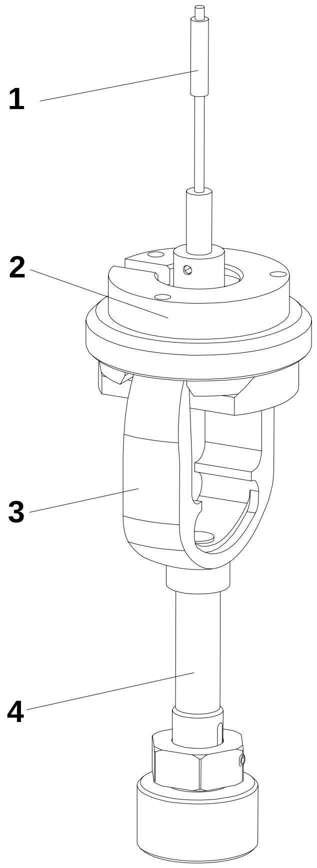
The SDTA thermocouple is inserted in the hole marked **4** and fixed in place with the Torx screw **5**.

♣ The sample supports and the end disk are marked with an identification number, in order to identify them as parts of a particular tension clamp.

LCA

Large clamping assembly (LCA)

Fig. 2-14 shows the design of the large clamping assembly.



1 Displacement sensor core

2 Clamp holder, fixed part

3 Clamp holder, movable part

4 Drive shaft

Fig. 2-14. Large clamping assembly

The large clamping assembly consists of two parts: a movable part and a fixed part.

The movable part is connected to the drive motor and sets the sample in oscillation. It consists of drive shaft **4**, the movable part of the clamp holder **3** and the displacement sensor core **1**.

The other part, the fixed part of the clamp holder **2**, is rigidly connected to the displacement sensor and fixes the sample in the clamp. The force induced in the sample is transferred via this part to the force sensor.

In the large clamping assembly you can use the following clamps:

- 3-point bending clamp
- Single/Dual cantilever clamp
- Tension clamp large clamping assembly
- Compression clamp

LCA

Bending clamp

bending measurement modes

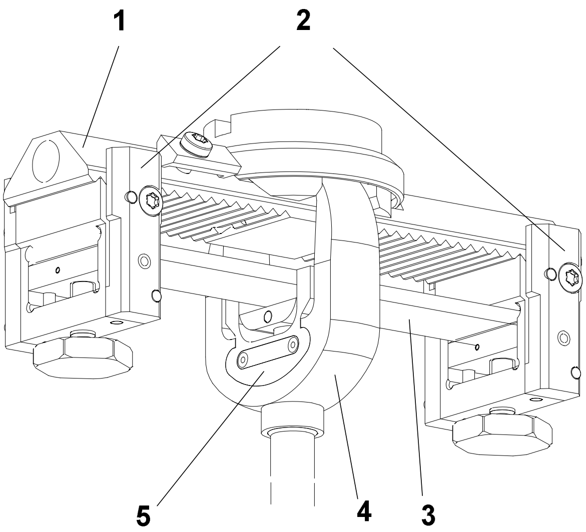
The bending clamp allows you to perform bending experiments and determine the dynamic modulus of elasticity or Young's Modulus. Three types of bending measurements are possible: 3-point bending, single cantilever and dual cantilever.

The 3-point bending mode is particularly suitable for hard materials such as reinforced thermosets, composites, metals and alloys.

The single or dual cantilever mode is especially suitable for materials that would otherwise bend excessively under static stress, for example unreinforced thermoplastics and thermosets.

The single cantilever mode is very similar to the dual cantilever mode, except that only one side of the sample is fixed. The middle of the sample is clamped to the movable part of the clamp holder providing the oscillatory force. This mode is suitable for materials that expand or shrink strongly along their length during the measurement. This is in particular the case with thermoplastics.

Fig. 2-15 shows the design of the bending clamp.



1 Bending beam (fixed part)
2 Riders (fixed part)
3 Sample
4 Clamp holder, movable part
5 Bending clamp, movable part

Fig. 2-15. Bending clamp (with sample and clamp holder)

A long sample is fixed in place with two clamps, the riders **2**, between the ends on a toothed supporting bending beam **1** that forms the fixed part of the bending clamp **1**. The bending beam and the riders form the fixed part of the bending clamp.

The movable part of the bending clamp **5** that fixes the sample **3** in the middle is connected directly to the drive motor via the clamp holder **4** and the drive shaft and transfers the dynamic bending stress to the sample.

A different set of riders **2** and a special movable part **5** is used for each type of bending measurement.

In the 3-point bending mode, the ends of the sample rests on two knife-edges in the riders and an oscillatory force is applied to the middle by a moving knife-edge in the movable part of the clamp holder. A preload force is applied to fix the sample in place. This way of mounting samples interferes least with the actual sample measurement.

In the dual cantilever mode, the ends of the sample are fixed in the riders **2** and its middle is clamped to the movable part of the clamp holder **4** providing the oscillatory force.

The same riders are used for single and dual cantilever bending but the right rider remains unused in the single cantilever mode. It should, however, still be mounted.

SDTA thermocouple

The SDTA thermocouple is clamped to the cheek of the left rider **2** by a plate to fix it in position.

♣ The longer the sample the more accurate the measured modulus of elasticity (Young’s modulus) is likely to be. The contribution of the shear modulus and clamping effects are stronger with short samples.

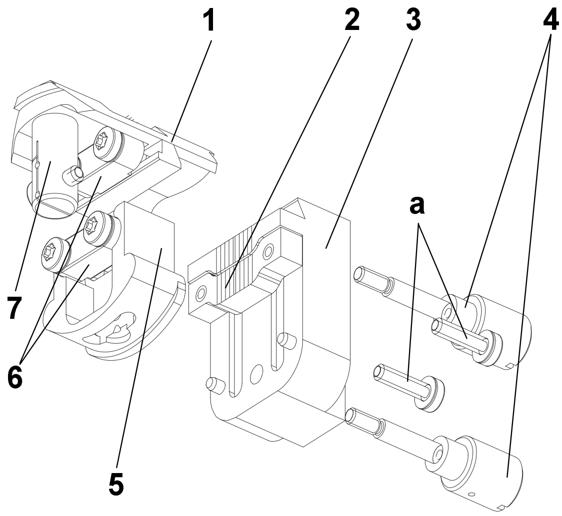
LCA

Tension clamp (large clamping assembly)

The large tension clamp allows you to perform measurements in tension and determine the dynamic modulus of elasticity or Young's Modulus. In this mode, the sample is subjected to oscillating tensile forces. One end of the sample is fixed and the oscillation is applied to the other end. A preload force must be applied to the sample so that buckling does not occur during the oscillation. When the offset control is set to Auto, a static tensile load is maintained.

With the tension clamp, the sample can be prepared and mounted in the clamp separately outside the DMA module, for example while a different sample mounted in another clamp is being measured. Once you have mounted the sample in the clamp, the clamp can be quickly installed and the next experiment started.

The tension clamp is most suitable for measuring films, fibers and thin rods or bars. The advantage is that sample clamping has practically no influence on the deformation.



1 Tension clamp, fixed part
2 Sample mounting plate
3 Sample mounting aid
4 Support pins
5 Tension clamp, movable part
6 Clamping plates
7 SDTA Thermocouple holder
a Torx screws for sample mounting plate

Fig. 2-16. Tension clamp for the large clamping assembly

The tension clamp for the large clamping assembly consists basically of a fixed part **1**, and a movable part **5**. The sample is mounted between the movable part and the fixed part by means of two clamping plates **6**. The supplied short or long torx screws can be used to fix the clamping plates, depending on the thickness of the sample.

The tension clamp can be adjusted to the length of the sample with the help a sample mounting plate **2** which is secured by two torx screws **a**. For sample lengths of 10.5 mm and 19.5 mm there are suitable sample mounting plates. For a sample length of 5.5 mm you do not need a sample mounting plate.

The movable part of the tension clamp is directly coupled to the drive motor via the movable part of the clamp holder and the drive shaft and transfers the tensile forces into the sample.

The support pins **4** serve to fix the two parts of the tension clamp when it is mounted in the clamping assembly in the sample mounting aid **3**. They are screwed into the threaded holes in the movable and fixed parts of the tension clamp. The support pins and the sample mounting aid are removed for the measurement.

SDTA thermocouple

The SDTA thermocouple holder **7** serves to fix the SDTA thermocouple in place.

sample mounting aid

In order to install and then remove the clamp from the clamping assembly, the sample mounting aid **3** must always be fixed to the clamp so that its parts are securely fixed together. The function of the sample mounting aid is therefore similar to that of the guide pins of the small clamp.

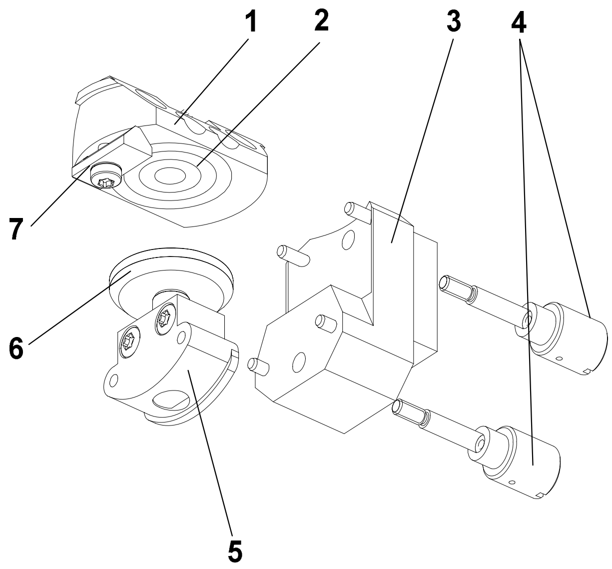
Compression clamp

The compression clamp allows you perform measurements in compression and to determine the dynamic modulus of elasticity or Young's modulus. In this mode the sample is subjected to oscillatory compressive forces. When the offset control is set to Auto, a static compressive load is automatically maintained.

With the compression clamp, the sample can be prepared and mounted in the clamp separately outside the DMA module, for example while a measurement of a different sample mounted in another clamp is being measured. Once you have mounted the sample in the clamp, the clamp can be quickly installed and the next experiment started.

We recommend that you perform measurements in compression mode only with soft samples. The measurement range of Young's modulus is 0.1 MPa to 10 GPa. In this mode, however, the Young's modulus cannot be determined with the same high accuracy as in the tension mode.

Fig. 2-17 shows the design of the compression clamp.



1 Compression clamp, fixed part
2 Sample mounting plate
3 Sample mounting aid
4 Support pins
5 Compression clamp, movable part
6 Sample support
7 SDTA thermocouple holder

Fig. 2-17. Compression clamp

The compression clamp consists basically of a fixed part **1** and a movable part **5**. The sample support **6** is inserted in the movable part. The sample is mounted between the sample support and the contact surface of the fixed part. There are laser markings on the sample mounting plate **2** to help you position the sample.

The movable part of the compression clamp is directly coupled to the drive motor via the movable part of the clamp holder and the drive shaft and transfers the compressive forces into the sample.

The support pins **4** serve to fix the two parts of the compression clamp when it is mounted in the clamping assembly in the sample mounting aid **3**. They are screwed into the threaded holes in the movable and fixed parts of the compression clamp. The support pins and the sample mounting aid are removed for the measurement.

SDTA thermocouple

The SDTA thermocouple holder **7** serves to fix the SDTA thermocouple in place.

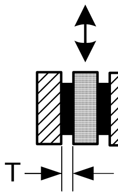
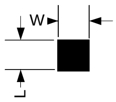
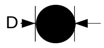
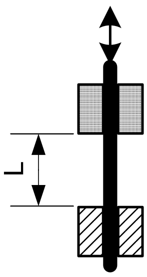
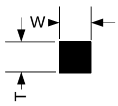
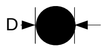
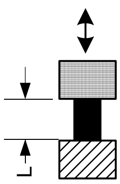
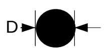
sample mounting aid

In order to mount and then demount the clamp from the clamping assembly, the sample mounting aid **3** must always be fixed to the clamp so that its parts are securely fixed together. The function of the sample mounting aid is therefore similar to that of the guide pins of the small clamp.

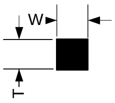
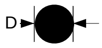
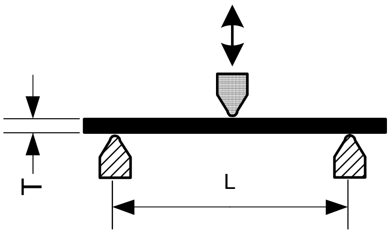
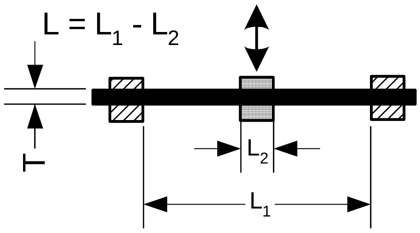
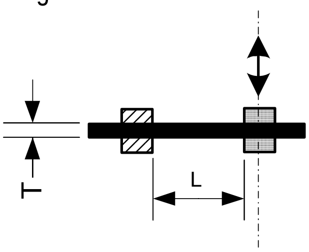
Sample dimensions and geometry factors

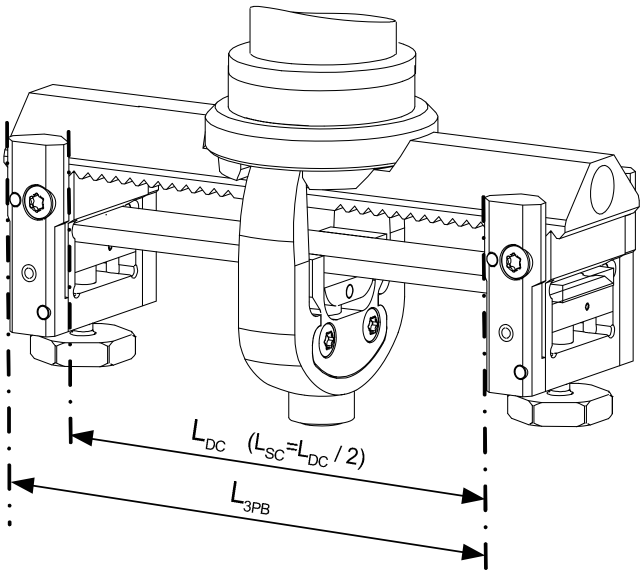
As explained in the previous section, the stiffness is the quantity that is directly measured. The modulus is calculated from the stiffness and the geometry factor, which includes the sample dimensions.

Table of sample dimensions and geometry factors

Mode	Rectangular samples	Round samples
Shear (small CA only) 	thickness, T : < 6.5 mm width, W : < 15 mm Length, L : < 18 mm $G = \frac{T}{2\,LW}$ 	thickness, T : < 6.5 mm diameter, D : < 15 mm $G = \frac{2\,T}{\pi\,D^2}$ 
Tension (small and large CA) 	Small clamping assembly length, L : 9.0 width, W : < 5 mm thickness, T : < 2 mm Large clamping assembly length, L : 19.5, 10.5, 5.5 mm width, W : < 7 mm thickness, T : < 3 mm $G = \frac{L}{W \cdot T}$ 	Small clamping assembly length, L : 9.0 diameter, D : < 2 mm Large clamping assembly length, L : 19.5, 10.5, 5.5 mm diameter, D : < 3 mm $G = \frac{4L}{\pi\,D^2}$, where $\frac{D}{L} \leq \frac{1}{20}$ 
Compression (large CA only) 	Rectangular samples are not recommended with the compression clamp.	length, L : < 9 mm diameter, D : < 20 mm $G = \frac{4 \cdot L}{\pi\,D^2}$ 

LCA

Mode	**Rectangular samples**	**Round samples**
		
Bending (large CA only)		
• 3-point bending 	length, L : 30 ... 90 mm width, W : < 15 mm thickness, T : < 5 mm $G = \frac{L^3}{4\,WT^3}$	length, L : 30 ... 90 mm diameter, D : < 5 mm $G = \frac{4 \cdot L^3}{3\pi\, D^4}$
• dual cantilever 	length, L : 20 ... 80 mm width, W : < 15 mm thickness, T : < 5 mm $G = \frac{L^3}{16\,W\,T^3}$	length, L : 20 ... 80 mm diameter, D : < 5 mm $G = \frac{L^3}{3\pi\, D^4}$
• single cantilever 	length: half the length for dual cantilever is used $G = \frac{L^3}{W\,T^3}$	length: half the length for dual cantilever is used $G = \frac{16 \cdot L^3}{3\pi\, D^4}$



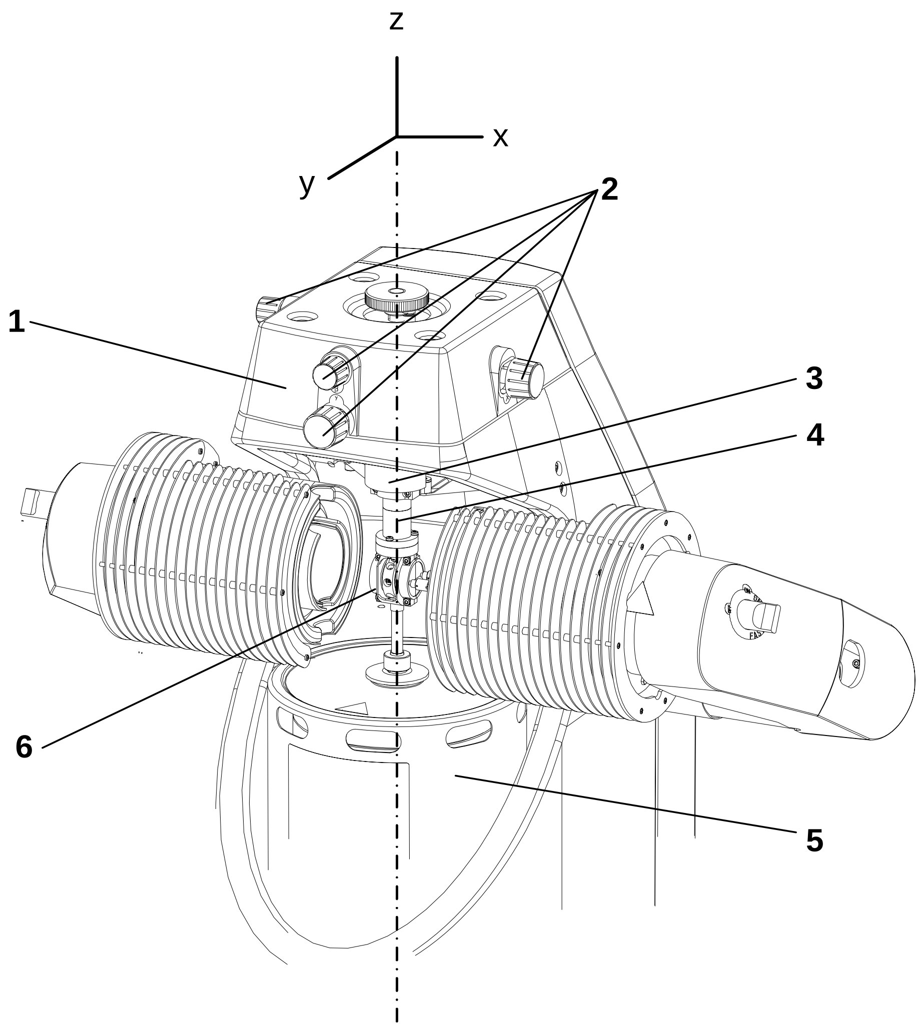
The sample length L is measured for dual cantilever, single cantilever and 3-point bending experiments as shown on the left:

- dual cantilever bending: L_{DC}
- single cantilever bending: $L_{SC}=L_{DC}$ / 2
- 3-point bending: L_{3PB}

Please note
Although round samples can be measured in the bending mode, you may have to take into account clamping effects (due the higher deformation of the sample in the clamps) that will possibly reduce measurement accuracy.

Four axes alignment device

The four axes alignment device allows you to align the position of the measurement system in order to guarantee correct measurement. You have to align the system properly with the four axes alignment device every time you change the clamping assembly.



Parts

1 Four axes alignment device

2 Knobs

3 Force sensor

4 Displacement sensor

5 Drive motor

6 Clamping assembly

Fig. 2-18. Arrangement of the four axes alignment device

Fig. 2-18 shows the arrangement of the four axes alignment device **1** above the clamping assembly **6.** It illustrates the position of the x-, y- and z-axes.

The four axes alignment device is a mechanism made of slides and spindles. By adjusting the slides and spindles with the four adjustment knobs **2**, you can change the position of the displacement sensor **3** with respect to the clamping assembly **6**. This however allows you to change the position only with regard to the x- and the y-axes. You can set the position on the z-axis by moving the z-axis table.

The four axes alignment device allows you to perform the following movements in order to align the measurement system properly with the clamping assembly:

- Movement in the x-direction
- Movement in the y-direction
- Rotation around the x-axis (solid angle β)
- Rotation around the y-axis (solid angle α)

♣ The displacement sensor housing moves in the opposite direction of the x and y knobs: If you screw the knobs in, the housing moves outward and vice versa.

Fast cooling device

The fast cooling device consists of two **fast cooling valves** which are operated manually by the two knobs on the outer sides of the furnace arms. The fast cooling device is used to rapidly cool the clamping assembly and sample down to low start temperatures before the actual measurement. When the fast cooling valves are switched on, the coolant from the cooler is directed into the furnace and the sample holder and the sample are cooled directly by the coolant vapor. fast cooling

External gas supply

The atmosphere (e.g. an inert or an oxidative atmosphere) in the furnace chamber can be created by means of an external gas supply. There is an inlet and a connector for the gas tubing on each furnace arm. gas supply

Keypad and LCD

The keypad is located on the right side of the front housing of the module. The keypad allows you to enter commands or to obtain information about the current status of the measuring cell and the experiment via the liquid crystal display (LCD).

Keypad on the DMA/SDAT861^{e} module

The OK key confirms instrument messages, e.g. commands requests, warnings and error messages.

The ROTATE key has several different functions:

- To adjust the contrast of the display (only possible when switching on). You set the contrast of the LCD display by immediately pressing the OK key after switching on the DMA module. You set the desired contrast of the LCD display using the ROTATE key. You quit this set-up procedure by pressing OK.
- To select the items in the second row line of the display (see the tables at the end of this section).

The FURNACE key opens and closes the furnace of the measuring cell.

- When the furnace is **closed**: Pressing the FURNACE key opens the furnace.
- When the furnace is **open**: Pressing the FURNACE key closes the furnace.

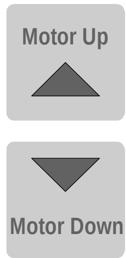
The GAS key allows you to view the flow rate in ml/min. The flow rates can only be observed if the TSO800GC or TSO800GC1 Gas Controller is connected (see the operating instructions for the gas controller).

The RESET key is a termination key.

After pressing the RESET key on the DMA module, the current measurement is aborted and the furnace chamber is cooled down or heated up until the temperature of the clamping assembly is between –10 °C and 60 °C. The DMA module then assumes the status "Furnace power off" and the furnace halves can be opened with the FURNACE key.

♣ This behavior is different to that of other TA modules. The End Behavior cannot be set in the Module Control Window of the STAR^e Software.

The MOTOR UP and MOTOR DOWN keys are used to drive the z-axis table up or down.



♣ The zero position of the z-axis table can be set with these keys at the beginning of the interactive Four Axes Alignment dialog for the fine alignment of the measuring sensors. See also Chapters 6 and 8, section *Fine alignment of the measuring sensors (four axes alignment)*.

measuring cell status

The **LCD display** shows status information and measurement values and messages from the measuring cell. The LCD display has two rows. The first row shows the current status of the DMA module measuring cell. The second row displays the current measurement quantities.

You can change to the desired item in the second row by repeatedly pressing the ROTATE key. The displayed status in the first row remains the same while you change between the values in the second row.

♣ The displays move only in one direction. If you jump over a display by mistake, you must press the ROTATE key repeatedly until you obtain the desired display.

Please note: Pressing the keys on the keypad during a measurement can cause vibrations and disturb a sensitive measurement.

Information shown in the first row of the LCD

♣ This information is also shown in the area at the bottom of the Module Control Window.

Status displayed on the LCD	Explanation
IDLE FURNACE OFF	The furnace is switched off. The text Standby (furnace power off) appears simultaneously in the Module Control Window.
RT CHECK	Clamping check at room temperature
ST CHECK	Clamping check at start temperature
GOING TO START TEMP.	Measuring cell goes to the start temperature (the CA temperature is used as reference).
SETTLING….	Settling of the CA temperature
MEASUREMENT SEG. : --	The measurement is in progress.
GOING TO ROOM TEMP.	Measuring cell goes to the remove tempera-ture (the CA temperature is used as refer-ence).

♣ See also Chapter 7, section *Preliminary stages before the measurement* for more details on the stages before the measurement.

Information shown in the second row of the LCD

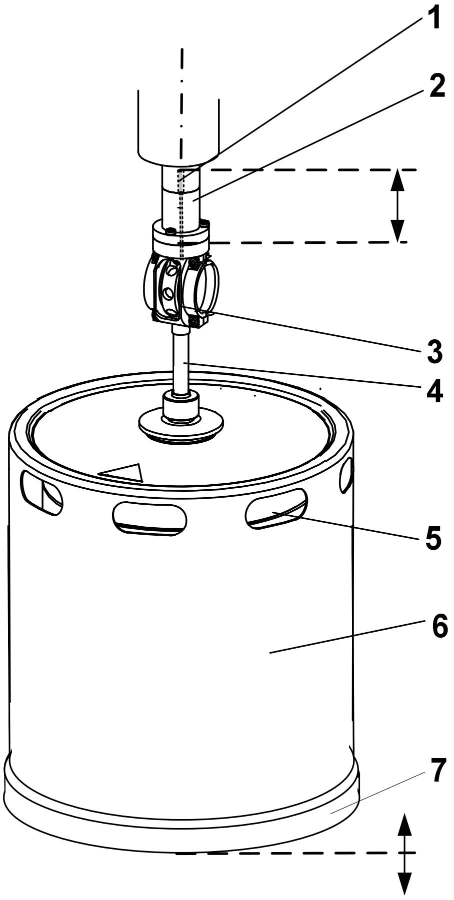
♣ This information (except for the items marked with *) can also be displayed in the monitor area of the Module Control Window.

Status displayed on the LCD	Explanation
Tca: … C	Clamping assembly temperature
Ts: … C	Current temperature of the sample in °C
Tcool: … C *	Current cooler temperature in °C
GAS 1: --ml GAS 2: -- ml *	Gas flow rate in ml per minute
Z-AXIS POS. -- *	Position of the z-axis table in µm (the unit is not displayed on the LCD)
FREQUENCY	Current frequency I Hz
DISPL. AMPL. -- um	Current displacement amplitude in µm
FORCE AMPL. -- mN	Current force amplitude in mN
PHASE -- rad	Current phase amplitude in rad
ENTER FAST CHECK *	Special mode for a fast adjustment (used for service and maintenance purposes only)

* This information cannot be displayed in the Module Control Window

Drive motor and z-axis table

Fig. 2-19 shows the arrangement of the drive motor, z-axis table and clamping assembly. Since the z-axis table is located below in the closed part of the stand, it is not visible from outside.



Parts

1 Displacement sensor core

2 Displacement sensor housing

3 Clamping assembly (without clamp)

4 Drive shaft

5 Drive motor

6 Drive motor housing

7 Z-axis table platform

Fig. 2-19. Arrangement of drive motor, z-axis table and clamping assembly. The movement of the z-axis table and the displacement sensor core is indicated by the up and down arrows.

The drive motor **5** is located in the drive motor housing **6** and mounted on the z-axis table platform. It generates the DMA oscillation that is transferred to the sample via the drive shaft **4** and clamping assembly **3**. There are two types of drive motors that can be installed in the factory for different force ranges.

The z-axis table allows you to adjust the position of the drive motor and the movable part of the clamping assembly in the z-direction. The z-axis table is driven by a high precision roller spindle powered by a step motor (see also *Fig. 2-4*). By moving the z-axis table platform **7** in the z-direction you can set the zero position of the displacement sensor core **1** in the displacement sensor coil (see *Fig. 2-19*). This is done during the four axes alignment procedure.

A round piece of felt is mounted on the drive motor surface to catch and absorb liquid nitrogen droplets that may drip down from the gap between the furnace halves.

Devices for calibration and adjustment

Special devices that serve as tools for calibration and adjustment are shipped as standard accessories with the DMA/SDTA861e.

The **alignment aid** allows the alignment of the measuring sensors and the clamping assembly to be performed quickly and accurately. More details are given in Chapters 6 and 8.

The **adjustment assembly** allows the movement of the roller spindle and the force sensor to be calibrated and adjusted. See Chapter 8 for more details.

2.2.7 Sample preparation

Proper sample preparation and sample mounting are of paramount importance to achieve good measurement results. The mounting of the sample is explained in detail in Chapter 6. This section includes some general hints for the preparation of samples.

- Samples should not be subjected to extreme stresses (mechanical, thermal, etc.) during preparation and mounting.
- A sample should have:
  - a straight and untwisted shape. This is particularly important in tension mode experiments.
  - a flat surface finish to make good mechanical contact with the clamping surfaces of the clamps.
  - accurately parallel surfaces perpendicular to the sides. This is important to accurately calculate the geometry factor. Dimensional tolerances should be better than ±0.1%.
- It is important to measure the sample dimensions accurately in order to obtain accurate geometry factor values. Wrong sample thicknesses can significantly affect the geometry factor and in turn the modulus values. This is particularly the case in the bending mode where the cube of the thickness is used to calculate the modulus.
- For bending measurements in particular, it is very important that the sample have a uniform width and thickness.
- Ideally, samples should be molded, machined, or otherwise formed into the shape suitable for the corresponding clamp.
- The dimensions of the sample must be chosen so that the sample stiffness is within the specifications of the instrument. If you are able to prepare a sample and mount it in a clamp, this does not necessarily mean that the modulus will be within the measuring range of the instrument.

Common problems with samples

- Moisture or solvents contained in the sample can affect the measurement data. Conditioning or drying the sample prior to the measurement can solve the problem.
- Elastomers such as carbon-filled rubbers can be susceptible to the strain or stress they suffer in the clamp. This may affect the repeatability of measurement results. Clamping the sample with torque values as low as possible and setting the torque values uniformly and accurately can solve the problem.
- Some samples may deform strongly under the pressure of the clamp. The deformation can lead to erroneous modulus values. This problem can occur in all measuring modes accept the 3-point bending mode. If possible, it is therefore advisable to use the 3-point bending mode for such samples, or to clamp the sample with as little torque as possible.
- The dimensions of some samples can depend strongly on temperature. Marked shrinkage or expansion can occur at low or high start temperatures, respectively. The shrinkage can be compensated for by reclamping at start temperature in the following measuring modes:
  - shear mode
  - dual and single cantilever bending mode
  - the tension mode

  The problem of marked expansion of the sample can be solved by using the single or the dual cantilever measuring mode where possible.

3 Cooling with the Liquid Nitrogen Dewar

Contents

3 Cooling with the Liquid Nitrogen Dewar

Your DMA module must be able to both heat and cool the sample in order to run temperature programs. This is necessary for reasons of temperature regulation and control. The sample is cooled using a special liquid nitrogen cooling system. The main part of this comprises a liquid nitrogen Dewar (Dewar) that is connected to your DMA module.

This chapter describes the operating principles of the liquid nitrogen Dewar, how you install the Dewar and how you put the liquid nitrogen cooling system into operation. In addition, it describes how you refill the empty Dewar.

3.1 Operating principles of the liquid nitrogen Dewar

The operating principles of the liquid nitrogen Dewar are explained with the help of Fig. 3-1.

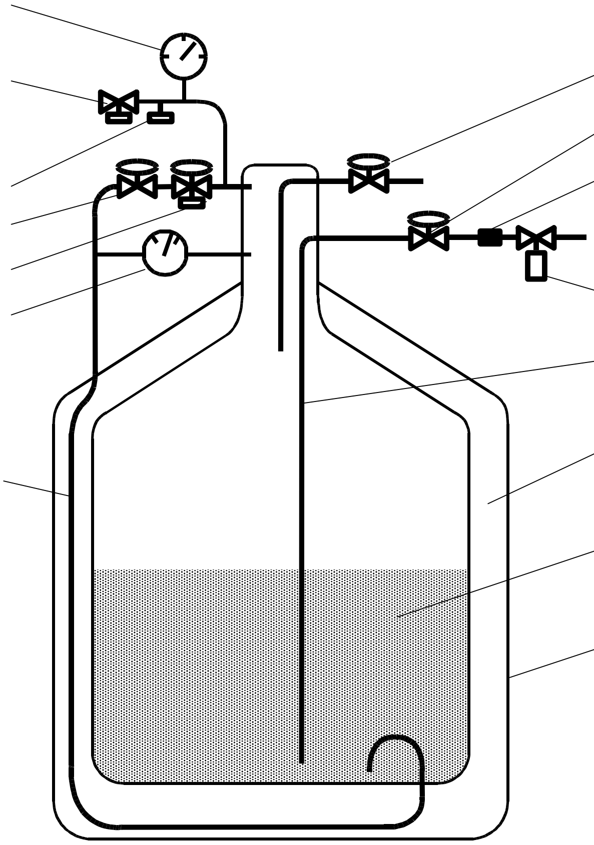


Fig. 3-1. Diagram of the liquid nitrogen Dewar

Nitrogen flows at the constant rated overpressure of 150 kPa from the liquid nitrogen Dewar via the rising pipe and the tube joint to the electromagnetic valve and then on to the heat exchanger in the furnace of the DMA module.

The pressure-building coil, pressure-building valve and pressure controller make up the **automatic pressure building system**. When the pressure-building valve and the pressure controller are open, liquid nitrogen flows into the pressure-building coil. There is good heat exchange between this coil and the under side of the Dewar. This causes the liquid nitrogen in the coil to vaporize and as a result of this the pressure in the Dewar increases.

When the rated overpressure is reached, the pressure controller closes and liquid nitrogen can no longer flow though the coil. The pressure remains constant. The rated pressure is typically 150 kPa; it can however be set by the pressure controller. The pressure controller also ensures that the pressure in the Dewar remains constant when nitrogen is discharged.

The purpose of the other fittings on the left side of Fig. 3-1 is to ensure that the Dewar operates safely. The level gauge indicates level of liquid nitrogen in the Dewar. The level is however only measured correctly when the pressure is stable and the pressure controller is practically closed.

3.2 Operating principles of the liquid nitrogen cooling system

The automatic pressure building system ensures that the liquid nitrogen Dewar is under a constant pressure (typically 150 kPa). Liquid nitrogen passes via the electromagnetic valve to the heat exchanger in the furnace of the DMA module and vaporizes. Nitrogen consumption is further reduced by taking advantage of the heat of vaporization.

A control system in the DMA module ensures that exactly the right amount of nitrogen flows at any phase of operation. This thereby minimizes the consumption of nitrogen. The control variable is the temperature in the heat exchanger of the DMA module. The DMA module controls the flow of liquid nitrogen to the furnace at the electromagnetic valve via an electrical connection. The control system also ensures that the temperature difference between the heat exchanger and the furnace does not exceed a certain value.

At high furnace temperatures or high heating rates the temperature difference between the furnace and the heat exchanger is large so that the electromagnetic valve closes completely.

Since the temperature of the heat exchanger must not exceed 400 °C, the control system switches the cooling on again at high furnace temperatures.

3.3 Delivery

METTLER TOLEDO supplies your DMA module complete with a liquid nitrogen Dewar from Cryofab, Inc.

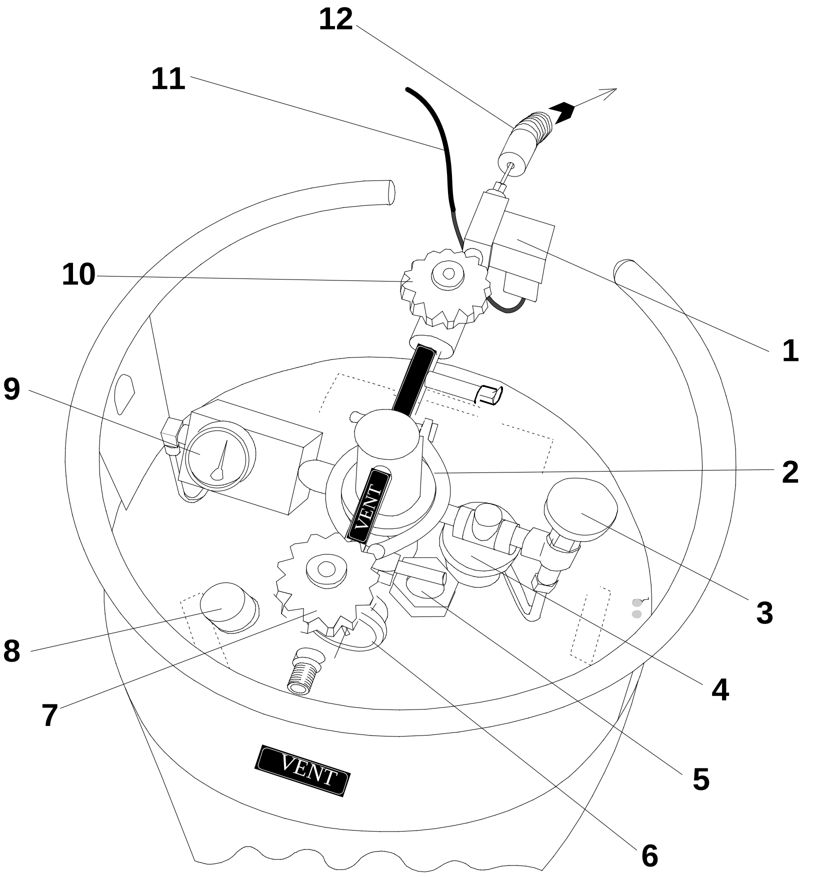
(This liquid nitrogen Dewar is also available as an accessory with order number ME 190 129.)

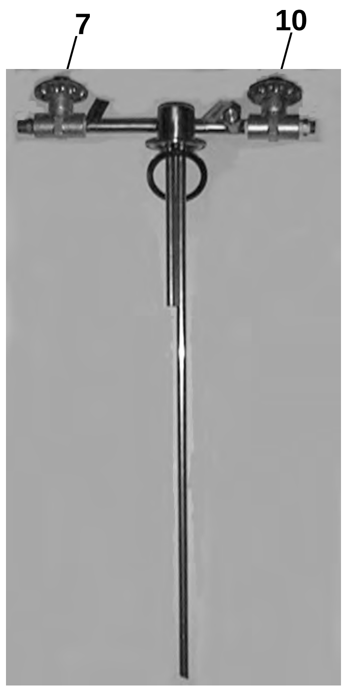
As an alternative, we also recommend the "Apollo" liquid nitrogen Dewar with pressure controller from Messer Griesheim GmbH, Düsseldorf, Germany.

3.4 Installation, start up and shut down

This section describes how you install the liquid nitrogen Dewar and how you put it into and out of operation.

3.4.1 Installing the liquid nitrogen Dewar





Siphon

Fig. 3-2 shows the liquid nitrogen Dewar

Parts:

1 Electromagnetic valve
2 Two-piece locking ring
3 Pressure build-up valve
4 Pressure control valve
5 Rupture disk (4.5 bar)
6 Pressure gauge
7 Pressure release valve (vent)
8 Safety valve (1.5 bar)
9 Level gauge
10 Liquid nitrogen valve
11 Electrical connection for the magnetic valve
12 Vacuum tubing

Fig. 3-2. The liquid nitrogen Dewar

To Install the liquid nitrogen Dewar:

Lift and transport the liquid nitrogen Dewar using a forklift. Make sure the Dewar is supported from below.

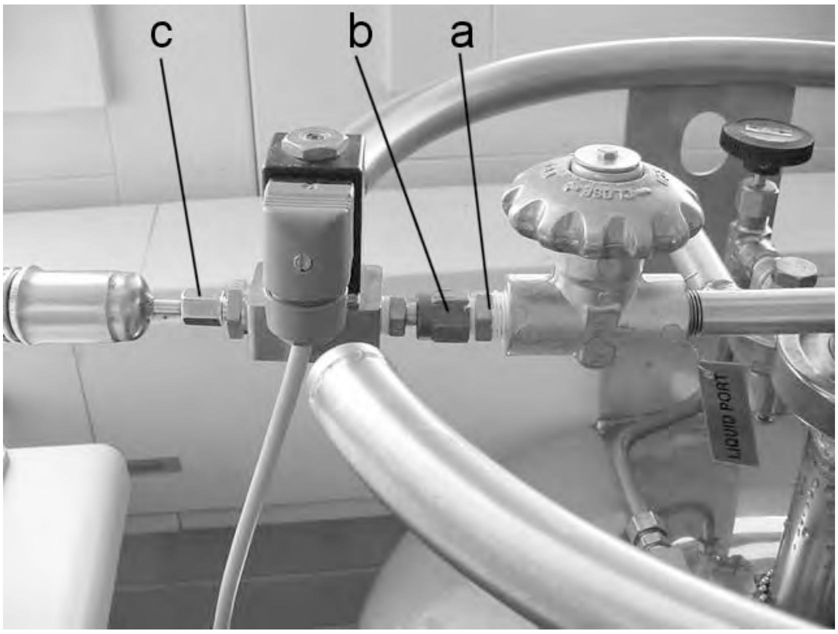
Never try to lift the liquid nitrogen Dewar with the handles or with hoisting loops wound round the outer part of the Dewar.

(1) Position the liquid nitrogen Dewar next to the DMA module so that vacuum tubing can be easily fitted.

⇨ *See page 3-5, Fig. 3-2*

(2) Screw the supplied fitting **a** into the inside thread in the end of the liquid nitrogen valve **10**.

♣ We recommend to use teflon thread or a similar sealing agent to seal the fittings.



(3) Connect the supplied electromagnetic valve **1** to the fitting on the end of the liquid nitrogen valve **10** by tightening the nut **b** firmly with a 17 mm open end wrench. The other end of the electromagnetic valve has a fitting for the connector **c** on the end of the vacuum tubing.

(4) Connect the vacuum tubing to the electromagnetic valve **1** by screwing the nut on the connector **c** tightly on to the fitting on the electromagnetic valve with a 12 mm open end wrench.

(5) Connect the electromagnetic valve control cable **11** to the electromagnetic valve by attaching the connector on the cable to the three prongs on the side of the electromagnetic valve. Fasten the connector securely by tightening the screws at the back of the connector.

The liquid nitrogen Dewar is installed.

3.4.2 Starting up and shutting down the liquid nitrogen Dewar

Always wear protective goggles and gloves when working with liquid nitrogen! It can cause severe burns on your skin.

Make sure that you have been trained in the correct operation of the liquid nitrogen Dewar.

Do not move the liquid nitrogen Dewar when measurements are in progress and if the electromagnetic valve is iced up! Frozen tubing could break and liquid nitrogen could flow out.

Ventilate closed rooms as frequently as possible! High concentrations of nitrogen are dangerous and can cause suffocation.

To start up the liquid nitrogen Dewar:

(1) Fully open the pressure build-up valve **3** with the control knob. Then turn it half a turn backward.

(2) Fully open the liquid nitrogen valve **10** with the control grip.

The liquid nitrogen Dewar is ready for operation

Always put the liquid nitrogen Dewar out of operation if it is not required for longer periods.

There is always the danger that liquid nitrogen could escape uncontrollably.

To shut down the liquid nitrogen Dewar:

(1) Completely close the pressure build-up valve **3** with the control knob.

(2) Completely close the liquid nitrogen valve **10** with the control grip.

The liquid nitrogen Dewar is out of operation.

3.5 Filling the liquid nitrogen Dewar

Always wear protective goggles and gloves when working with liquid nitrogen!

It can cause severe burns on your skin. Make sure that you have been trained in the correct operation of the liquid nitrogen Dewar.

Do not move the liquid nitrogen Dewar when measurements are being performed and if the electromagnetic valve is iced up! The frozen tubing could break and liquid nitrogen could flow out.

Ventilate closed rooms as frequently as possible! High concentrations of nitrogen are dangerous and can lead to suffocation.

To prepare the liquid nitrogen Dewar for filling:

preparing for filling

(1) Switch off the DMA module or put it in the standby mode, (indicated by the **IDLE FURNACE OFF** display on the LCD). In this state the electromagnetic valve is closed and no nitrogen can escape.

♣ The DMA module is in the **IDLE FURNACE OFF** mode, if an experiment has not been started in the Module Control Window. You can recognize an experiment in progress in the Module Control Window by the red bar and the experiment description that appears in bold type in the experiment buffer.

(2) Close the liquid nitrogen valve **10**.

(3) Unscrew the electromagnetic valve using a 17 mm open-end wrench.

The liquid nitrogen Dewar is disconnected from the DMA module and is ready for filling.

There are two ways to fill the liquid nitrogen Dewar.

➩ *See page 3-5, Fig. 3-2*

To fill the liquid nitrogen Dewar via a liquid nitrogen line:

(1) Close the pressure build-up valve **3**.

(2) Open the pressure release valve **7** **slowly** to release the pressure. Check the pressure drop on the pressure gauge **6**.

(3) Connect the filling line of the liquid nitrogen supply to the threaded tube fitting **a** (1/4" outer thread) at the liquid nitrogen valve **b** (see *Fig. 3-3*).

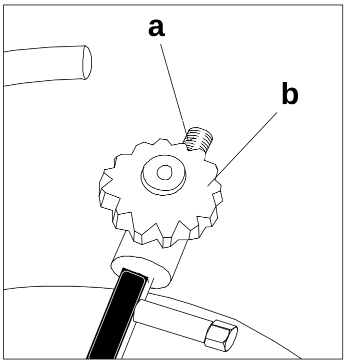


Fig. 3-3. Connection for the filling line

(4) Apply the filling pressure.

(5) Open the liquid nitrogen valve **10** carefully.

The filling process begins. You can see when the liquid nitrogen Dewar is full by observing the level gauge.

The cold nitrogen vapor that is formed on filling escapes via the open pressure release valve.

♣ You can follow the filling process on the filling gauge of the liquid nitrogen Dewar. The filling rate depends on the filling pressure (the filling rate at 0.5 bar overpressure is about 1.4 l/min).

(6) When the liquid nitrogen Dewar is full, shut off the filling pressure and close the liquid nitrogen valve **10**.

(7) Disconnect the filling line and screw on the electromagnetic valve **1** again.

(8) Close the pressure release valve **7**.

The liquid nitrogen Dewar is now full.

To fill the liquid nitrogen Dewar via the siphon opening:

⇨ *See page 3-5, Fig. 3-2*

(1) Close the pressure build-up valve **3**.

(2) Open the pressure release valve **7** **slowly** to release the pressure. Check the pressure drop on the pressure gauge **6**.

(3) Slacken off the two halves of the locking rings **2** and re-move the siphon.

Removing the siphon

Siphon

Fig. 3-4. Removing the siphon from the liquid nitrogen Dewar

♣ The actual filling process depends on your supply of liquid nitrogen. Ask you gas supplier for instructions.

(4) Insert the filling line into the liquid nitrogen Dewar and begin filling.

The filling process begins. The liquid nitrogen Dewar is full when the liquid nitrogen is filled up to the opening of the Dewar. Make sure that liquid nitrogen does spill over.

(5) Remove the filling line from the liquid nitrogen Dewar.

(6) Reinsert the siphon **carefully** into the Dewar. Make sure that the inlets are free of ice particles and that the sealing ring is properly located.

Liquid nitrogen can temporarily escape through the pressure release valve.

(7) Screw on the electromagnetic valve **1**.

(8) Close the pressure release valve **7**.

The liquid nitrogen Dewar is now full.

4 Installation

Contents

4 Installation

This chapter explains how you install your DMA module and how you set it up for operation with the S**TA**R**e** Software on your PC and the liquid nitrogen cooling system. The chapter also describes the procedures necessary to install the DMA module within the S**TA**R**e** Software.

Instructions for installing the S**TA**R**e** Software can be found in the Chapter *Installation Instructions* of the S**TA**R**e** Software Operating Instructions.

4.1 Installing your DMA module

This section explains how you install and connect your DMA module.

4.1.1 Location

The DMA/SDTA861^e is designed to operate trouble-free at room temperatures between +10 °C and 31 °C and at relative humidity of air below dewpoint.

Electromagnetic fields are present everywhere in our environment. These can cause interference and affect the performance of DMA/SDTA861e thereby creating artifacts.

When choosing the installation location for the DMA/SDTA861e, please avoid the following electromagnetic disturbances:

electromagnetic disturbances

- There should be no vertical power lines, motors, or similar devices near the module. Check the neighboring rooms as well.
- Make sure that the following minimum distances are kept between the DMA/SDTA861e and the following devices:
  - 0.5 m to a PC
  - 1.0 m to a printer or a plotter with a power supply transformer
  - 1.0 m to any lamp with a fluorescent tube
  - 5.0 m to a refrigerator or a deep freezer
  - All other types of electrical equipment are possible sources of interference and should therefore kept away from the DMA/SDTA861e.
- Do not use a mobile phone, radio transmission equipment or similar devices during measurements.

mechanical disturbances

The DMA/SDTA861e is a very sensitive, high resolution instrument for dynamic mechanical analysis. The DMA/SDTA861e should therefore be protected from mechanical shocks and vibrations. To eliminate the effects of possible vibrations, it should be installed on a solidly constructed bench, ideally a stone bench.

The dimensions and material of the bench should be chosen so that it can easily bear the weight of the DMA modules and PC.

The **weight** of the DMA/SDTA861e is approx. 120 kg

The **dimensions** of the DMA/SDTA861e are:

Width:	80 cm
Depth:	65 cm
Height:	75 cm

There should be at least 20 cm free space at the rear and on both sides of the DMA module for cables and tubing.

Stone bench

If the instrument and the PC are installed together on the same bench, the bench should have the following dimensions:

Length x width x height: 150 x 90 x 90 cm

The above picture shows a suitable stone bench on which the DMA/SDTA861e can be installed.

vibration damping

For most measurement conditions it is sufficient to use a stone bench without vibration dampers. To meet high demands on measurement accuracy vibration dampers can be inserted between the supports and the stone table slab. Please note however that the resonance behavior of the DMA module will be different after this measure.

4.1.2 Delivery

After receiving your DMA/SDTA861^e from your METTLER TOLEDO representative, unpack it carefully and check the following points:

- Is the equipment complete and does it contain the desired accessories? Have you received the right version of the clamping assembly?
- Have you received all the necessary tubing and fittings for connection of gases?
- Is the accessory box complete according to your order?

Please contact your METTLER TOLEDO representative if anything is missing or obviously damaged.

Parts supplied

The parts supplied with your DMA/SDTA861^e depend on the configuration of your DMA/SDTA86^e you specified in your order. For further details on accessories see chapter 12, Accessories.

- The stand and the body containing the electronics are basically the same in all configurations.
- Depending on the force range you specified in your order small or the large drive motor is included. For force ranges up to 18 N the small drive motor is installed, for force ranges from 20 N to 40 N the large drive motor is installed.
- The DMA/SDTA861^e can be configured for two frequency ranges: for 200 Hz or 1000 Hz.
- Depending on the clamping assembly you ordered, different clamps are supplied as the following table shows:

Clamping assembly	Clamp
Small clamping assembly	Shear clamp
Large clamping assembly	3-point bending Dual/Single cantilever Compression clamp Tension clamp (films, fibers, rods and bars)
Adjustment assembly	None (No clamp is used with the adjustment assembly.)

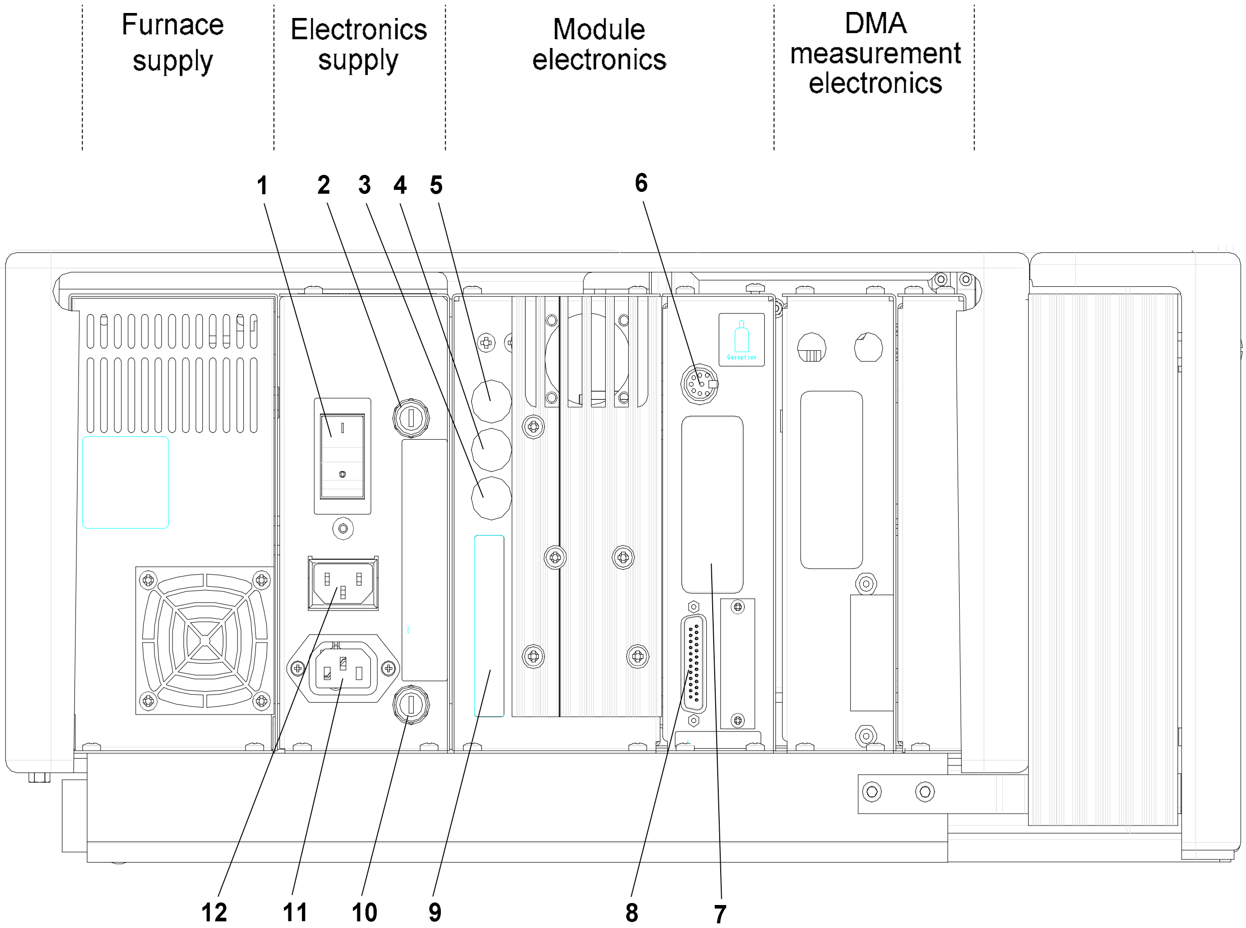
- The accessories box always contains the alignment aid and the adjustment assembly as well as a standard set of accessories. See also chapter 12, *Accessories*.
- Normally a liquid nitrogen container from Cryofab, Inc. is shipped with the instrument.
- The TSO800GC or TSO800GC1 gas controller for the dry gas supply is optional.

4.1.3 Setting up and making connections

The DMA/SDTA861e is very heavy. At least four people should be available to lift it.

Never try to lift the DMA/SDTA861e alone – you could injure yourself.

When setting up the DMA module, take account of the information in the section *Location*.



Parts:

1	Mains switch	**7**	Module software cartridge
2	Fuse for electronics supply	**8**	RS232 interface
3	Connection for synchronization with external devices	**9**	Rating plate
4	Connection for output from gas controller for gas 1	**10**	Fuse for line output socket
5	Connection for control of liquid nitrogen cooling	**11**	Mains connection
6	Connection for output from gas controller for gas 2	**12**	Mains output (for "Switched Line Output " option

Fig. 4-1. Connections on the back of the DMA module

Electronic connections

Fig. 4-1 shows the back of the DMA/SDTA861^e with the electronic connections. The different electronic units furnace supply, electronics supply, module electronics and DMA measuring electronics are visible at the back.

Set up the DMA module and make connections

The following procedure shows you how to set up the the DMA/SDTA861^e and make the electronic and gas connections.

To set up the DMA module and make connections:

(1) Set up the DMA/SDTA861^e (unpacked) at the desired location.

♣ We recommend that you use a fork lift to transport the DMA module to its location and lift it up to the level of the stone bench.

(2) Adjust the DMA/SDTA861^e horizontally by using the leveling screws and a bubble level.

(3) Make the following electrical and electronic connections (see Fig. 4-1):

- Using the power cable supplied, connect the power connector at the rear of the instrument to a power supply receptacle.
- Using the RS232 cable supplied, connect the connector **8** (5-pole, female) of the RS232 interface at the rear of the DMA module to the connector (9-pole, male) of the RS232 interface (COM1 or COM2) at the rear of your PC (desktop or laptop).

♣ If the R232 interface of your PC has a 25-pole connector, you can order a suitable 25-pole RS232 cable from your METTLER TOLEDO representative.

(4) Make the following gas connections:

- Connect the liquid nitrogen Dewar to the vacuum tubing as explained in chapter 3 in section *Installing the liquid nitrogen Dewar*.
- Connect the dry gas supply as explained in section *Installing the dry gas supply*.

Installing the dry gas supply

A supply of inert or oxidative gas can be installed in addition to the supply of liquid nitrogen required for cooling the sample. Dry gas (for example nitrogen) prevents condensation of moisture on the inside surfaces of the furnace. In some experiments, however, it is necessary to create an oxidative atmosphere.

A flowmeter is needed to install the gas supply. The TSO800GC or TSO800GC1 gas controller can also be used instead of a flowmeter.

To install the gas supply:

(1) Connect the inlet of the flowmeter to the gas supply.

♣ The gas is supplied from a bottle or from a permanently installed in-house supply.

(2) Connect the outlet of the flowmeter to the dry gas inlet on one of the arms of the furnace of the DMA module.

(3) Set the gas flow at the flowmeter to about 200 ml/min.

♣ The flowmeter should not indicate any fluctuation of flow. A fluctuation of the flow rate indicates possible problems in the gas supply (e.g. gas bottle empty or problems with the in-house supply).

4.2 Installing your DMA module in the STARe Software

Before you start up your DMA/SDTA861e you have to make some preparations in the the STARe Software. This section describes the functions of the STARe Software Install Window that are specific to the DMA module. The full description of the Install Window can be found in the online Help to the the Install Window.

Chapter 5, S*witching on and switching off,* explains how to switch on your DMA/SDTA861e.

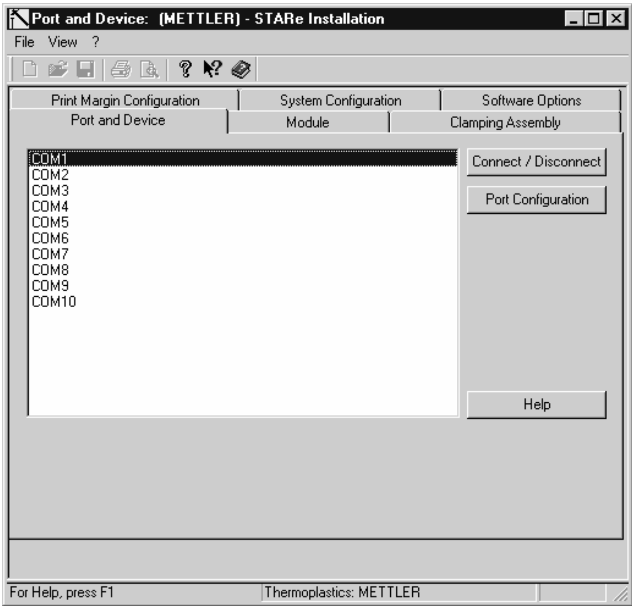
4.2.1 Open the Install Window

opening the Install Window

- Click Functions/Install Window in the main menu bar of the STARe Software.

  An Install Window opens and the symbol 

 appears in the Windows task bar.



4.2.2 Installing the DMA module in the STARe Software

To install your DMA module in the STARe Software, you have to open a dataset belonging to your module in the STARe Software. You can import the dataset from the module floppy disk supplied and open it in the STARe Software. You can also create a new individual dataset and perform the installation in the STARe Software step by step.

To import the module dataset of your STARe module from the module floppy disk supplied

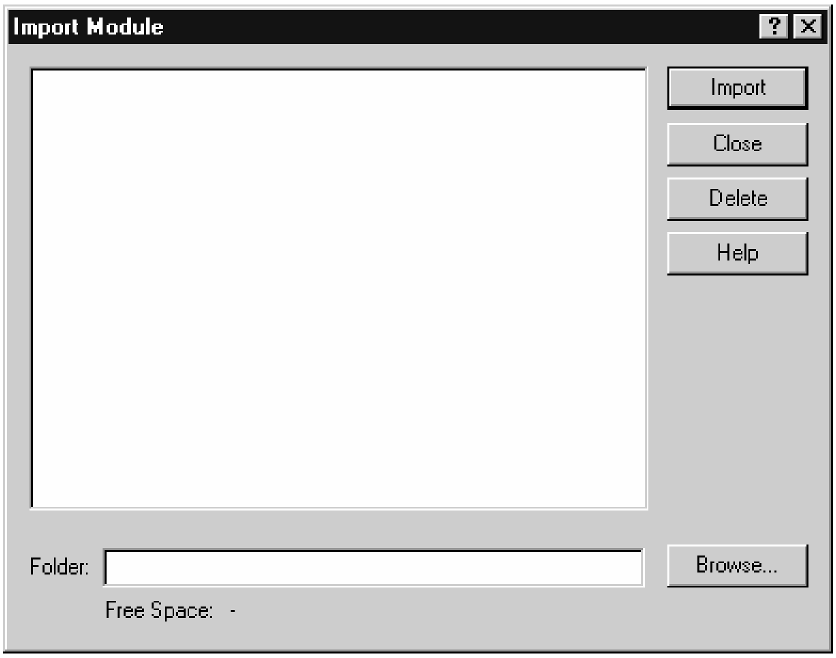
installation with the module floppy disk

♣ For data import you must make sure that the floppy disk is not write-protected.

(1) Insert the module floppy disk in the disk drive.

(2) In the Install Window, click the Module tab and then click Import on the File menu.

The Import Module dialog box appears.



(3) Enter the path in the Folder box, e.g. A:\

The name of your DMA module dataset appears in the list. This dataset was created during adjustment in the factory and contains the default settings.

(4) Mark the dataset of your DMA module and click Import.

The module dataset of your DMA module is imported from the floppy disk together with the settings and adjustment data.

The module dataset of your DMA module is imported in your STARe Software database. You can now open the database.

To create a new module dataset without an installation floppy disk:

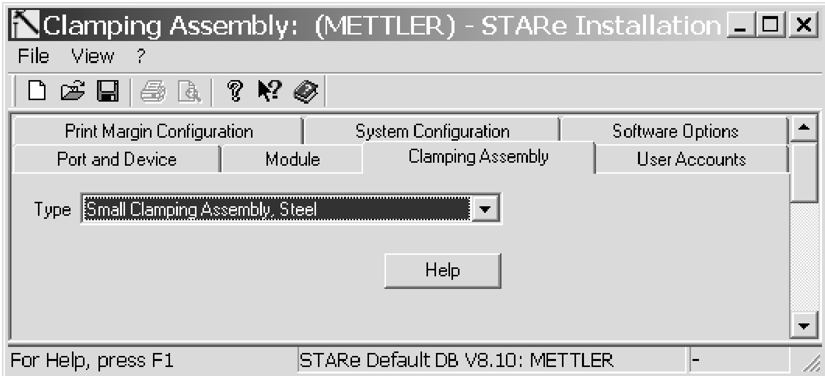
creating a new clamping assembly dataset

(1) Open a new dataset for a clamping assembly for your DMA module in the database first as follows:

♣ The Clamping Assembly box is per default set for the small steel clamping assembly and is marked Small Clamping Assembly, Steel.

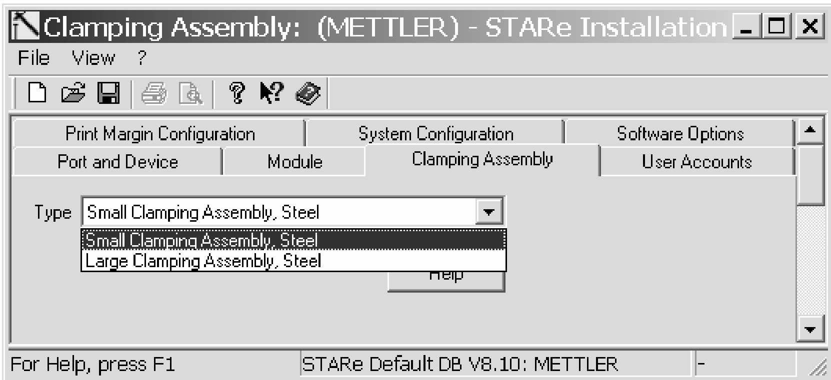
- In the Install Window, click the Clamping Assembly tab.

  The Clamping Assembly tabbed page appears.



- Click in the Type box.

  A list of the different types of clamping assemblies appears.



- Select the desired clamping assembly in the list.

- Click File/Save As...

  The Save As dialog box appears.

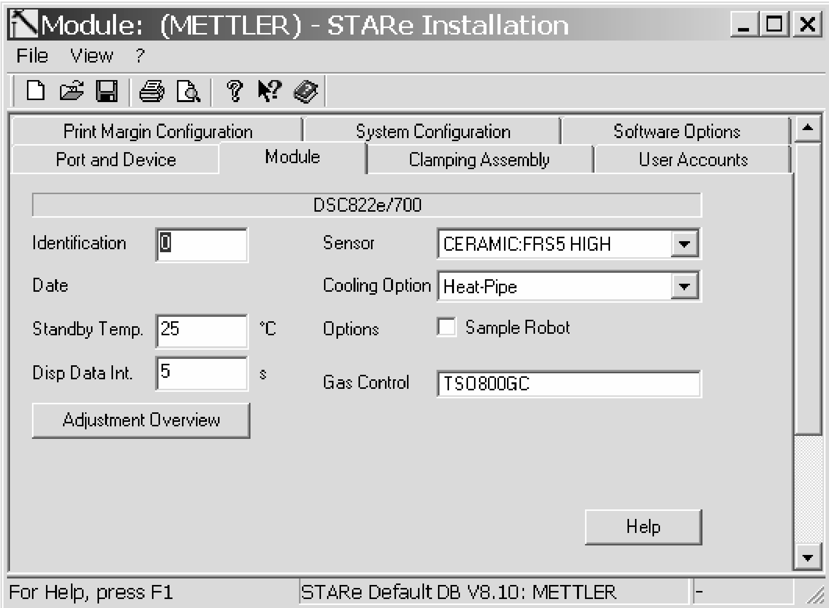
- Give the clamping assembly a meaningful name (for example SCA for small clamping assembly) and click OK.

The clamping assembly is entered in the database.

creating a new module dataset

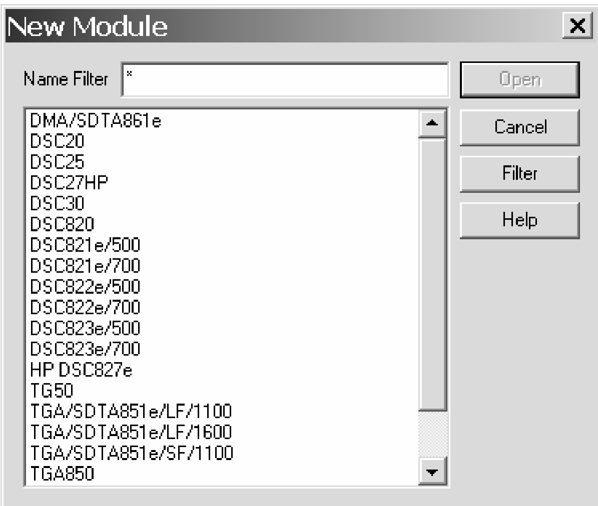
(2) Selct the Module.tabbed page.

The Module dialog box appears. An DSC822e/700 module dataset always appears by default.



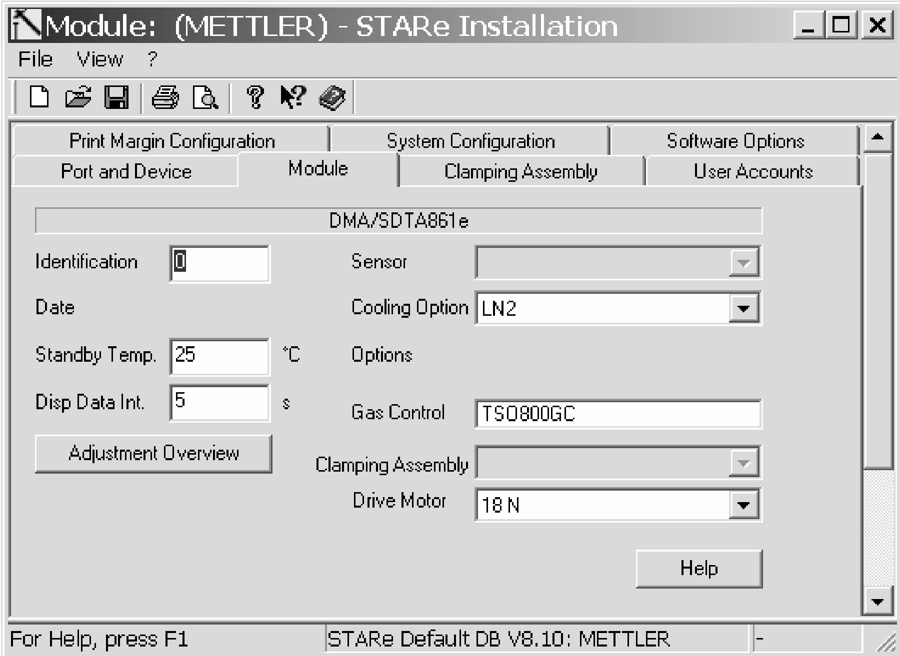
(3) Click File/New:

The New Module dialog box appears.



(4) Select DMA/SDTA861e as the type of measuring module and confirm with OK.

The Module dialog box opens and the default dataset for a DMA module appears in the Module dialog box. You can enter data relevant to the configuration of your DMA module.



♣ For DMA modules, the Sensor button is not active and the Cooling Option button is only configured for liquid nitrogen cooling and is labeled LN2.

(5) Enter the following information for your DMA module in the text boxes in the left half of the dialog box:

Identification number

- Enter the identification number of your DMA in the Identification text box. This is always necessary because the STARe Software identifies your DMA module on the basis of this entry.

♣ The identification number appears in the LCD display on starting the measuring module.

Standby temperature

- If you want to change the standby temperature: Enter the desired standby temperature for your measurements in the Standby Temp. text box. The default value is 25 °C.

Data display interval

- If you want to change the data display interval: Enter the interval for the data display in the Module Control Window in the Disp Data Int. text box. The default value is 5 s).

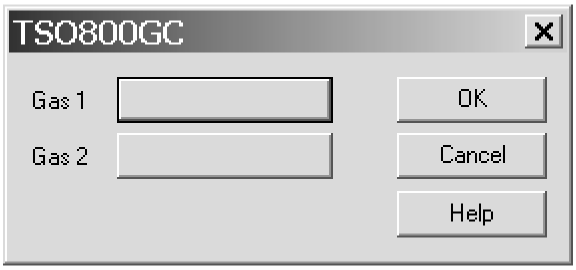
♣ The data display interval does not influence the measurement. It has to do with the updating of the display of data in the Module Control Window (refresh rate).

♣ By clicking the Adjustment Overview button you get an overview of the current adjustment data. More information on this is available in the section *Adjustment data in the Install Window*.

(6) If you have installed a gas controller: Click the Gas Control button to define the gases.

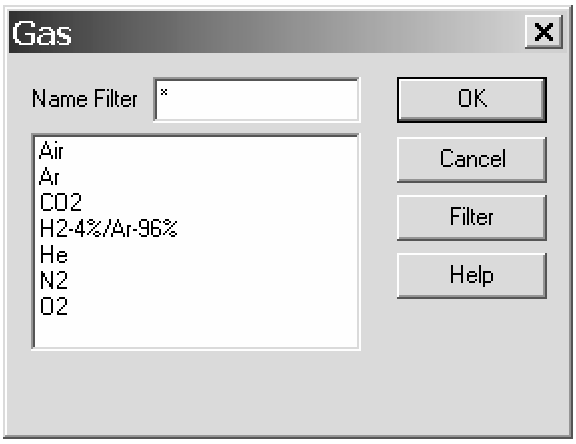
Gas Controller

The TSO8000GC dialog box appears.



- Double-click the Gas 1 box.

The Select Gas dialog box appears with a list of all the gases.



- Select the desired Gas 1 in the list.
- Double-click the Gas 2 button.

The Select Gas dialog box appears with a list of all the gases.

- Select the desired Gas 2 in the list.
- Confirm with OK.

The gas controller settings are defined.

(7) Select a clamping assembly:

♣ The clamping assembly dataset must be entered in the database according to step (1).

- Click the Clamping Assembly button in the Module dialog box.

  A list of the clamping assemblies entered appears.

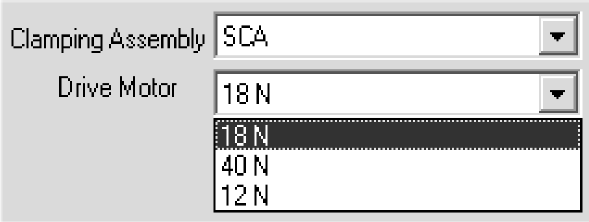
- Click the name of the desired clamping assembly.

The desired clamping assembly dataset is linked to the dataset of your module.

(8) Select the dataset of your drive motor as follows:

- In the Module tabbed page, click in the Drive Motor box.

A list of drive motor forces (12 N, 18 N and 40 N appears.



- Click the desired drive motor force.

The dataset of the selected drive motor is linked with the dataset of your module.

(9) Save the dataset of your DMA module in the database as follows:

save dataset

- Choose File/Save as…

  The Save as dialog box appears.

- Enter a name for your module (for example DMA/SDTA861e ID3).

♣ Include only the module type (in this case DMA/SDTA861e) and the ID of your module (ID3) in the name. Do not include information about other devices such as the type of clamping assembly used in the name since the module can be linked to different devices (e.g. small or large clamping assembly).

- Click OK.

The dataset of the module is stored in the database.

♣ The name entered appears as the title of the Module Control Window after you have established communication between your DMA module and the STARe Software. The same name also appears in the Experiment Window as the label of the button next to the text Send Experiment.

The module dataset created in this way contains only the default adjustment data. If you want to use a module dataset created in this way for your measurements, you must first of all adjust your DMA module. You can find instructions on how to adjust your DMA module in the Chapter *Calibration and Adjustment*. During the adjustment, the STARe Software saves the new adjustment dataset and at the same time overwrites the previous adjustment data.

4.2.3 Establishing communication between the STAR^e^ Software and your DMA module

If you have opened a dataset for your DMA module in the STARe Software, you can establish communication with your DMA module by connecting it to an interface of your PC.

To establish communication between the STARe Software and your DMA module:

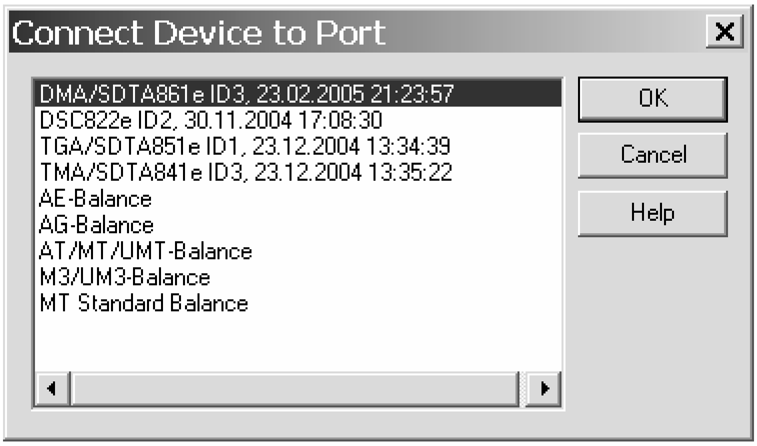
(1) Click the Port and Device tab.

connecting interfaces

(2) Connect your module to the desired port as follows:

- Click one of the entries in the list (e.g. COM1). A TA-module is usually connected to the first or second serial ports COM1 or COM2.
- Then click Connect/Disconnect.

The Connect Device to Port dialog box appears containing a list of the module datasets stored in the database appears.



- Select the desired DMA module dataset and then click OK.

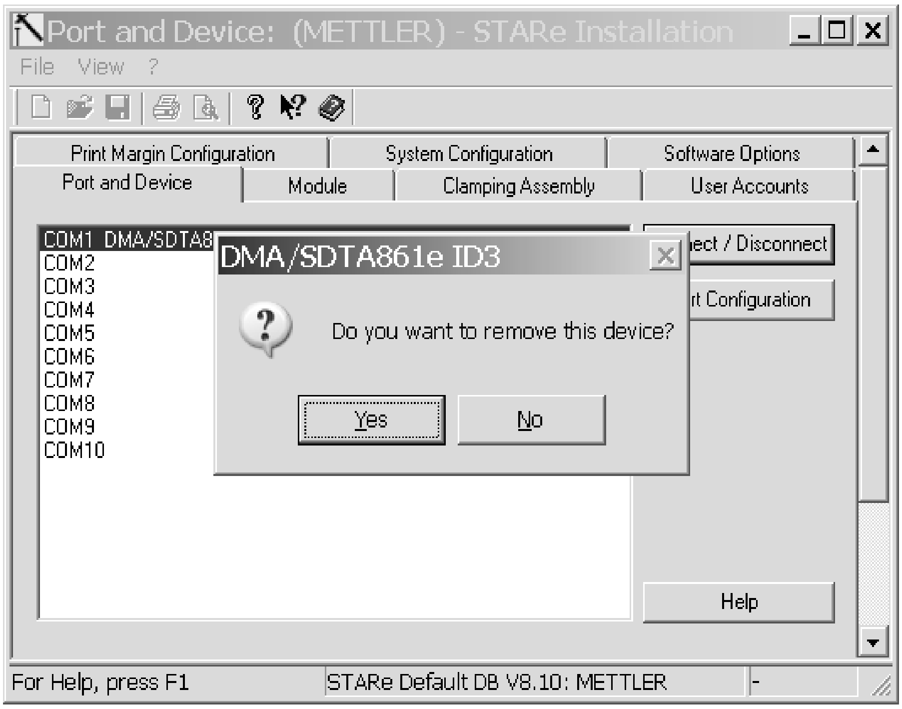
The DMA module is connected to the port and communication is established after a short while. The Module Contol Window opens automatically.

disconnect a module from a port

To disconnect your DMA module from a port:

- In the list, click the entry with the corresponding device name and installation date.

  A dialog box appears with the prompt: Do you want to remove this device?



- Click Yes to disconnect the device from the port.

4.2.4 Adjustment data in the Install Window

In the Install Window, you can look at the current adjustment data of your DMA module, but you cannot change the adjustment values. The data corresponds to the most recent adjustment of your DMA modules. It is identical to the calibration data displayed in the information boxes at the end of the individual calibration runs.

You can find instructions on how to calibrate and adjust your DMA module in the Chapter *Calibration and adjustment*.

Your DMA module was adjusted in the factory before delivery. If you have not performed any adjustments, the adjustment data corresponds to the factory adjustment.

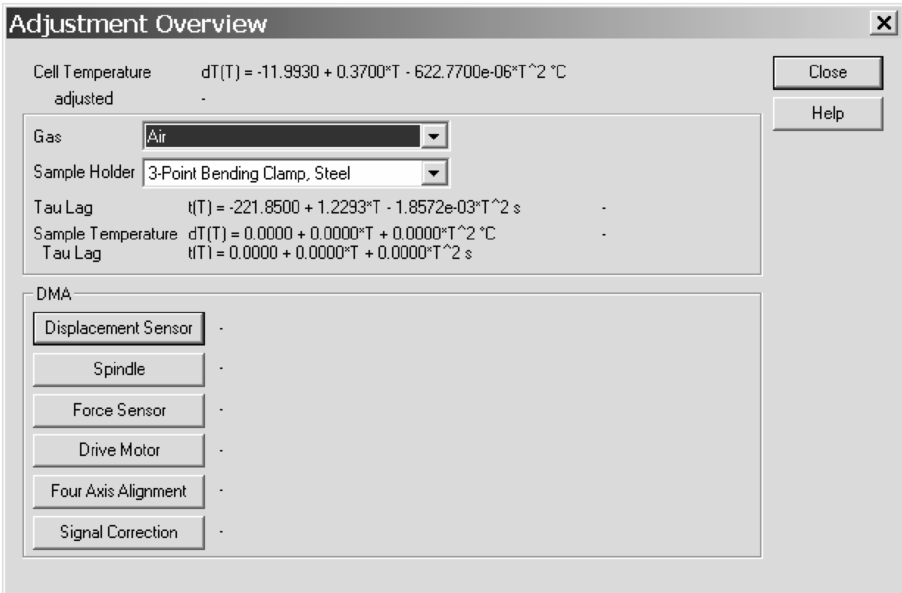
To look at your DMA module adjustment data:

(1) Open the dataset of the desired DMA module in the Install Window. To do this, click the Module tab, click File/Open…, select the desired module dataset and click OK.

The selected module dataset appears in the Module tabbed page.

(2) Click the Adjustment Overview button.

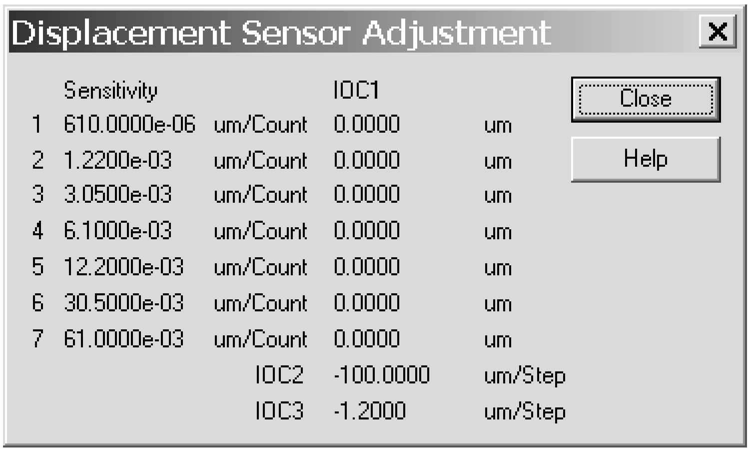
The Adjustment Overview dialog box appears. The thermal adjustment data is displayed in the upper half of the dialog box. The lower half contains buttons for initiating mechanical adjustments.



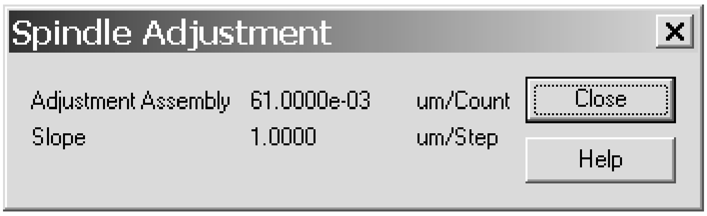
♣ **Please note:** The data displayed next to the text Cell Temperature represents the parameters for the CA temperature (clamping assembly temperature). The label Cell Temperature is incorrect.

(3) To look at the mechanical adjustment data, click one of the buttons in the lower half of the dialog box:

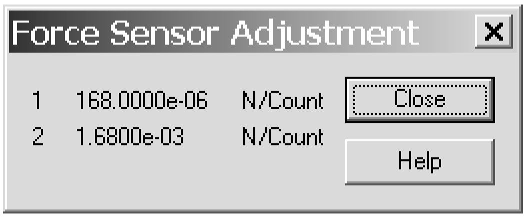
- the Displacement Sensor button:



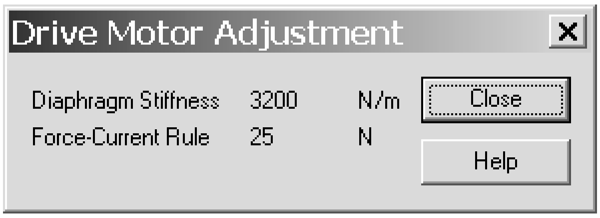
- the Spindle button:



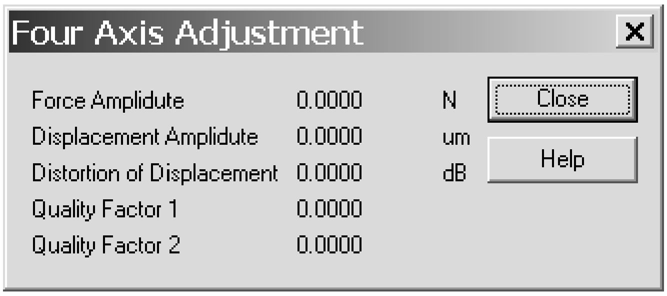
- the Force Sensor button:



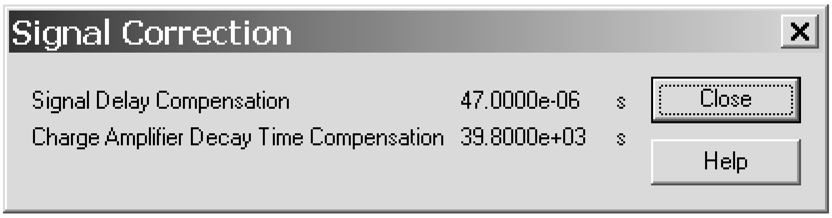
- the Drive Motor button:



- the Four Axis Alignment button:



- the Signal Correction button :



Explanation:

Signal Delay Time Compensation: Delay time or phase shift of the displacement signal with respect to the force signal

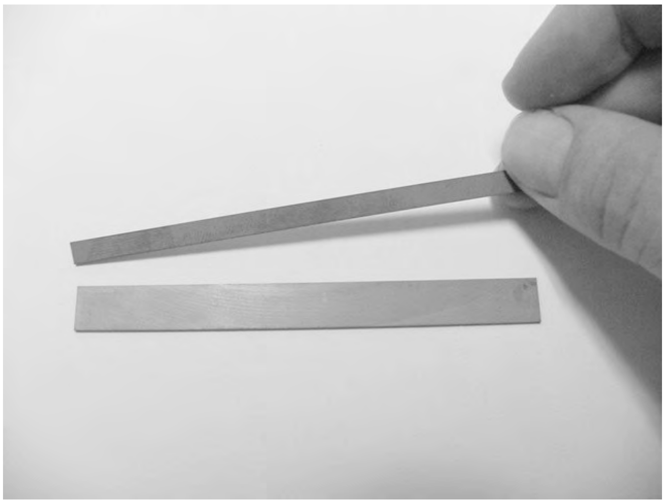
Charge Amplifier Decay Time Compensation: Compensation for signal delay of the force sensor's charge amplifier at low frequencies compensation

♣ The dialog boxes displayed above contain the default data of the STARe database. The STARe Software uses this data if no other adjustment data for your DMA module is available.

4.2.5 Reference measurement after installation

measurement accuracy

- Check the measurement accuracy of the module after you have completed the installation. We recommend that you perform the reference measurement in the 3-point bending mode with the standard silicon samples (shown below) available from METTLER TOLEDO. The modulus values for this material are well documented and are easy to reproduce.



If the measurement results are outside the limits of permissible error, then you have to adjust your module. You can find the instructions of how to do this in the Chapter *Calibration and Adjustment*.

You can get an overview of the current adjustment data by clicking the Adjustment Overview button in the Topic/Module dialog box. More information is available on this in the section *Adjustment data in the Install Window*.

5 Switching On and Switching Off

This chapter explains how you switch your DMA module on, and how you switch it off. The order in which you switch the PC and DMA module on or off does not matter.

Never switch your DMA module off during a measurement.

If you switch the DMA module off, all the data belonging to the current measurement are lost.

♣ If you use a **HP DeskJet** printer, you must switch this on before you switch on the other instruments.

To switch on your DMA module:

(1) Switch on the DMA module by actuating the main switch (red). The main switch is located above at the rear of the instrument.

After it has been switched on, the DMA module performs a start-up routine that lasts about 20 seconds. The following messages appear one after the other on the LCD display:

DMA V1.00 **************	Version of the firmware
DMA V1.00 **SELFTEST**	Performs a self test
DMA V1.00 **SELFCALIBRATION**	Calibration routine
Module starting up **DMA INITIALIZATION...**	Module initializes DMA initialization
IDENT. NO: **SYSTEM: OK**	Display of the identification number of the module
IDLE FURNACE OFF **Ts:**	Instrument ready: furnace switched off, current sample temperature

♣ The identification number is required for the installation of the DMA module in the S**TA**R^{e} Software.

(2) If the text **IDLE FURNACE OFF** appears for longer than 30 seconds on the LCD display:

Your DMA module is ready for operation.

If you have not already done so, you must now switch on the PC, start the STARe Software, and set up the DMA module in the STARe Software in order to begin the measurement. The instructions can be found in the section *Installation* and in the Chapter *Installation Window* of the STARe Software User Manual.

communication problems

If the text: ***COMMUNICATION TIMEOUT*** appears after 30 seconds, there is a communication problem between the DMA module and the STARe Software installed on your PC. In this case, proceed as follows:

- Check that the PC is switched on.
- Check whether the RS232 cable is properly connected at both ends and that the screws on the connectors are properly tightened.
- Check whether the DMA module is properly installed in the Installation Window of the STARe Software under Port and Device, and whether the connection to the RS232 interface has been made. The text with the name of your module should appear in bold next to the corresponding port. If necessary, interrupt the connection of the DMA module to the port under Port and Device and reinstate the connection.
- Switch the DMA module off and then on again.

If the message ***COMMUNICATION TIMEOUT*** still remains after you have checked these points, contact your METTLER TOLEDO service engineer.

To switch off your DMA module:

(1) If you want to access the sample after switching off the module: open the furnace before you switch off the DMA module.

♣ When the module is switched off, you cannot open the furnace and cannot therefore access the sample.

(2) Switch off the DMA module. The main switch is located above at the rear of the instrument.

(3) If you leave the DMA module unattended for a longer period of time after switching it off, put the liquid nitrogen container out of operation. Follow the instructions given in Chapter 4, *Installation* in the section *Installation, start up and shut down.*

The DMA module is switched off.

6 Preparing for a Measurement

Contents

6 Preparing for a Measurement

This chapter describes how to prepare the DMA/SDTA861e for a measurement. We explain how to mount and remove a sample. The procedures vary according to the type of measurement and the clamp or clamping assembly used.

Abbreviations:

For better orientation, the margins of the pages in this chapter include the following signs to indicate the device to which the text in the corresponding section refers:

ALA Alignment aid, used for the text relating to the alignment aid in Chapter 6.1.

SCA Small clamping assembly, used for the text relating to the small clamping assembly in Sections 6.2 and 6.3

LCA Large clamping assembly, used for the text relating to the large clamping assembly in Sections 6.4 and 6.5

4 Axes 4 axes alignment, used for the text relating to the Fine alignment (or 4 axes alignment) procedure in Section 6.6

tools

The tools required for tightening and loosening nuts and screws in the procedures are supplied with the DMA/SDTA861e and are shown in Fig. 6-1. See also *Chapter 12, Accessories*.

Always use the Torx screwdrivers and wrenches supplied to tighten and loosen the Torx screws. The use of a different tool could damage the Torx screws or even parts of the DMA/SDTA861e.

Use the supplied torque wrenches where indicated in these operating instructions to set the correct torque. Parts of the DMA/SDTA861e could suffer damage if different tools are used.

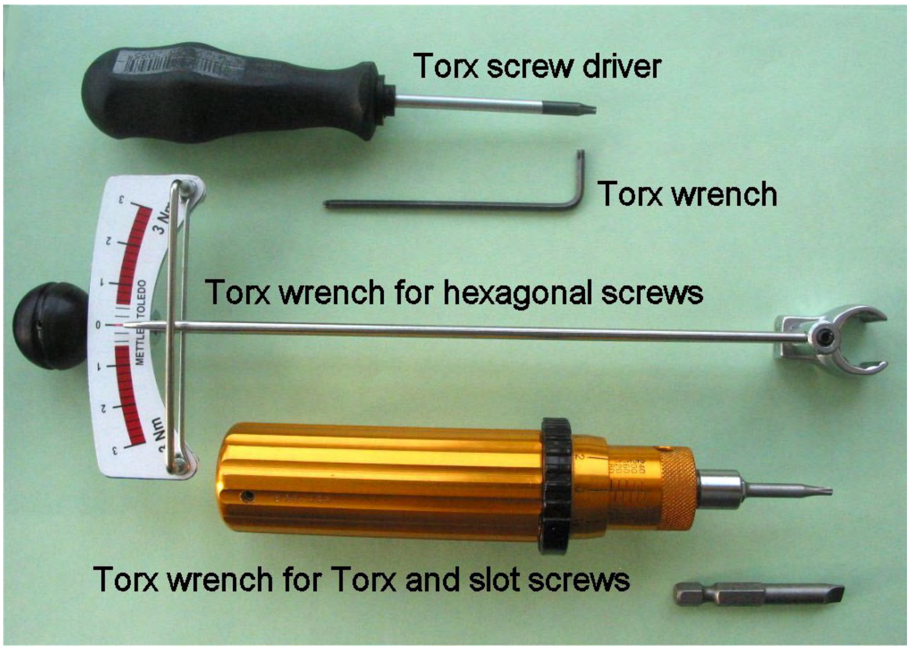


Fig. 6-1. Tools supplied with the DMA/SDTA861e

Message **Risk of damage!** appears on the LCD

If the force sensor measures an unallowed value, the error message **Risk of damage!** appears on the LCD. You can however acknowledge the message by pressing the OK key on the keypad of the module. The third time the message occurs, however, it is no longer possible to acknowledge the message. In this case, you have to switch off the DMA module using the mains switch at the rear of the instrument and then restart it. See also Chapter 10*, Error messages and warnings*.

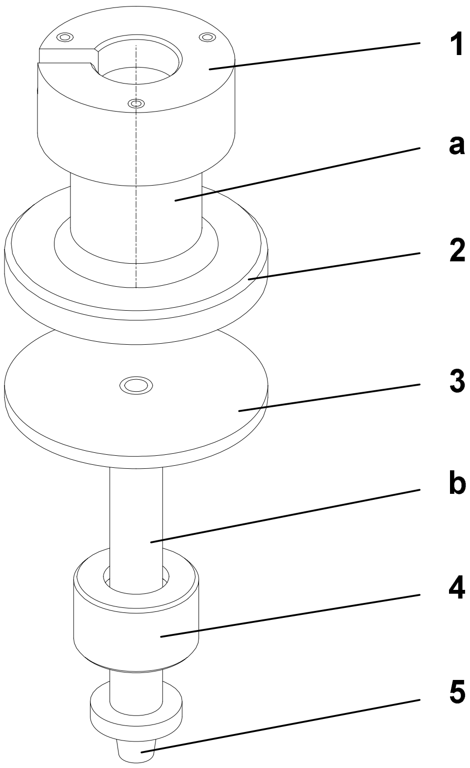
6.1 Pre-aligning the measuring sensors before installing a clamping assembly

Before you install a clamping assembly for the first time, you must adjust the four axes alignment device manually to pre-align the displacement and the force sensors with the clamping assembly.

This alignment procedure has already been performed in the METTLER TOLEDO factory with the clamping assembly you ordered. We recommend that you repeat the alignment procedure described below when you install a new clamping assembly.

6.1.1 Pre-aligning the measuring sensors with the alignment aid

The alignment aid shipped with the DMA/SDTA861^e module serves the purpose of pre-aligning the displacement and the force sensors with the clamping assembly. It is shown in Fig. 6-2:

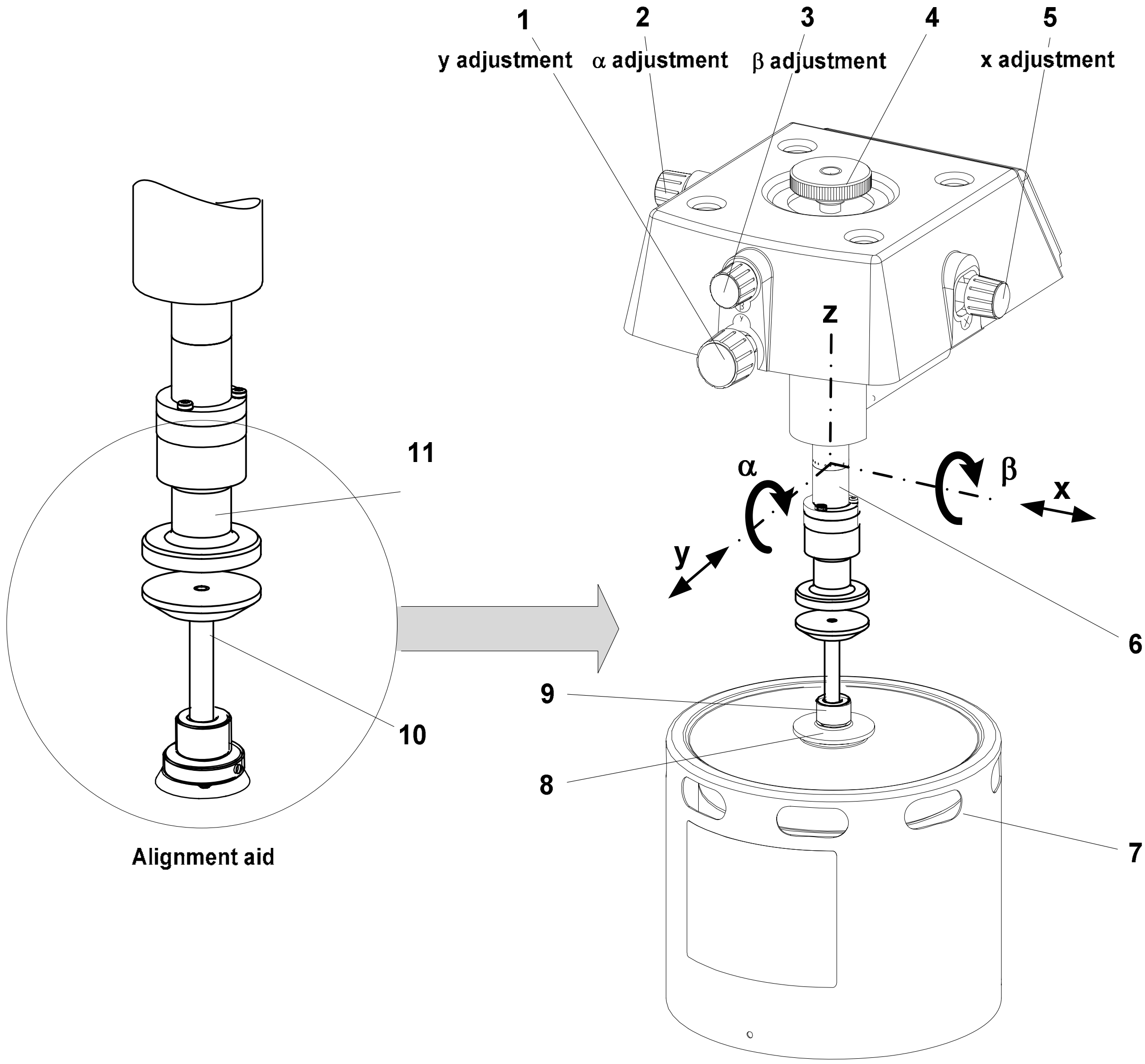


a	Alignment aid, upper part
1	Flange
2	Plate
b	Alignment aid, lower part
3	Plate
4	Nut
5	Conical end

Fig. 6-2. Alignment aid

♣ The fine alignment of the measuring sensors is carried out after the manual pre-alignment using a dialog in the DMA module software (see the section *Fine alignment of the measuring sensors (Four axes alignment)*.

Fig. 6-3 shows the four axes alignment device, the measuring sensors, the alignment aid and the positions and angles to adjust. With the help of the alignment aid, the positions and angles of the measuring sensors can be adjusted quickly so that the sensors are in the right position when you install a clamping assembly.



1	Knob for adjusting the y -position	**7**	Drive motor
2	Knob for adjusting the α angle	**8**	Threaded fitting
3	Knob for adjusting the β angle	**9**	Nut
4	Locking screw	**10**	Alignment aid, lower part
5	Knob for adjusting the x -position	**11**	Alignment aid, upper part
6	Displacement sensor housing		

Fig. 6-3. Pre-alignment with the alignment aid

ALA

To pre-align the measurement sensors with the alignment aid:

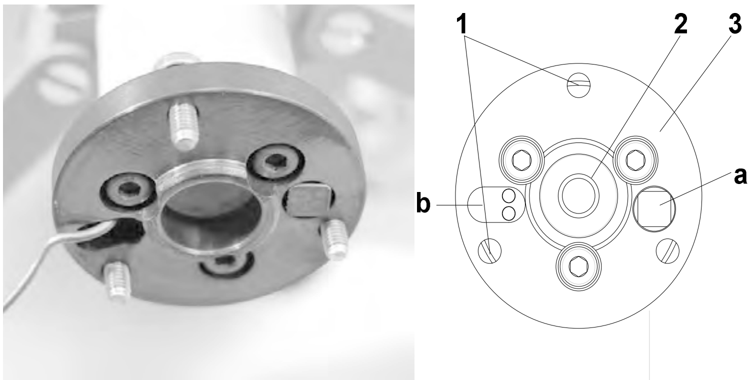
⇨ See page 6-5, Fig. 6-3

Start: - Alignment aid is ready

♣ The mechanism of the 4-axes alignment device operates best when it is pressed together slightly by the locking screw.

Install alignment aid

(1) Fasten the upper part of the alignment aid as follows to the flange of the displacement sensor, ❶:



1 Torx screws
2 Displacement sensor core
3 Displacement sensor flange
a CA thermocouple
b Hole for SDTA thermocouple

Fig. 6-4. Displacement sensor flange (view from below)

- Hold the upper part of the alignment aid under the flange of the displacement sensor. Ensure that the thermocouple projecting from hole **b** is positioned freely in the notch (Fig. 6-4). Make sure you do not wedge the thermocouple while fastening the upper part of the alignment aid.

♣ The Torx screws must be aligned with the tapped holes in the upper part of the alignment aid.

- Screw in the three Torx screws and tighten them firmly with the same torque using the Torx Allen wrench supplied.

The upper part of the alignment aid is now fixed to the flange of the displacement sensor.

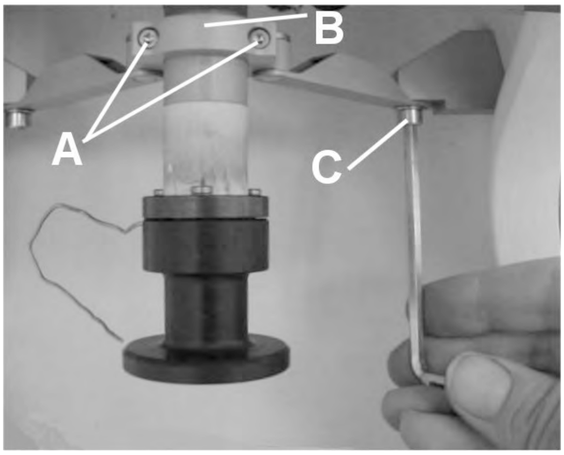
(2) Set the lower part of the alignment aid into the seat in the threaded fitting of the drive motor. The conical end fits snugly into the conical bore. Tighten the nut on the fitting, ❷.

(3) Loosen the support structure of the measuring sensors as follows:

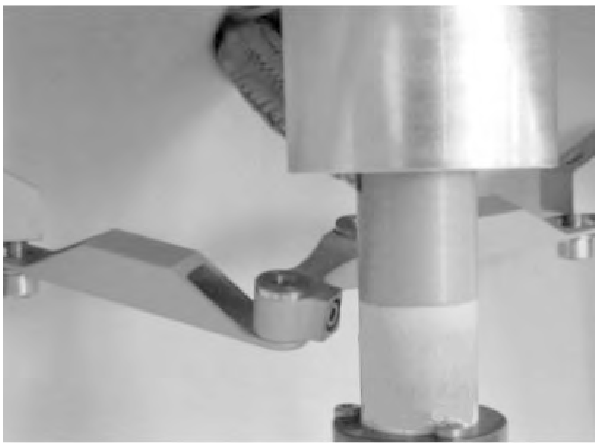
- Remove the Torx screws (A) with the Torx wrench and remove the front clamp (B). Loosen the Allen screws (C) with a 4-mm Allen wrench, ❸, but do not completely remove them so that the support structure is still attached.
- Fold the support structure back so that it is no longer in contact with the displacement sensor flange, ❹.

❶

❷



❸



❹

(4) Loosen the locking screw of the four axes alignment device, ❺, then slightly tighten it again to the point where you feel slight resistance.

Do not drive the lower part of the alignment aid mounted on the z-axis table too far up toward its upper part.

The drive motor could suffer damage.

---

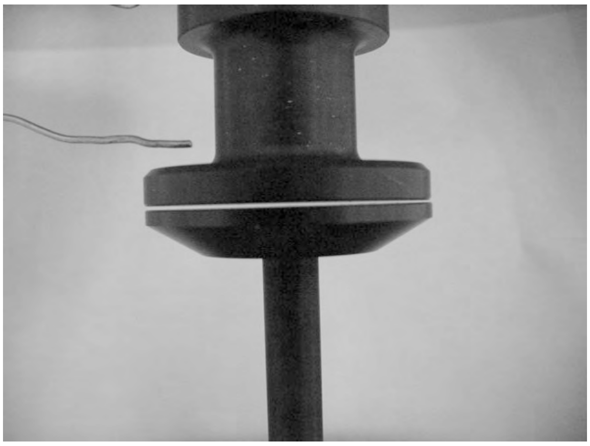
(5) Drive the z-axis table upward as follows.

- Press the MOTOR UP key once. The z-axis table moves upward and stops at an intermediate position. The two plates of the alignment aid are still well apart in this position, **❻-a** and **❻-b**.
- Press the MOTOR UP key and hold it down to slowly move the lower plate of the alignment aid toward the upper plate until the gap between the two plates is about 0.5 mm, **❻-c**.

❺

❻-a

❻-b



❻-c

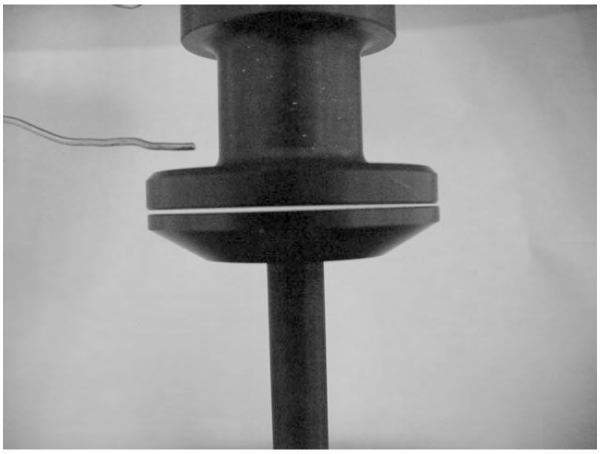
ALA

(6) Check visually in the x and y directions whether the upper and the lower plate are flush with each other, ❼**-a** and ❼**-b**. Using knobs **1** and **5**, adjust the x and y positions so that the plates are flush, ❽**-a** and ❽**-b**.

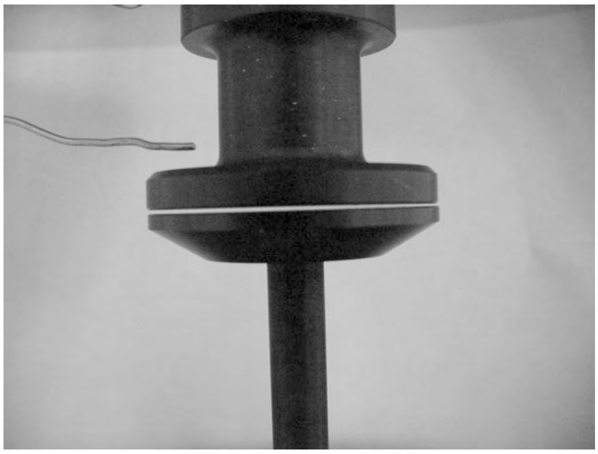
♣ The gap can be more clearly seen if you insert a sheet of white paper behind the adjustment aid.

(7) Check visually whether the upper plate is parallel to the lower plate by looking at the gap in the x and y directions, ❼**-a** and ❼**-b**. Using knobs **2** and **3**, adjust the α and β angles so that the two plates are parallel, ❽**-c** and ❽**-d**.

♣ Knobs **2** and **3** are not mounted when you receive the DMA/SDTA861^e from the factory. You have to mount the knobs first to adjust the angles.



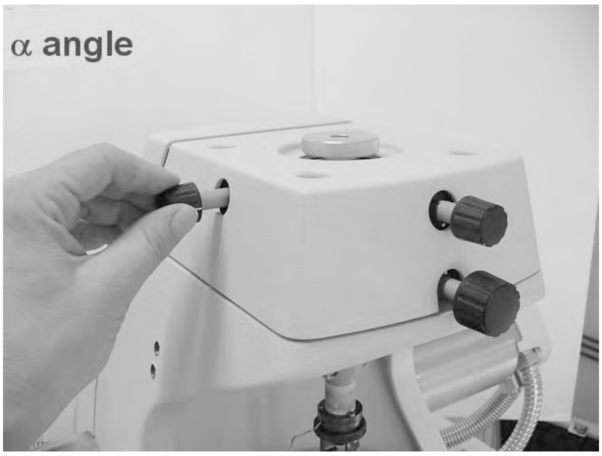
❼-a



❼-b

❽**-a**

❽**-b**



❽-c

❽-d

❾

❿

(8) Drive the z-axis table fully downward. Press the MOTOR DOWN key until the z-axis table stops in the intermediate position. Briefly press the MOTOR DOWN key once more to move the z-axis table into the lower position. The z-axis table stops automatically in the lower position, ❾.

♣ The travel speed of the z-axis table changes from low to high at the intermediate position.

(9) Tighten the locking screw **4**, ❿.

(10) Remove both parts of the alignment aid. To remove the upper half, loosen the three Torx screws on the flange of the displacement sensor core. Loosen the nut and take the lower half out of the seat of the drive motor.

End:
- Pre-alignment completed
- Alignment aid removed

6.2 Installing and removing the small clamping assembly

This section shows you how to assemble, align and install the small clamping assembly. It also explains how to remove the clamping assembly.

Before the clamping assembly can be installed, the measuring sensors must be pre-aligned as described in the section *Pre-aligning the measuring sensors before installing a clamping assembly*. This adjustment ensures that the measuring sensors are optimally aligned before installation. This step is essential for achieving good results.

6.2.1 Assembling and aligning the small clamping assembly

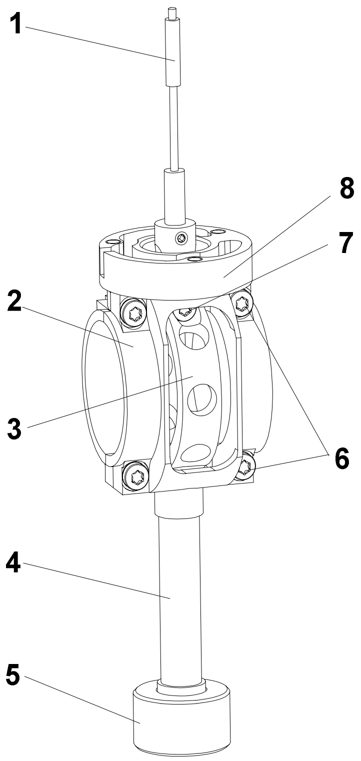
To install the small clamping assembly in the DMA module, you have to align the clamping assembly within itself. If you have previously disassembled the small clamping assembly, you have to first assemble it again.

The following sections describe these steps.

Assembling the small clamping assembly

To align the small clamping assembly within itself, it should be assembled as shown in Fig. 6-5.

The movable part of the small clamping assembly, comprising the displacement sensor core **1**, the movable part of the clamp holder **3** and the drive shaft **4**, is installed in the fixed part of the clamp holder **2**.



1 Displacement sensor core
2 Clamp holder, fixed part
3 Clamp holder, movable part
4 Drive shaft
5 Nut
6 Torx screws in the fixed part of the clamp holder
7 Torx screw in the movable part of the clamp holder
8 Flange

Fig. 6-5. Assembled small clamping assembly

SCA

Normally there is no need to disassemble the small clamping assembly into its various parts. However, you can disassemble it, for example for repair. The following procedure shows you how assemble the small clamping assembly after it has been disassembled.

To assemble the small clamping assembly:

⇨ *See page 6-13, Fig. 6-5*

Start:
- Fixed part of the clamp holder **2** (consisting of two halves) has been removed
- Drive shaft **4**, movable part of clamp holder **3** and displacement sensor core **1** are joined up together

(1) Remove the four Torx screws **6**.

(2) Remove the front half of the clamp holder **2**.

(3) Put the movable part of the small clamping assembly in the half of the clamp holder. Guide the displacement sensor core through the flange **8** of the clamp holder. The middle Torx screw **7** must face outward (so that it is accessible).

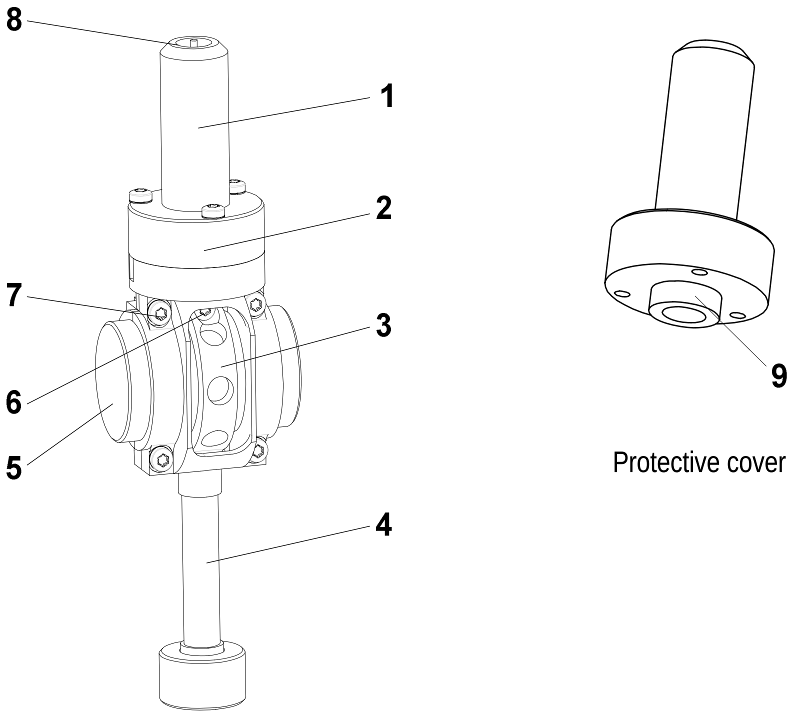
(4) Remount the front half of the clamp holder **2**. Make sure that it is correctly orientated: It is correctly orientated when the middle Torx screw **7** is clearly visible. Fasten the four Torx screws **6** so that the fitting is loosely mounted.

End: Small clamping assembly assembled

Aligning the small clamping assembly within itself

Before you can install the small clamping assembly in the DMA module, you have to align it within itself. This is done with the help of the alignment insert and the protective cover.

Fig. 6-6 shows the small clamping assembly in the assembled state with the alignment inset and the protective cover installed.



- **1** Protective cover
- **2** Clamp holder, fixed part
- **3** Clamp holder, movable part
- **4** Drive shaft
- **5** Alignment insert
- **6** Middle Torx screw
- **7** Torx screws in the fixed part of the clamp holder
- **8** Protective cover
- **9** Rim

Fig. 6-6. Small clamping assembly with alignment insert and protective cover

To install the small clamping assembly in the DMA module, the alignment insert must be inserted. In measurement operation, the alignment insert is replaced with one of the small clamps containing a sample.

To align the small clamping assembly within itself:

➩ *See page 6-15, Fig. 6-6*

Start: Small clamping assembly in assembled state

Always use the Torx screwdriver supplied to tighten and loosen the Torx screws. Using a different tool could damage the Torx screws.

(1) Make sure that all five Torx screws **6**, **7** in the movable and the fixed clamp holders **2**, **3** have been loosened. Loosen Torx screw **6** of the movable part of the clamp holder by about one full turn only.

♣ If you loosen the Torx screw too much, it can hit the fixed part of the clamp holders and jam the clamp holder.

(2) Slide the alignment insert **5** carefully from one side into the clamp holder. If necessary, loosen the Torx screws a bit more.

(3) Position the movable part of the clamp holder **3** symmetrically in the middle of the fixed part of the clamp holder **2** and alignment insert **5** symmetrically to the middle of the clamp holder.

(4) Firmly tighten Torx screw **6** of the movable part of the clamp holder.

(5) Mount the protective cover **1** from above onto the flange of the clamp holder. Guide the displacement sensor core carefully into the bore of the protective cover **8**.

The lower guide rim **8** of the protective cover projects into the gap between the flange of the clamp holder and the displacement sensor core. The displacement sensor core is completely inserted in the bore of the protective cover.

(6) Align the movable parts of the small clamping assembly so that the end of the displacement sensor core is positioned concentrically in the bore of the protective cover **1**.

(7) Tighten the four Torx screws **7** of the fixed clamp holders. Make sure that the movable part of the clamp holder **3** does not shift.

(8) Remove the protective cover **1** by carefully lifting it upward over the displacement sensor core.

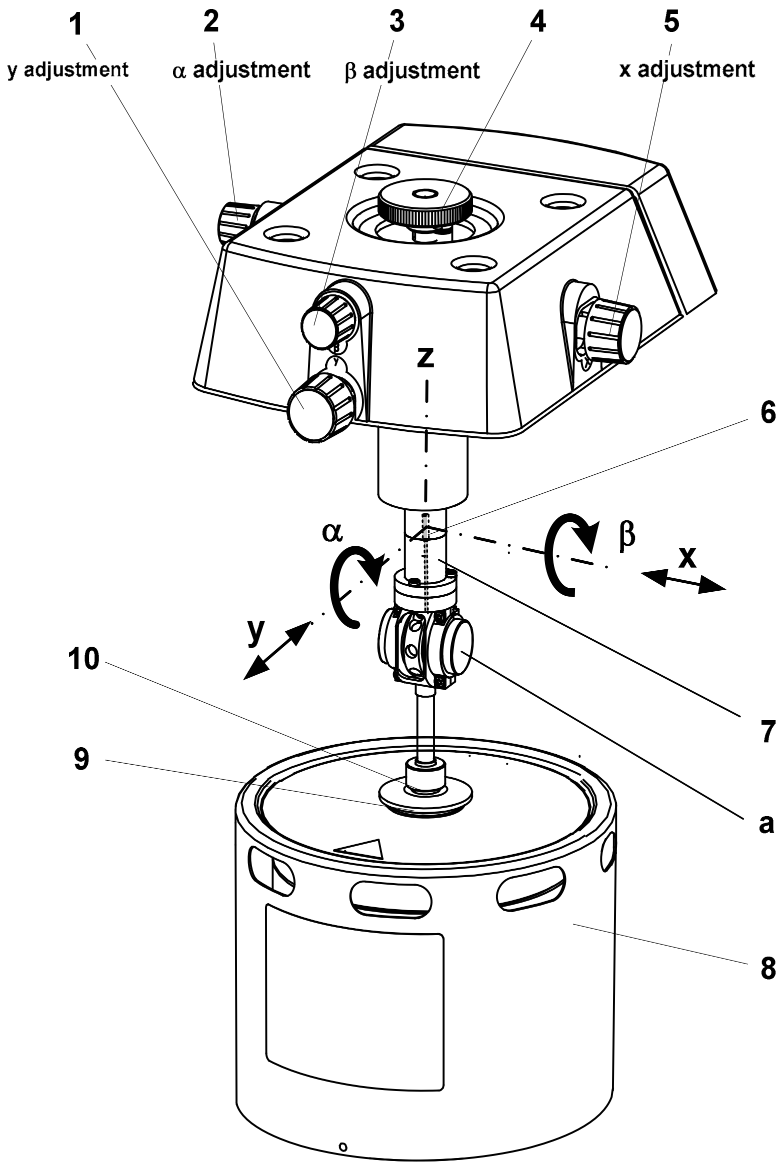
End:
- Small clamping assembly aligned within itself
- Alignment insert is mounted and protective cover removed
- Small clamping assembly ready to install in DMA module

6.2.2 Installing the small clamping assembly

This section shows you how to install the small clamping assembly. Before the clamping assembly can be installed, the measuring sensors must be pre-aligned as described in the section *Pre-aligning the measuring sensors before installing a clamping assembly*.

After this manual alignment has been performed, you may have to readjust the x and y positions slightly while installing the small clamping assembly. The installation procedure of the clamping assembly therefore consists of two steps: the actual installation of the clamping assembly and the alignment of the measuring sensors (four axes alignment).

Fig. 6-7 gives an overview of the adjustment possibilities with the four axes alignment device.



- **1** Knob for adjusting the y -position
- **2** Knob for adjusting the α angle
- **3** Knob for adjusting the β angle
- **4** Locking screw
- **5** Knob for adjusting the x -position
- **6** Displacement sensor core
- **7** Displacement sensor housing
- **8** Drive motor
- **9** Threaded fitting
- **10** Nut
- **a** Alignment insert

Fig. 6-7. Small clamping assembly installed

SCA

To install the small clamping assembly:

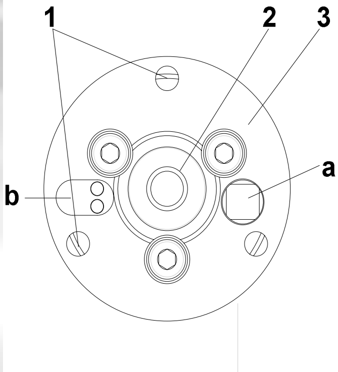
➩ *See page 6-17, Fig. 6-7*

Start:
- Small clamping assembly aligned within itself
- Alignment insert mounted * and protective cover removed

(1) Hold the small clamping assembly up at an angle below the displacement sensor flange with the heads of the Torx screws pointing toward you. Guide the displacement sensor core **6** into the displacement sensor housing **7** by raising the entire adjustment assembly, ❶.

Install small clamping assembly

(2) Fasten the small clamping assembly to the flange of the displacement sensor, ❷ as follows:



1 Torx screws
2 Displacement sensor core
3 Displacement sensor flange
a CA thermocouple
b Hole for SDTA thermocouple

Fig. 6-8. Displacement sensor flange (view from below)

- Hold the clamping assembly under the flange of the displacement sensor. Ensure that the thermocouple projecting from the notch is positioned freely in the notch. Make sure you do not wedge the thermocouple while fastening the adjustment assembly.

♣ The Torx screws must be aligned with the tapped holes in the flange of the clamping assembly.

- Screw in the three Torx screws and tighten them firmly with the exactly the same torque using the Torx Allen wrench supplied.

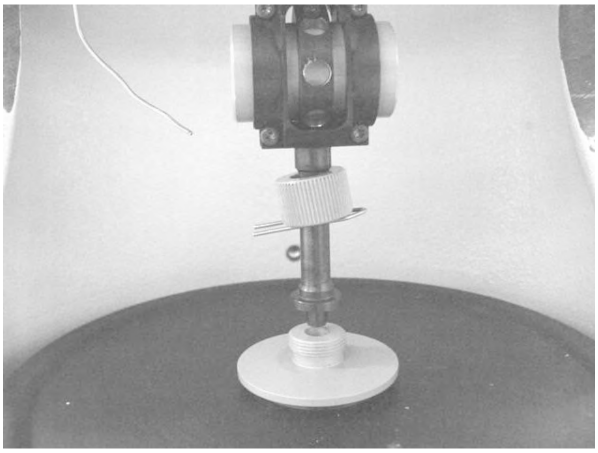
The small clamping assembly is now fixed to the flange of the displacement sensor.

* See the section .*Installing and removing the alignment insert of the small clamping assembly.*

❶

❷

❸



❹

Do not drive the z-axis table too far toward the clamping assembly.
The drive motor could suffer damage.

(3) Drive the z-axis table upward by pressing the MOTOR UP key once. The z-axis table stops automatically in a position in which the conical end of the drive shaft is just over the bore of the threaded fitting **9** of the drive motor, ❸ and ❹.

♣ The travel speed of the z-axis table changes from high to low in this intermediate position.

SCA

(4) If you have previously not done so, loosen the support structure of the measuring sensors as follows:

- Remove the Torx screws (A) with the Torx wrench and remove the front clamp (B). Loosen the Allen screws (C) with a 4-mm Allen wrench, ❺, but do not completely remove them so that the support structure is still attached.
- Fold the support structure back so that it is no longer in contact with length sensor flange, ❻.

(5) Loosen the locking screw of the four axes alignment device, ❼, then slightly tighten it again to the point where you feel slight resistance.

Readjust x and y positions

(6) Readjust the x and y positions as follows:

- Slide the nut **10** upward and fix it with a paper clip, ❹ (previous page).
- If the conical end of the drive shaft is not concentrically above the bore in the threaded fitting **9** of the drive motor: Use knobs **1** and **5**, to adjust the x and y positions until the conical end of the drive shaft is positioned concentrically above the bore, ❽**-a** and ❽**-b**.
- Drive the z-axis table upward carefully by pressing the MOTOR UP key. Continuously check visually that the position of the conical end of the drive shaft in the bore is concentric. If necessary, adjust the x and y positions again.

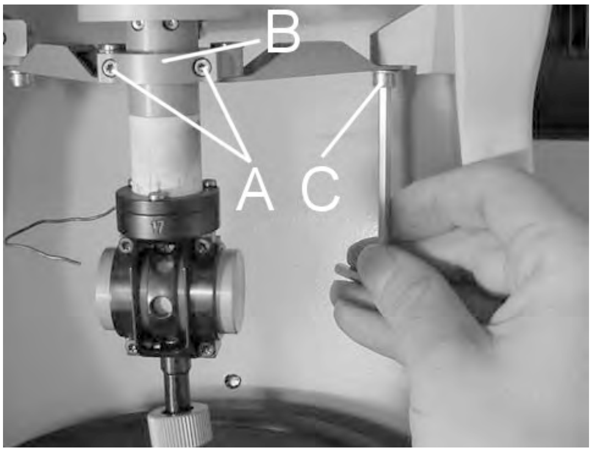
(7) Check the play of the drive motor diaphragm before tightening the nut **10**, by pulling the diaphragm upward with the threaded fitting **9** using your fingers, ❾. Drive the z-axis table upward until the play of the diaphragm is less than 0.5 mm. Do not pull the diaphragm while the z-axis table is moving.

(8) Tighten the nut **10** firmly.

End:
- Small clamping assembly installed
- Support structure loosened

The pre-alignment of the measuring sensors has been completed. You can now begin the fine alignment of the measuring sensors. See the *section Fine alignment of the measuring sensors (four axes alignment) on page 6-102*.

If you have newly installed the small clamping assembly, you must perform a displacement adjustment after the fine alignment. This is explained in *Chapter 8, Calibration and adjustment* in the section *Adjusting the displacement*.



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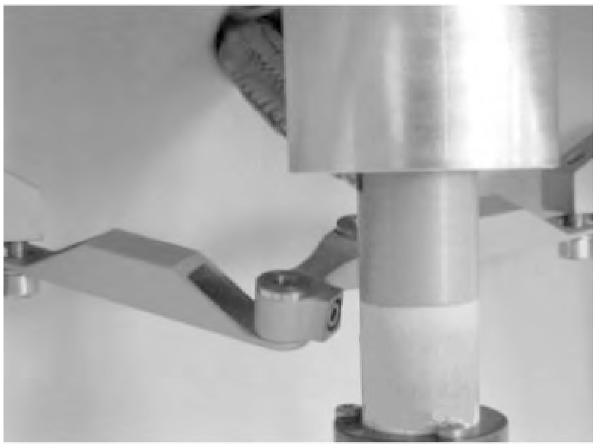

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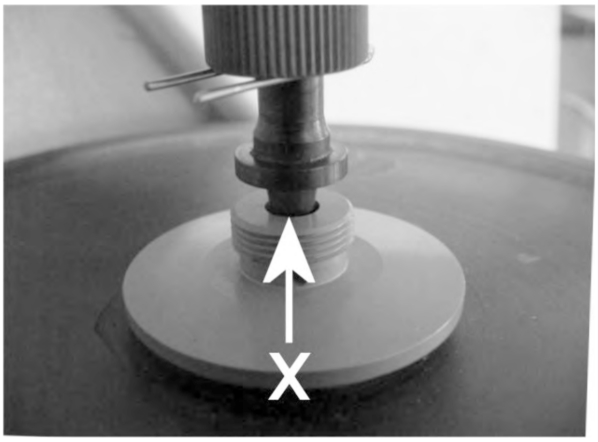

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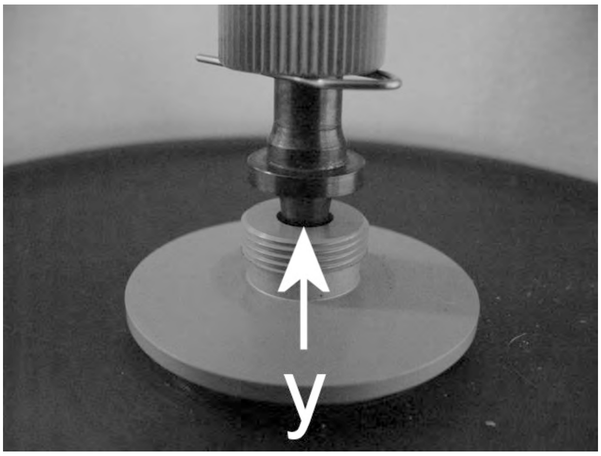

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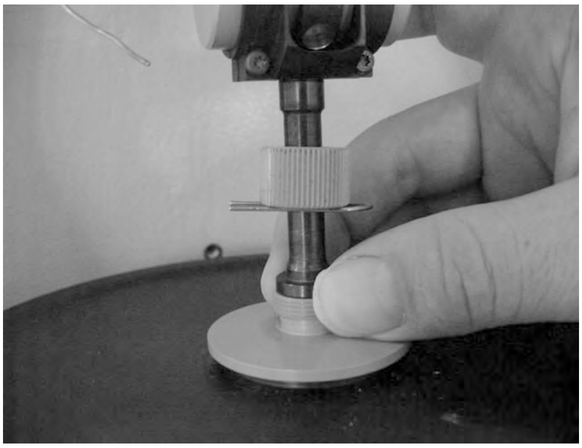

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SCA

6.2.3 Removing the small clamping assembly

To remove the small clamping assembly from your DMA module, the alignment insert must be mounted in the clamping assembly.

To remove the small clamping assembly

➩ *See page 6-17, Fig. 6-7*

Start:
- Alignment insert mounted *
- Two halves of the furnace are fully open and swung to the rear

(1) Fully loosen the three Torx screws on the displacement sensor, ❶. Use the Torx Allen wrench supplied. Make sure that the clamping assembly is completely loose and can move freely downward when you drive the z-axis table downward.

(2) Drive the z-axis table fully downward. Depress the MOTOR DOWN key until the z-axis table stops in the intermediate position. Briefly depress the MOTOR DOWN key once more to move the z-axis table to the lower position. The z-axis table stops automatically at the lower position.

♣ The travel speed of the z-axis table changes from low to high in the intermediate position.

The clamping assembly has been driven right down with the z-axis table. There is now sufficient room above the displacement sensor core to remove the clamping assembly.

(3) Loosen the nut **10** of the drive shaft from the threaded fitting of the drive motor.

(4) Lift the clamping assembly upward out of the drive motor fitting. Remove the clamping assembly carefully in a downward direction. Make sure that you do not damage the displacement sensor coil and the displacement sensor core, ❷**-a** and ❷**-b**.

(5) Screw on the protective cover, ❸.

End: Small clamping assembly removed

* See the section .*Installing and removing the alignment insert of the small clamping assembly.*

❶

❷-a

❷-b

❸

6.2.4 Installing and removing the alignment insert of the small clamping assembly

Before you install or remove the clamping assembly, the alignment insert must always be installed to ensure that the parts of the clamping assembly are firmly fixed together.

Never use excessive force to move the alignment insert. Exerting excessive force on the clamp could damage the force sensor, the displacement sensor or the drive motor.

To install the alignment insert:

➪ *See page 6-15, Fig. 6-6*

Start:
- Small clamping assembly installed, clamp removed
- Two halves of the furnace are fully open and swung to the rear

(1) Make sure that all five Torx screws **6**, **7** in the movable and the fixed clamp holder **2**, **3** have been loosened.

(2) Slide the alignment insert, ❶, carefully from one side into the clamp holder, ❷. If necessary, loosen the Torx screws even more. Make sure that the alignment insert is positioned symmetrically in the clamp holder, ❸.

(3) Tighten the Torx screws **6**, **7** of the clamp holder.

End: Alignment insert installed in small clamping assembly

To remove the alignment insert:

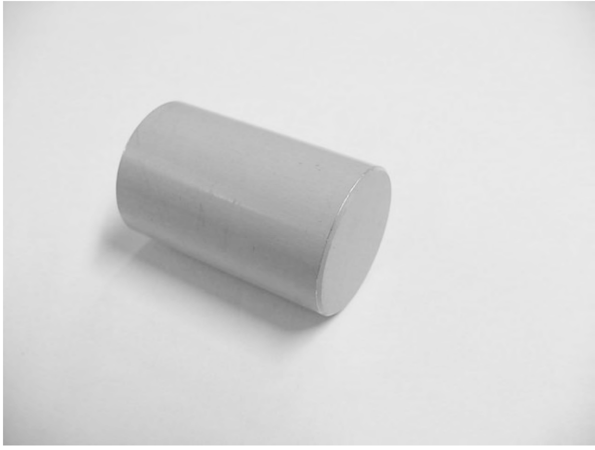
➪ *See page 6-15, Fig. 6-6*

Start:
- Alignment insert installed in small clamping assembly
- Two halves of the furnace are fully open and swung to the rear

(1) Loosen the Torx screws **6**, **7** of the clamp holder.

(2) Pull the alignment insert carefully toward the right side out of the clamp holder, ❸.

End: Alignment insert removed from small clamping assembly.



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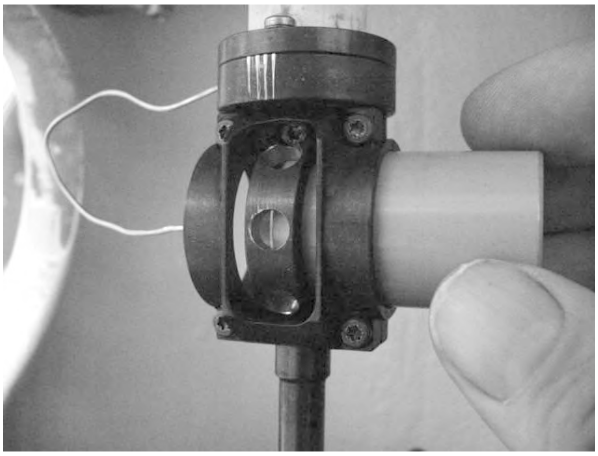

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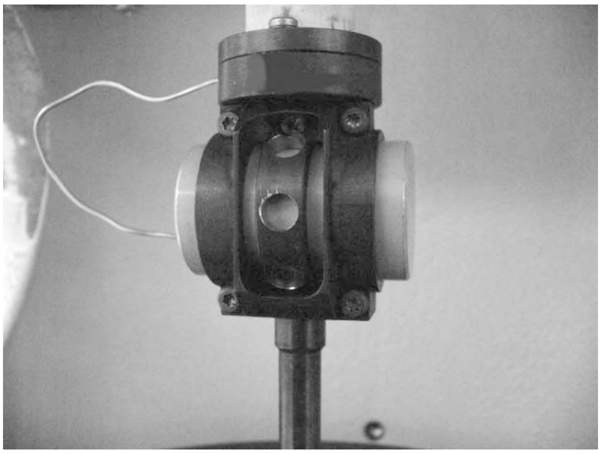

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6.3 Mounting the sample in the small clamping assembly

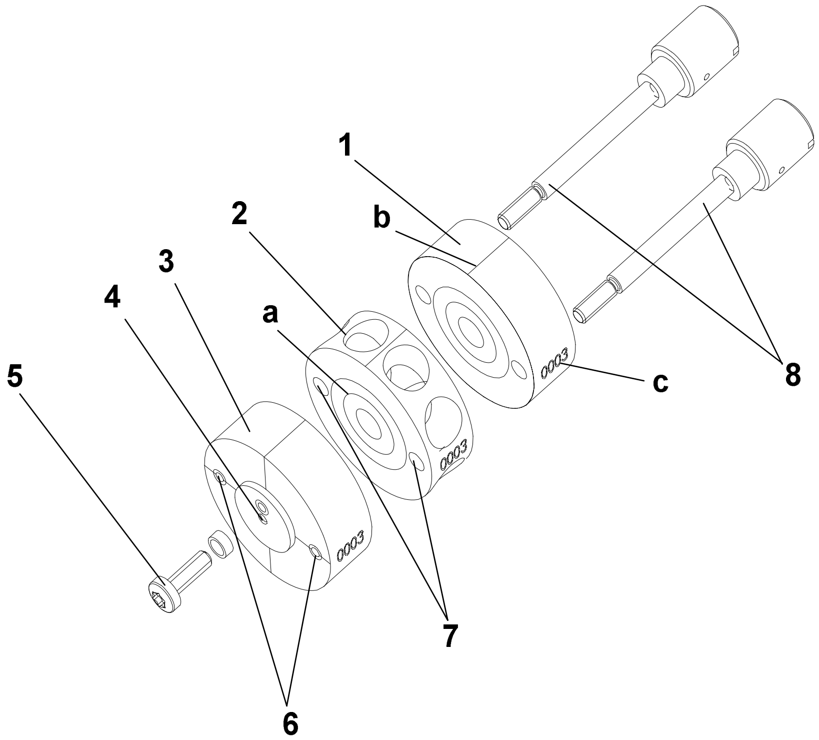
Mounting the sample in the small clamping assembly consists of two steps: First of all you have to mount the sample in the clamp before you then install the clamp in the clamp holder. The following sections describe this two-step procedure.

Several different clamps are available for the small clamping assembly. The clamp you use depends on the measuring mode and the type of sample.

6.3.1 Disassembling the standard shear clamp and mounting the sample

The instructions in this section refer to the standard shear clamp for highly viscous materials.

Before you can mount the sample in the standard shear clamp, you must first disassemble the shear clamp. Fig. 6-9 shows the design of the standard shear clamp.



- **1,2** Sample supports
- **3** End disk
- **4** Hole for SDTA thermocouple
- **5** Torx screw for SDTA thermocouple
- **6** Tapped holes
- **7** Guide holes
- **8** Guide pins
- **a** Laser markings
- **b** Line marks
- **c** Clamp identification number

Fig. 6-9. Design of the standard shear clamp

Two pairs of guide pins **8** of different length are supplied with the standard shear clamp. Either pair of the guide pins can be used, depending on the thickness of the sample.

SCA

To disassemble the standard shear clamp:

➩ *See page 6-27, Fig. 6-9*

Start: Shear clamp removed from small clamping assembly

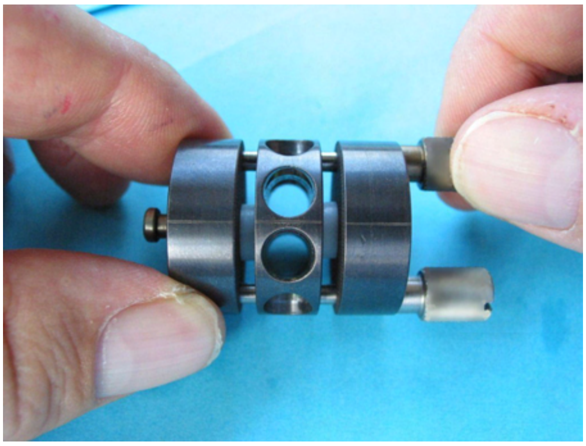
(1) Carefully unscrew and remove the two guide pins **6**, ❶. Hold the sample support disks **1**, **2** and the end disk **3** with one hand so that they do not fall apart.

(2) Now take apart the two sample support disks **1**, **2** and the end disk **3** and remove the previous sample (if present), ❷, ❸ and ❹.

(3) Clean the surfaces of the sample support disks **1**, **2** and the end disk **3** from any residues of the previous sample. Use suitable cleaning agents or an ultrasonic bath. The surfaces are very precisely machined. Make sure you do not damage them.

♣ To remove stubborn residues, we recommend that you bake out the sample supports and the end disk in a muffle furnace at 400 °C for half an hour.

End:
- Shear clamp is in parts
- Sample has been removed
- Surfaces of sample supports and end disk are clean



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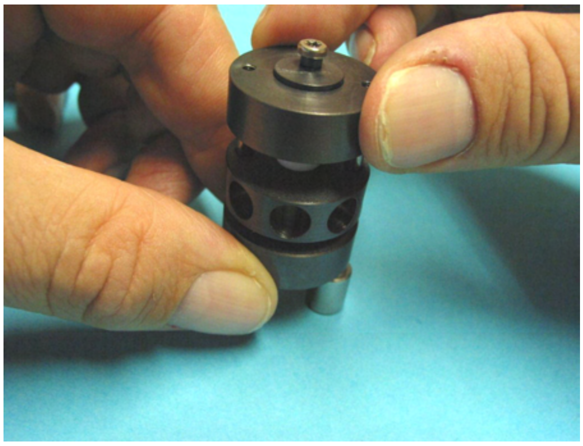

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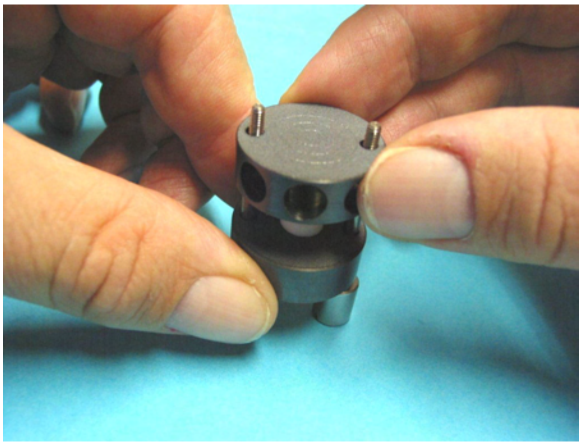

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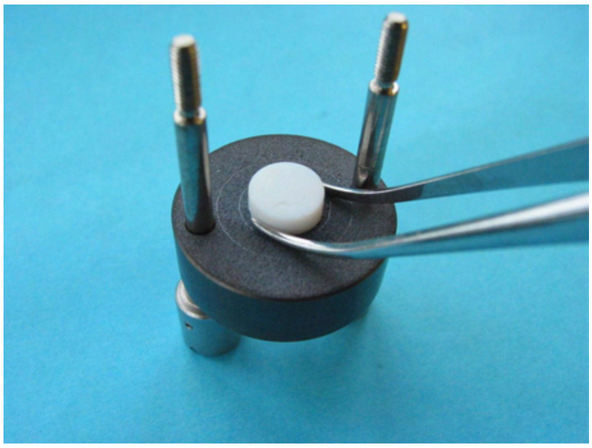

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SCA

To mount the sample in the shear clamp:

⇨*See page 6-27, Fig. 6-9*

Start:
- Shear clamp is in parts
- Two samples are prepared for measurement

(1) Punch out two, as far as possible, identical samples: Measure the thickness of each sample with a micrometer and use the average value as reference value (e.g. the value you enter in the S**TA**R^e software). Additionally, measure the length of the empty shear clamp by placing the three disks on top of each other. Note this value. You will need it later on to calculate the length of the shear clamp when the sample is compressed in step (8).

♣ The best way to accurately punch out the samples is to use a boring machine with a punch tool.

♣ The dimensions of the sample depend very much on the type of material used and there is no general rule as to what the shape and size of the samples should be. As a rough guideline, we recommend a geometry factor of 30 to 50.

(2) Select the guide pins **8** that are suitable for the thickness of your sample and push the two pins **8** through the guide holes **7** of one of the sample supports **1**.

(3) Then place the sample support on a horizontal surface so that it rests on the ends of the guide pins **8** and the guide pins project upward, ❶.

(4) Place a sample on the load-bearing surface of the sample support in the center of the surface, ❷. Use the markings on the surface to help position the sample.

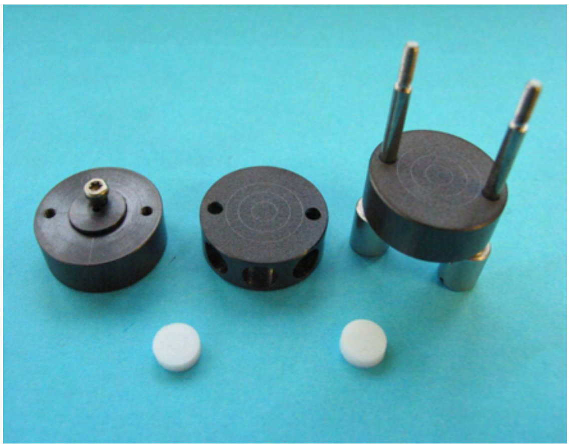
(5) Slip the second sample support **2** over the guide pins **8** until it rests on the first sample. There are marks and numbers on the sides of both sample support disks for orientation which should coincide when the disks in position, ❸.

♣ If your sample has an internal structure, you can use these markings to take into account the orientation of the structure when installing the clamp.

(6) Mount the second sample in the center of the surface, ❹. Use the markings on the surface to position the sample.

(7) Place the end disk **3** over the guide pins **8** so that the ends of the threads are positioned in contact with the tapped holes **6** of the end disk **3**, ❺. There are marks and numbers on the sides of both sample support disks and the end disk for orientation that should coincide when the disks are in position.

(8) Screw in the two guide pins **8** by hand so that the two samples are fixed in the spaces of the clamp and no gap remains, ❻.



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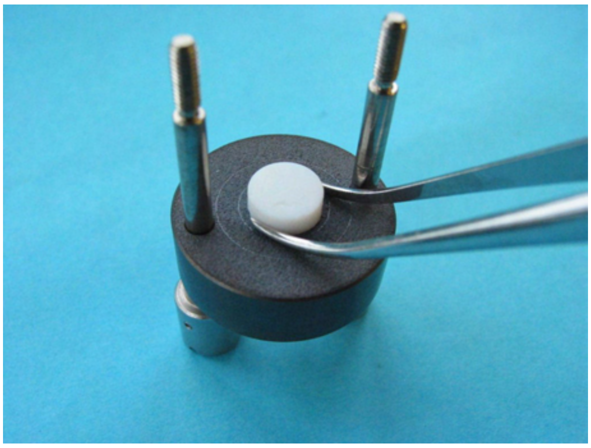

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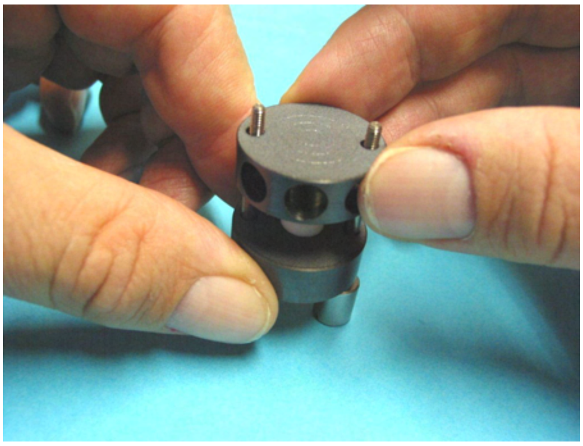

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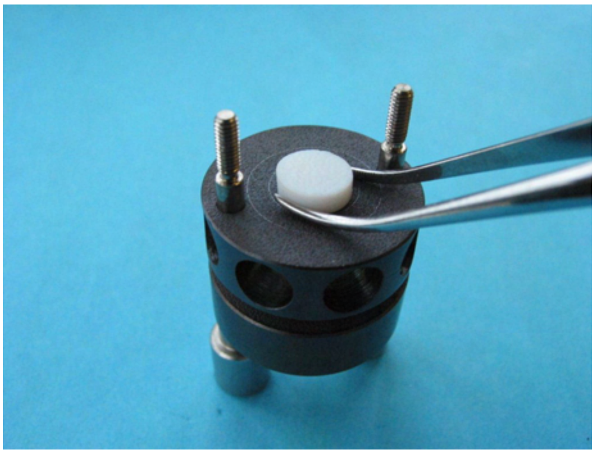

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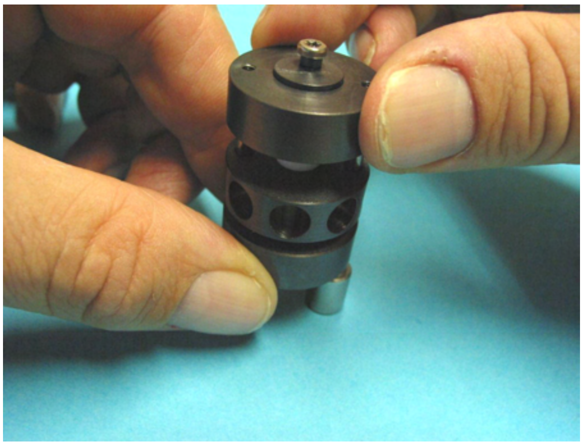

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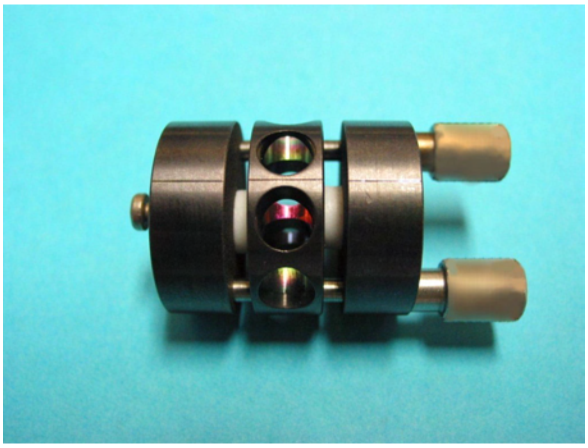

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SCA

(9) For elastomer samples: Using a micrometer, measure the length of the shear clamp containing the sample and subtract the length of the empty clamp to calculate values for the uncompressed sample thickness. Then tighten the guide pins **8** so that each of the two samples is compressed by about 10%.

or

For stiff samples: Tighten the guide pins with the torque wrench supplied using its slot screw blade, but do not set a torque of more than 0.4 Nm.

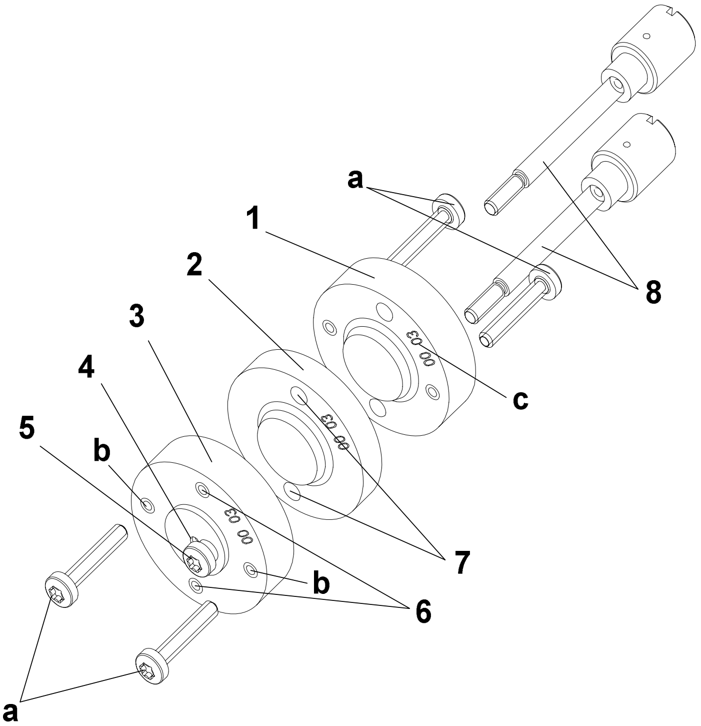
♣ The amount of torque set to fasten the guide pins depends very much on the type of sample you are measuring. The sample must be fixed in place to prevent it from slipping during the measurement. But at the same time it must be loose enough to avoid excessive clamping pressure that would affect the measurement or even damage the sample.

End:
- Both samples are mounted in the shear clamp
- Shear clamp is ready for installing in the clamping assembly

6.3.2 Disassembling the special shear clamp and mounting the sample

The instructions in this section refer to the special shear clamp for low viscosity liquids.

Before you can mount the sample in the special shear clamp, you must first disassemble the shear clamp. Fig. 6-10 shows the design of the special shear clamp.



- **1, 2** Sample supports
- **3** End disk
- **4** Hole for SDTA thermocouple
- **5** Torx screw for SDTA thermocouple
- **6** Tapped holes
- **7** Guide holes
- **8** Guide pins
- **a** Distance screws
- **b** Tapped holes for distance screws
- **c** Clamp identification number

Fig. 6-10. Special shear clamp for low viscosity liquids

A thickness gage is needed to mount the samples with the correct distance between the two sample supports, and to adjust the middle sample support and the end disk:

Thickness gage

SCA

To disassemble the special shear clamp:

➩ *See page 6-33, Fig. 6-10*

Start: Shear clamp removed from small clamping assembly

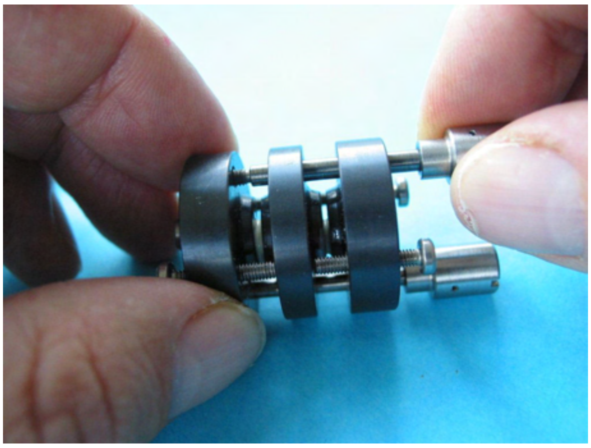
(1) Carefully unscrew and remove the two guide pins **8**, ❶. Hold the sample support disks **1**, **2** and the end disk **3** with one hand so that they do not fall apart.

(2) Now take apart the two sample support disks **1**, **2** and the end disk **3** and remove the previous samples (if present) as far as possible, ❷, ❸ and ❹.

(3) Clean the surfaces of the sample support disks **1**, **2** and the end disk **3** from residues of the previous sample. Use suitable cleaning agents or an ultrasonic bath. The surfaces are very precisely machined. Make sure you do not damage them.

♣ To remove stubborn residues, we recommend that you bake out the sample supports and the end disk in a muffle furnace at 400 °C for half an hour.

End:
- Shear clamp is in parts
- Samples are removed
- Surfaces of sample supports and end disk are clean



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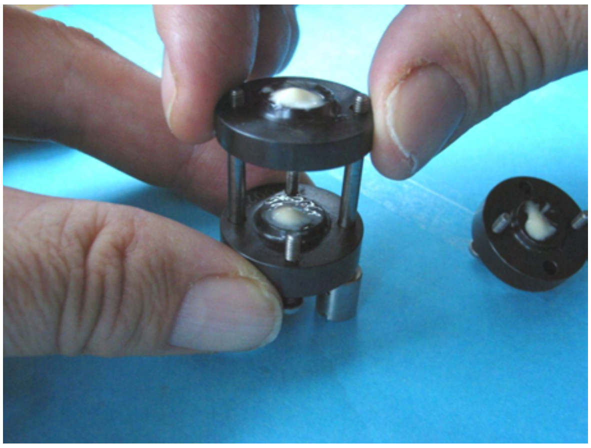

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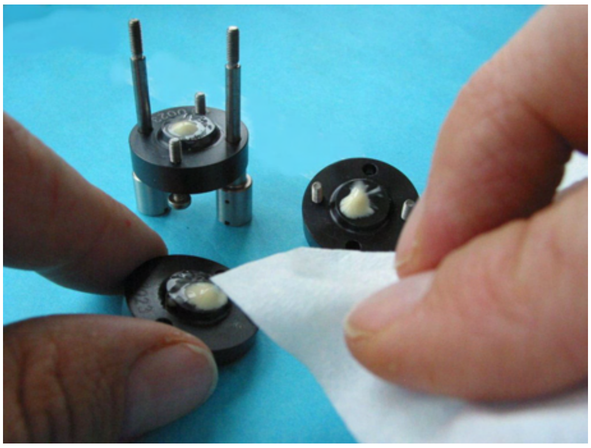


❹

SCA

To mount the sample in the special shear clamp for low viscosity liquids:

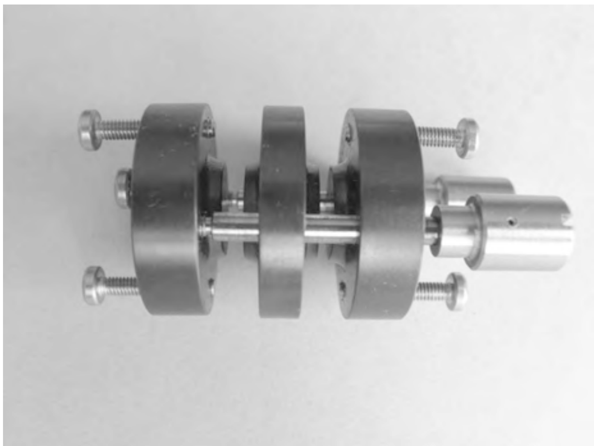
➪ *See page 6-33, Fig. 6-10*

Start:
- Shear clamp assembled
- Thickness gage is ready
- Sample is prepared for the measurement

(1) Loosen the four distance screws **a** and the guide pins **8** as shown in ❶, but without removing them completely.

(2) Adjust the two spaces for the sample between the disks using the thickness gage:

- First adjust the sample space that is nearer the head of the guide pins. Insert an appropriate gage corresponding to the sample thickness into the space. Manually tighten the guide pins to the same extent, ❷. Make sure that the distance screws **a** are sufficiently loose.
- Screw in the two distance screws of the sample support disk using the Torx screwdriver supplied, ❸. Make sure the gage does not interfere with the distance screws. Loosen the guide pins and pull out the gage.
  The thickness of the first sample space is now set.
- Repeat the above two steps in the same sense for the other sample space, ❺ and ❻. If necessary, you will have to loosen the guide pins more in order to insert the gage, ❹. Make sure the distance screws are sufficiently loose. Finally leave the guide pins tightened.
  The thickness of the second sample space is now set.



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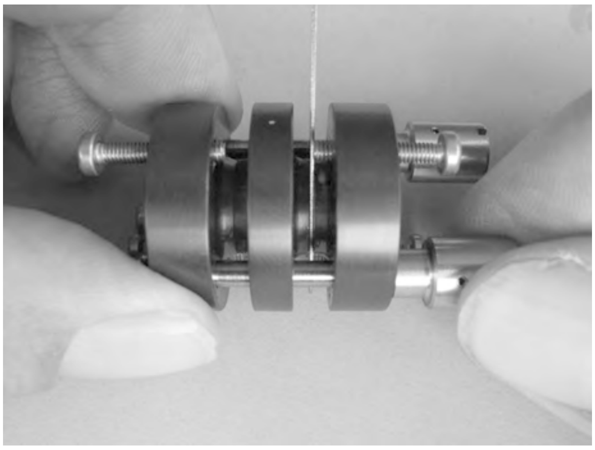

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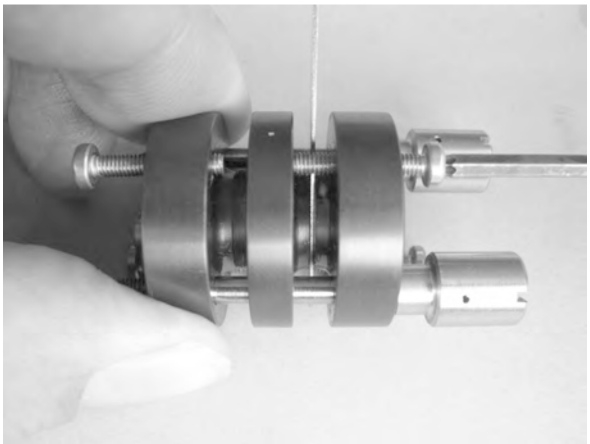

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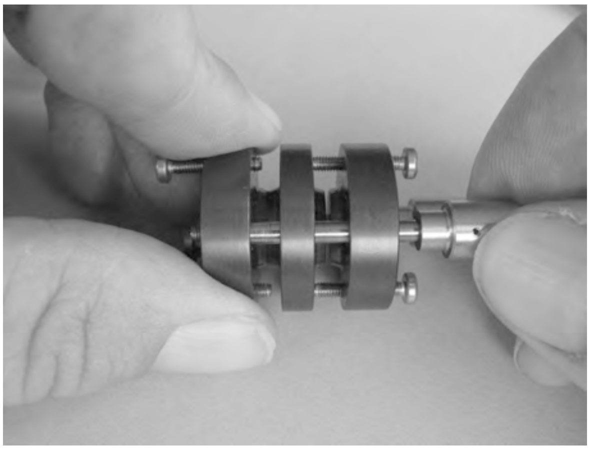

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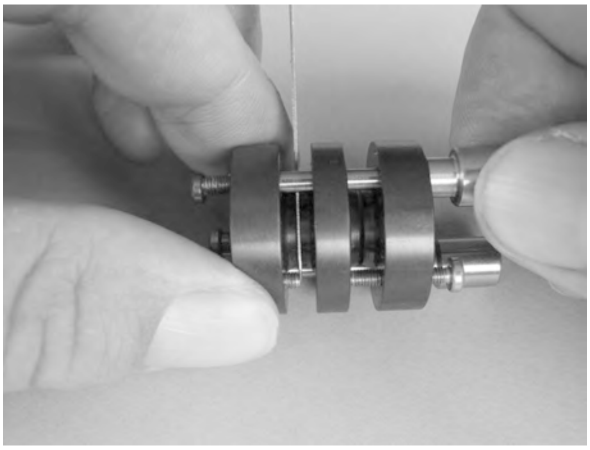

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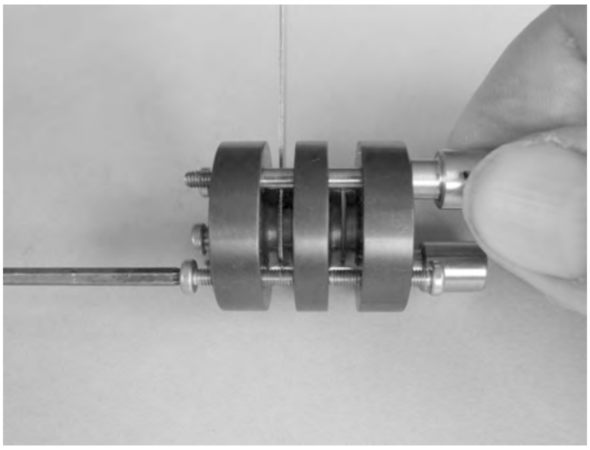


❻

(3) Check the thickness of the two spaces using the thickness gage. If necessary, correct the thickness of the spaces by repeating step (2).

(4) Loosen the guide pins **8** and remove the end disk **3** and the middle sample support disk **2**.

Make sure that you do no alter the distance screws and thereby change the thickness of the space.

A change in the thickness of the space leads to a change in sample thickness and results in measurement errors.

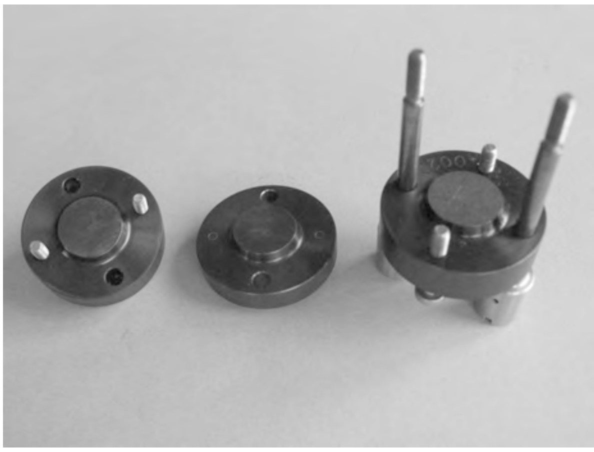
---

(5) Place the sample support disk with the guide pins on the heads of the guide pins so that the pins point upward, ❼.

(6) Apply the sample and mount the sample support and end disk as follows:

- Make sure with the second sample support and with the end disk that the numbers on the surface point upward and coincide.
- Make sure you do not apply too much sample material and that it does not run over.
- Apply the sample first to the surface of the first sample support disk and to the corresponding surface of the middle sample support disk. You can use a spatula or a pipette, ❽, for this, depending on the viscosity of the liquid.
- Mount the middle sample support disk (guide the guide pins through the guide holes).
- In the same way as described above, apply the sample material to the surface of the end disk and to the corresponding surface of the middle sample support disk, ❾.
- Mount the end disk and manually tighten the guide pins, ❿.
- Remove excess sample material with a spatula or a suitable means.

End: - Sample is mounted in the shear clamp
- Shear clamp is ready for installing in the clamping assembly



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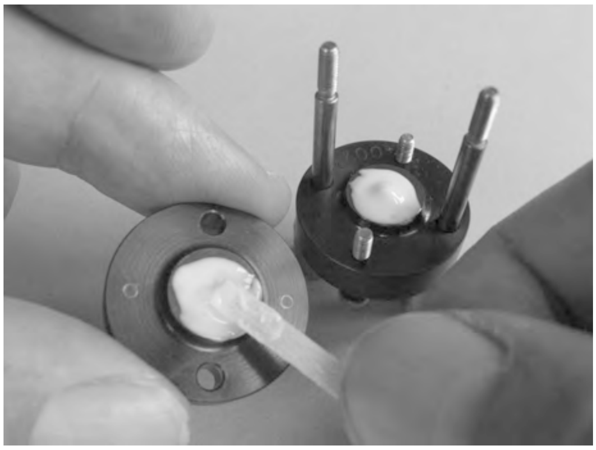

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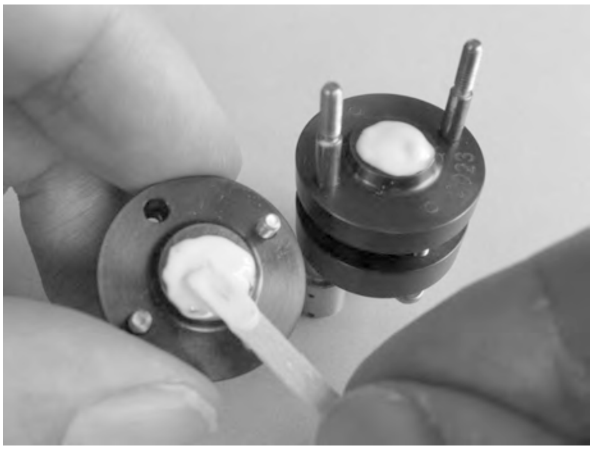

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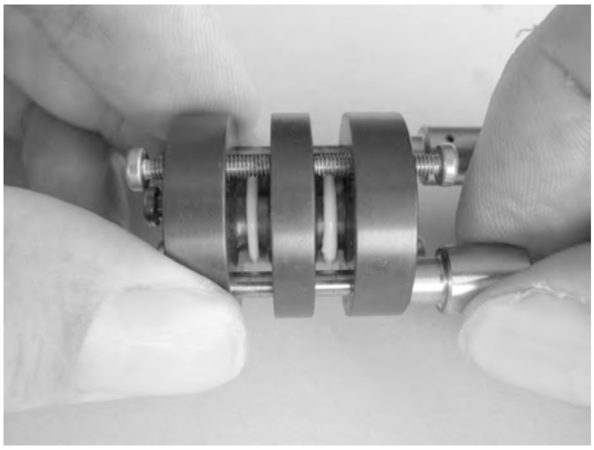


❿

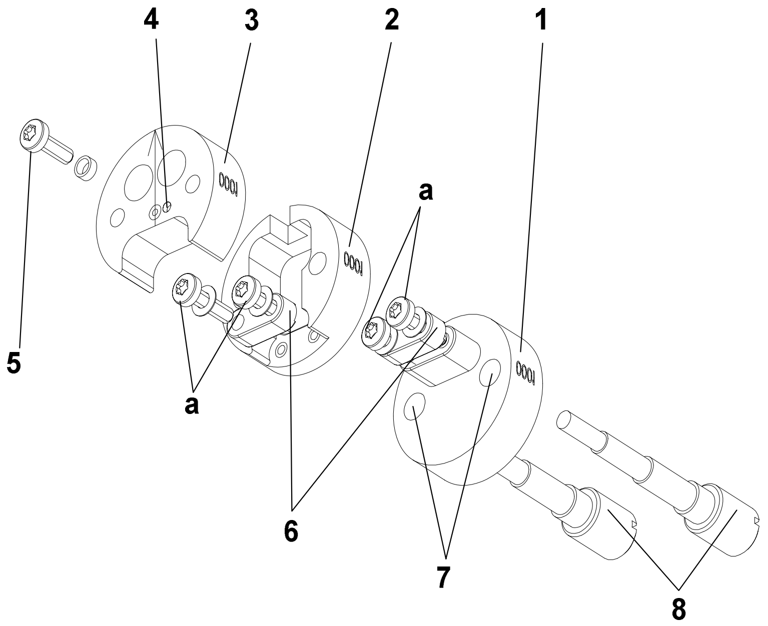
Note on installing the special shear clamp for low viscosity liquids in the small clamping assembly:

Before the measurement, in addition to removing the guide pins, you must also remove the four distance screws. The guide pins should be removed before the distance screws.

If the samples are cooled, they can solidify and shrink through crystallization or vitrification. In this case, to ensure that the samples are properly mounted, the guide pins can be tightened again shortly before the measurement. This is done in the so-called reclamping stage at the start temperature of the experiments.

6.3.3 Disassembling the tension clamp and mounting the sample

Before you can mount the sample in the tension clamp, you must first disassemble the tension clamp. Fig. 6-11 shows the design of the tension clamp.



Key

1, 2	Sample supports
3	End disk
4	Hole for the SDTA thermocouple
5	Torx screw for the SDTA thermocouple
6	Clamping plates
7	Guide holes
8	Guide pins
a	Torx screws for the clamping plates

Fig. 6-11. Design of the tension clamp

To disassemble the tension clamp:

➩ See page 6-40, Fig. 6-11

Start: Tension clamp removed from small clamping assembly

(1) Place the tension clamp on a horizontal surface so that it stands on the guide pins **8**, which point upward, ❶.

(2) Remove the end disk upward over the guide pins.

(3) Loosen the Torx screws on the clamping plates and remove the sample with tweezers, ❷, ❸. If necessary, completely loosen the screws and remove the clamping plates.

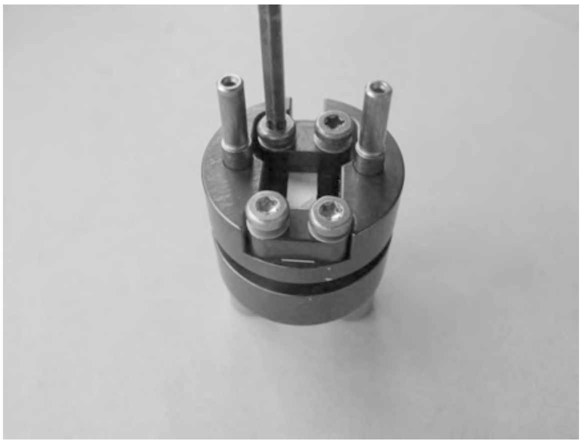
(4) Remove the second sample support **2** and the end disk **3**, ❹.

(5) Clean the surfaces of the sample support disks **1**, **2** and the end disk **3** from any residues of samples. Use a suitable cleaning agent or an ultrasonic bath. The surfaces are very precisely machined. Make sure you do not damage them.

♣ To remove stubborn residues, we recommend that you bake out the sample supports and the end disk in a muffle furnace at 400 °C for half an hour.

End:
- Tension clamp is in parts
- Sample removed
- Surfaces of the sample support and the end disk cleaned

❶



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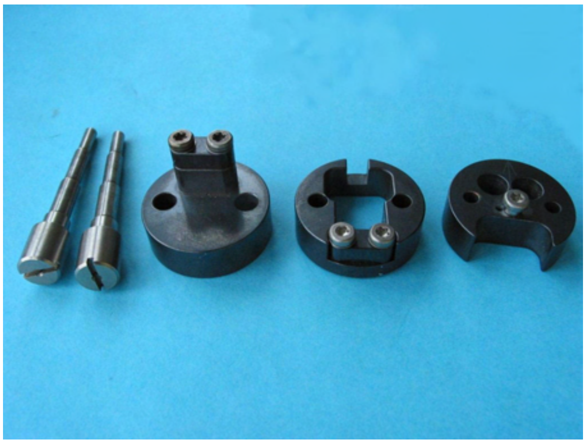


❹

SCA

To mount the sample in the tension clamp:

➩ *See page 6-40, Fig. 6-11*

Start: - Tension clamp is in parts
- Sample material prepared for measurement

(1) Cut a piece of sample material about 5 cm long. The sample should not be more than 4 mm wide and 2 mm thick, ❶.

♣ The dimensions of the sample depend very much on the type of material. There are no general rules for the shape and size of the sample. The sample must not be more than 4 mm and 2 mm thick.

(2) Slide the two sample supports over the guide pins and place the tension clamp without the end disk on a horizontal surface so that it stands on the guide pins **8** and these point upward, ❶.

(3) Loosen the Torx screws on both clamping plates **6**, ❷. Slide the sample from one side under the clamping plates through to the other side, ❸. If necessary, remove the clamping plate completely in order to insert the sample. Make sure that the spring washers of the Torx screws do not fall out.

(4) Position the sample parallel to the symmetry axis of the clamping plate, ❹. Hold the sample with one hand in this position so that it is under slight tension.

(5) If you have removed the clamping plates, first remount the clamping plates and the Torx screws.

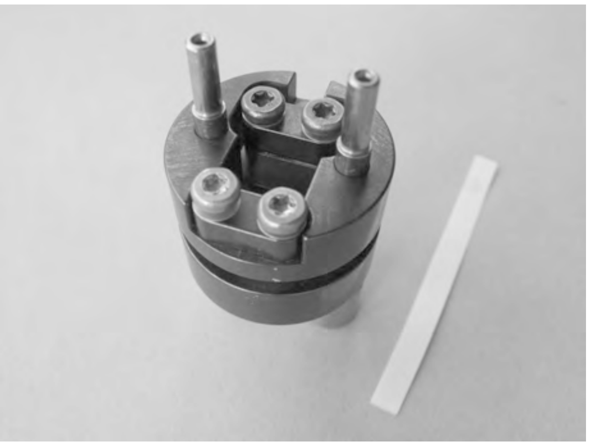
(6) Slightly tighten the four Torx screws. Make sure that you do not move the sample, ❺.

♣ The torque set to tighten the clamping plates depends very much on the type of sample you are measuring. The clamping plates must be fixed to prevent the sample from slipping during the measurement. But at the same time it must be loose enough and must not squeeze the sample together, which would falsify the measurement or even damage the sample.

(7) Cut off the excess ends of the sample, ❻.

(8) Slide the end disk **3** over the guide pins.

End: - Sample is mounted in the clamp
- Tension clamp is ready to install in the clamping assembly



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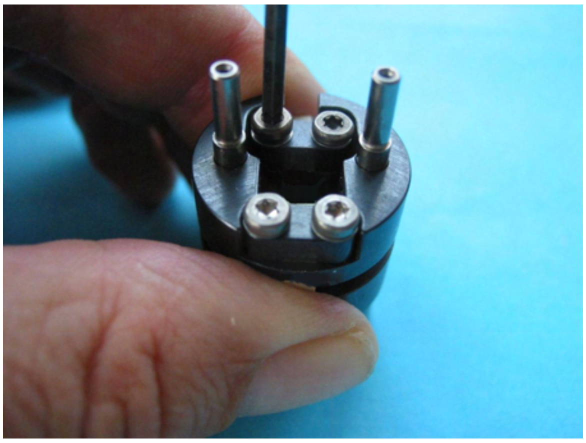

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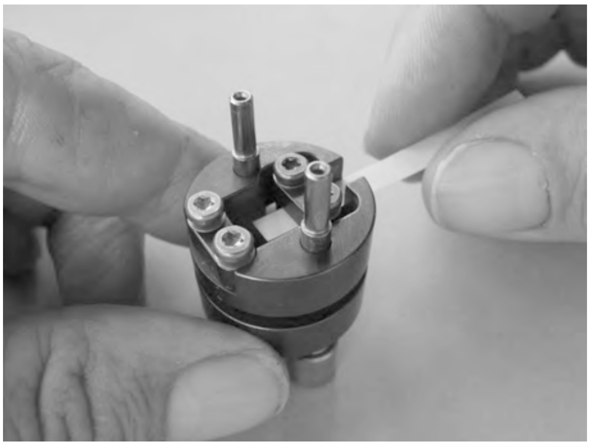

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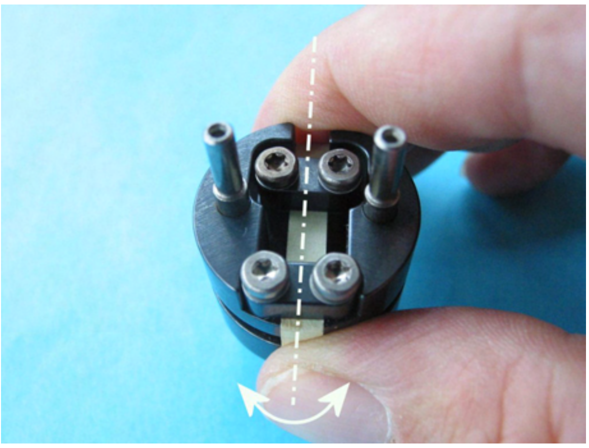

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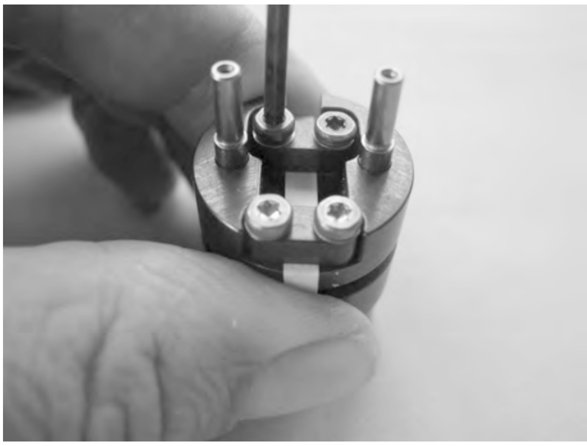

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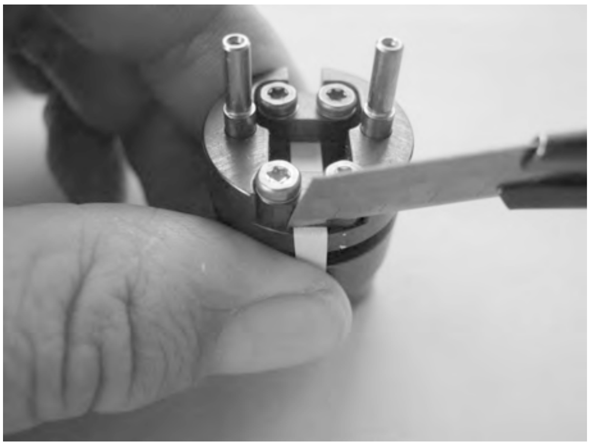


❻

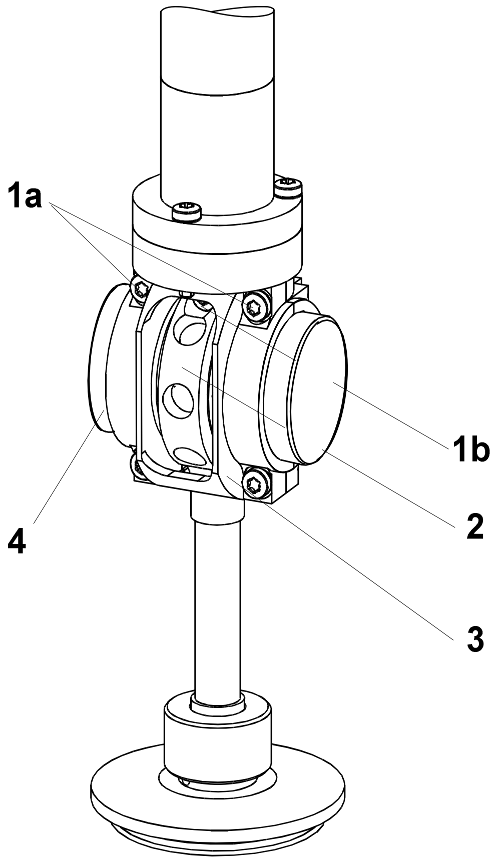
6.3.4 Installing the small clamps

Various types of clamps can be installed in the small clamping assembly, depending on the particular type of measurement and sample. The different clamps are interchangeable in the clamping assembly and it is not necessary to remove and reinstall the entire clamping assembly to run a measurement with a different sample. The sample can be prepared and mounted in the clamp separately outside the DMA module, for example while a measurement of a different sample that is mounted in another clamp is being measured by the instrument.

When you have mounted the sample in the clamp, the clamp can be quickly installed and the next experiment started.

This section explains how install the shear clamps and the tension clamp. The section *Removing the small clamps* explains how to remove these clamps.

Fig. 6-12 shows the shear clamp installed in the clamp holder **2** and **3** of the small clamping assembly.



1a Torx screws in the fixed part
1b Torx screw in the movable part
2 Clamp holder, movable part
3 Clamp holder, fixed part
4 Clamp

Fig. 6-12. Small clamping assembly

In order to mount or remove the sample from the small clamping assembly, the clamp is removed in a separate step.

With the small clamping assembly, the procedure for installing or removing the various clamps is the same and is described in the following section.

Please pay attention to the following note concerning magnitude of the displacement amplitude when using the small clamping assembly:

When using the clamps of the small clamping assembly, the value for the displacement amplitude must not be greater than 1200 µm.

In experiments using the clamps of the small clamping assembly, the displacement amplitude must not be greater than 1200 µm. If the displacement amplitude is greater than 1200 µm, the screw **1b** hits the clamp holder. The measurement results are then wrong. These considerations must be taken into account when defining the maximum displacement amplitude in the Method Window of the STARe Software.

SCA

To install a shear clamp:

⇨ *See page 6-45, Fig. 6-12*

Start:
- Small clamping assembly installed
- Fine alignment and displacement adjustment performed
- No clamp or alignment insert in the clamping assembly
- Furnace arms fully open and swung to the rear

(1) Make sure that all five Torx screws **1a**, **1b** in the movable and the fixed clamp holder **2**, **3** have been loosened, ❶.

Never use excessive force to move the clamp. Exerting excessive force on the clamp could damage the force sensor, the displacement sensor or the drive motor.

---

(2) Slide the clamp carefully from one side into the clamp holder, ❷. If necessary, loosen the Torx screws even more.

♣ If your sample has an internal structure, you can use the line markings on the side of the clamp to take into account the orientation of the structure when installing the clamp.

(3) Slide the middle disk of the clamp completely into the movable part of the clamp holder **2**. Make sure that the clamp is positioned symmetrically in the clamp holder, ❸.

The middle part of the clamp must not touch the fixed part of the clamp holder.

Otherwise there is a fixed connection between the drive motor and the measuring sensors and measurement of the sample becomes impossible.

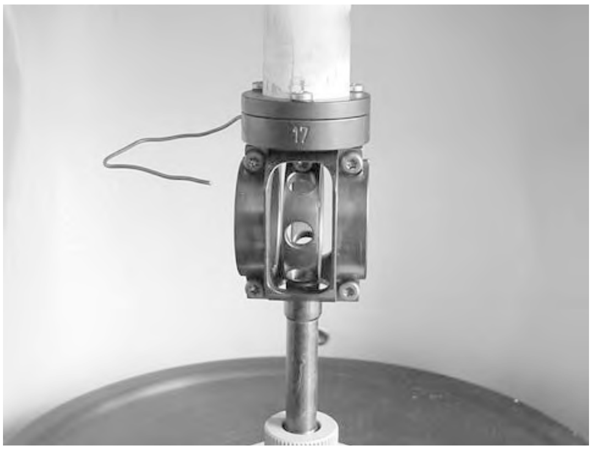
---

(4) At first, tighten the inner Torx screw **1b** gently, ❹, to make sure the clamp does not slip while tightening the screws.

(5) If no sample shrinkage is expected at start temperatures:

- Tighten each of the outer Torx screws **1a** crosswise with a torque of 0.8 Nm, ❺. Finally tighten the inner Torx screws with a torque of 0.8 Nm, ❹.
- With the special shear clamp for low viscosity liquids: Remove the all distance screws before the guide pins.
- Screw the guide pins out of the tapped holes and then pull them out of the guide holes, ❻.

♣ Step (8) shows you how you can reclamp a sample that shrinks with the guide pins.



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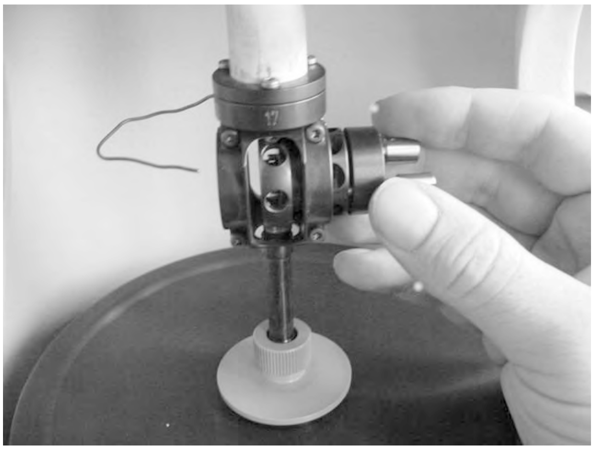

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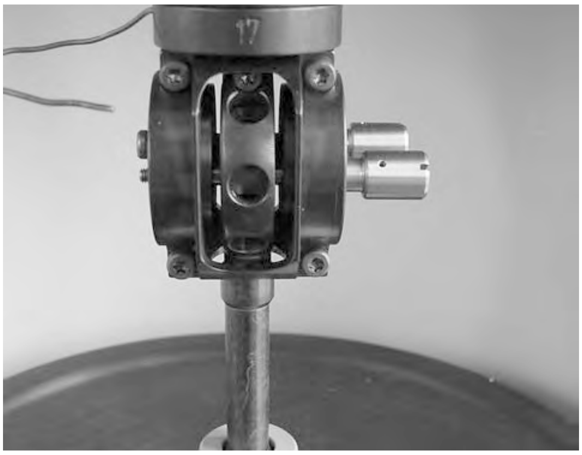

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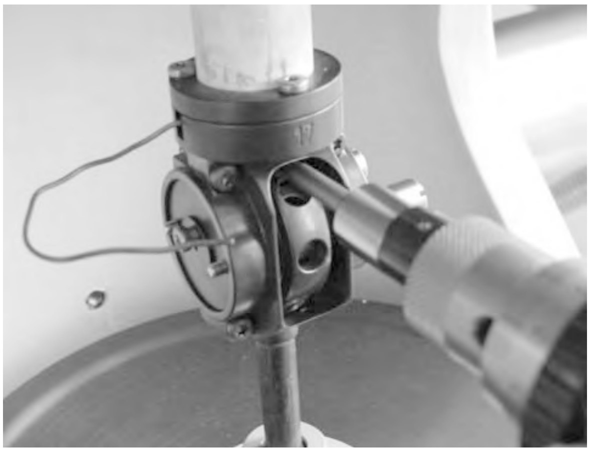
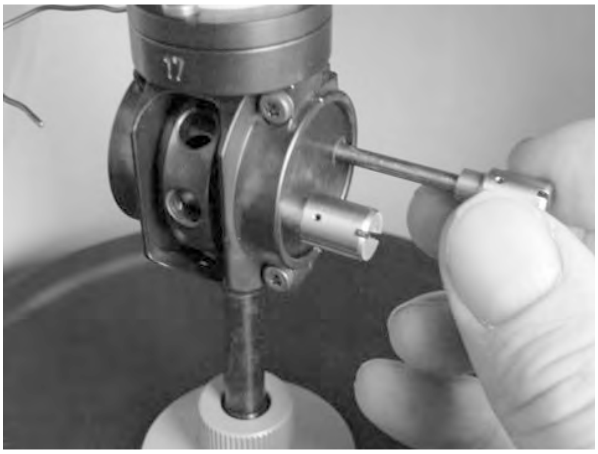


❹

❺



❻

Never touch the furnace halves or the clamping assembly during or immediately after a measurement. Always wear protective gloves or let the furnace and the clamping assembly cool down to ambient temperature before you open the furnace halves. Parts of the furnace and the clamping assembly can reach temperatures down to –150 °C or up to +500 °C. There is a risk of burns.

♣ The amount of torque set to fasten the guide pins depends very much on the type of sample you are measuring. The sample must be fixed in place to prevent it from slipping during the measurement. But at the same time it must be loose enough to avoid excessive clamping pressure that would affect the measurement or even damage the sample. As a rough guideline, we recommend a torque value of about 0.4 Nm.

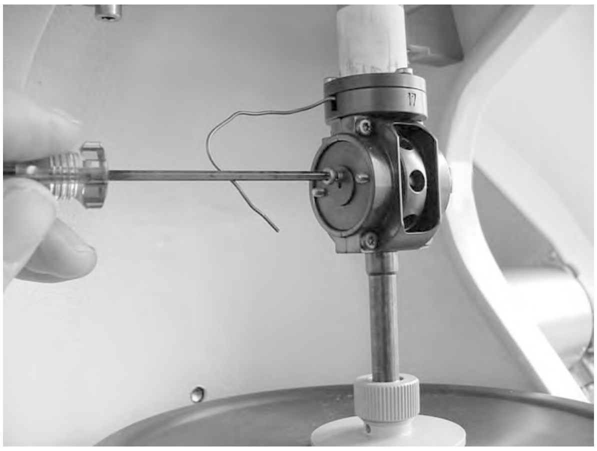
(6) Loosen the clamping screw for the SDTA thermocouple in the surface of the end disk, ❼.

(7) Insert the end of the SDTA thermocouple into the hole in the surface of the end disk, ❽, and make sure you push it through to the end of the bore so that it is as close as possible to the sample. Tighten the clamping screw to fix the thermocouple in its position.

(8) If sample shrinkage is expected at start temperature:

- For low temperature measurements: Leave the guide pins inserted and retighten them setting a torque of 0.4 Nm with the torque wrench supplied during the reclamping phase at start temperature of the experiment, ❾. The message **WAITING RECLAMP STARTTEMP** appears on the LCD during this phase.
- Then tighten the outer Torx screws **1a** firmly crosswise setting a torque of 0.8 Nm with the torque wrench on each screw, **❿-a**. Finally, retighten the inner Torx screw with a torque of 0.8 Nm, **❿-b**.
- First unscrew the guide pins from the tapped holes then pull them out of the guide holes. Be sure to wear protective gloves for this. With the special shear clamp for low viscosity liquids: Remove the all distance screws before the guide pins.

End: - Clamp installed in clamping assembly
- SDTA thermocouple installed



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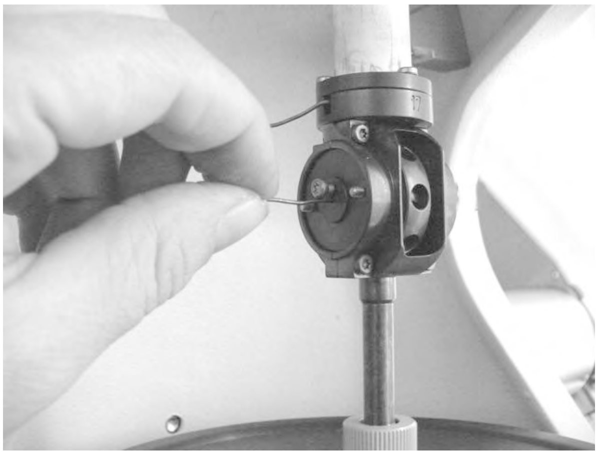

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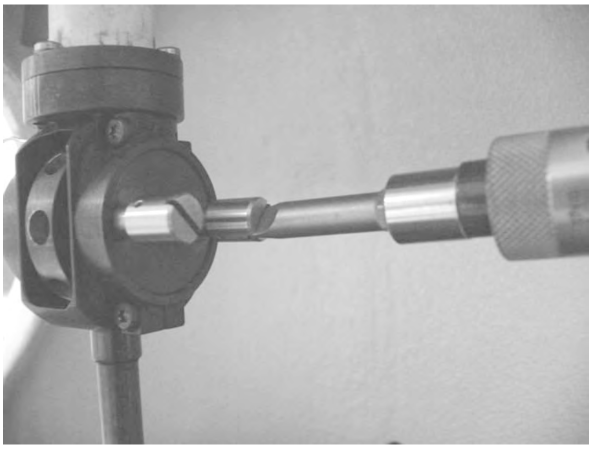
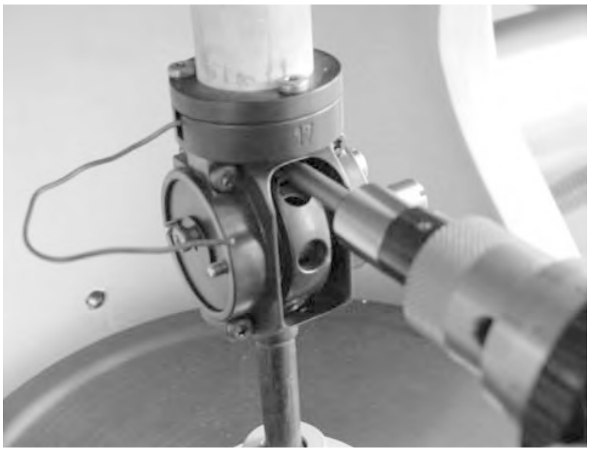


❾

❿-a



❿-b

To install the small tension clamp:

➩ *See page 6-45, Fig. 6-12*

Start:
- Small clamping assembly without the clamp installed
- Fine adjustment of the measuring sensors and displacement adjustment performed
- No clamp or alignment assembly in the clamping assembly
- Furnace arms fully open and swung to the rear

(1) Make sure that the five Torx screws **1a**, **1b** in the movable and fixed clamp holder **2**, **3** are loosened.

Never pull or push the clamp to move it. Exerting excessive force on the clamp could damage the force sensor, the displacement sensor or the drive motor.

(2) Slide the clamp carefully from one side into the clamp holder, ❶. If necessary, loosen the Torx screws even more.

(3) Slide the middle disk of the clamp **2** fully into the movable part of the clamp. Make sure that the clamp is positioned symmetrically in the clamp holder, ❷.

(4) Rotate the clamp so that the arrow on the right side points vertically upward and coincides with the separation line of the halves of the clamp holder, ❸

The middle part of the clamp must not touch the fixed part of the clamp holder.

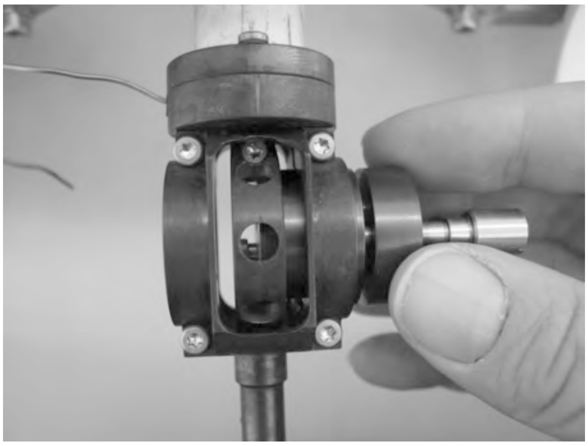
Otherwise there is a fixed connection between the drive motor and the measuring sensors and measurement of the sample becomes impossible.

(5) First slightly tighten the inner Torx screw, ❹.

(6) Then slightly tighten the two right outer Torx screws **1a**.

(7) Pull out the guide pins, ❺.

(8) Remove the end disk by pulling it out from the left side, ❻.



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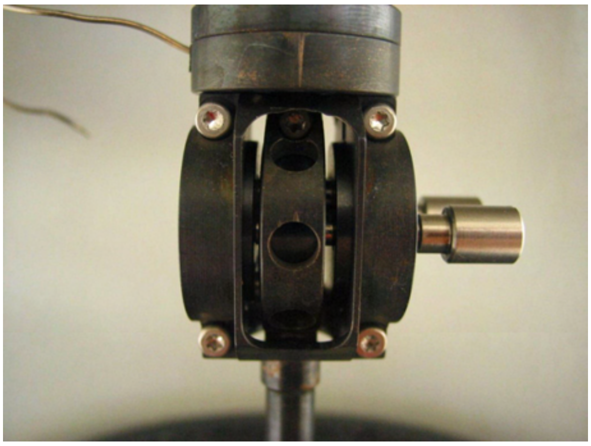

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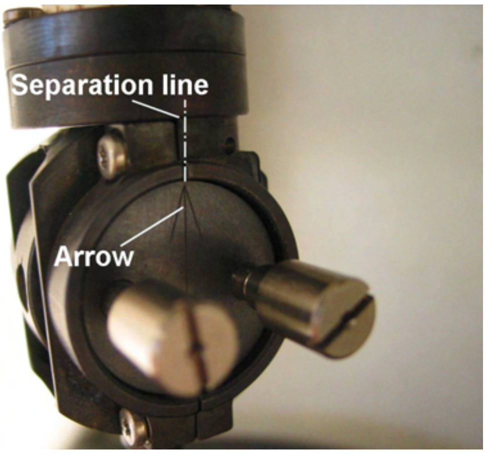

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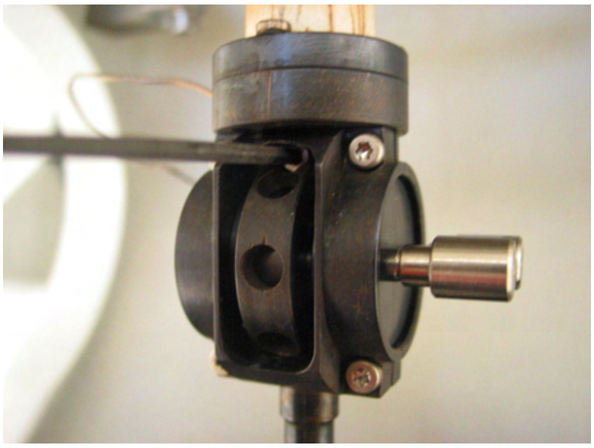

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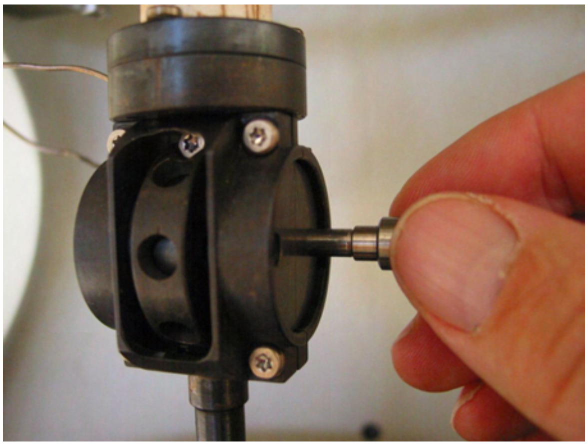

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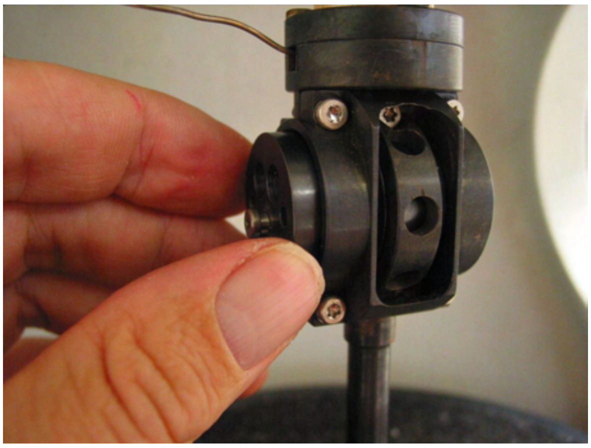


❻

(9) Check visually that the sample is taught and its edges are parallel. If this is not the case, carefully drive down the z-axis table a little by briefly pressing the MOTOR DOWN-key. Repeat this until the sample appears to be taught, ❼.

(10) Push the guide pins in again and insert the end disk by pushing it in from the left side.

(11) First slightly tighten the inner Torx screw **1b**, ❽**-a**, to make sure that the clamp does not slip when you tighten the other screws.

(12) Tighten each of the outer Torx screws **1a** crosswise with a torque of 0.8 Nm, ❽**-b**. Finally tighten the inner Torx screw with a torque of 0.8 Nm, ❽**-c**. Then pull out the guide pins.

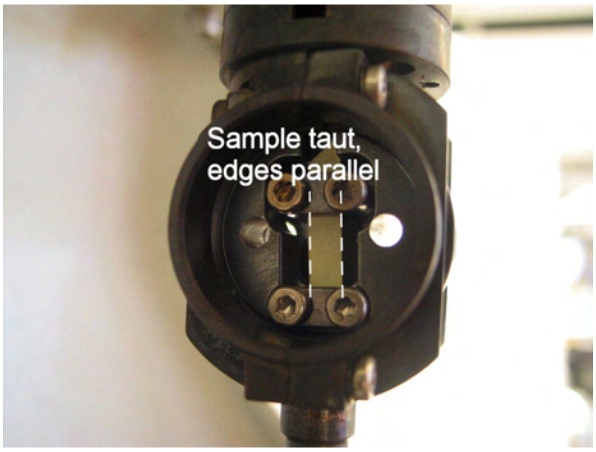
(13) Loosen the clamping screw of the SDTA thermocouple in the surface of the end disk, ❾**-a**.

(14) Insert the SDTA thermocouple into the hole in the surface of the end disk, ❾**-b**, and make sure you push it through to the end so that it is as close as possible to the sample. In general, we recommend that you push the tip in about 10 mm. With thick samples this value is somewhat less, ❾**-c**.

♣ You can make a mark on the thermocouple with a felt-tipped pen.

(15) Tighten the clamping screw to fix the SDTA thermocouple in position.

End:
- Clamp is installed in the clamp holder
- SDTA thermocouple is installed



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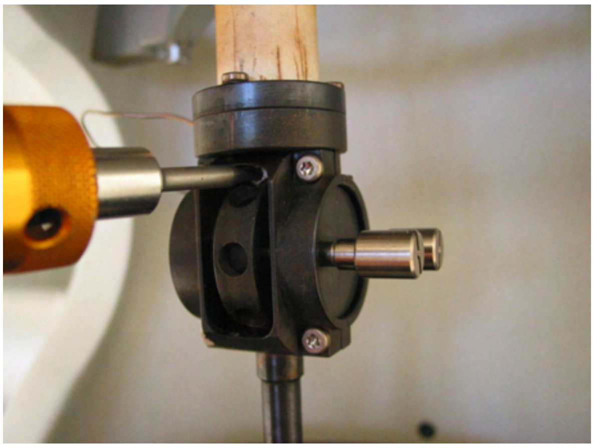

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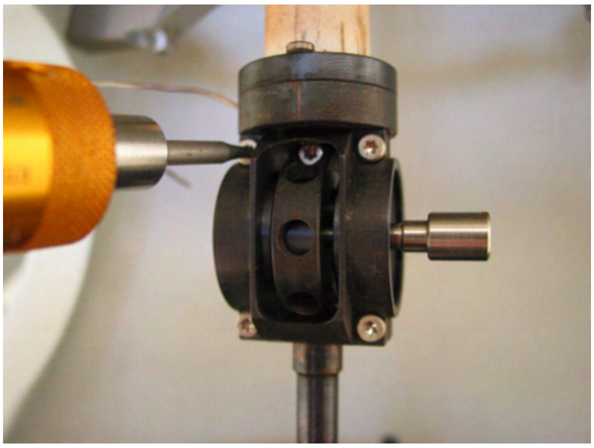

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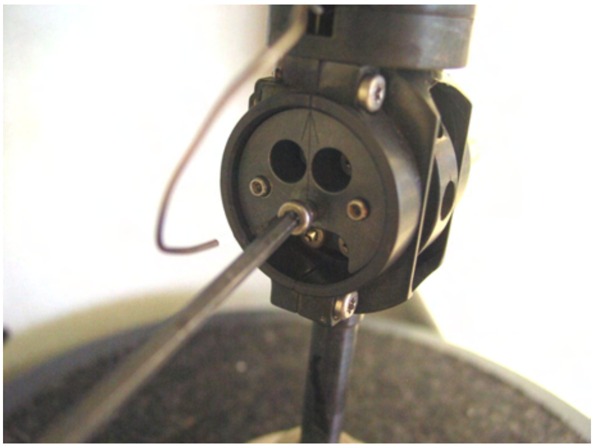
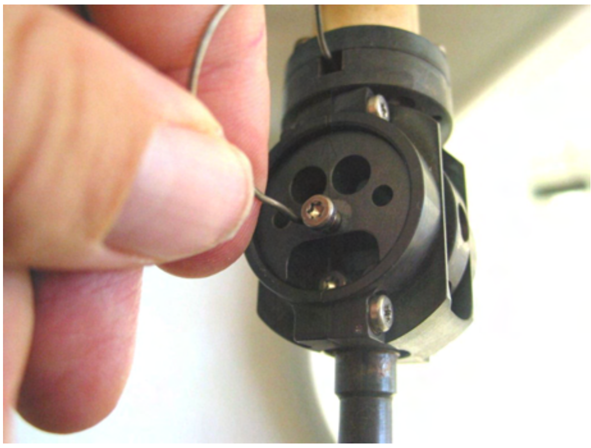
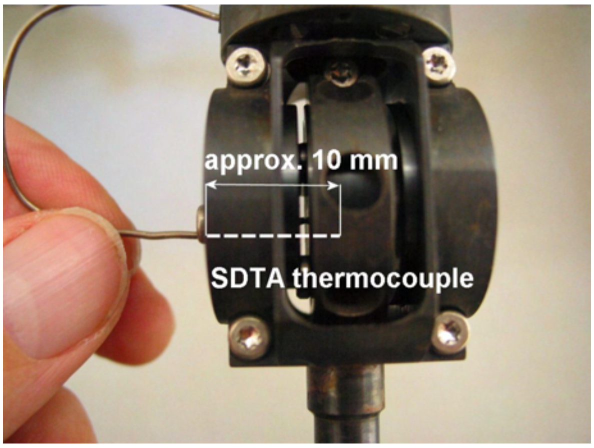


❾-a



❾-b



❾-c

SCA

6.3.5 Removing the small clamps

This section describes how you remove the small clamps. The following instructions apply to the two shear clamps (for highly viscous materials and for low viscosity liquids) as well as for the small tension clamp.

To remove a small clamp:

⇨ *See page 6-45, Fig. 6-12*

Start: - Clamp installed in clamping assembly
- Furnace arms fully open and swung to the rear

(1) Loosen the clamping screw for the SDTA thermocouple in the surface of the end disk.

(2) Pull the SDTA thermocouple out of its hole in the surface of the end disk, ❶.

(3) Slide the guide pins into the guide holes of the clamp and screw the guide pins finger tight in the tapped holes, **❷-a**.

(4) With the special shear clamp for low viscosity liquids: Screw in the four distance screws, **❷-b**.

(5) Loosen all five Torx screws **1a**, **1b** with the Torx screw-driver, ❸.

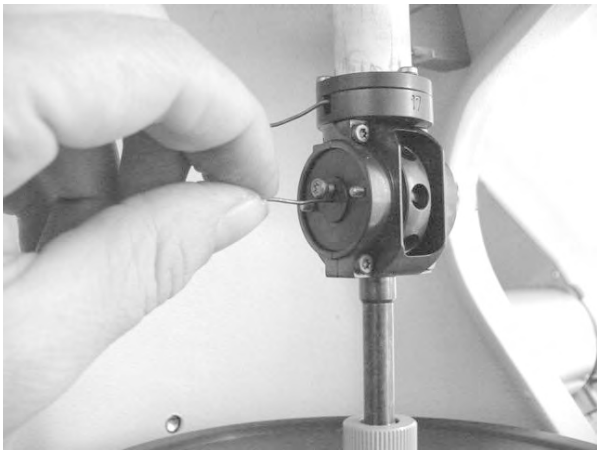
Never pull or push the clamp to move it. Exerting excessive force on the clamp could damage the force sensor, the displacement sensor or the drive motor

(6) Remove the clamp carefully from the clamp holder, ❹.

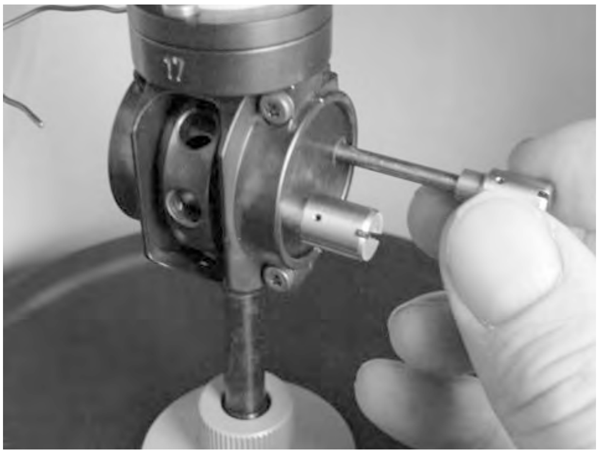
With the tension clamp: Press against the end disk so that it does not slip off the guide pins.

Rotating the clamp carefully and gentle pushing and pulling, helps you to remove the clamp.

End: Shear clamp removed from small clamping assembly

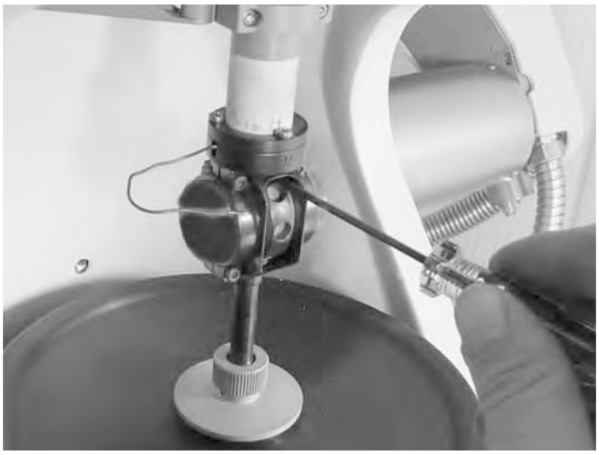


❶

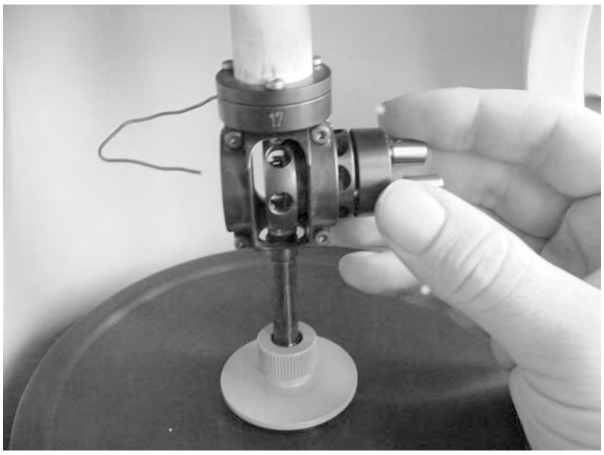


❷-a

❷-b



❸



❹

6.4 Installing and removing the large clamping assembly

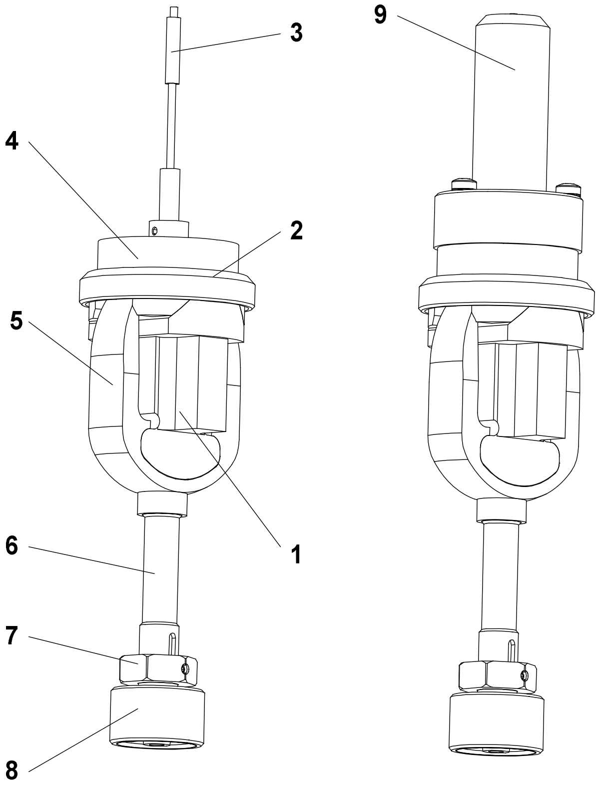
This section shows you how to assemble, align and install the large clamping assembly. It also explains how to remove the clamping assembly.

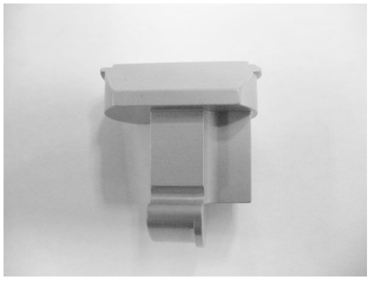
Before the clamping assembly can be installed, the measuring sensors must be pre-aligned as described in the section *Pre-aligning the measuring sensors before installing a clamping assembly*. This adjustment ensures that the measuring sensors are optimally aligned before installation. This step is essential for achieving good results.

6.4.1 Aligning the large clamping assembly within itself

Before you can install the large clamping assembly in the DMA module, you have to align it within itself. This is done with the help of the alignment insert and the protective cover.

Fig. 6-6 shows the large clamping assembly in the pre-installed state with the alignment insert and the protective cover installed.





Alignment insert

1	Alignment insert	**6**	Drive shaft
2	Clamping ring	**7**	Hexagonal nut
3	Displacement sensor core	**8**	Nut
4	Clamp holder, fixed part	**9**	Protective cover
5	Clamp holder, movable part		

Fig. 6-13. Large clamping assembly with alignment insert and protective cover

To install the large clamping assembly in the DMA module, the alignment insert must be inserted. In measurement operation, the alignment insert is replaced with one of the small clamps holding a sample.

Before you align the large clamping assembly within itself, you must assemble it by placing on the fixed part of the clamp holder **5** and the clamping ring **2**.

assembly

To align the large clamping assembly within itself:

➩ *See page 6-57, Fig. 6-13*

Start: Large clamping assembly in assembled state

Always use the Torx screwdriver supplied to tighten and loosen the Torx screws. Using a different tool could damage the Torx screws.

(1) Make sure that clamping ring **2** and the nut **8** have been fully loosened.

(2) Slide the alignment insert **1** carefully from one side into the clamp holder.

(3) Push the alignment insert into the movable part of the clamp holder **5** from the side of its shoulder shaped edge as follows:

- Press the beveled side of the alignment insert **1** against the inner side of the clamp holder.
- At the same time fit the alignment insert into the shoulder-shaped edge of the clamp holder. The alignment insert has a rim that lies flush with the shoulder-shaped edge.

(4) Place the fixed part of the clamp holder **4** on the alignment insert **1** so that its beveled cutout sits snugly on the beveled side of the alignment insert.

(5) Screw the clamping ring **2** so that it is hand-tight.

The alignment insert is fixed in the clamp holder.

(6) Mount the protective cover **9** from above on the flange of the clamp holder. Guide the displacement sensor core carefully into the bore of the protective cover.

The lower guide rim of the protective cover projects into the gap between the flange of the clamp holder and the displacement sensor core. The displacement sensor core is completely inserted in the bore of the protective cover.

(7) Align the movable parts of the large clamping assembly so that the end of the displacement sensor core is positioned concentrically in the bore of the protective cover **9**.

(8) Tighten the hexagonal nut **7** slightly to fix the movable part of the clamp holder in its position.

(9) Remove the protective cover **1** by carefully lifting it upward over the displacement sensor core.

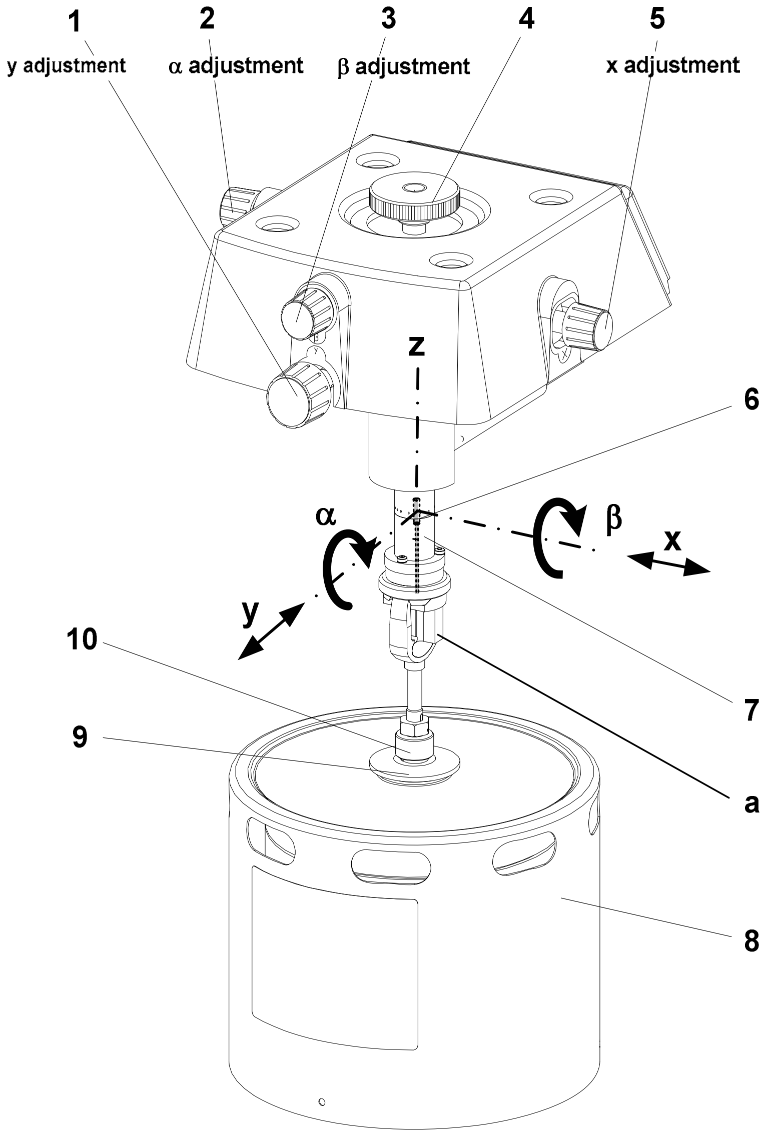
End:
- Large clamping assembly aligned within itself
- Alignment insert is mounted and protective cover removed
- Large clamping assembly ready to install in DMA module

6.4.2 Installing the large clamping assembly

This section describes how to install the large clamping assembly. Before the clamping assembly can be installed, the measuring sensors must be pre-aligned as described in section *Pre-aligning the measuring sensors before installing a clamping assembly*.

After this manual alignment has been performed, you may have to readjust the x and y positions slightly while installing the large clamping assembly. The installation procedure of the clamping assembly therefore consists of two steps: actually installing the clamping assembly and aligning the measuring sensors (four axes alignment).

Fig. 6-14 shows the installed large clamping assembly and gives an overview of the adjustment possibilities with the four axes alignment device.



1 Knob for adjusting the y -position
2 Knob for adjusting the α angle
3 Knob for adjusting the β angle
4 Locking screw
5 Knob for adjusting the x -position
6 Displacement sensor core
7 Displacement sensor housing
8 Drive motor
9 Threaded fitting
10 Nut
a Alignment insert

Fig. 6-14. Large clamping assembly installed

LCA

To install the large clamping assembly:

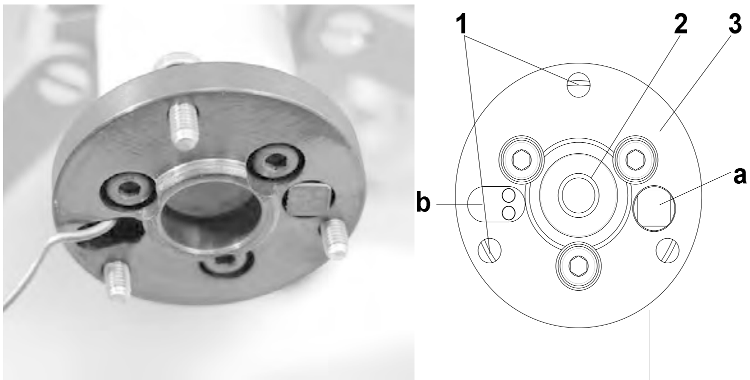
➩ *See page 6-59, Fig. 6-14*

Start:
- Large clamping assembly pre-installed
- Clamping assembly aligned within itself
- Alignment insert mounted *

(1) Hold the large clamping assembly up at an angle below the displacement sensor flange so that the heads of the Torx screws are pointing toward you. Guide the displacement sensor core **6** into the displacement sensor housing **7** by raising the entire clamping assembly, ❶.

Install large clamping assembly

(2) Fasten the large clamping assembly as follows to the flange of the displacement sensor, ❷:



1 Torx screws
2 Displacement sensor core
3 Displacement sensor flange
a CA thermocouple
b Hole for SDTA thermocouple

Fig. 6-15. Displacement sensor flange (view from below)

- Hold the upper part of the clamping assembly under the flange of the displacement sensor. Ensure that the thermocouple projecting from hole **b** is positioned freely in the notch (Fig. 6-15). Make sure you do not wedge the thermocouple while fastening the clamping assembly.

♣ The Torx screws must be aligned with the tapped holes in the flange of the clamping assembly.

- Screw in the three Torx screws and tighten them firmly with the same torque (as far as possible) using the Torx Allen wrench supplied.

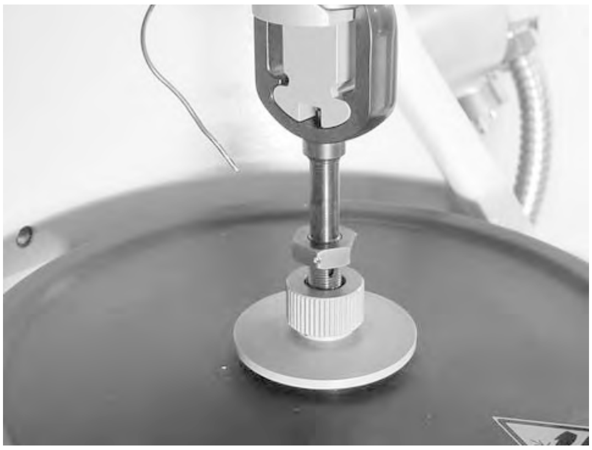
The large clamping assembly is now fixed to the flange of the displacement sensor.

* See the section *Installing and removing the alignment insert of the large clamping assembly.*

❶

❷

❸



❹

Do not drive the z-axis table too far toward the clamping assembly. The drive motor could suffer damage.

---

(3) Drive the z-axis table upward by pressing the MOTOR UP key once. The z-axis table stops automatically at a position in which the conical end of the drive shaft is just over the bore of the threaded fitting **9** of the drive motor, ❸ and ❹.

♣ The travel speed of the z-axis table changes from high to low in this intermediate position.

LCA

(4) If you have previously not done so, loosen the support structure of the measuring sensors as follows:

- Remove the Torx screws (A) with the Torx wrench and remove the front clamp (B). Loosen the Allen screws (C) with a 4-mm Allen wrench, ❺, but do not completely remove them so that the support structure is still attached.
- Fold the support structure back so that it is no longer in contact with length sensor flange, ❻.

(5) Loosen the locking screw of the four axes alignment device, ❼, then slightly tighten it again to the point where you feel slight resistance.

Readjust x and y positions

(6) If necessary, readjust the x and y positions as follows:

- Slide the nut **10** upward and hold it up with your fingers.
- If the conical end of the drive shaft is not concentrically above the bore in the threaded fitting **9** of the drive motor: Using knobs **1** and **5**, adjust the x and y positions until the conical end of the drive shaft is positioned concentrically above the bore, ❽**-a** and ❽**-b**.
- Drive the z-axis table upward carefully by pressing the MOTOR UP key. Continuously check visually that the position of the conical end of the drive shaft in the bore is concentric. If necessary, adjust the x and y positions again.

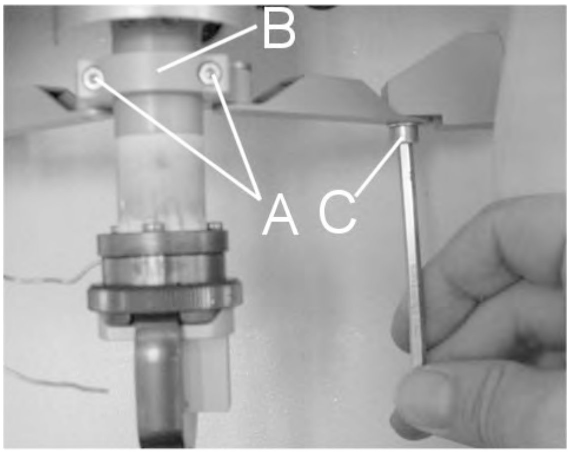
(7) Check the play of the drive motor diaphragm before tightening the nut **10**, by pulling the diaphragm upward with the threaded fitting **9** using your fingers, ❾. Drive the z-axis table upward until the play is less than 0.5 mm. Do not pull the diaphragm while the z-axis table is moving.

(8) Tighten the nut **10** firmly.

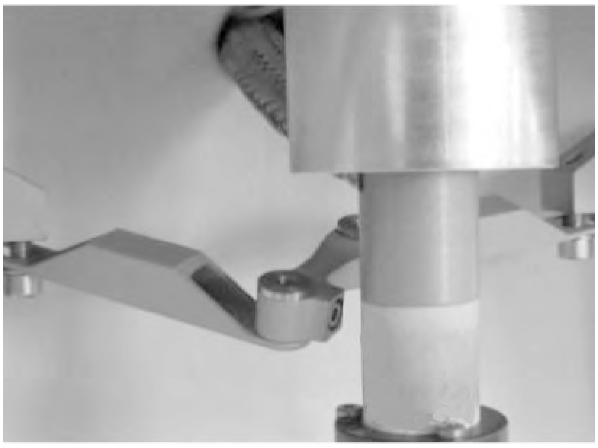
End: - Large clamping assembly installed.
- Support structure loosened

The pre-alignment of the measuring sensors has been completed. You can now begin the fine alignment of the measuring sensors. See the section *Fine alignment of the measuring sensors (four axes alignment) on page 6-102*.

If you have installed the large clamping assembly for the first time, you must perform a displacement adjustment after the fine alignment. This is explained in *Chapter 8, Calibration and adjustment* in the section *Adjusting the displacement*.

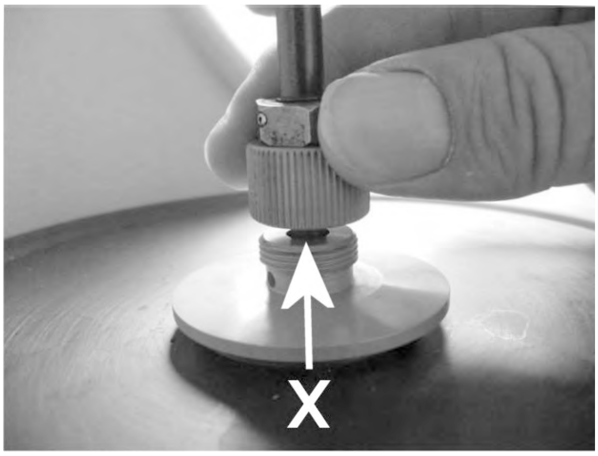


❺

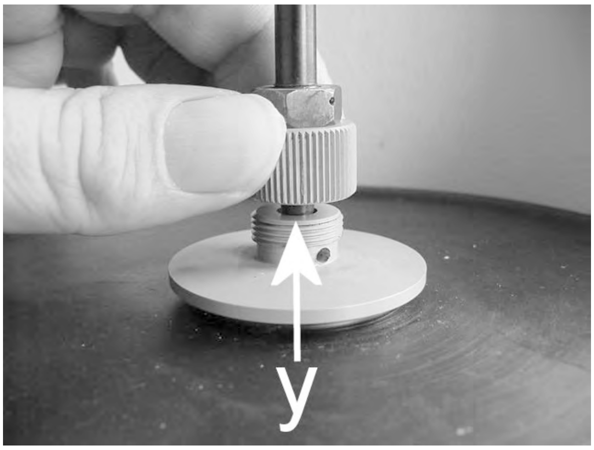


❻

❼



❽-a



❽-b

❾

LCA

6.4.3 Removing the large clamping assembly

To remove the large clamping assembly from your DMA module, the alignment insert must be mounted in the clamping assembly.

To remove the large clamping assembly:

⇨ *See page 6-59, Fig. 6-14*

Start:
- Alignment insert mounted *
- The two halves of the furnace are fully open and swung to the rear

(1) Fully loosen the three Torx screws on the displacement sensor, ❶. Use the Torx Allen wrench supplied. Make sure that the clamping assembly is completely loose and can move freely downward when you drive the z-axis table downward.

(2) Drive the z-axis table fully downward. Depress the MOTOR DOWN key until the z-axis table stops in the intermediate position. Briefly depress the MOTOR DOWN key once more to move the z-axis table to the lower position. The z-axis table stops automatically at the lower position.

♣ The travel speed of the z-axis table changes from low to high in the intermediate position.

The clamping assembly has been driven right down with the z-axis table. There is now sufficient room above the displacement sensor core to remove the clamping assembly.

(3) Loosen nut **10** of the drive shaft from the threaded fitting of the drive motor.

(4) Lift the clamping assembly upward out of the drive motor fitting. Remove the clamping assembly carefully in a downward direction. Make sure that you do not damage the displacement sensor coil and the displacement sensor core, ❷**-a** and ❷**-b**.

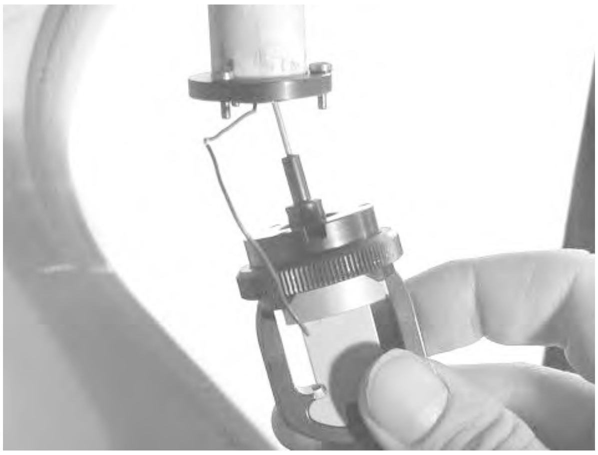
(5) Screw on the protective cover, ❸.

End: Large clamping assembly removed

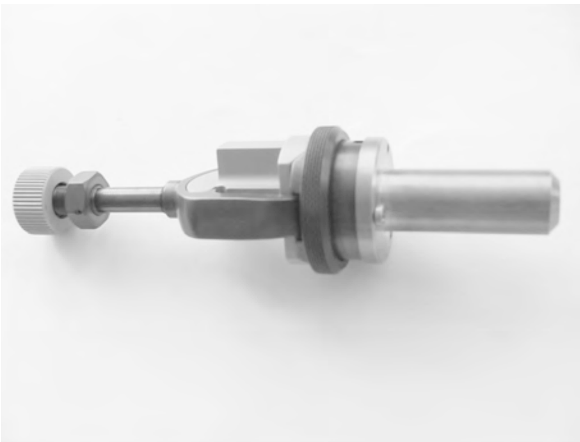
* See the section *Installing and removing the alignment insert of the large clamping assembly.*

❶

❷-a



❷-b



❸

6.4.4 Installing and removing the alignment insert of the large clamping assembly

Before you install or remove the clamping assembly, the alignment insert must always be installed to ensure that the parts of the clamping assembly are fixed together firmly.

Never use excessive force to move the alignment insert. Exerting excessive force on the clamp could damage the force sensor, the displacement sensor or the drive motor.

To install the alignment insert:

➩ *See page 6-57, Fig. 6-13*

Start:
- Large clamping assembly installed, clamp removed
- The two halves of the furnace are fully open and swung to the rear

(1) Make sure that the clamping ring **2** of the fixed part of the clamp holder **4** is loosened.

(2) Make sure that the hexagonal nut **6** on the drive shaft is screwed fully down so that the stem connected with it is sunk in the clamp holder.

(3) Push the alignment insert into the movable part of the clamp holder **5** from the side of its shoulder shaped edge as follows, ❷:

- Press the beveled side of the alignment insert **1** against the inner side of the clamp holder **4**.
- At the same time fit the alignment insert **1** into the shoulder-shaped edge of the clamp holder. The alignment insert has a rim that lies flush on the shoulder-shaped edge.

(4) Place the fixed part of the clamp holder **4** on the alignment insert **1** so that its beveled cutout sits snugly on the beveled side of the alignment insert.

(5) Screw the clamping ring **2** so that it is hand-tight.

(6) Tighten the hexagonal nut **7** on the drive shaft with your fingers.

End: Alignment insert is installed in large clamping assembly, ❸.

LCA

To remove the alignment insert:

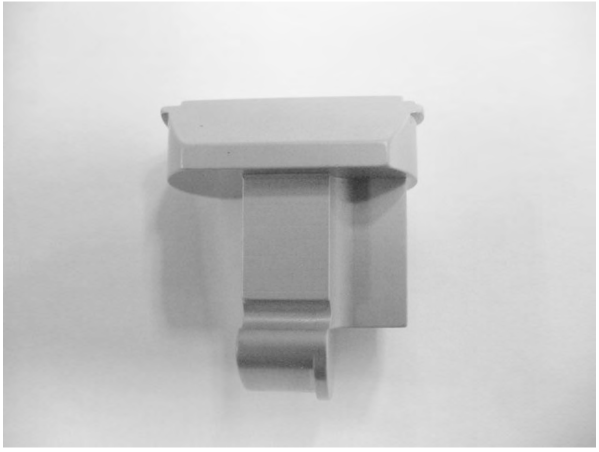
Start: - Alignment insert installed in large clamping assembly ⇨ *See page 6-57, Fig. 6-13*
- Two halves of the furnace are fully open and swung to the rear

(1) Loosen the clamping ring **2**.

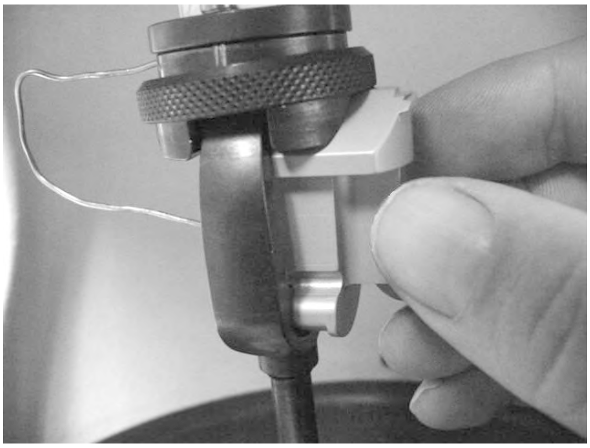
(2) Loosen the hexagonal nut **7** on the drive shaft.

(3) Pull the alignment insert carefully toward the right side out of the clamp holder, ❷.

End: Alignment insert removed from large clamping assembly.



❶



❷

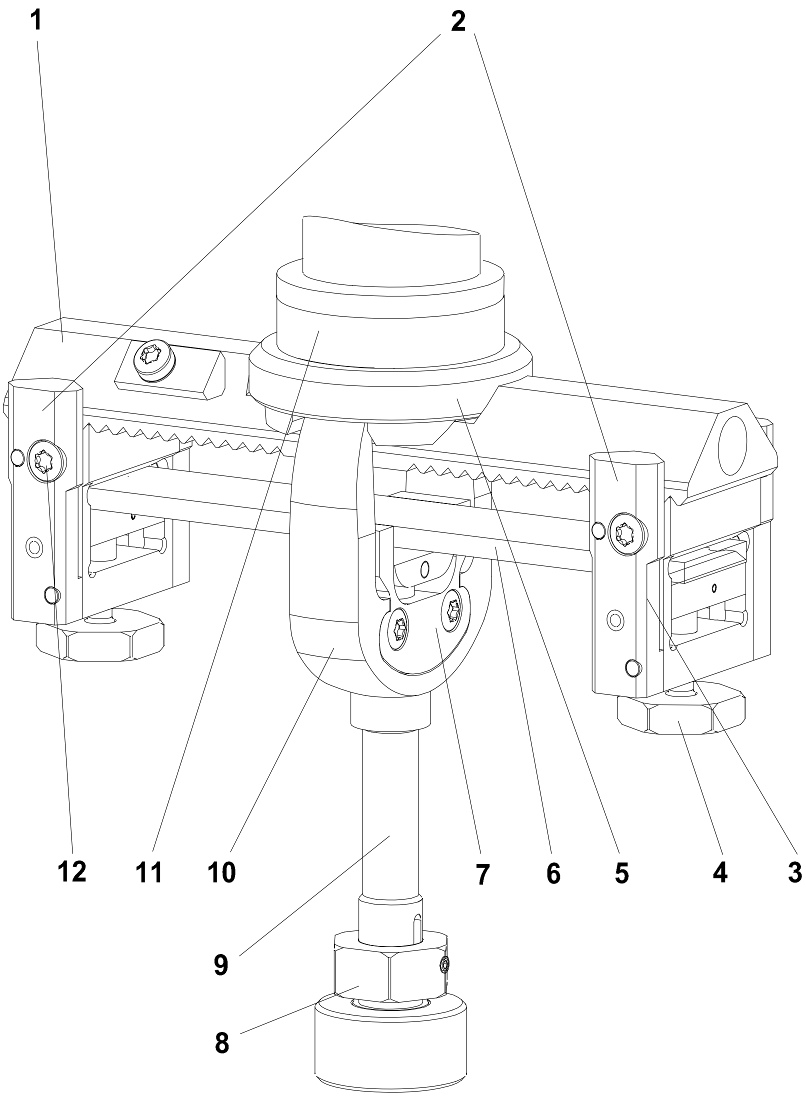
❸

6.5 Mounting the sample in the large clamping assembly

Several different clamps are available for the large clamping assembly. The clamp you use depends on measuring mode and the type of sample. The following section describes how you install and remove different clamps with the sample in and from the large clamping assembly.

6.5.1 Installing the bending clamp with the sample in the large clamping assembly

With the bending clamp, the sample is mounted directly in the clamp in the clamping assembly, and not first outside the clamping assembly as it is with the other clamps of the large clamping assembly.



1 Bending beam
2 Rider
3 Cheek of the rider
4 Hexagonal screw
5 Clamping ring
6 Sample
7 Movable part of bending clamp
8 Hexagonal nut
9 Drive shaft
10 Clamp holder, movable part
11 Clamp holder, fixed part
12 Torx screw

Fig. 6-16. Bending clamp with clamp holder

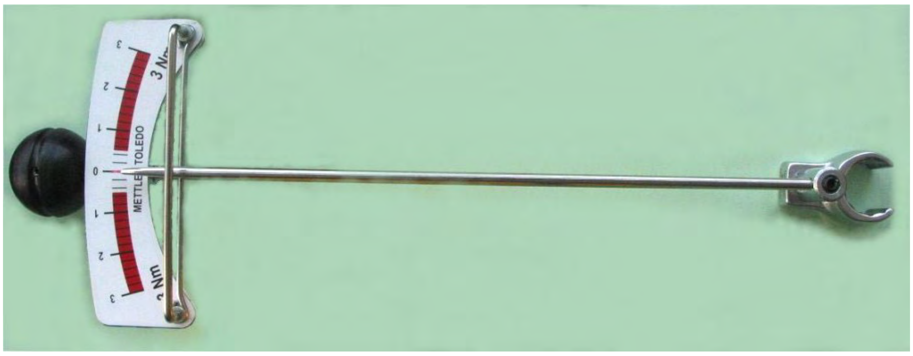
The two phases of the installation procedure

In the procedure for installing the bending clamp there are two distinct phases: The first phase, which includes the installation of the bending beam **1** and the mounting of the riders **2** and sample clamps **7**, is the same for all bending modes. The second phase, the actual mounting of the sample, depends on the particular bending mode.

The type of riders **2** and the movable part of the clamp **7** used also depends on the bending mode of the measurement. There are special interchangeable types of riders and clamps for cantilever type bending and for 3-point bending.

Additional tool required

The following torque wrench, which is supplied as standard accessory, is additionally required to install the large clamping assembly. The torque wrench is used to exert the correct torque on the hexagonal nut **8** and screws **4**.



Torque wrench

LCA

To install the bending clamp and mount the sample:

➪ *See page 6-68, Fig. 6-16*

Start:
- Large clamping assembly installed without a clamp or alignment insert
- Bending clamp with riders and the sample are ready for installation
- Fine alignment and displacement adjustment performed
- Two halves of the furnace are fully open and swung to the rear

(1) Make sure that the clamping ring **5** of the fixed part of the clamp holder **11** has been loosened.

(2) Slide the bending beam **1** with the beveled side upward through the movable part of the clamp holder **10** pressing it against the inside of the fixed part of the clamp holder **11**, **❶-a.**

(3) Mount the fixed part of the clamp holder **11** on the bending beam **1** so that its angled recess fits snugly on the beveled side of the bending beam.

(4) Tighten the clamping ring **5** by hand, **❶-b**.
The bending beam is now fixed in the clamp holder.

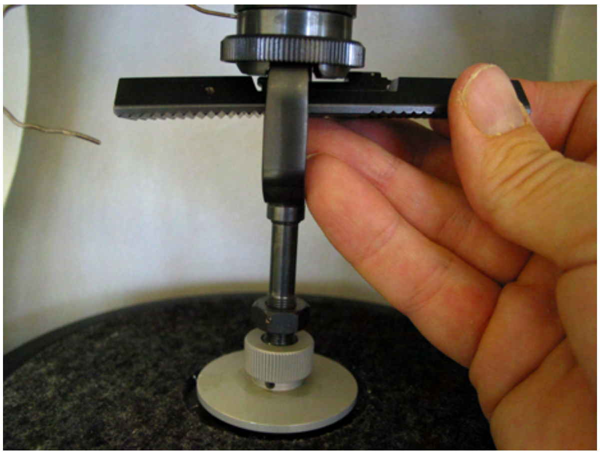
(5) Select the desired type of riders, **❷-a** and **❷-b**. On both riders **2**, make sure that the Torx screws **12** on the cheeks have been loosened, **❷-a.**

♣ The Torx screws fix the riders **2** on bending beam **1**. If they are not loose enough, the riders cannot be slid over the bending beam.

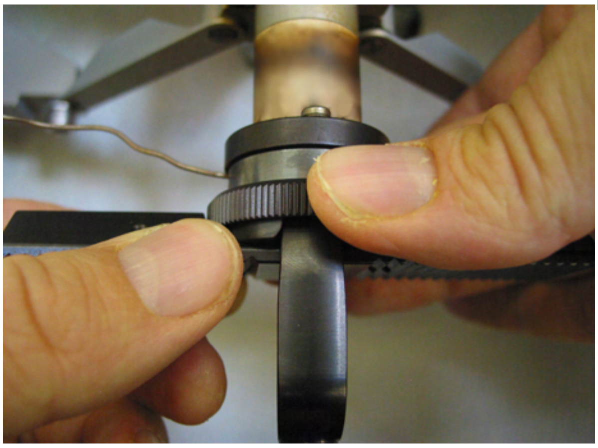
(6) Guide a rider **2** over each of the ends of the bending beam **1** and position the riders **2** symmetrically at the desired point on the toothed surface of the bending beam. Make sure that the cheek of each rider with the Torx screw is facing toward you, **❸**. The position of the riders depends on the length of the sample.

(7) Slightly tighten the Torx screws of both cheeks **4** so the riders do not fall off, **❸**.

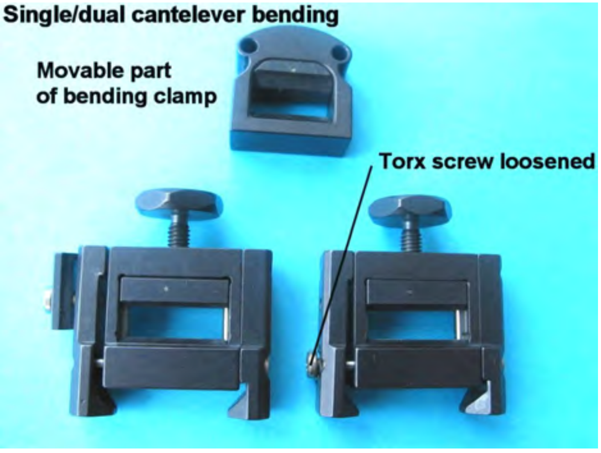
(8) Make sure that the hexagonal nut **8** of the drive shaft **9** has been loosened.



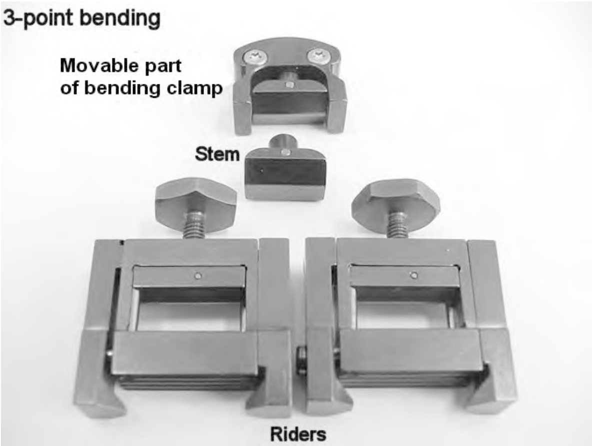
❶-a



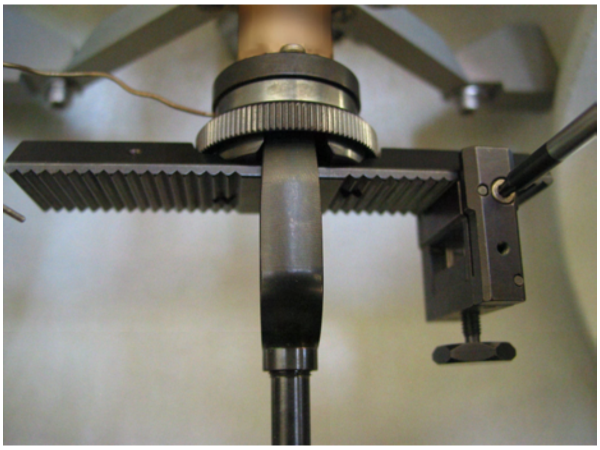
❶-b


Single/dual cantelever bending
Movable part
of bending clamp
Torx screw loosened

❷-a


3-point bending
Movable part
of bending clamp
Stem
Riders

❷-b



❸

LCA

(9) Select the movable part of the clamp, ❹**-a**. you require for your measurement mode.

- For 3-point bending measurement: Select the stem that corresponds to the desired sample thickness and insert it in the bore in the movable part of the clamp. Adjust the movable part of the clamp to the thickness of the sample by placing it upside down on the sample and fix the stem in its position by fastening the two Torx screws, ❹**-b**.

(10) Place the movable part of the clamp **7**, ❹**-a**, in the recess of the movable part of the clamp holder **10**, ❹**-c**. The movable part of the clamp has a rim that fits flush in the shoulder shaped edge on one side of the clamp holder **10**. Tighten the hexagonal nut **8** a little so that the movable part of the clamp **7** can no longer fall out.

(11) Slide the sample **6** from one side through the rider **2**. Continue to slide the sample further through the movable part of the clamp **7**, ❺**-a**.

(12) For 3-point bending and dual cantilever bending:

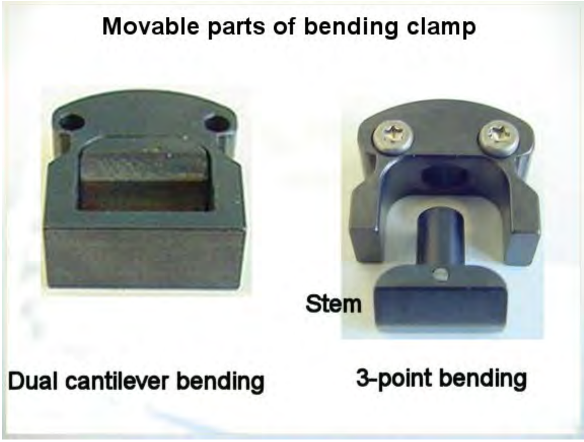
- Continue to slide the sample through the other rider. Position the sample symmetrically between the left and the right rider, ❺**-b**.

or (for single cantilever bending):

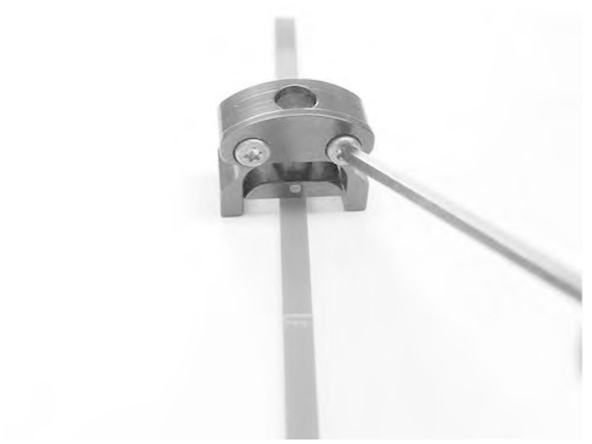
- Position the sample symmetrically between the left rider and the movable part of the clamp holder.

♣ You can check whether the riders are symmetrically positioned by counting the number of teeth between the riders **2** and the ends of the bending beam **1**.

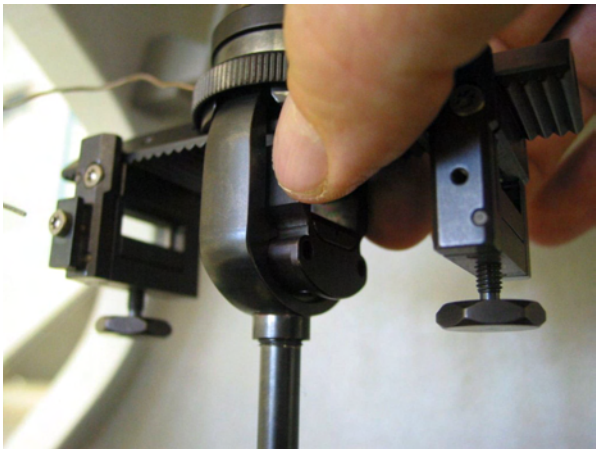
(13) Fix both riders **2** by tightening the Torx screws **12** with the torque wrench supplied setting a torque of 0.8 Nm, ❻.



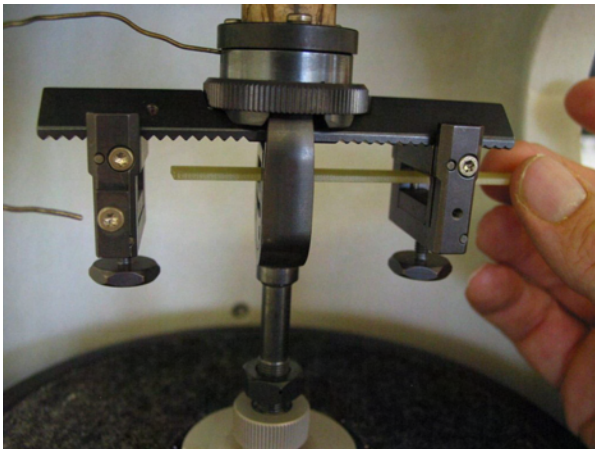
❹-a



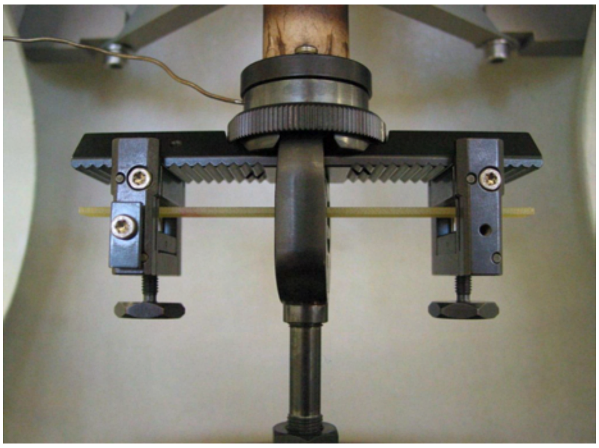
❹-b



❹-c



❺-a



❺-b

❻

LCA

(14) For the single cantilever mode, make sure that the sample is positioned in the middle of both riders, ❼. For the single cantilever mode, the sample must be positioned in the middle of the rider and the movable part of the clamp.

(15) For the single and dual cantilever bending modes, fasten the sample as follows:

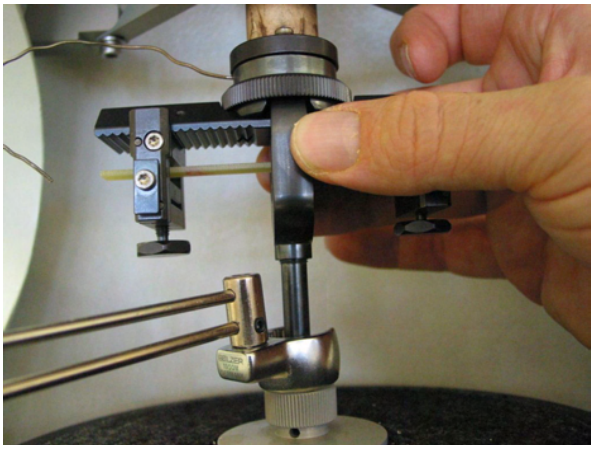
- First tighten the hexagonal nut **8** as far as possible with your fingers.
- Set the torque wrench supplied in position on the hexagonal nut, ❽**-a**. Hold the movable part of the clamp holder firmly with one hand so that it does not turn when you tighten the hexagonal nut. Using your other hand, tighten the hexagonal nut with a maximum torque of 0.5 Nm, ❽**-b**.
- For dual cantilever measurement: Tighten the hexagonal screws on both riders **4** with the torque wrench to ensure that you do not set a torque of more than 0.5 Nm, ❽**-b**, ❾. For single cantilever measurement: Tighten the hexagonal screw of only one rider with a torque of up to 0.5 Nm.

♣ The amount of torque set to fasten the nut and screws depends very much on the type of sample you are measuring. The sample must be fixed in place to prevent it from slipping during the measurement. But at the same time it must be loose enough to avoid excessive clamping pressure, which would affect the measurement or even damage the sample. As a rough guideline, we recommend a torque value of about 0.5 Nm. The torque value should not be greater than 1.0 Nm.

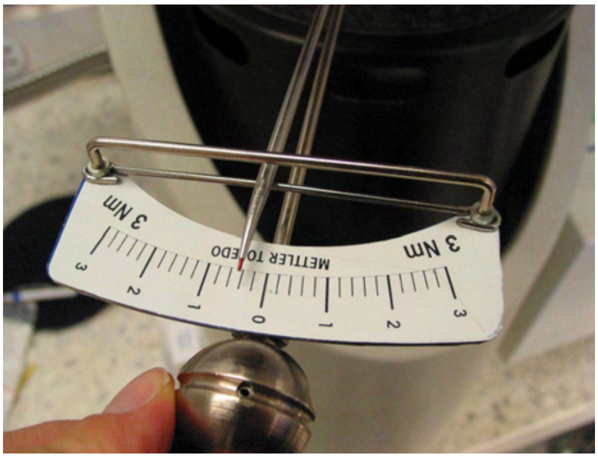
(16) Fix the SDTA thermocouple in position by wedging it between the cheek of the rider and the plate screwed on to it. Bend the thermocouple carefully to position it as close as possible to the sample without making direct contact, ❿. Be careful not to damage the thermocouple.

End: Bending clamp with sample installed in large clamping assembly

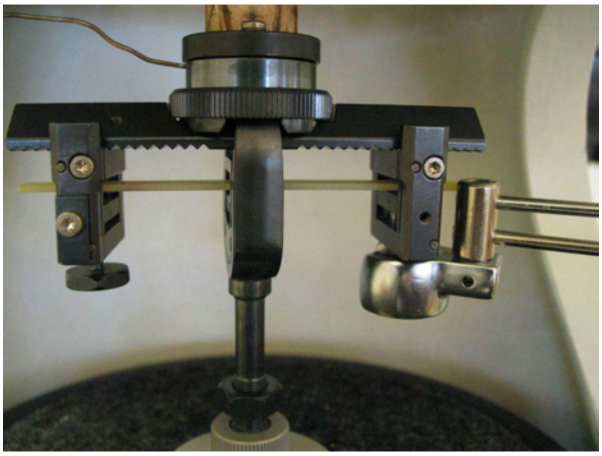
❼



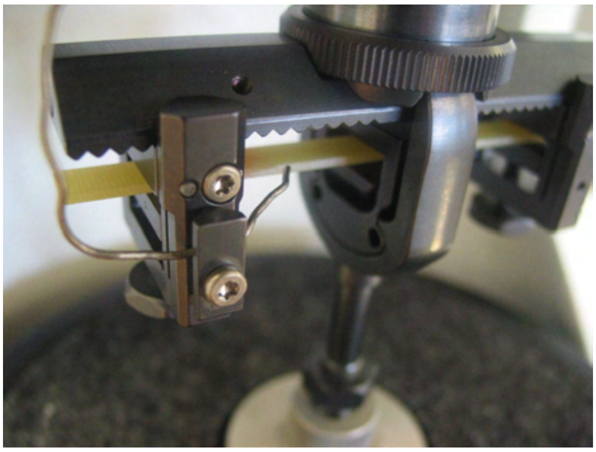
❽-a



❽-b



❾



❿

Changing the sample in the bending clamp

The following procedure explains how you change the sample in the bending clamp. You do not have to remove the bending clamp to change the sample between measurements in the same bending mode.

To change a sample in the bending clamp:

⇨ *See page 6-68, Fig. 6-16*

Start: Sample mounted in bending clamp

(1) Loosen both the hexagonal screws **4** and the hexagonal nut **8** on the drive shaft **9** with the supplied torque wrench, ➊.

(2) Slide the sample **6** out of the bending clamp from one side.

(3) Slightly loosen the Torx screws **12** on both riders **2**.

♣ The Torx screws fix the riders **2** on the bending beam **1**. If they are not loose enough, the riders cannot be slid over the bending beam.

(4) If you want to change the bending mode from cantilever type bending to 3-point bending:

- Loosen the Torx screws **12**, remove the riders **2** and replace them with the riders for the desired bending mode. See the section *Installing the bending clamp with the sample in the large clamping assembly*, step (6) on page 6-70 for this.
- Remove the movable part of the bending clamp **7** and replace it with the part for the desired bending mode. See the section *Installing the bending clamp with the sample in the large clamping assembly*, step (10) on page 6-72 for this.

(5) Slide the riders **2** to the desired position for the new sample **6**. Make sure that both riders **2** are mounted symmetrically on the bending beam **1**.

♣ You can check whether the riders are symmetrically positioned by counting the number of teeth between the riders **2** and the ends of the bending beam **1**.

(6) Slightly tighten the Torx screws of both cheeks **4** so the riders do not fall off.

(7) Slide the sample **6** from one side through the rider **2** over the angled clamping jaw. Continue to slide the sample further through the movable part of the clamp holder **10**, **➋-a**.

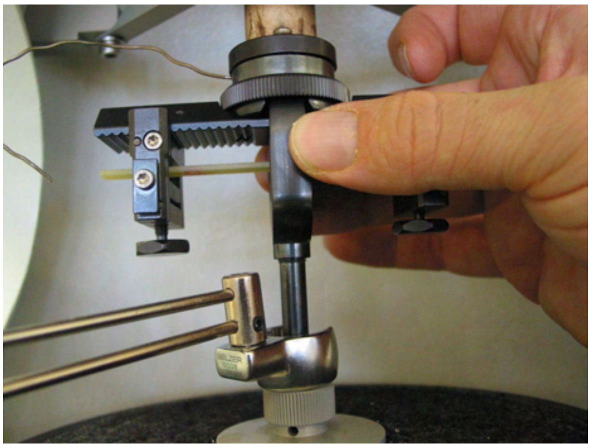
(8) For 3-point bending and dual cantilever bending:

- Continue to slide the sample through the other rider. Position the sample symmetrically between the left and the right rider. The sample should be symmetrically positioned below the bending beam between the left and the right the rider, ❷-b.

or for single cantilever bending:

- Position the sample symmetrically between the left rider and the movable part of the clamp holder.

(9) Fix both riders **2** by tightening the Torx screws **12** with the torque wrench supplied setting a torque of 0.8 Nm, ❸.



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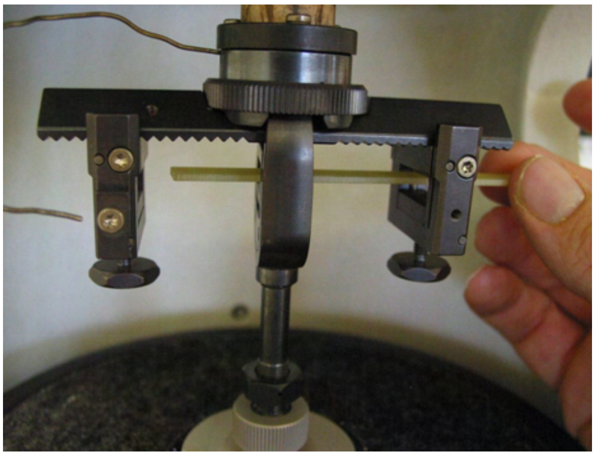
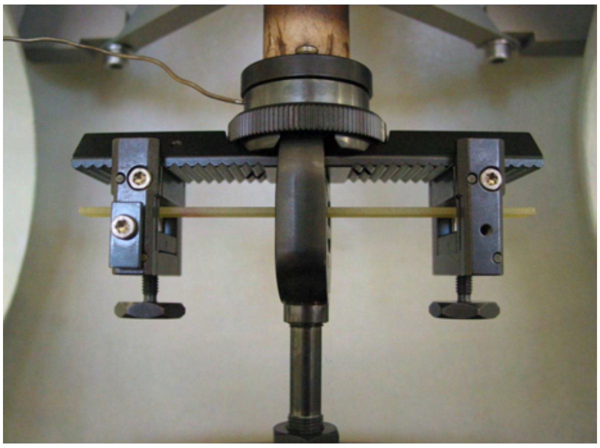
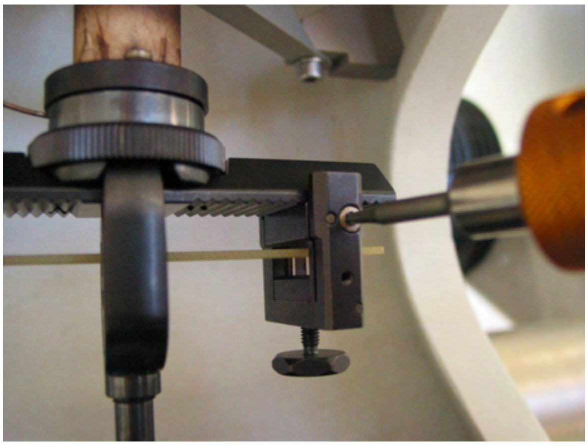


❷-a



❷-b



❸

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(10) Make sure that the sample is positioned in the middle of both riders. For the 3-point bending mode, the sample must be positioned in the middle of the rider and the movable part of the clamp. Check this visually from the ends of the bending clamp, ❹.

(11) For the single and dual cantilever bending modes, fasten the sample as follows:

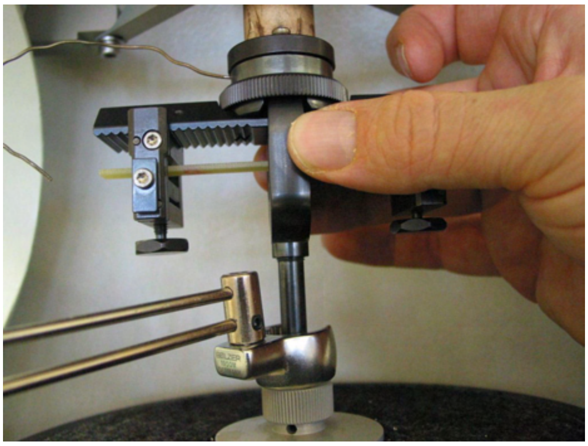
- First tighten the hexagonal nut **8** as far as possible with your fingers.
- Set the torque wrench supplied in position on the hexagonal nut, **❺-a**. Hold the movable part of the clamp holder firmly with one hand so that it does not turn when you tighten the hexagonal nut. Using your other hand, tighten the hexagonal nut with a maximum torque of 0.5 Nm, ❻.
- For dual cantilever measurement: Tighten the hexagonal screws on both riders **4** with the torque wrench to ensure that you do not set a torque of more than 0.5 Nm, **❺-b**, ❻. For single cantilever measurement: Tighten the hexagonal screw of only one rider with a torque of up to 0.5 Nm.

♣ The amount of torque set to fasten the nut and screws depends very much on the type of sample you are measuring. The sample must be fixed in place to prevent it from slipping during the measurement. But at the same time it must be loose enough to avoid excessive clamping pressure, which would affect the measurement or even damage the sample. As a rough guideline, we recommend a torque value of about 0.5 Nm. The torque value should not be greater than 1.0 Nm.

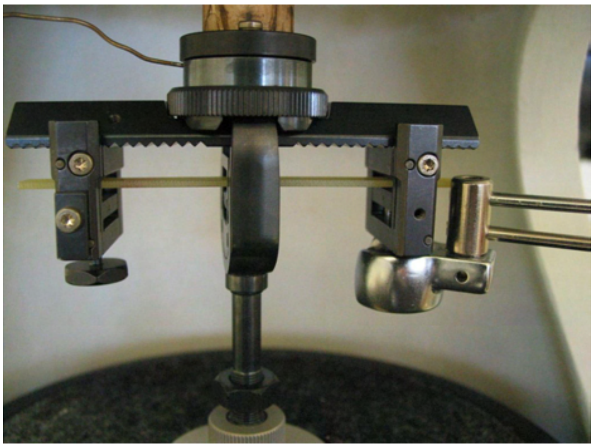
(12) Fix the SDTA thermocouple in its position by wedging it between the cheek of the rider and the plate screwed on to it. Bend the thermocouple carefully to position it as close as possible to the sample without making direct contact, ❼. Be careful not to damage the thermocouple.

End: New sample mounted in the bending clamp.

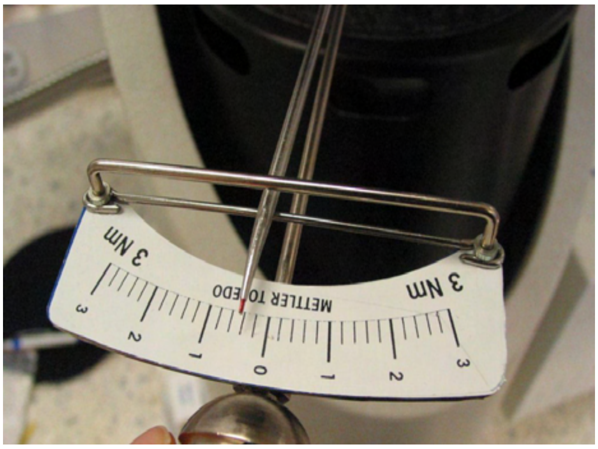
❹



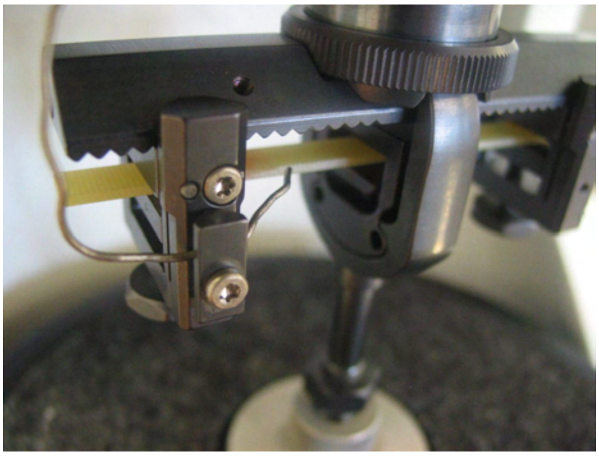
❺-a



❺-a



❻



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6.5.2 Removing the bending clamp and sample

If you want to perform measurements in a different mode than the bending modes, you must first remove the sample and then, if necessary, the bending clamp.

To remove the bending clamp and sample:

➩ *See page 6-68, Fig. 6-16*

Start: - Bending clamp installed
- Sample mounted

(1) Loosen both the hexagonal screws **4** and the hexagonal nut **8** on the drive shaft **9** with the supplied torque wrench, ➊.

(2) Slide the sample **6** out of the bending clamp from one side, ➋.

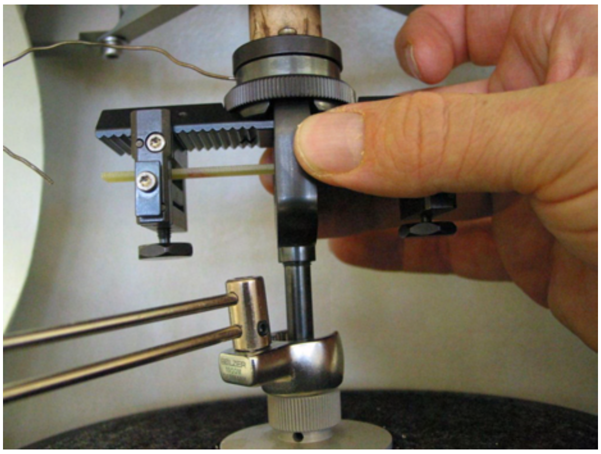
(3) Remove both riders **2** by loosening the Torx screws **12** and sliding both riders off of the bending beam **1**, ➌.

♣ The Torx screws fix the riders on the bending beam. If they are not loose enough, the riders cannot be slid over the bending beam.

(4) Remove the movable part of the clamp **6** from the cutout of the movable part of the clamp holder **7**. Loosen the hexagonal nut **8** first until the movable part of the clamp can be easily removed, ➊ and ➍.

(5) Loosen the clamping ring **5** by hand and pull the bending beam **1** at one end out of the clamp holder **7**, ➎ and ➏.

End: Bending clamp and sample removed



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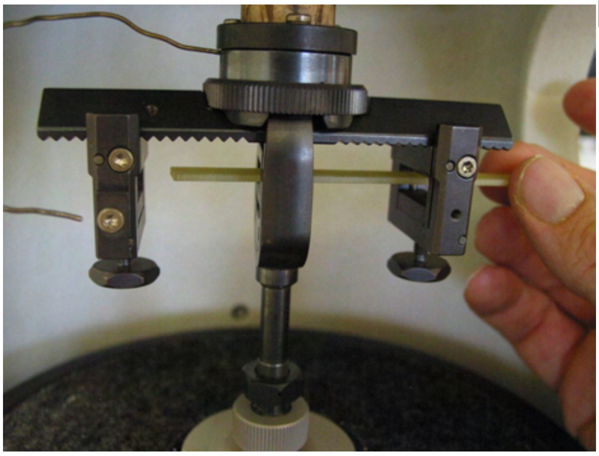
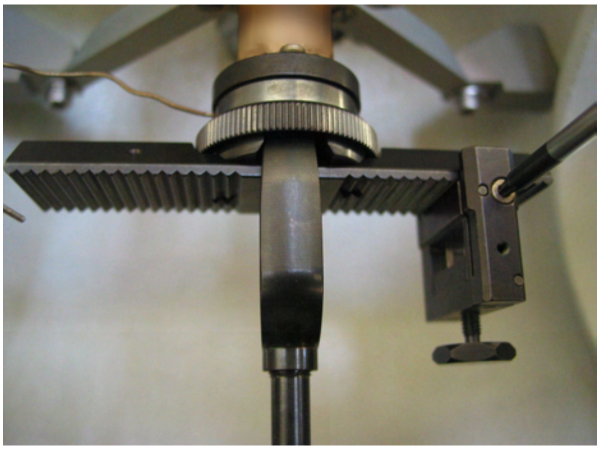


❷



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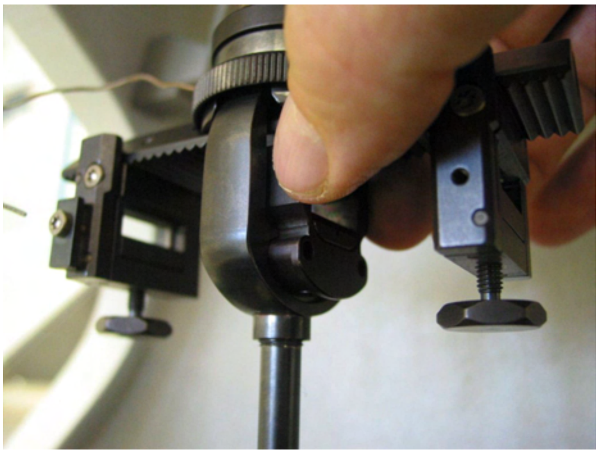

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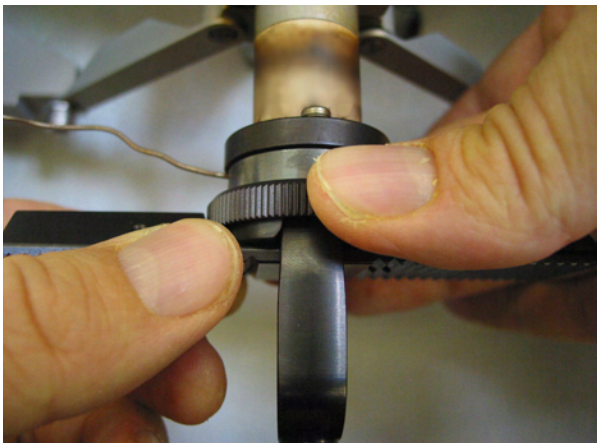

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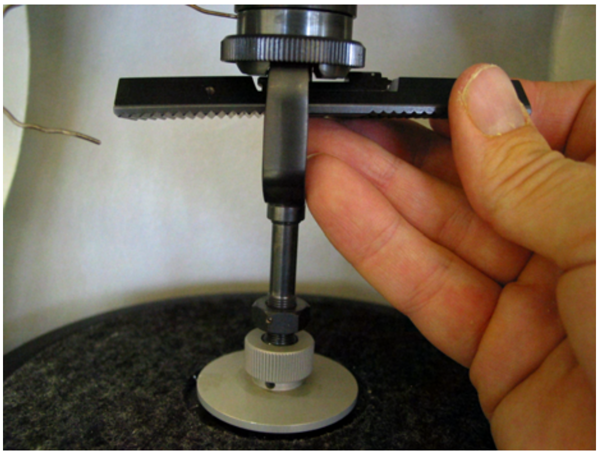

❻

6.5.3 Installing the tension and compression clamps with the sample in the large clamping assembly

Several different types of clamps can be installed in the large clamping assembly, depending on the particular type of measurement and sample. The clamps are interchangeable in the clamping assembly and it is not necessary to remove and reinstall the entire clamping assembly to perform a measurement with a different sample. With the tension clamp and the compression clamp, the sample can be prepared and mounted in the clamp separately outside the DMA module, for example while a different sample mounted in another clamp is being measured.

When you have mounted the sample in the clamp, the clamp can be quickly installed and the next experiment started.

The sections *Mounting a sample in the tension clamp* and *Mounting a sample in the compression clamp* explain how to mount the sample in the tension and compression clamps.

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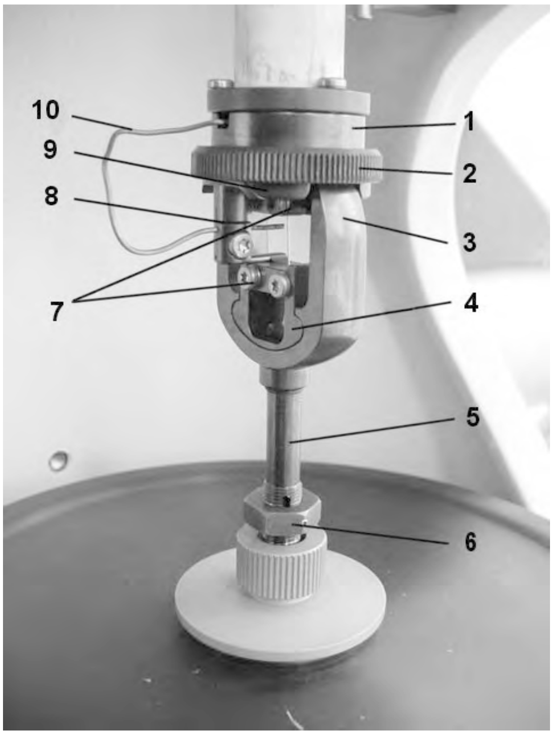
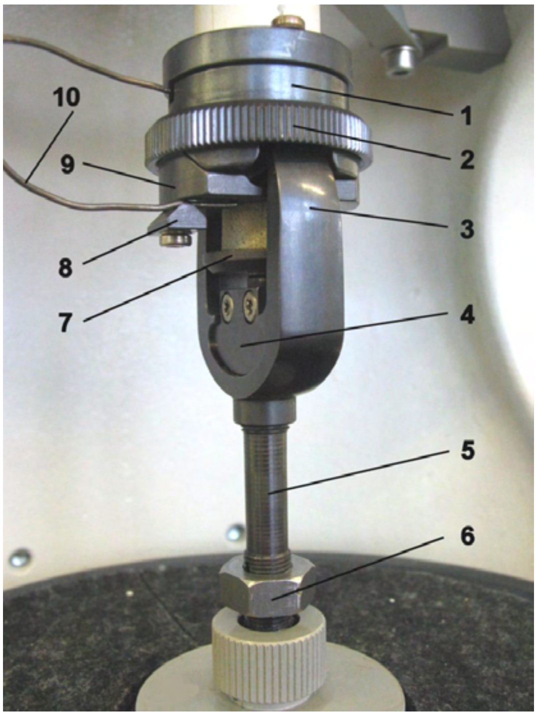
Fig. 6-17 shows the tension clamp installed in the large clamping assembly with a film sample.



1 Clamp holder, fixed part
2 Clamping ring
3 Clamp holder, movable part
4 Movable part of tension clamp
5 Drive shaft
6 Hexagonal nut
7 Clamping plates
8 SDTA thermocouple holder
9 Fixed part of tension clamp
10 SDTA thermocouple

Fig. 6-17. Tension clamp installed in large clamping assembly

Fig. 6-18 shows the compression clamp installed in the large clamping assembly with a compression sample.



1 Clamp holder, fixed part
2 Clamping ring
3 Clamp holder, movable part
4 Movable part of compression clamp
5 Drive shaft
6 Hexagonal nut
7 Sample support
8 SDTA thermocouple holder
9 Fixed part of tension clamp
10 SDTA thermocouple

Fig. 6-18. Compression clamp installed in large clamping assembly

Sample mounting aids

To remove the clamps from or install them in the clamping assembly, the parts of the clamps must be firmly fixed to the sample mounting aid supplied.

The sample mounting aid therefore serves a similar purpose as the guide pins of the small clamps. The sample mounting aids are also used to mount the samples in the clamps. The samples are mounted in the clamps in a separate step before installing the clamps in the large clamping assembly

There are suitable sample mounting aids for both the tension and the compression clamps.

Fig. 6-19 and Fig. 6-20 show the tension and the compression clamps in the mounting aids with samples ready to install in the large clamping assembly.

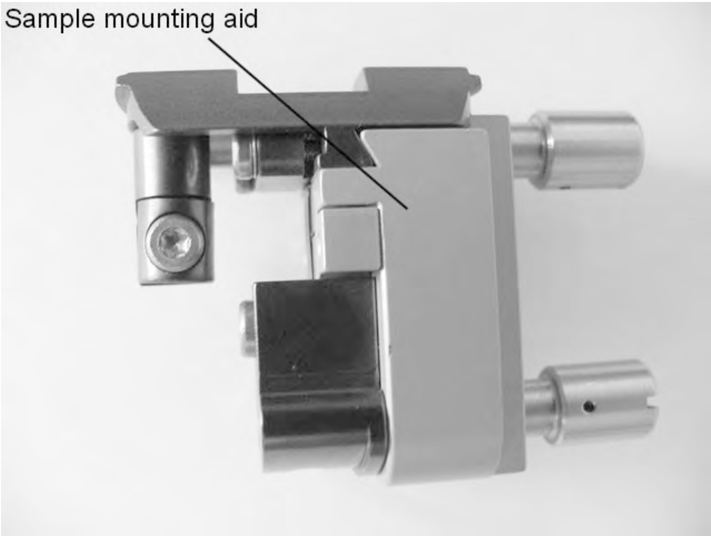


Fig. 6-19. Tension clamp with sample mounting aid

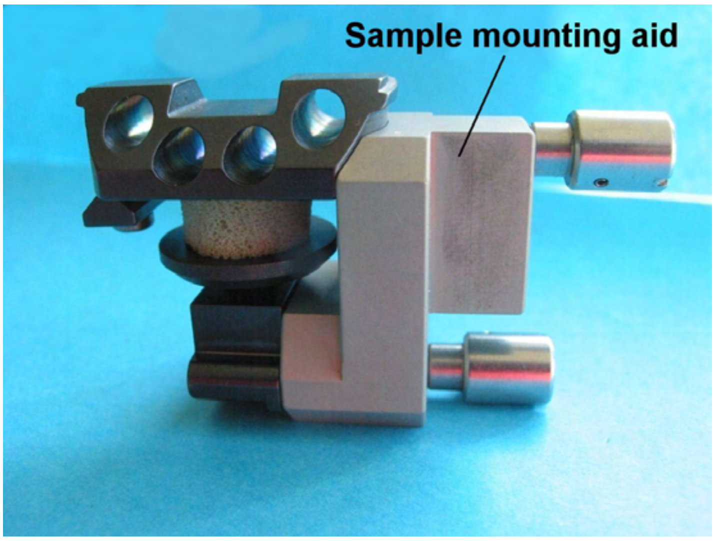


Fig. 6-20. Compression clamp with sample mounting aid

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Mounting a sample in the tension clamp

Before you can mount the sample in the tension clamp, you may have to remove an old sample that is still mounted in the clamp. This step is explained first in the following section. You can skip this step if the sample has already been removed.

To remove the previous sample:

➪ *See page 6-83, Fig. 6-17*

Start:
- Previous sample mounted in tension clamp
- Tension clamp is fixed in the sample mounting aid

(1) Mount the tension clamp fixed in the sample mounting aid in a vise so that the ends of the support pins point downward and the sample is easily accessible from above.

(2) If necessary remove the SDTA thermocouple holder: Loosen the Torx screw that fixes the holder of the SDTA thermocouple to the tension clamp and remove the holder, ❶.

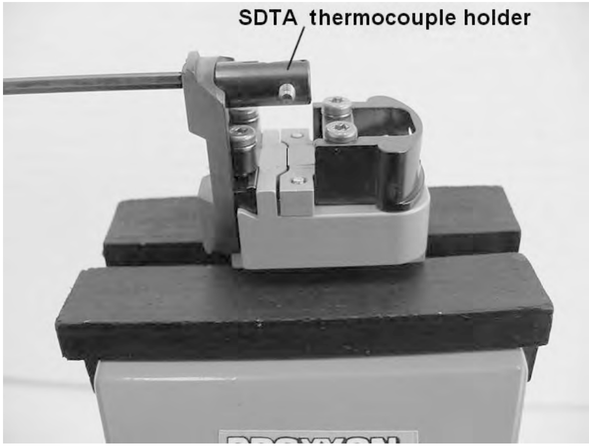
(3) Remove the sample as follows:

- Loosen the Torx screws on both clamping plates **7** of the tension clamp with the Torx screwdriver. Completely remove both clamping plates, ❷, if necessary.
- Remove the sample by carefully pulling it out with tweezers, ❸.

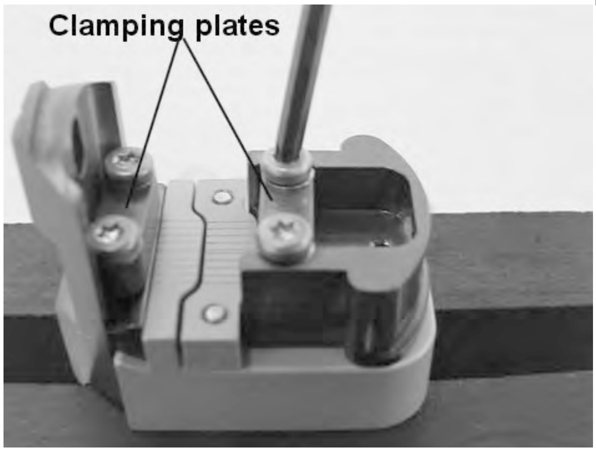
(4) Remove the tension clamp together with the sample mounting aid from the vise.

♣ If you no longer require the tension clamp for measurements, you can loosen the support pins and remove the fixed and movable parts of the tension clamp from the sample mounting aid, ❹.

End: Sample is removed from the tension clamp

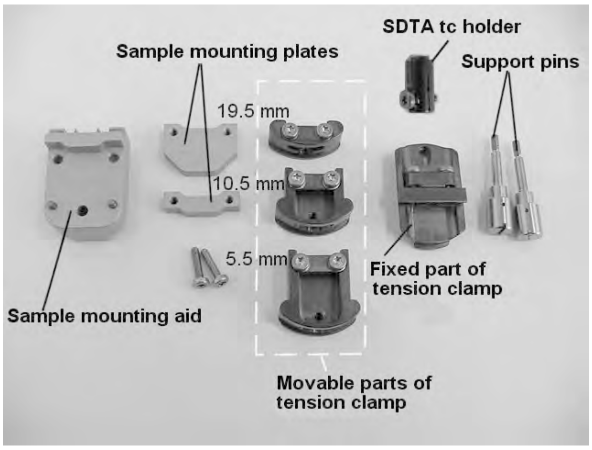

SDTA thermocouple holder

❶


Clamping plates

❷

❸


SDTA tc holder
Sample mounting plates
Support pins
19.5 mm
10.5 mm
5.5 mm
Fixed part of
tension clamp
Sample mounting aid
Movable parts of
tension clamp

❹

LCA

To mount a new sample in the tension clamp:

➪ *See page 6-83, Fig. 6-17*

Start:
- Tension clamp removed from large clamping assembly
- Previous sample removed from the tension clamp
- New sample prepared and ready for mounting
- Tension clamp disassembled

(1) Adapt the tension clamp to the length of your sample. Select the appropriate sample mounting plate. For sample lengths of 10.5 mm and 19.5 mm there are suitable sample mounting plates. For a sample length of 5.5 mm you do not need a sample mounting plate, ❶.

(2) If a sample mounting plate is already in the tension clamp, remove it by loosening the Torx screws and removing the sample mounting plate.

(3) Place the appropriate sample mounting plate in the sample mounting aid, ❷. Lead the Torx screws from the back (the side of the support pins) into the sample mounting aid and tighten them to secure the sample mounting aid.

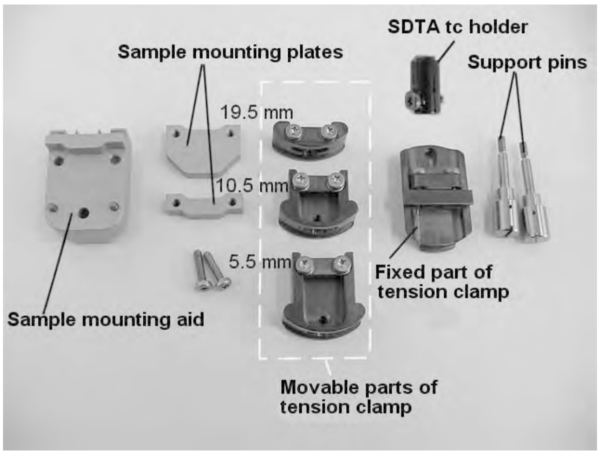
(4) Insert the movable part **4** and the fixed part **9** of the tension clamp and then screw them tight with the corresponding support pin, ❸.

(5) Mount the tension clamp fixed in the sample mounting aid in a vise so that the ends of the support pins point downward and you can insert the sample easily from above.

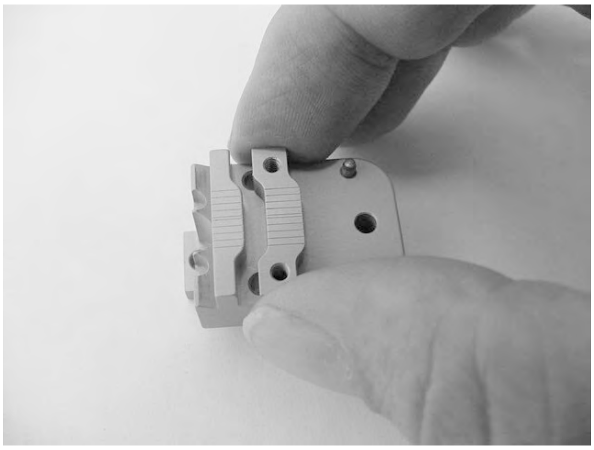
(6) If necessary, remove the SDTA thermocouple holder. Loosen the Torx screw that fixes the holder of the SDTA thermocouple to the tension clamp and remove the holder, ❹.

(7) Loosen the Torx screws on both clamping plates **7** of the tension clamp with the Torx screwdriver and completely remove both clamping plates. Make sure that you do not lose the spring washers belonging to the four Torx screws, ❺.

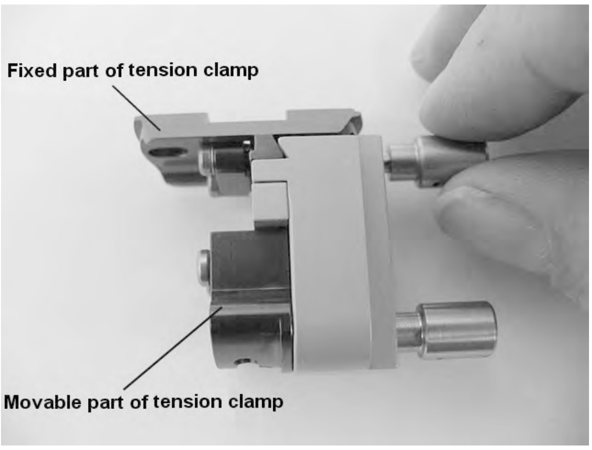
(8) Make sure the Torx screws are suitable for the thickness of your sample. The supplied short or long torx screws can be used, depending on the thickness of the sample. If necessary, exchange the screws.


SDTA tc holder
Sample mounting plates
Support pins
19.5 mm
10.5 mm
5.5 mm
Fixed part of tension clamp
Sample mounting aid
Movable parts of tension clamp

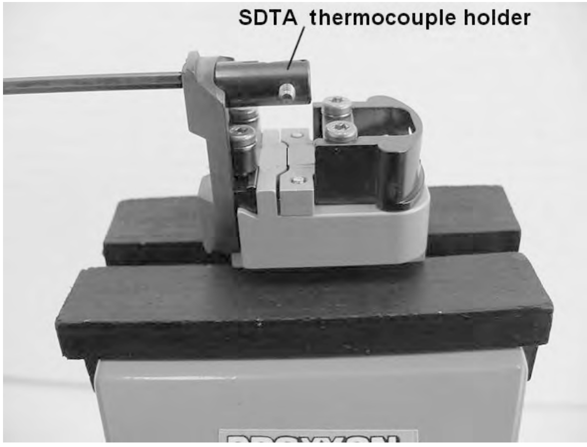
❶



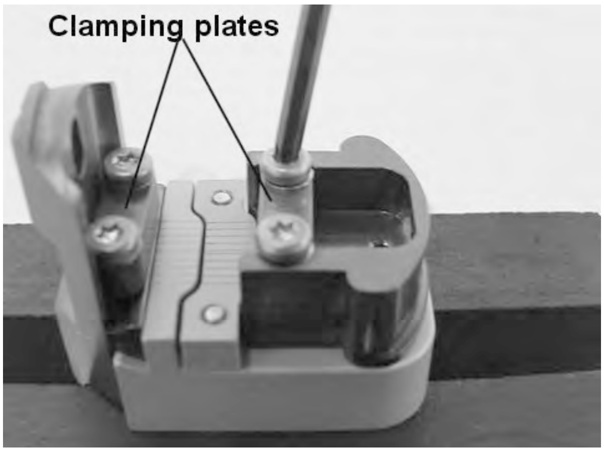
❷


Fixed part of tension clamp
Movable part of tension clamp

❸


SDTA thermocouple holder

❹


Clamping plates

❺

(9) Mount the sample as follows:

With films and bars:

- Place the sample on the surface of the sample mounting plate and position parallel to the guide lines, **❻-a** and **❼-a**.
- Insert both clamping plates carefully into the tension clamp again. Make sure you do not move the sample. Fix the sample by slightly tightening the four Torx screws, **❻-b** and **❼-b**.

♣ The flat side of clamping plate must be pointing upward.

With fibers:

- Place the sample on the surface of the sample mounting plate and position its ends over the guide notches on the fixed and movable parts of the tension clamp. Hold the sample in this position with one hand so that it is under slight tension and not buckled, **❽-a**.
- Insert both clamping plates with the other hand and slightly tighten the four Torx screws. Check whether the sample is between the two middle guide lines, **❽-b**.

♣ The flat side of clamping plate must be pointing upward.

- If necessary, loosen one of the clamping plates and reposition the sample. Re-tighten the two Torx screws slightly.

♣ The amount of torque set to fasten the clamping plates depends very much on the type of sample you are measuring. The clamping plates must fixed in place to prevent the sample from slipping during the measurement. But at the same time they must be loose enough to avoid excessive clamping pressure that would affect the measurement or even damage the sample. As a rough guideline, we recommend a torque value of about 0.5 Nm.

(10) Re-fit the SDTA thermocouple holder **8**, by inserting the end in the oval hole in the fixed part of the tension clamp, inserting the Torx screw from the other side and tightening it securely. Make sure when installing the tension clamp in the clamping assembly that the Torx screw on the holder points forward and is easily accessible.

End:
- Sample mounted in the tension clamp.
- Tension clamp ready to be installed in the clamping assembly

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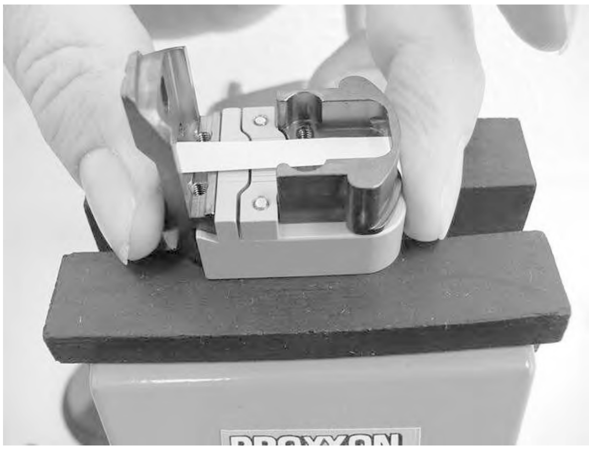
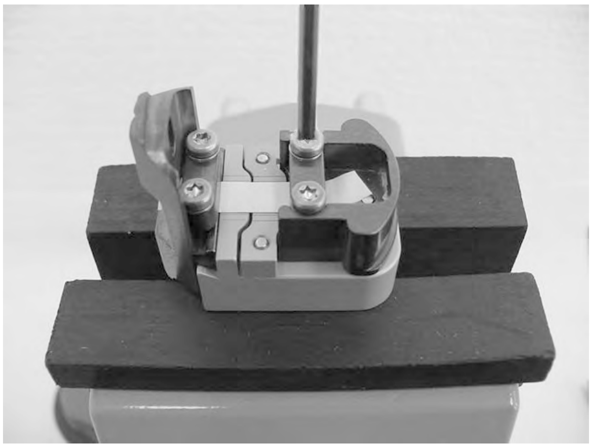
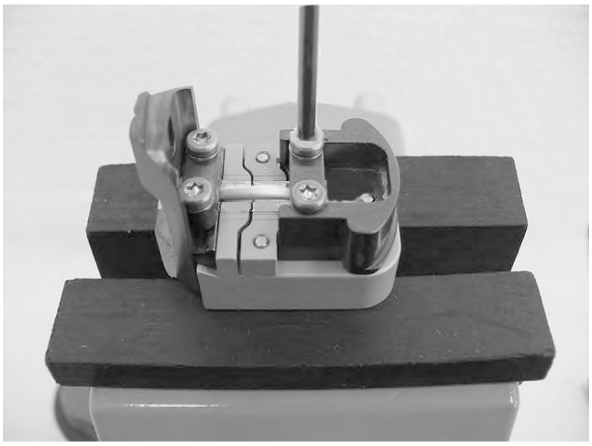




Films ❻-a ❻-b



Bars ❼-a ❼-b

Fibers ❽-a ❽-b

LCA

Mounting a sample in the compression clamp

Before you mount a sample in the compression clamp, you must remove any previous sample that is still mounted. This step is described in the following section. You can skip this if the sample has already been removed.

To remove a previous sample and disassemble the compression clamp:

➪ *See page 6-85, Fig. 6-18*

Start: Compression clamp removed from the large clamping assembly.

(1) Loosen the two Torx screws **1** in the movable part **2** of the compression clamp and move the sample support **3** a little to the rear.

(2) Remove the previous sample by pulling or pushing it out from the side.

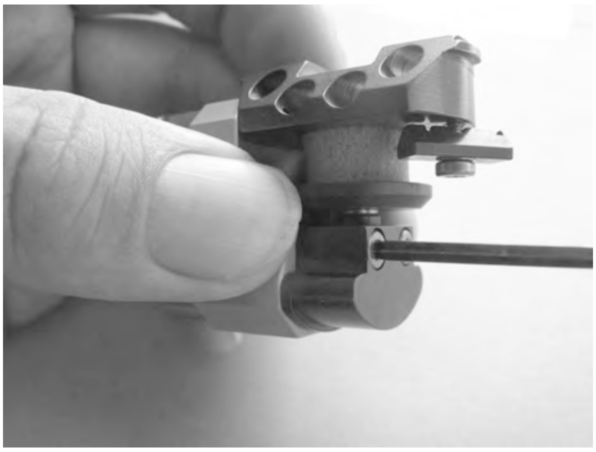
(3) Screw out the two support pins **6**, ➊.

(4) First remove the fixed part **1** of the compression clamp by pilling it slowly from the sample mounting aid. The remove the movable part **2** of the compression clamp in the same way.

(5) Clean the surface of the sample support **3** and the contact surface of the fixed part **1** of the compression clamp from sample residues. Use suitable cleaning agents or if necessary an ultrasonic bath. The surfaces are very precisely machined. Make sure you do not damage them.

♣ To remove stubborn residues, we recommend that you bake out the sample supports and the end disk in a muffle furnace at 400 °C for half an hour.

End:
- Compression clamp disassembled in its parts
- Previous sample is removed
- Surfaces of the sample support and the movable part are clean



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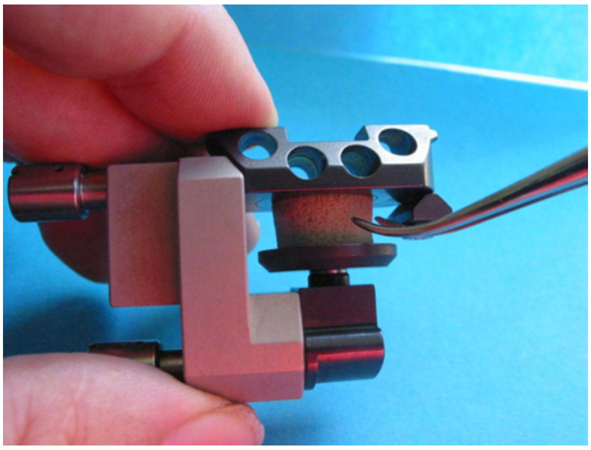
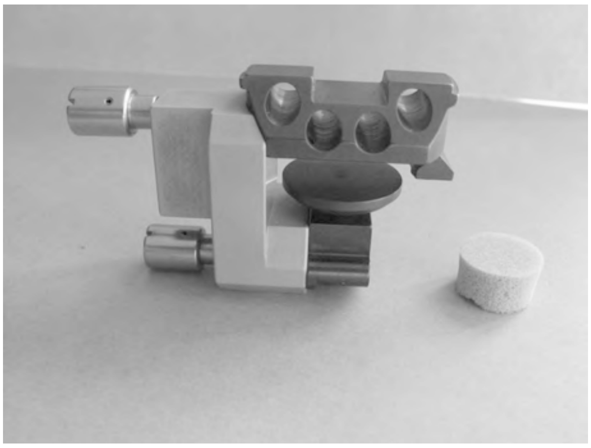
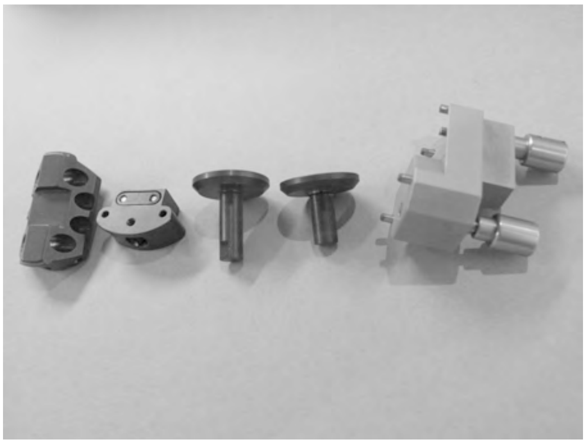


❷



❸



❹

To mount a new sample in the compression clamp:

➩ *See page 6-85, Fig. 6-18*

Start:
- Compression clamp removed from the clamping assembly
- Previous sample removed from the compression clamp
- Compression clamp disassembled in its parts

(1) Use a suitable sample support that corresponds to the size of the sample in the movable part of the compression clamp, ❶, ❷.

♣ Two sample supports of different length are supplied with the compression clamp.

(2) Fix the movable part of the compression clamp to the sample mounting aid by pushing it onto the pins and tightening the support pin, ❸. Fix the fixed part in the same way, ❹.

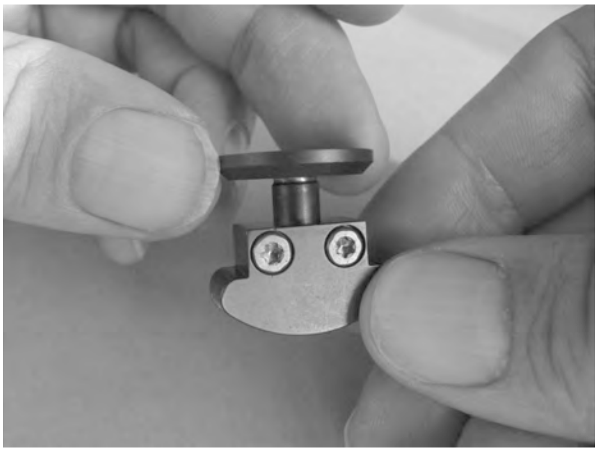
(3) Mount the sample as follows:

- If necessary move the sample support slightly downward.
- Mount the sample. Use the circular laser markings on the contact surface of the movable part to position the sample, ❺.
- Make sure that no air gap occurs between the contact surfaces of the sample. An air gap indicates that the sample is not in proper contact with the surfaces, which can lead to measurement errors.

(4) Fix the sample support by tightening both Torx screws on the movable part of the compression clamp, ❻.

End:
- Sample is mounted in the compression clamp
- Compression clamp is ready for installation in the clamping assembly

❶



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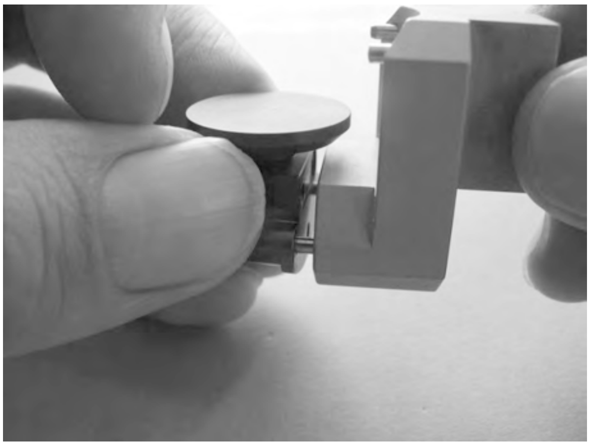

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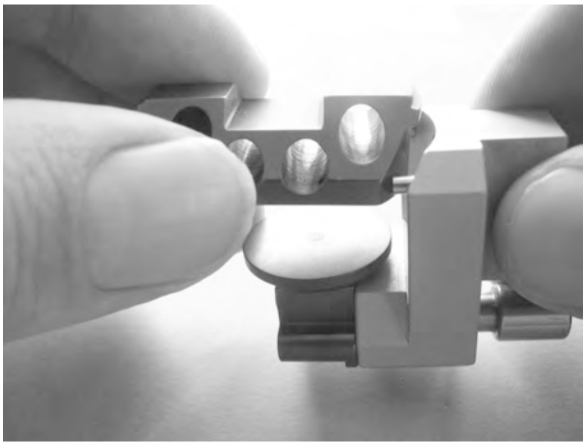

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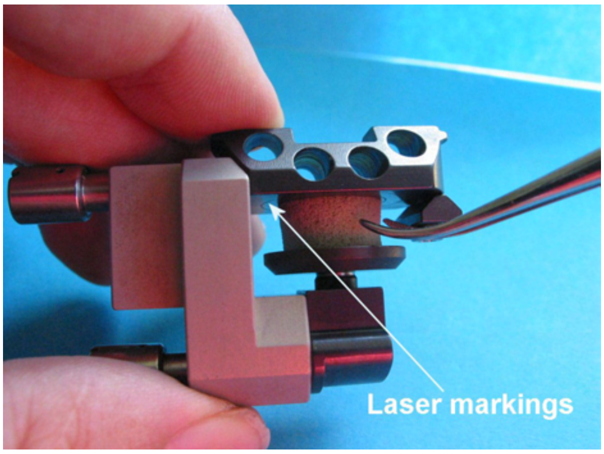

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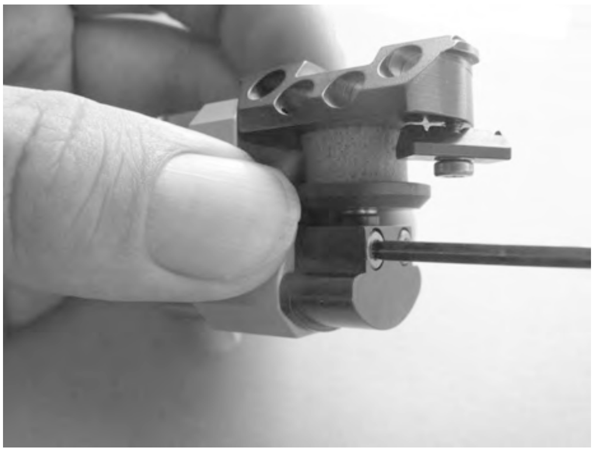


❻

LCA

Installing and removing the tension or compression clamp with sample

The following procedures explain how to install and remove the tension or the compression clamp with the sample.

To install the tension clamp with the sample:

➩ *See page 6-85, Fig. 6-17 and Fig. 6-18*

Start:
- Large clamping assembly without clamp installed
- Fine alignment and displacement adjustment performed
- Tension clamp with sample and sample mounting aid mounted ready for installation (see Fig. 6-19)
- Furnace halves driven fully apart and swung to the rear

(1) Make sure that the clamping ring **2** of the fixed part of the clamp holder **1** is loosened.

(2) Make sure that the hexagonal nut **6** on the drive shaft **5** is screwed fully down so that the stem connected with it is sunk in the clamp holder, ❶.

(3) Push the tension clamp fixed in the sample mounting aid into the movable part of the clamp holder **3** from the right side as follows:

- Press the beveled side of the fixed part of the tension clamp against the inner side of the fixed part of the clamp holder **1**, ❷.
- At the same time fit the movable part of the tension clamp **4** into the shoulder-shaped edge of the clamp holder **3**. The movable part of the tension clamp has a rim that lies flush on the shoulder-shaped edge, ❸.

(4) Place the fixed part of the clamp holder **1** on the tension clamp **9** so that its beveled cutout sits snugly on the beveled side of the tension clamp.

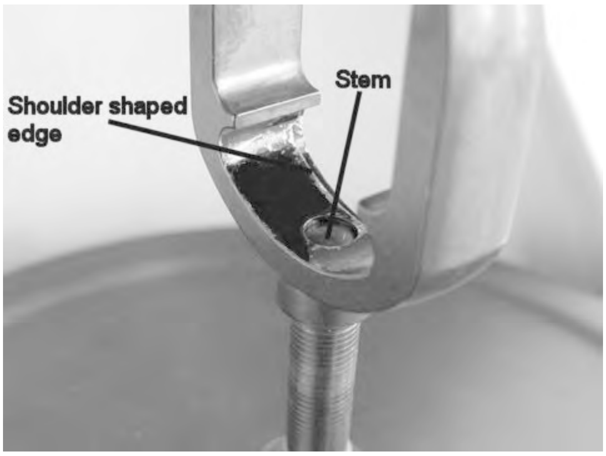
(5) Screw the clamping ring **2** so that it is hand-tight.

The fixed part of the tension clamp **9** is fixed.

(6) Tighten the hexagonal nut **6**, with the supplied Torx wrench, ❹, setting a torque of about 0.5 Nm. Hold the movable part of the clamp holder tightly so that it cannot turn.

The movable part of the tension clamp is fixed.

(7) Loosen support pins completely and pull the sample mounting aid away carefully from the side, ❺.



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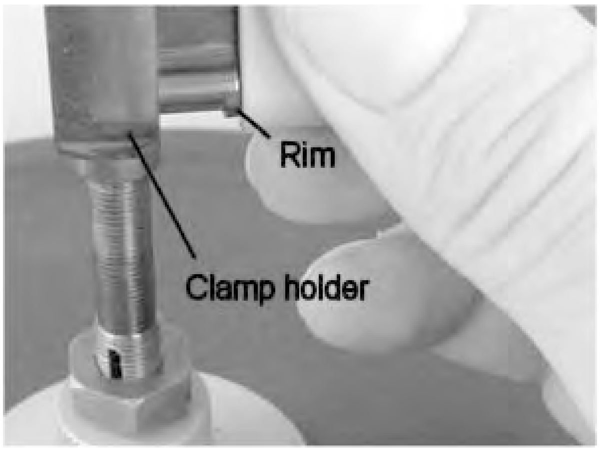
❷



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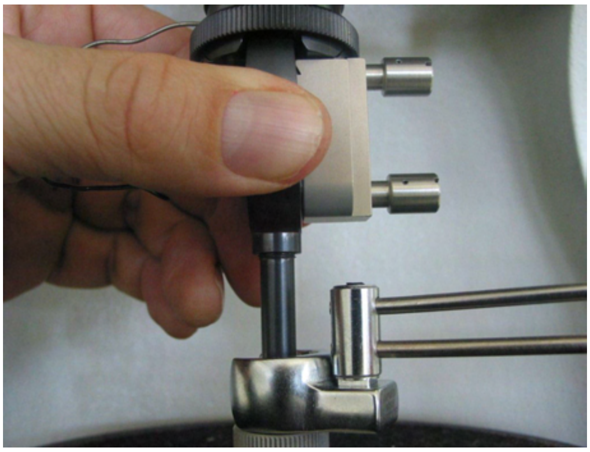


❹

❺

LCA

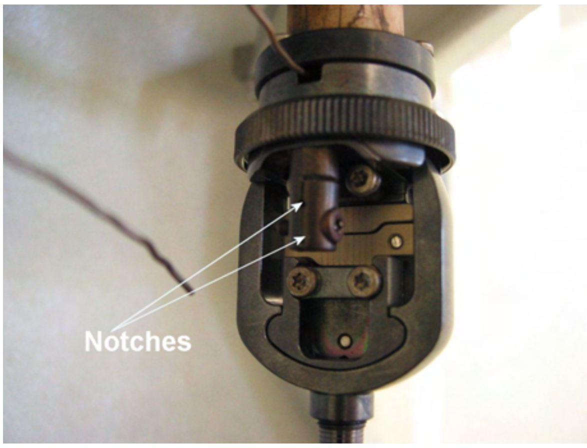
(8) Fix the SDTA thermocouple **10** in position as follows:

- Bend the thermocouple carefully to position its tip as close as possible to the sample without making direct contact, ❻-a, ❻-b. Make sure the thermocouple does not kink.
- Clamp the thermocouple tightly in the SDTA holder. There are two notches fix the thermocouple, **❼-a**, **❼-b**

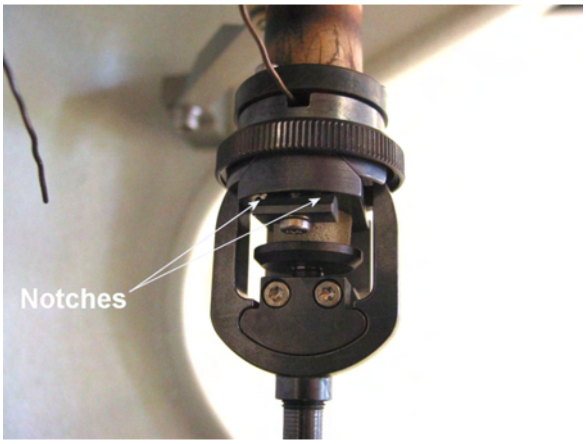
End: Clamp with sample is installed in the large clamping assembly

❻-a

❻-b



❼-a



❼-b

To remove the tension or compression clamp:

⇨ See page 6-85, Fig. 6-17 and Fig. 6-18

Start:
- Tension clamp installed in clamping assembly
- Furnace arms fully open and swung to the rear

(1) Loosen the clamping screw for the SDTA thermocouple **10** in the SDTA thermocouple holder **8**.

(2) Pull the SDTA thermocouple out of the holder.

(3) Fix the sample mounting aid on to the tension clamp. Insert the support pins in the holes of the sample mounting aid and screw them finger tight, ❶.

(4) Loosen the clamping ring **2**.

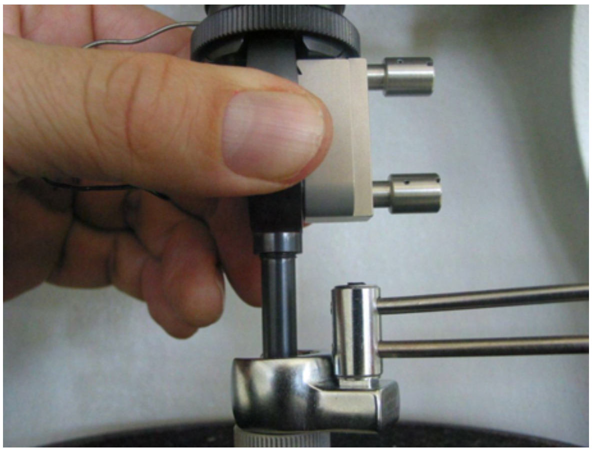
(5) Loosen the hexagonal nut **6** on the drive shaft **5** with the supplied Torx wrench, ❷.

Never use excessive force to move the clamp. Exerting excessive force on the clamp could damage the force sensor, the displacement sensor or the drive motor.

(6) Pull the tension clamp carefully sideways out of the clamp holder, ❸.

End: Tension clamp removed from large clamping assembly.

❶



❷

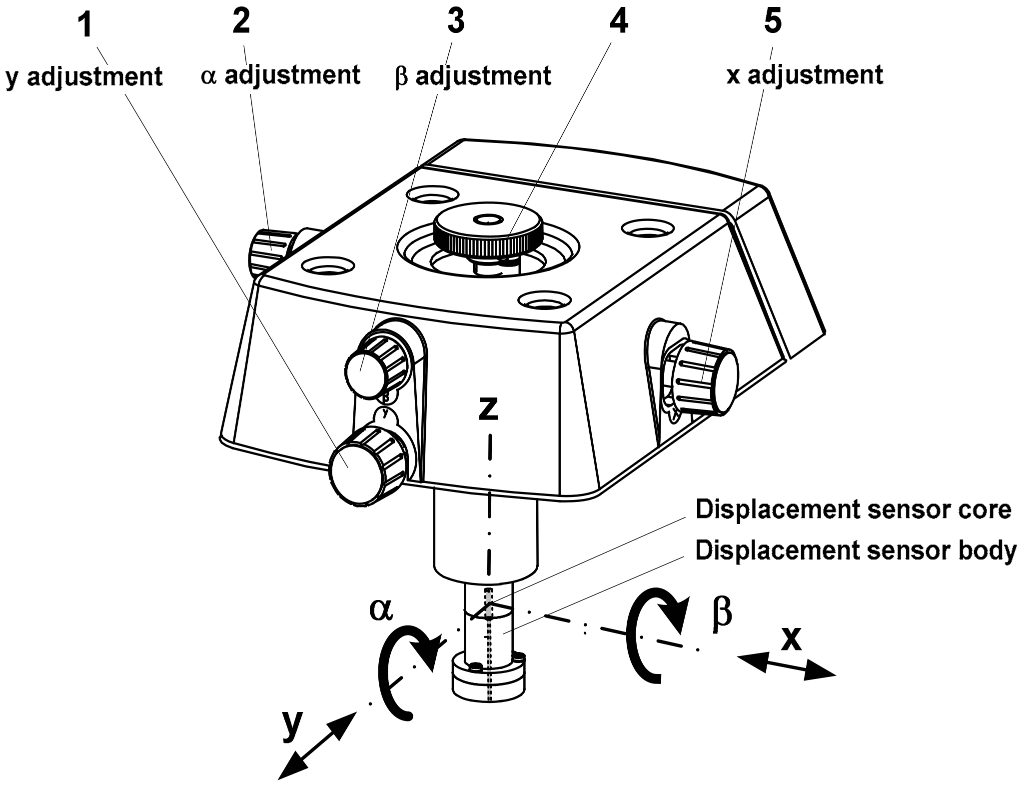
❸

6.6 Fine alignment of the measuring sensors (four axes alignment)

To measure properly, you must follow a special procedure to perform fine alignment of the measuring system after installation of the clamping assembly.

By rotating the knobs on the four axes alignment device you can position the displacement sensor core within its coil.

Fig. 6-21 shows the function of the knobs on the four axes alignment device:



1 Knob for adjusting the y -position

2 Knob for adjusting the α angle

3 Knob for adjusting the β angle

4 Locking screw

5 Knob for adjusting the x position

Fig. 6-21. Knobs on the four axes alignment device

The following diagram shows the procedure for fine alignment of the measuring sensors:

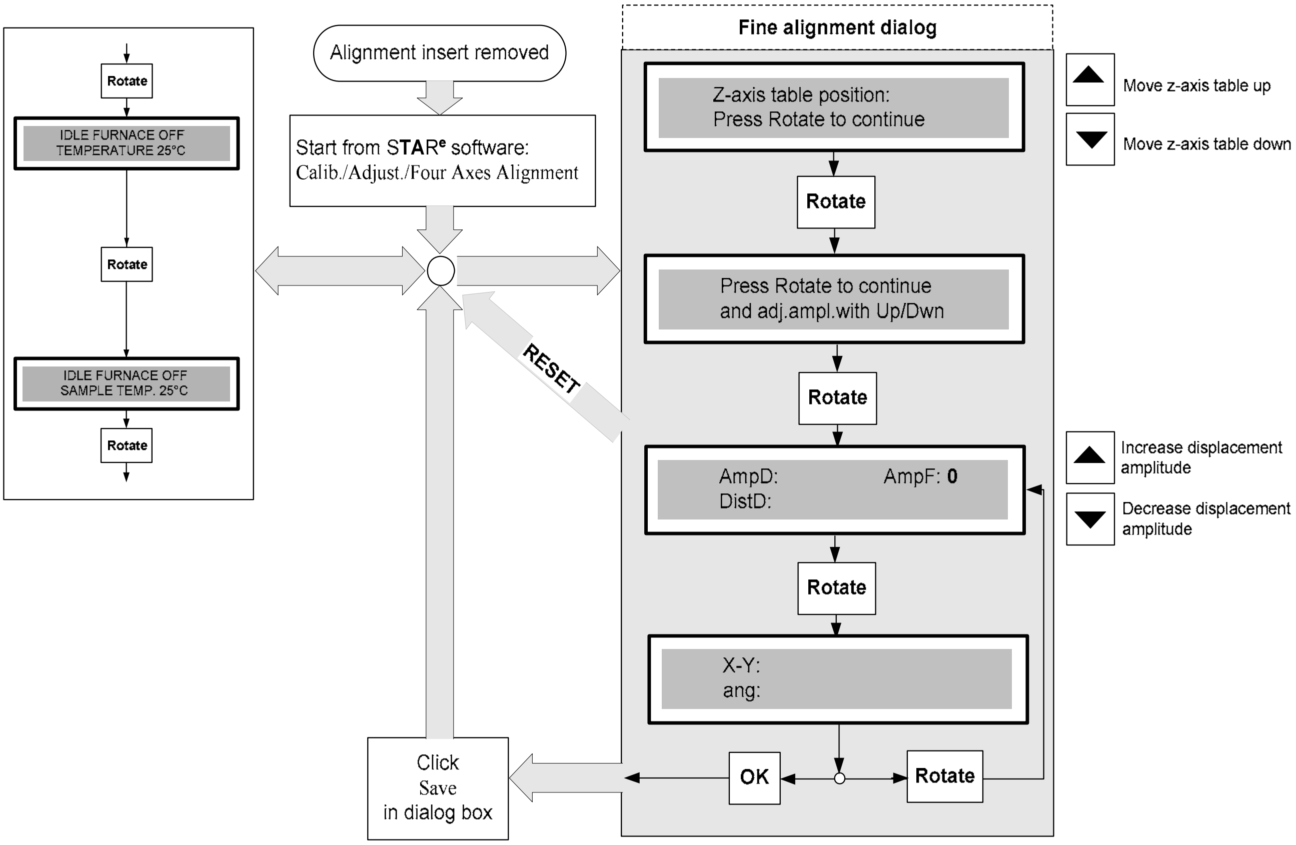


Fig. 6-22. Procedure for fine alignment of the measuring sensors

First the x position is adjusted, then, if necessary, the y position.

The α and β angles have already been adjusted during pre-alignment with the alignment aid and need not be changed.

To perform fine alignment of the measuring sensors:

⇨ *See page 6-102, Fig. 6-21*

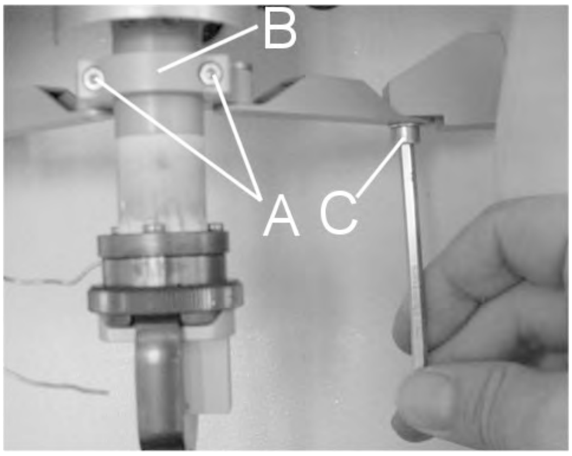
Start:
- Furnace arms moved apart
- Clamping assembly installed

(1) If you have previously not done so, loosen the support structure of the measuring sensors as follows:

- Remove the Torx screws (A) with the Torx wrench and remove the front clamp (B). Loosen the Allen screws (C) with a 4-mm Allen wrench, ❶, but do not completely remove them so that the support structure is still attached.
- Fold the support structure back so that it is no longer in contact with the displacement sensor body, ❷.

(2) Loosen the locking screw of the four axes alignment device, ❸, then slightly tighten it again to the point where you feel slight resistance.

♣ The mechanism of the 4-axes alignment device operates best when it is pressed together slightly by the locking screw.



❶

❷

❸

(3) In the STAR^e software Module Control Window, start the dialog for the four axes alignment procedure by choosing Calib./Adjust./Four Axes Alignment Device.

The current value of the Offset Position of the z-axis table (**z-axis table position**) is shown on the LCD. This value represents the current position of the z-axis table and can now be changed using the arrow key.

♣ The value for the z-axis table is only meaningful if you have fully adjusted your DMA module as shown in Chapter 8, *Calibration and adjustment*.

(4) If the position of the z-axis table deviates more than 50 µm from the zero position: Move the z-axis table into the zero position by pressing the MOTOR UP and MOTOR DOWN keys. The zero position is sufficiently accurate when a value between –50 µm and +50 µm is displayed.

The z-axis table is now in the zero position.

♣ The z-axis table automatically moves into the current zero position at the end of a displacement adjustment procedure.

(5) Press ROTATE to continue.

The following message appears:

Press Rotate to continue and adj.ampl.with Up/Dwn

or:

Press RESET, to return to quit the dialog. You can always quit the dialog by pressing the RESET key from this point onward without saving the alignment settings.

♣ The MOTOR UP and MOTOR DOWN arrow keys are multi-function keys. From this point onward in the dialog they serve to increase or decrease the displacement amplitude of the drive motor.

(6) Press ROTATE to proceed with the adjustment of the four axes alignment device.

The current settings of the measuring system appear on the LCD. The drive motor produces a default oscillation with an amplitude of about 23 µm and 2 Hz.

Quality factor	Definition	Adjust to
AmpD	Current value of the displacement amplitude	1500 µm
AmpF	Current value of the force amplitude	0 (zero) mN
DisD	Current value of the distortion factor	Minimum value

♣ If the mechanical pre-adjustment has been done well, the value of the force amplitude should be 0 (zero) mN.

⇨ *See page 6-102, Fig. 6-21*

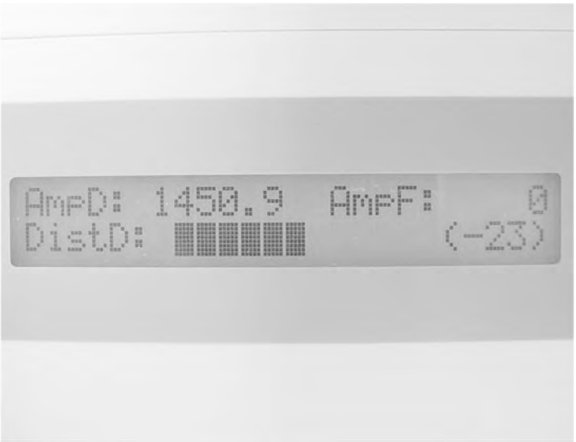
(7) Increase the displacement amplitude in steps to about 1500 µm by pressing the ▲ key. Take note of the force amplitude display. If the force amplitude becomes greater than 0 (zero) mN:

adjust x and y position

- Adjust the x-position of the displacement sensor by rotating knob **5** until the force amplitude returns to 0 (zero) mN, ❹**-a**. Try rotating the knob in the other direction. If the force amplitude does not decrease, adjust the y-position by rotating knob **1** in the same way, ❹**-b**.
- In this way, increase the displacement amplitude in steps to about 1500 µm. This value corresponds to the maximum displacement of the displacement sensor core, ❺.

❹**-a**

❹**-b**



❺

❻

♣ When you press the ▲ and ▼ keys, the value of the force amplitude changes each time by a factor of 1.3. It is therefore difficult to set an exact value. For the adjustment, however, it is sufficient to set a value close to the desired value.

(8) If the force amplitude is not 0 (zero) mN: Press the RESET key to terminate the alignment procedure. Repeat the mechanical pre-adjustment. We recommend that you remove and reinstall the adjustment assembly for this (see the section *Installing and removing the small clamping assembly* or *Installing and removing the large clamping assembly*) and repeat the fine alignment procedure described in this section

The force amplitude is 0 (zero) mN at the maximum displacement.

(9) For the small clamping assembly: Readjust the y-position of the measuring sensors, if necessary, as follows:

adjust y-position

- Drive the displacement amplitude back to the lowest value by pressing the ▼ key.
- Slide the alignment insert carefully from the side into the clamp holder, ❻. If the alignment insert cannot be inserted or if it is difficult to insert, adjust the y-position slightly with the y-position knob **1** until the alignment insert can be easily inserted, **❹-b**. Then remove the alignment insert again.
- Increase the displacement amplitude again by pressing the ▲ key to about 1500 µm. Note the force amplitude display.
- If the force amplitude is 0 (zero) mN: Drive the displacement amplitude back to the lowest value by pressing the ▼ key.
- If you cannot bring the force amplitude to 0 (zero) mN: press the RESET key to terminate the alignment procedure. Repeat the mechanical pre-adjustment. You must remove and reinstall the clamping assembly for this (see the sections *Installing and removing the small clamping assembly* or *Installing and removing the large clamping assembly*) and the fine adjustment procedure for the four axes alignment device described in this section.

(10) Press OK to quit the four axes alignment procedure.

(11) In the STARe software Module Control Window, click Save to save the current adjustment data of the four axes alignment device. Then start the dialog for the four axes alignment procedure again by clicking Calib./Adjust./Four Axes Alignment Device.

(12) Press ROTATE twice on the DMA module keypad to display the values of DistD again.

The value of the distortion factor DistD should be as small as possible and always less than 30 dB.

(13) Then press OK to terminate the four axes alignment procedure.

The measurement sensors have now been aligned and the dialog returns to the starting point and displays **Idle furnace off**. A dialog box with the new adjustment data appears in the Module Control Window of the STARe software. The data for the previous adjustment are given in brackets.

(14) In the STARe software Module Control Window, click Save to save and activate the current adjustment data of the four axes alignment device.

(15) Fasten the locking screw **4** first, ❼, and then fasten the support structure as follows:

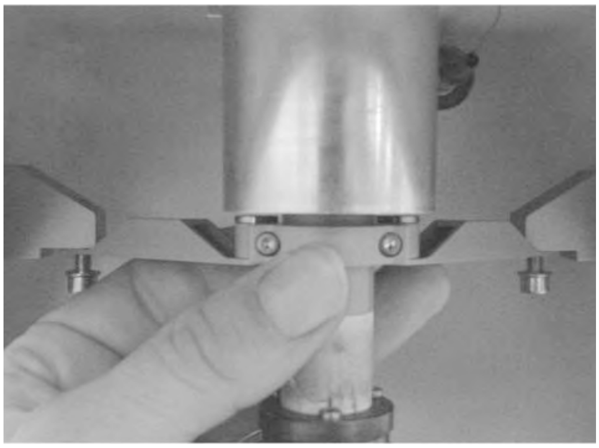
- Screw in the two Torx screws (A) and then attach the clamp (B) while holding the support structure at the same level as the two cantilevers on the stand, ❽ and ❾ (see next page).
- Fasten the Allen screws (C) with the 4-mm Allen wrench, ❾.

(16) Make a final check of the alignment as follows:

- In the STARe software Module Control Window, start the dialog of the four axes alignment device again by choosing Calib./Adjust./Four Axes Alignment Device.
- Press ROTATE twice to display the settings of the measuring system and increase the displacement amplitude in steps to about 1500 µm by pressing the ▲ key.
- Note the force amplitude display:
  - If the force amplitude is 0 (zero) mN, press RESET to quit the for axes alignment dialog without saving the adjustment data.
  - If the force amplitude is greater than 0 (zero) mN, repeat the alignment procedure with step (1).

End: Fine alignment completed. Measuring sensors aligned with the clamping or adjustment assembly.

❼



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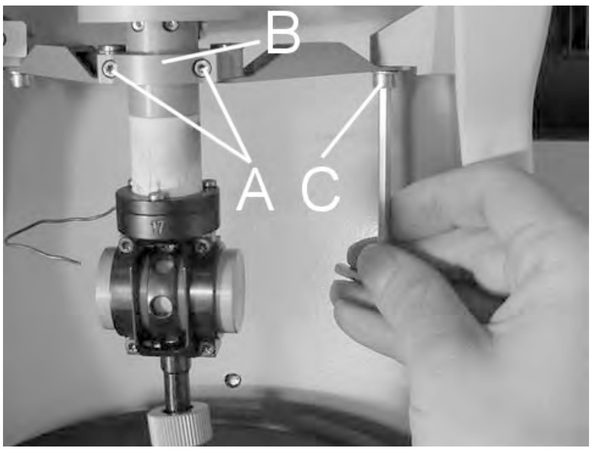


❾

Adjustment of the x and y position knobs for high demands on accuracy

If the pre-adjustment is good, you should achieve good alignment of the measuring sensors by following the above adjustment procedure. If you have higher demands on measurement accuracy, however, we recommend that you perform an additional adjustment with the x and y position knobs at the beginning of step (7) as follows.

- Rotate the knob in one direction until a low value for the force (less than 10 µN) appears on the LCD. Note this position of the knob as position 1.
- Rotate the knob in the opposite direction until the value for the force on the LCD is 0 (zero) mN. Note the position of the knob at which the force signal is 0 (zero) mN by its angle as position 2. The angle between position 1 and position 2 is the mechanical play of the knob.
- Rotate the knob further in the same direction until a low value for the force (less than 10 µN) appears on the LCD again. Note this position of the knob by its angle as position 3 of the knob.
- Determine the middle position of the knob between position 2 and position 3. Rotate the knob to the middle position.

  The displacement sensor has now been adjusted.

♣ There is a certain amount of play in the movement of the knob. The procedure described above, in which the knob is rotated backward and forward, eliminates this play.

Problems that may occur while aligning the measuring sensors

Force value is not 0 (zero) mN

If you do not succeed in aligning the measuring sensors by the above procedure, the reason for this might be due to one of the following causes:

- The pre-alignment on installing the clamping assembly was not performed correctly at first.
- The X and Y positions or the α and β angles have been changed by accident.
- Some of the screws were not tightened enough during the installation of the clamping assembly.
- The clamping assembly was not properly aligned within itself at first.

To solve the problem you must re-align the clamping assembly within itself (see the sections *Aligning the large clamping assembly within itself* or *Aligning the small clamping assembly within itself*) and also re-align the measuring sensors. To do this you must remove and reinstall the clamping assembly (see the sections *Installing and removing the small clamping assembly* or *Installing and removing the large clamping assembly*) and repeat the procedure for the fine alignment of the measuring sensors described above.

Message **Risk of damage!** appears on the LCD

If the force sensor measures an unallowed value, the error message **Risk of damage!** appears on the LCD. You can however acknowledge the message by pressing the OK key on the keypad of the module and continue with the four axes alignment. The third time the message appears, however, it is no longer possible to acknowledge the message. In this case, you have to switch off the DMA module using the mains switch at the rear of the instrument and restart it. See also Chapter 10*, Error messages and warnings*.

7 Performing an Experiment

Contents

7 Performing an Experiment

This chapter explains how you perform a measurement with the DMA/SDTA861^{e} module. We show you what preparation to make before you start the experiment, how to enter your experiment in the Experiment Window of the S**TA**R^{e} Software and how to start the measurement on the DMA/SDTA861^{e}.

Further information and instructions on how to prepare a measurement can be found in Chapter 6, *Preparing for the measurement.*

Never remain in close proximity of the DMA module during a measurement without hearing protection. Always wear hearing protection when working near the DMA module. The DMA module can produce excessive noise that can impair your hearing. The noise level can exceed 85 dB(A) during certain measurements.

7.1 Preparations before you setup an experiment

Before you setup an experiment and start it on the DMA module it is important to check that all the devices connected to the DMA module are set up properly and the settings in the S**TA**R^{e} Software are made correctly.

We strongly recommend that you take note of the points explained in the following sections and perform the checks described to make sure that your DMA module is set up properly for the experiment.

DMA experiments can take considerably longer than experiments on other TA modules. We recommend that you take some time to check the measurement set up properly before the experiment to avoid having to repeat the measurement because of erroneous data.

7.1.1 Notes on creating DMA methods in the Method Window

To define a DMA experiment you must first of all create a DMA method in the S**TA**R**e** Software Method Window. The instructions for doing this can be found in the online Help to the Method Window in the S**TA**R**e** Software.

DMA oscillation

amplitude control

The sinusoidal oscillation in a DMA experiment is realized either by the displacement or force amplitude control defined in the method.

This is done in the different dialog boxes that can be called up under DMA on the Segment menu. The force and displacement amplitude settings can be defined, the temperature program entered and type of offset control selected.

In a DMA temperature segment, a temperature program can be defined in parallel to the DMA oscillation which consists of up to 10 frequencies. It is also possible to overlay oscillation frequencies (multi-frequency mode). Up to 4 overlaying frequencies are possible.

Preliminary checks

preliminary clamping check

You can define a pre-measurement DMA oscillation immediately before an experiment to make a preliminary check of the clamping. The check is performed at room temperature and at start temperature. If necessary you can reclamp the sample during the oscillation at this point. See also section *Preliminary stages before the measurement.*

Offset control

Offset control of displacement or force can be defined for the measurement modes. The type of offset control and amount of offset used depends very much on the measuring mode and the sample itself. As general rule of thumb large offset values are used for elastic samples and small offset values are used for hard samples.

The following types of offset control are implemented:

types of offset control

- Zero Displacement: The dimensions of the clamping assembly and drive shaft change slightly with temperature. This deformation is automatically compensated during the experiment by repositioning of the z-axis table. The z-axis table is automatically moved into the zero position determined by the displacement adjustment and 4 axes alignment adjustment. This is the position in which samples can be introduced free of force. Please note that a displacement offset of the sample may occur with this type of offset control if the sample is not introduced free of force initially or when the dimensions of the sample change in the course of the experiment. Zero displacement offset control is only available for shear and cantilever sample clamps.
- Constant Current Mode: During the measurement, the drive motor generates a static force (force offset) in addition to the actual dynamic oscillation. This force is distributed between the sample and the membrane according to the ratio of the modulus and spring constant. As a sample softens, its deformation therefore increases while the force exerted on the sample decreases. This mode is recommended for samples whose elasticity modulus is high compared to the elasticity of the drive motor 's diaphragm (i.e. for "hard" samples) throughout the experiment, so that the force on the sample corresponds approximately to the force set for the drive motor. This is also the case in compression experiments. The force offset is entered in units of N. Constant current mode offset control is only available for 3-point bending, tension and compression clamps.
- Auto: The dynamic oscillation at the drive motor is overlaid with a static force. The static force (minimum value) is chosen so as to ensure perfect contact between the sample and the sample holder at an oscillation of 3 Hz and with the maximum values selected for force and displacement amplitude. (Inadequate contact between the sample and sample holder in a tension experiment can for example result in so-called sample "buckling".) The additional static force can be selected by entering a %-value depending on the amplitude. 100% corresponds to the minimum static force. The auto offset mode is especially recommended for fragile samples. The auto mode offset control is only available for 3-point bending, tension and compression clamps.

- None: No offset control. The oscillation does not have a static component; the z-axis table remains in the position set before the start of the experiment. Note that the dimensions of the clamping assembly and drive shaft change slightly with temperature, which results in quasi static changes in force (hard samples) or changes in displacement (soft samples). This mode of offset control is recommended only for special studies and for measurements at room temperature. This mode of offset control is available for all sample holders.

The type of offset control selected is marked with a dot under Offset Control. In the window, the annotation of the segment includes the value of the offset with the appropriate unit.

The type of offset control depends on the measurement setup and on the sample holder you have selected in the Sample Holder box of the Method Window:

	None	Zero Displ.	Const.Curr. Mode	Auto
Shear clamp (small clamping assembly)	✓	✓✳	-	-
3-point bending clamp (large clamping assembly)	✓	-	✓	✓✳
Dual cantilever clamp (large clamping assembly)	✓	✓✳	-	-
Single cantilever clamp (large clamping assembly)	✓✳	✓	-	-
Tension clamp (large clamping assembly)	✓	-	✓	✓✳
Tension clamp (small clamping assembly)	✓	-	✓	✓✳
Compression clamp (large clamping assembly)	✓		✓	✓✳

✓ : available
- : not available
✳ : default setting

Settling

settling condition

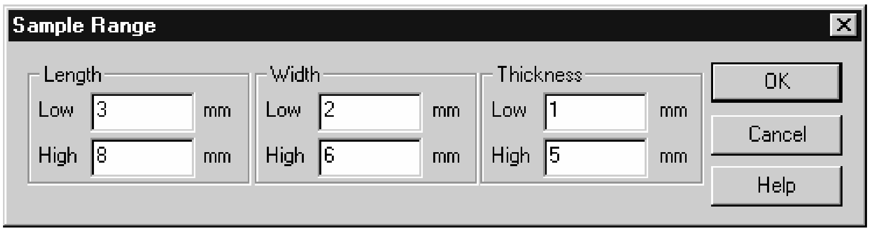
The settling condition demands that temperature oscillations must not exceed the limits defined by the entries for the Band Width and Time parameters in the Settling dialog box in the Method Window. The DMA module checks this condition during the settling procedure until timeout is reached and starts the experiment as soon as the settling condition is satisfied. It is possible to define settling for individual segments on DMA modules.

Sample limits

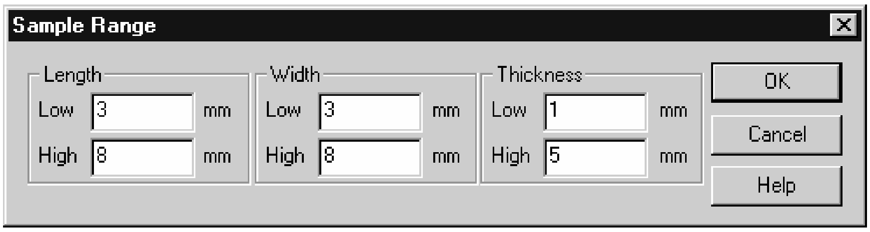
You can define the sample limits in the STARe Software Method Window in the Sample Range dialog box on the Miscellaneous menu. You can enter the range of the sample dimensions for the different clamps as follows:

- **Shear clamp**:

  Cuboid samples: Click in the Length, Width and Thickness boxes and enter the range for the length, width and thickness of the sample in mm. The length is the dimension parallel to the shear force.

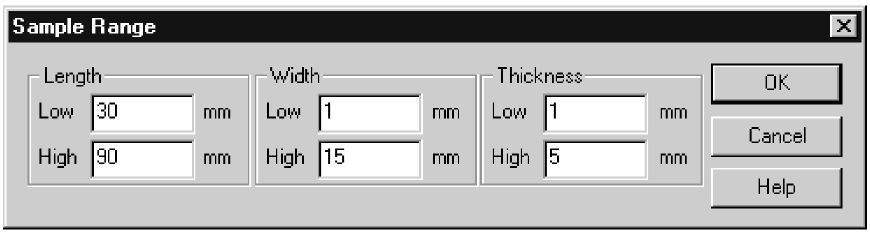


  Round, disk shaped samples: Enter the diameter range of the sample twice, once in the text boxes under Length and once under Width. The entries under Length and Width must be identical for disk shaped samples.
  Enter in the text boxes under Thickness the thickness range of the sample in mm.

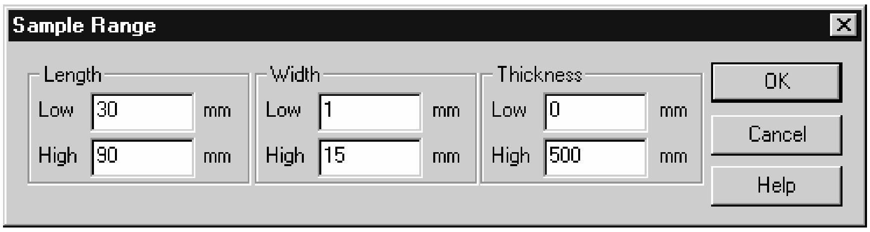


- **3-point bending, dual and single cantilever, large tension clamps**:

  Bar shaped samples: Enter in the text boxes under Length the length range of the sample and in the text boxes under Width and Thickness the range for the width and thickness of the sample in mm.



Round, cylindrical samples: Enter in the text boxes under Length the length range of the sample and in the text boxes under Width the diameter range of the sample in mm. The entries under Thickness have no relevance for cylindrical samples.



These setting can also be changed in the Experiment Window by clicking Sample Limits… on the Miscellaneous menu.

Method names

Make sure to save your method to have it available at a later time. When you save your method include abbreviations in the name you give it denoting e.g. the temperature program and the type of method. This enables you to identify the method immediately even after a long time. We recommend that you use the following conventions.

D	Dynamic temperature program	**A**	Amplitude
I	Isothermal	**f**	Frequency
L	Length ("L" : length)	**Off**	Offset
F	Force ("F" : force)		

Example:	**D +20..120/2 f1000..1 LA10 LOff:**
D +20..120/2:	Dynamic temperature segment from 20 °C to 120 °C with a heating rate of 2 °K/min
f1000..1:	Frequency range of 1000 to 1 Hz
LA10:	Displacement amplitude of 10 µm
LOff:	Displacement offset of 0 (zero)

7.1.2 Adjustment of liquid nitrogen Dewar

Automatic adjustment of liquid nitrogen Dewar

To achieve optimum dosing of liquid nitrogen at low temperatures, the liquid nitrogen Dewar is adjusted automatically. This ensures that the minimum temperature is reached without excessive coolant consumption. In particular it prevents the escape of liquid coolant.

The liquid nitrogen Dewar is adjusted automatically when you run experiments whose start temperatures are lower than -60 °C. The cooler is cooled down to a constant temperature during the second reclamping stage (when the message **ST CHECK** appears on the LCD) or the start temperature stage (when the message **GOING TO START TEMP** appears on the LCD). The constant temperature is maintained for about 5 minutes and the Dewar adjustment is performed during this time. Subsequently, the start temperature is approached and the measurement is started.

You can skip the automatic adjustment by pressing the OK key on the DMA module's keypad. After switching on the DMA module, the automatic adjustment is always performed during the first experiment whose start temperature is lower than -60 °C. You cannot skip the automatic adjustment by pressing OK during this experiment. The OK key is deactivated until the adjustment is completed (the message **WAIT FOR LN2 ADJUST...** appears on the LCD).

To check the Dewar adjustment

- Check the Dewar adjustment while the measurement is running: On approaching low temperatures (e.g. –80 °C), check that the cooler temperature (**Tcool** on the LCD) sinks to at least –170 °C without liquid coolant escaping from the furnace chamber.
- Also check the following possibilities:
  - Are the Dewar filling level and the pressure in the Dewar two low?
  - Is the pressure in the Dewar stable?
  - Did a forced start occur?

7.1.3 Segment start temperatures ≤ -150 °C

If you have defined start temperatures equal to or lower than –150 C in the method, the furnace chamber is only cooled but not heated on approach of these temperatures. The furnace heating remains switched off for the duration of the approach to ensure that the temperature is reliably reached. The fast cooling feature must be switched on manually (see also section *Starting the experiment and performing the measurement*. To start the measurement, you must switch off the fast cooling valve and press the OK key (forced start).

7.1.4 Icing of the clamping assembly

artifacts because of icing

Depending on the humidity, time and the need to open the furnace to adjust clamping, it is possible that ice forms on the clamping assembly at low temperatures. This can lead to the following artifacts occurring in the measurement results:

Noise of the loss factor (Tan Delta)

On heating, the temperature sensor in the clamping assembly detects the thawing of ice and evaporation of water. This affects the temperature control.

wrong modulus and loss angle values

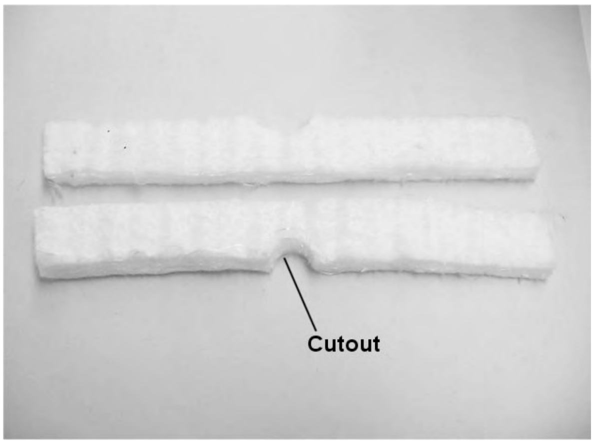
If ice touches the sides of the furnace, the displacement and force measurement signal are wrong. This consequently leads to wrong modulus and cause noise in loss factor values (Tan Delta).

Remedy

insulation strips

For optimum signal quality, the clamping assembly must not touch the furnace. Problems occurring with icing of the clamping assembly must be avoided. The insulation strips, ❶, supplied (ME 51141509) can be inserted on the upper side of the furnace in the gap between the cooler and the glass Dewar. You should make sure that the cutout fits neatly around the clamping assembly.

The insulation strips stop the inflow of moist air from the surroundings to the clamping assembly and thus prevents the formation of ice. Depending on the type of measurement, if the insulation touches the clamping assembly, the modulus and the loss angle values can also be affected. Normally, this disturbance is smaller than that of icing.



❶

❷

❸

To install the insulation strips:

- On the upper side of both furnace halves, insert the insulation strips, ❶, into the gap between the cooler and the glass Dewar, ❷.
- Make sure that the cutout fits neatly around the clamping assembly and that the lower cutout in the furnace halves is not filled with the insulation strips.

♣ It is sufficient to insulate the upper part of the furnace halves. Filling the part around the lower cutout can cause a larger disturbance of the measurement.

The insulation strips are installed in the furnace halves.

7.1.5 Checking the furnace height

furnace height

For optimum signal quality, it is essential that the clamping assembly does not touch the sides of the furnace. The correct position of the furnace height is marked on the rear side of the furnace arm. You should check this mark periodically. See chapter 9, *Control and adjustment of furnace height*.

7.1.6 Operation of the fast cooling device

The fasts cooling device consists of two fast cooling valves which are installed in the furnace arms. The fast cooling valves are operated manually by the two knobs on the outer sides of the furnace arms. The fast cooling device is used to rapidly cool the clamping assembly and sample to low start temperatures before the actual measurement.

When the fast cooling valve is switched off, the coolant from the cooler is directed to the exhaust outlet. The coolant vapor escapes at the rear side of the furnace arm through the silencer into the laboratory atmosphere. This ensures that no disturbances occur in the furnace chamber due to convection currents. The heat is transferred primarily by radiation. This is the setting in measurement mode.

reduce cooling times

When the fast cooling valve is switched on, the coolant from the cooler is directed into the furnace. The coolant vapor escapes above and below through the furnace openings into the laboratory atmosphere. The sample holder and the sample are cooled directly by the coolant vapor; cooling times are thereby significantly reduced. This position is used to cool to the start temperature.

The valves should be switched back to the OFF position **about 5 °C above the start temperature**.

disturbance from fast cooling

Switching fast cooling on or off causes a large disturbance in the system. It requires a certain amount of time for the system to restabilize - typically about 15 to 20 minutes. For this reason, we recommend that you do not switch the cooling valve on or off unnecessarily.

7.1.7 Final checks before the measurement

Before you start your experiment we recommend that you make a few final checks to see if the clamp and the clamping assembly have been installed properly.

These checks do not take long and we recommend you to take some time here to avoid running a measurement that will produce erroneous data due to improper mounting of the sample.

To perform the final checks:

- Check that the nut on the drive shaft that connects it firmly to the drive motor is fastened properly.
- Check that the drive shaft is firmly connected to the clamp holder.
- Check that the clamp is firmly fastened in the clamp holder.
- Check that the fixed part of the clamping assembly is firmly connected to the flange of the displacement sensor
- Check that the locking screw on the four axes alignment device is fastened.
- Start the four axes alignment procedure from the S**TA**R^{e} Software again to check if the force reading of the force sensor is 0 (zero). See chapter 6, *Fine alignmnet of the measuring sensors*.
- Make sure that the SDTA thermocouple does not touch the furnace half on closing the furnace.

If you detect any problem with the installation of the clamping assembly, clamp or sample (loose clamps, loose screws and nuts etc.), you must correct it by reclamping or, if necessary, remounting the sample. The corresponding instructions can be found in chapter 6.

7.1.8 Creating DMA Experiments in the Experiment Window

To perform a measurement in your DMA/SDTA861e, you have to create an experiment data set in the S**TA**R**e** Software Experiment Window that contains the desired settings for your measurement. This data set that you send to the DMA module is called an “Experiment”. It also includes the information about the DMA method that you have created in the Method Window for your DMA measurement.

The appearance of the S**TA**R**e** Software Experiment Window depends on the type of module used – the text boxes are different depending on the TA technique. The functions and input possibilities described here for the Experiment Window are specific to the DMA. Further information can be found in Chapter 11, *Experiment Window*, of the S**TA**R**e** Software Operating Instructions*.*

To create an experiment and send it to the DMA module:

Start:
- DMA module ready for measurement
- DMA method for your experiment is created and saved in the STARe software database

♣ To create a new experiment you must always select a method and define a sample name.

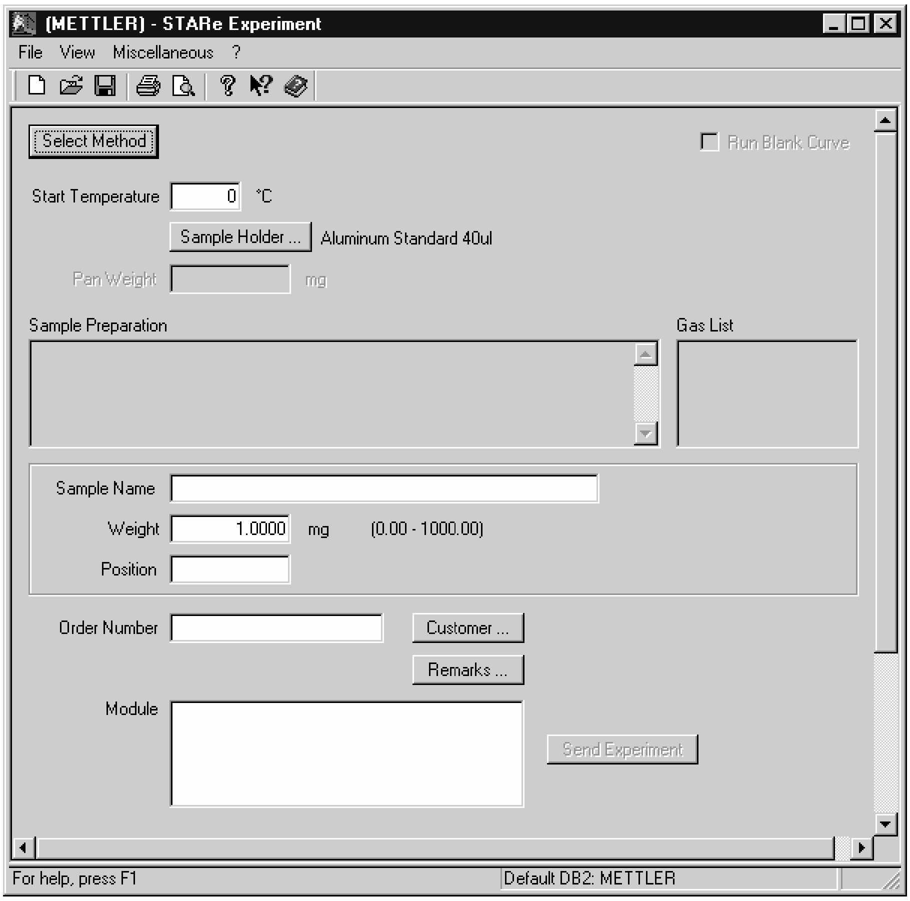
(1) Open an Experiment Window in the STARe Software by choosing Functions/Experiment in the main menu bar.

The STARe software opens a new Experiment Window. The Experiment Window is per default set up for a DSC module.

♣ The Direct Method function is not available for DMA experiments.

(2) Click the Select Method button and select the desired DMA method in the drop-down list that appears. select method

The data for the selected DMA method and the DMA specific text boxes appear in the Experiment Window.



The upper half of the Experiment Window is the same as for other STARe modules. The Pan Weight box is however is not active because it is not used for DMA experiments. The DMA specific text boxes are in the lower half of the window.

(3) To enter a start temperature for your experiment that is different to that specified in the method: Enter the desired start temperature in the Start Temperature box.

start temperature

♣ The default value in this box is the start temperature of the method.

(4) To enter or modify a text on sample preparation: Enter the appropriate details in the text box. The text box is located above the Sample Name text box.

(5) Enter the name of the sample in the Sample Name text box. Use a name that identifies the sample (e.g. PET or W007).

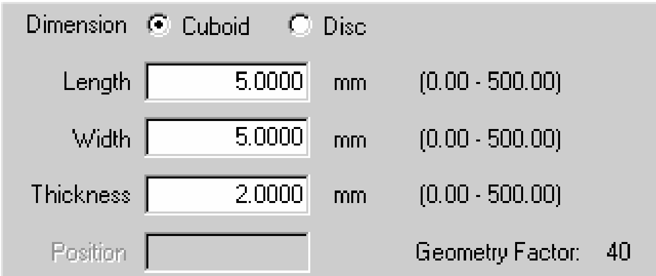
dimensions of the sample

(6) Enter the dimensions of the sample as follows:

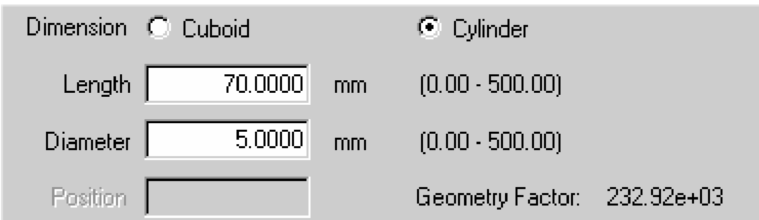
♣ For DMA experiments you can enter the dimensions of the sample in the Experiment Window. You can enter data for cuboid, disc shaped or cylindrical samples.

▸ Click the description that corresponds to your sample (i.e. Cuboid, Disc or Cylinder, next to Dimension).

If your sample is cuboid, text boxes for the length, width and height of the sample are displayed.



If your sample is disc shaped or cylindrical, text boxes for the diameter and thickness or length, respectively, of the sample are displayed.



The size range for the dimensions of the sample is shown to the right of the text boxes. This was specified in the Method Window when you created the method. It is also possible to change the size range for the dimensions of the sample in the Experiment Window. To do this, see the instructions in the section *Defining the range of sample size and limiting the force and displacement amplitudes*.

- Click the boxes and enter the dimensions of your sample according to the definitions the following section *Sample dimensions and geometry factors*.

You have entered the dimensions of your sample. The STARe software automatically calculates the sample geometry factor from the values entered. The value of the geometry factor appears below the boxes next to the text Geometry Factor.

♣ The geometry factor takes into account the effect of sample geometry in the calculation of the Young's modulus or the shear modulus (or compliance). See the following section *Sample dimensions and geometry factors*.

(7) To give your experiment a number: Click the Order Number box and enter a number for your experiment.

♣ You can associate your experiment with the name of a customer. This allows you later on to select all the experiments belonging to the same customer by using the Filter function when you open an experiment with File/Open. You can enter the name of the customer in the Installation Window under Topic/Customer und enter the appropriate data.

(8) To assign your experiment to a customer name entered in the STARe database:

- Click the Customer button
- Select the name of the desired customer in the list in the box under List:

  The name appears in the Selection box
- Click OK or press ENTER.

The experiment is assigned to the name of the customer.

(9) To save your experiment:

- Choose File/Save under...

  The Save under dialog box appears
- Enter a name for the experiment and click OK. The sample name appears per default. The name of the experiment cannot contain more than 40 characters.

  The experiment is saved in the database under the name you enter.

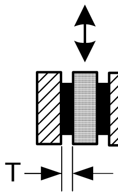
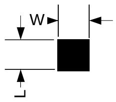
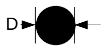
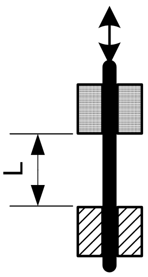
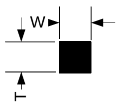
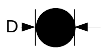
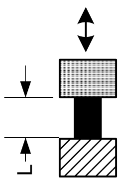
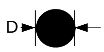
♣ Every experiment you send to the STARe module is automatically saved in the database, even if you have not saved it manually yourself. By default, the STARe software uses the name entered in the Sample Name box.

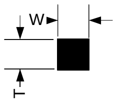
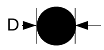
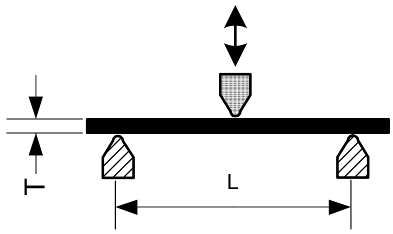
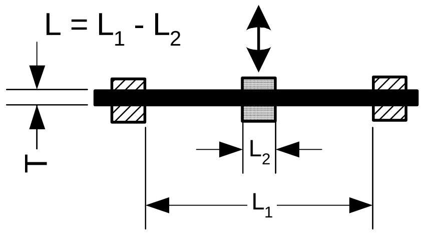
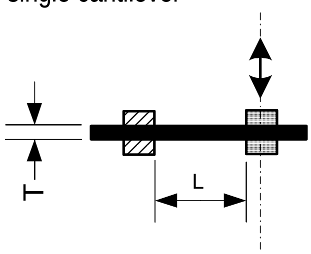
End: Experiment is set up in the Experiment Window

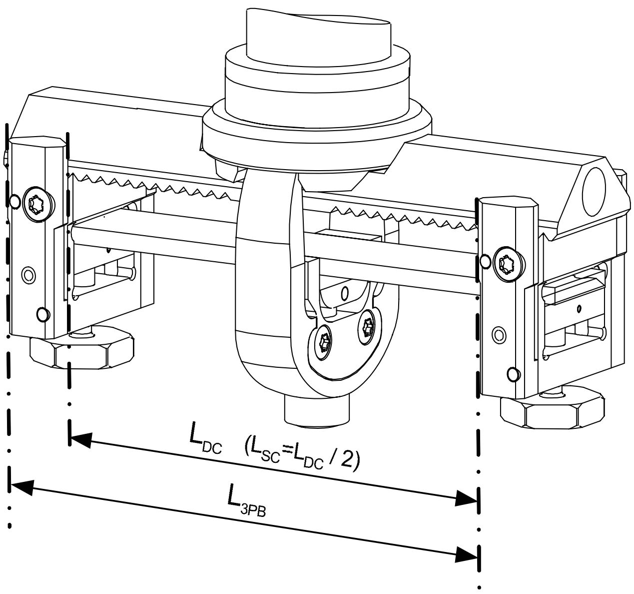
Sample dimensions and geometry factors

As explained in chapter 2, the stiffness is the quantity that is directly measured. The modulus is calculated from the stiffness and the geometry factor which includes the sample dimensions.

Table of sample dimensions and geometry factors

Mode	Rectangular samples	Round samples
Shear (small CA only) 	thickness, T : < 6.5 mm width, W : < 15 mm Length, L : < 18 mm $G = \frac{T}{2\,LW}$ 	thickness, T : < 6.5 mm diameter, D : < 15 mm $G = \frac{2\,T}{\pi\,D^2}$ 
Tension (small and large CA) 	Small clamping assembly length, L : 9.0 width, W : < 5 mm thickness, T : < 2 mm Large clamping assembly length, L : 19.5, 10.5, 5.5 mm width, W : < 7 mm thickness, T : < 3 mm $G = \frac{L}{W \cdot T}$ 	Small clamping assembly length, L : 9.0 diameter, D : < 2 mm Large clamping assembly length, L : 19.5, 10.5, 5.5 mm diameter, D : < 3 mm $G = \frac{4L}{\pi\,D^2}$, where $\frac{D}{L} \leq \frac{1}{20}$ 
Compression (large CA only) 	Rectangular samples are not recommended with the compression clamp.	length, L : < 9 mm diameter, D : < 20 mm $G = \frac{4 \cdot L}{\pi\,D^2}$ 

Mode	Rectangular samples	Round samples
		
Bending (large CA only)		
• 3-point bending 	length, L : 30 ... 90 mm width, W : < 15 mm thickness, T : < 5 mm $G = \frac{L^3}{4\,WT^3}$	length, L : 30 ... 90 mm diameter, D : < 5 mm $G = \frac{4 \cdot L^3}{3\pi\,D^4}$
• dual cantilever 	length, L : 20 ... 80 mm width, W : < 15 mm thickness, T : < 5 mm $G = \frac{L^3}{16\,W\,T^3}$	length, L : 20 ... 80 mm diameter, D : < 5 mm $G = \frac{L^3}{3\pi\,D^4}$
• single cantilever 	length: half the length for dual cantilever is used $G = \frac{L^3}{W\,T^3}$	length: half the length for dual cantilever is used $G = \frac{16 \cdot L^3}{3\pi\,D^4}$



The sample length L is measured for dual cantilever, single cantilever and 3-point bending experiments as shown on the left:

- dual cantilever bending: L_{DC}
- single cantilever bending: $L_{SC}=L_{DC} / 2$
- 3-point bending: L_{3PB}

Please note
Although round samples can be measured in the bending mode, you may have to take into account clamping effects (due the higher deformation of the sample in the clamps) that will possibly reduce measurement accuracy.

Defining the range of sample size and limiting the force and displacement amplitudes

The Experiment Window uses the limits defined in the Method Window under Miscellaneous/Range of Sample Size for the sample dimensions, the force and the displacement. You can however change these limits afterward in the Experiment Window under Extras/Sample Limits.

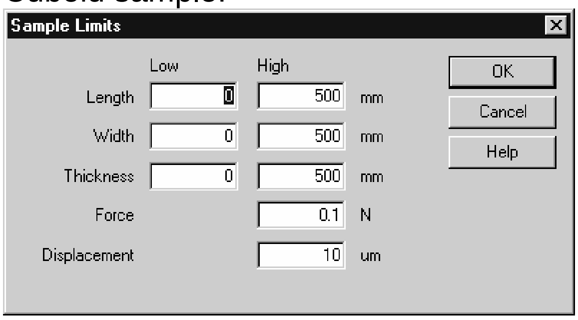
♣ For a DMA experiment you can set upper limit for the force and the displacement amplitude and limit the force or displacement amplitude created in the experiment by the drive motor. This protects your sample from possible damage.

To define sample size range or limit force and displacement amplitudes in a DMA experiment:

(1) Choose Miscellaneous/Sample Limits.

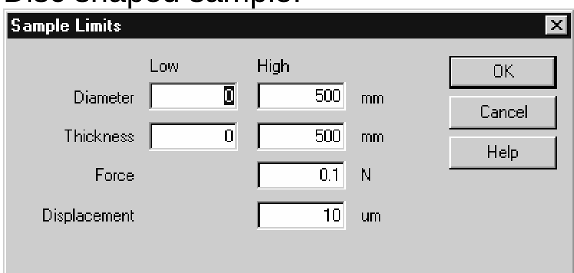
The Sample Limits dialog box appears: The text boxes vary according to whether have selected Cuboid, Disc or Cylinder as the sample geometry.

Cuboid sample:

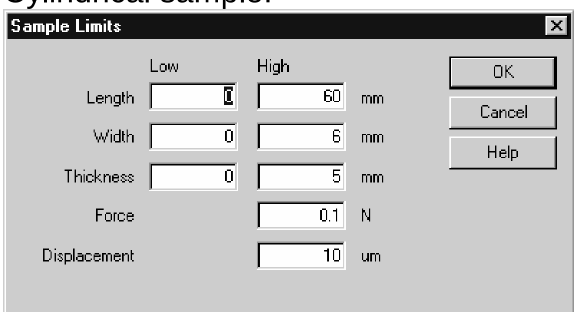


♣ Further details on entering sample dimensions can be found in sections *Sample limits and Sample dimensions and geometry factors*.

Disc shaped sample:



Cylindrical sample:



(2) Enter the limits for the geometry of your sample in the Length, Width and Thickness boxes for a cuboid sample, or in the Diameter and Length boxes for a cylindrical sample. Enter the upper limits for the Force and Displacement amplitude in the Force and Amplitude boxes

♣ The default range of sample dimensions is 0 to 500 mm. The upper limits for the force and displacement amplitude are by default 100 N and 50 10+3 µm respectively

(3) Click OK.

The sample limits are saved with the dataset of the experiment.

7.2 Preliminary stages before the measurement

The experiment is processed on the DMA module as soon as it has been sent from the Experiment Window of the S**TA**R**e** Software. Information about the progress of the experiment is displayed in the Module Control Window. Before the actual measurement there are several preliminary stages. These stages are similar to those on other TA modules except for the first two stages which are specific to the DMA module. The clamping of the sample is checked during these two stages. This is explained in the following.

Preliminary checks

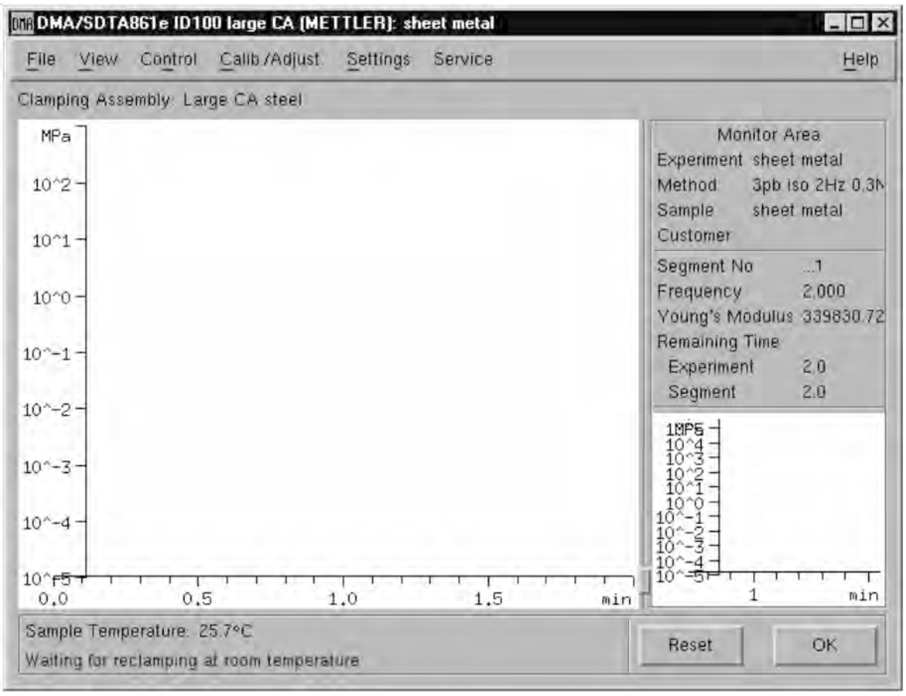
The two first stages are preliminary checks of the clamping of the sample at room and start temperature. The sample is subjected to the force and displacement amplitudes specified in the method for the measurement. You can reclamp the sample if necessary at these two stages by opening the furnace and following the corresponding instructions in chapter 6, section *Installing and removing the shear clamp* or section *Installing the bending clamp with the sample in the large clamping assembly*.

Autostart without reclamping

♣ When you select the Autostart check box in the Module Control Window, the DMA module skips the two reclamping stages at the beginning of an experiment. This is useful, for example to run repeat experiments, when you have already checked the clamping.

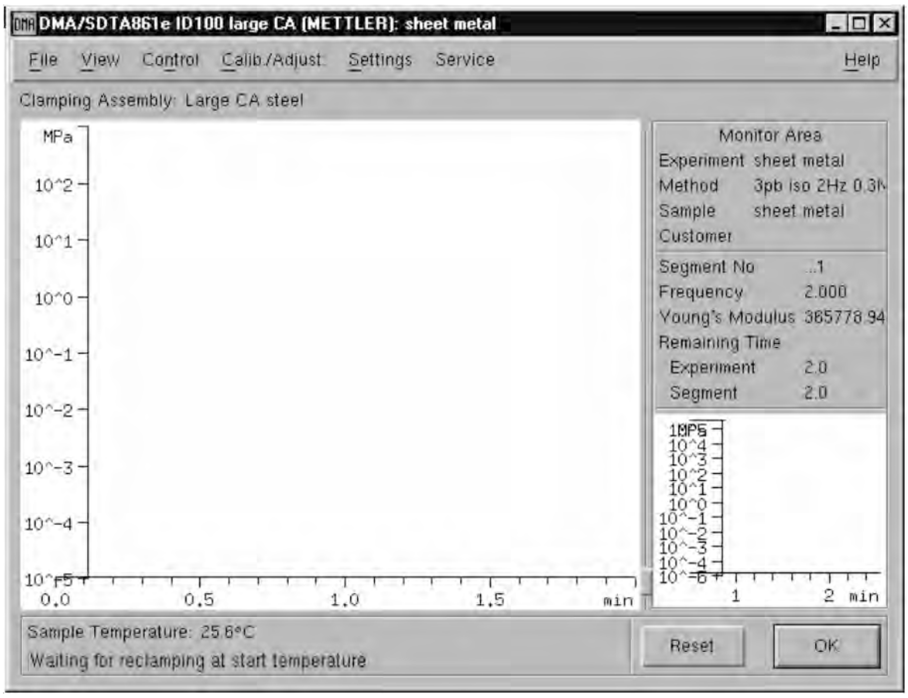
Reclamping stage at room temperature

1 **First reclamping stage:** In the bottom area of the Module Control Window under Sample temperature, the text Waiting for reclamping at room temperature appears. The message **RT CHECK** appears simultaneously on the LCD on the DMA module.



2 **Second reclamping stage:** In the bottom area of the Module Control Window under Sample temperature, the text Waiting for reclamping at start temperature appears. The message **ST CHECK** appears simultaneously on the LCD on the DMA module.

Reclamping stage at start temperature



The fast cooling can be switched on at this stage to reduce cooling time if the start temperature lies significantly below ambient temperature. The knobs for the fast cooling valves on the outer sides of the furnace arms are used for this. The fast cooling has to be switched off again before the start temperature stage.

3 **Start temperature and settling stages:** These two preliminary stages are similar to those on other TA modules. In the bottom area of the Module Control Window under Sample temperature, the text Going to start temperature or Settling appears, respectively. The color of the area changes from green to red and remains red throughout the measurement. See chapter 8 of the STARe Software User Manual for further information.

Going to start temperature and Settling

4 **Measurement:** The actual measurement is started when the start temperature is reached and the settling has been completed. The text Measurement appears in the bottom area of the Module Control Window on a red background.

Measurement

7.3 Starting the experiment and performing the measurement

Never remain in close proximity of the DMA module during a measurement without hearing protection. Always wear hearing protection when working near the DMA module. The DMA module can produce excessive noise that can impair your hearing. The noise level can exceed 85 dB(A) during certain measurements.

To start the experiment and perform the measurement

Start: Experiment is set up in the Experiment Window.

send experiment

(1) To send the experiment to the DMA module: In the Experiment Window, click the button with the name of your DMA module next to the text Send Experiment.

The experiment is sent to the DMA module.

♣ The experiment is sent to the DMA module is listed in the Experiment buffer of the Module Control Window. An experiment starts automatically if the there are no other experiments queued in the buffer or if the Autostart feature is switched on.

start experiment

(2) To start the experiment manually if the Autostart feature is switched off: Choose Control/Start Experiment in the Module Control Window.

The name of the experiment appears in the buffer of the Module Control Window in bold type. The first reclamping stage is reached. The text Waiting for reclamping at room temperature appears in the area at the bottom of the Module Control Window. The modulus values are displayed in the monitor area on the right.

check modulus values

(3) To check the clamping do the following:

- Check the modulus values in the monitor area of the Module Control Window.
- Check whether there are any unusual noises that indicate loose screws or loose clamps.

If necessary correct the clamping as follows:

reclamp sample at room temperature or start temperature

- Reclamp the sample:
  - ▸ Open the furnace by pressing the FURNACE key.
  - ▸ Follow the corresponding instructions in chapter 6.

remount sample if necessary

- Remount the sample:
  - ▸ Terminate the experiment by pressing the RESET key on the keypad or by clicking Reset. Follow the corresponding instructions in chapter 6.

(4) At the end of the first reclamping stage: Press the OK key or click OK to start the second reclamping stage.

The text Waiting for reclamping at start temperature appears in the area at the bottom of the Module Control Window.

Never touch the furnace halves or the clamping assembly during or immediately after a measurement. Always wear protective gloves or let the furnace and the clamping assembly cool down to ambient temperature before you open the furnace halves. Parts of the furnace and the clamping assembly can reach temperatures down to –150 °C or up to +500 °C. There is risk if burns.

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fast cooling

To use the fast cooling feature:

- Switch on the fast cooling by turning the two knobs into the “ON” position.

- Cool to **about 5 °C above the start temperature**, then switch off fast cooling by turning the two valves into the “OFF” position.

♣ Switching fast cooling on or off causes a large disturbance in the system. It requires a certain amount of time for the system to re-stabilize - typically about 15 to 20 minutes. For this reason, we recommend that you do not switch the cooling valve on or off unnecessarily.

♣ The fast cooling feature can be used during the second reclamping stage (Waiting for reclamping at start temperature) or during the start temperature stage (Going to start temperature).

(5) When the start temperature is reached: Repeat step (3). If the furnace has to be opened for reclamping and the sample temperature has risen you may want to use the fast cooling again as explained in step (4).

Going to start temperature

Settling

Measurement

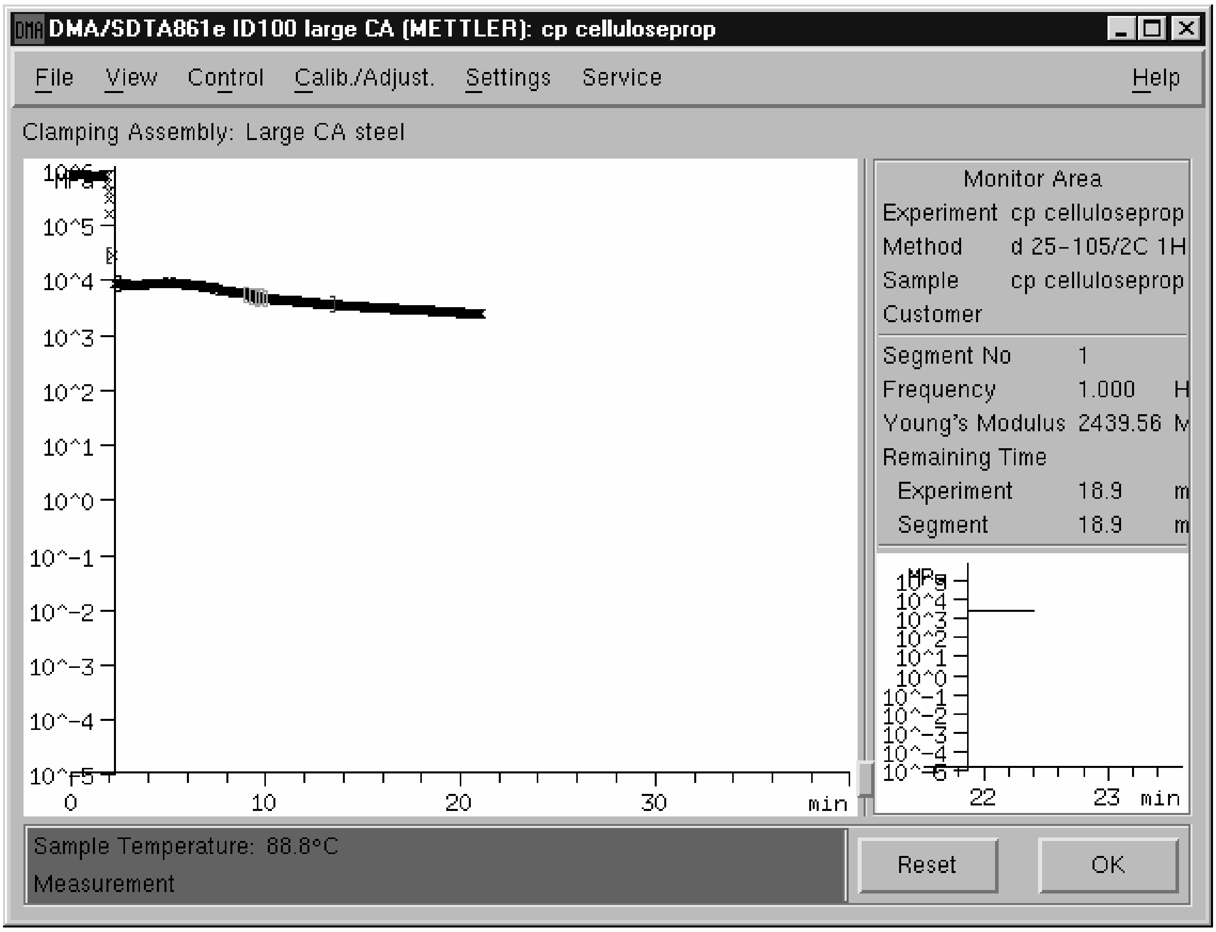
(6) At the end of the second reclamping stage: Press the OK key or click OK go on to the start temperature stage.

The text Going to start temperature appears in the area at the bottom of the Module Control Window. The temperature control loop approaches the start temperature and the settling stage automatically begins. When the settling is completed the measurement stage starts automatically.

End: Measurement started on the DMA module.

7.3.1 DMA measurement in the Module Control Window

You have sent your experiment to the DMA module. The measurement begins as soon as the DMA module passes the preliminary stages before the measurement. A red bar appears at the bottom of the Module Control Window with the text Measurement and the display of the current sample temperature. The window area displays the online curve of a measurement quantity. In the default setting this is the value of Young's modulus E* as a function of time.



Module Control Window during a DMA measurement

Monitor Area

The monitor area on the right side of the Module Control Window contains the current DMA experiment data that is not contained in the measurement curve of the current measurement. Here, you can for example trace the course of a measurement quantity during the reclamping stage or settling for up to two minutes.

You can display this window area using the View/Monitor Area command. In addition the command calls up an upper and a lower monitor area with the following data:

- The upper right corner the Module Control Window shows data on the current measurement in progress. As with other STARe modules, the monitor area contains the experiment, method, sample and customer names.
- The lower monitor area displays the segment number and the time remaining for the current segment and experiment. Besides this it displays the values of the frequency and the absolute value of the modulus (of shear or elasticity) corresponding to the current measurement point.

♣ With other STARe modules, the Module Control Window displays the heating rate and the sample weight here.

online curve of measurement quantities

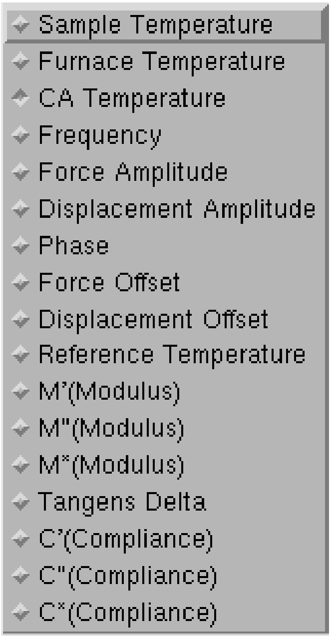
Other measurement quantities can also be displayed as online curves. In the menu View under Online Curves there is a drop-down-list in which you can select the desired curve. The following table presents an overview of the measurement quantities and their units that can be displayed in the window area:

Measurement quantity	**Unit**
Sample temperature	° C
Furnace temperature	° C
CA temperature	° C
Frequency	Hz
Force amplitude	N
Displacement amplitude	μm
Young's modulus: phase	rad
Force offset	N
Displacement offset	μm
Force distortion	dB
Displacement distortion	dB
Reference temperature	° C
E' (real part of Young's modulus)	MPa
E'' (imaginary part of Young's modulus)	MPa
E* (absolute value of Young's modulus)	MPa
Tangens Delta	-

♣ These measurement quantities are also available for the evaluation in the Evaluation Window when you open a DMA measurement curve.

To display a measurement quantity as an online curve:

(1) Choose View/Online Curves.



A drop-down list appears with the available measurement quantities.

(2) Click the desired measurement quantity in the drop-down list. You can only select one measurement quantity.

The selected online curve appears in the window area.

(3) To adapt the curve to the window area: choose View/Automatic Scaling,

The measurement is displayed in the window area. The STARe Software updates the online curve after each measurement point.

The Calib./Adjust. menu in the Module Control Window

The Module Control Window contains special functions for the calibration and adjustment of the DMA module in the Calib./Adjust. menu. Besides the temperature calibration and adjustment, with the DMA module it is also necessary to perform mechanical adjustments. Appropriate functions are implemented for the spindle, the force sensor and the displacement sensor. Detailed information on this can be found in Chapter 8, *Calibration and Adjustment* in this operating manual.

7.4 Evaluation of DMA measurements

You can evaluate the measurement data produced by your DMA module in the Evaluation Window of the S**TA**R**e** Software. This section gives a short summary of the DMA evaluation possibilities. Detailed information and instructions can be found in the online Help to the S**TA**R**e** Software Evaluation Window.

7.4.1 Basic evaluation of DMA measurements

A DMA measurement yields a large amount of data. It is not possible to display all this data at the same time in the Evaluation Window. It is therefore only sensible to concentrate on the measurement quantities of interest.

open DMA curves

thermoanalytical evaluations

The DMA and TA menus in the S**TA**R**e** Software Evaluation Window include the basic functions for opening, displaying and processing your DMA measurement curves. On the displayed DMA measurement curves you can perform the thermoanalytical evaluations given in the TA menu.

With DMA measurements, it is the components of the complex modulus of elasticity (E) or the compliance (C) that are above all of interest. It is possible to display these quantities versus time, temperature or frequency. The data for the modulus of elasticity and the compliance is derived from the primary force and displacement measurement quantities.

Direct measurement quantities

direct measurement quantities

A measurement with the DMA module provides the following measurement quantities. You can display these directly in the Evaluation Window:

- The force and displacement amplitudes and the phase shift (phase) between these signals
- The force and displacement offsets
- The reference temperature, sample temperature, furnace temperature and clamping assembly temperature

Derived measurement quantities

derived measurement quantities

The following data of interest for the evaluation are derived from the direct measurement quantities and the geometry entered in the Experiment window by the user:

- M': Storage component of modulus of elasticity or shear modulus (real part)
- M'': Loss component of modulus of elasticity or shear modulus (imaginary part)
  M*: Complex modulus of modulus of elasticity or shear modulus
- Tan Delta : Loss factor
- C': Storage component of modulus of elasticity or shear compliance (real part)
- C'': Loss component of modulus of elasticity or shear compliance (imaginary part)
- C*: Complex modulus of compliance of elasticity or shear compliance

You can display the above quantities versus time, temperature or frequency in the STAR^{e} Software Evaluation Window. You can open and display curves with DMA data just like other TA curves using the File/Open Curve command.

♣ You can define the measurement quantities to be displayed on opening the DMA curve under Settings/Individual DMA Curve Selection....

display of individual measurement points

In contrast to curves from other TA measurements, DMA data are displayed as individual measurement points plotted as symbols (crosses, circles, etc.) in the window area. The crosses correspond to the measurement points specified in the method. The DMA curves are therefore not continuous in contrast to the other continuous TA curves. The measurement points displayed by their symbols are, however, joined by straight lines. The time difference between two consecutive measurement points is not defined by a sampling rate, but is a result of the technique-dependent measurement duration of individual measurement points.

7.4.2 Time-Temperature Superposition

software option “DMA Evaluations”

The software option “DMA Evaluations” to the Evaluation Window offers the possibility of performing evaluations by the Time-Temperature Superposition method (see also chapter 12, *Accessories* for the corresponding order numbers).

The TTS procedure (Time-Temperature Superposition, TTS) is based on the principle of the equivalence of time and temperature. This principle describes the physical relationship between the time or frequency dependence of the viscoelastic properties of a polymer and its temperature dependence.

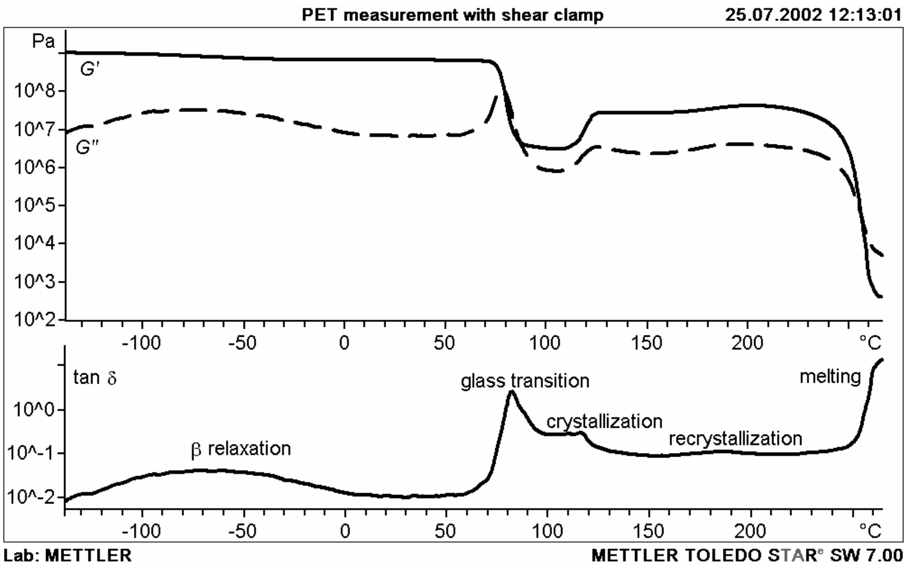
7.4.3 Evaluation names

Make sure to save your evaluation to have it available at a later time. When you save your evaluation include abbreviations in the name you give it denoting e.g. the sample name, the kind of evaluation used or information about the method. This enables you to identify the evaluation immediately even after a long time.

Examples:

- DMA/PET/shear/glass transition
- DMA/PVC/shear/freq. scan/offset none
- DMA/PP film/tension /freq. scan/offset 250%

7.4.4 Example of a DMA measurement:



The above measurement of a disk shaped PET sample was performed with the shear camp in the large clamping assembly. The curves represent the shear modulus G’ and G’’ and the loss factor tan delta.

The method included the following settings.

- Temperature scan from –180 to 280 °C at 1°C/min
- Displacement amplitude 10µm
- Force amplitude 30 N
- Frequency series: 1000 Hz, 100 Hz, 10 Hz, 1 Hz
- Automatic offset control: zero displacement

The modulus and tan delta curves depicted above represent the temperature scan at 1 Hz. As can be seen all the effects characteristic for the PET material are clearly visible in a single measurement.

8 Calibration and Adjustment

Contents

8 Calibration and Adjustment

8.1 Definitions and abbreviations

This chapter describes how you calibrate and adjust the measuring system and the z-axis table.

We use the terms "calibration" and "adjustment" according to the following definitions:

calibration For the module in question, the determination of the actual deviation of measured values of reference substances from the standard literature values by means of calibration measurements.

Reference substance: a substance that is suitable for the calibration measurement and whose thermoanalytical property values are well established in the literature

adjustment Adapting the specific module parameters so that the measured values of the calibration measurements performed afterward are within the limits of permissible error

Limits of permissible error: the specified extreme values for permitted deviations (positive and negative) from a set value.

Abbreviations:

For better orientation, the margins of the pages of this chapter include the following signs to indicate to which device the text is in the corresponding section refers:

ALA Alignment aid, used for the text relating to the alignment aid in section 8.3.

AAS Adjustment assembly, used for the text relating to the adjustment assembly in sections 8.2 and 8.4 to 8.5.1

CA Clamping assembly, used for the text relating to the small or the large clamping assembly in sections 8.5.2.

8.2 Mechanical adjustment procedure

The procedure for the mechanical adjustment of the DMA/SDTA861e serves the calibration and adjustment of the force and displacement sensors and comprises several individual steps. Different types of adjustment are performed in some of the steps. The steps are performed in the following order:

1	**Installation of the alignment aid ***	ALA
2	**Pre-alignment with the alignment aid**	ALA
3	**Installation of the adjustment assembly ***	AAS
4	**Fine alignment of the measuring sensors ***	AAS
5	**Adjustment of the spindle and the force**	AAS
6	**Installation of the small/large clamping assembly**	CA
7	**Fine alignment of the measuring sensors**	CA
8	**Displacement adjustment****	CA

* these steps are only necessary on full adjustment of the DMA/SDTA861e

** this step is only necessary on first installation of a clamping assembly

8.2.1 Overview of the adjustment steps

The following table gives an overview of the adjustment steps with the alignment aid, adjustment assembly or a clamping assembly.

Alignment aid
ALA

Adjustment assembly
AAS

Small clamp. assemb.
SCA

Large clamp. assemb.
LCA

Adjustment	ALA	AAS	SCA	LCA
Pre-alignment with alignment aid *	✓	-	-	-
Adjust four axes alignment device **	-	✓	✓	✓
Adjust spindle and force	-	✓	-	-
Adjust displacement ***	-	-	✓	✓

ALA : Alignment aid

AAS : Adjustment assembly

SCA : Small clamping assembly

LCA : Large clamping assembly

✓ means "is necessary"

* The pre-alignment with the alignment aid should be performed before a complete mechanical adjustment of the DMA module or if the measuring sensors are strongly misaligned and cannot be corrected by the four axes alignment procedure. It should be performed about once a year.

** The four axes alignment must be performed each time you change the clamping assembly or install the adjustment assembly. It should normally be performed about once a week.

*** The displacement adjustment should be performed separately when you install a new clamping assemlby.

AAS

8.3 Adjustment assembly

For the mechanical adjustment you need the adjustment assembly that is supplied with the instrument.

We recommend you to use the supplied Torx Allen wrench and the Torx screwdriver to fasten and loosen the Torx screws in the procedures in the following sections.

Fig. 8-1 shows the design of the adjustment assembly.

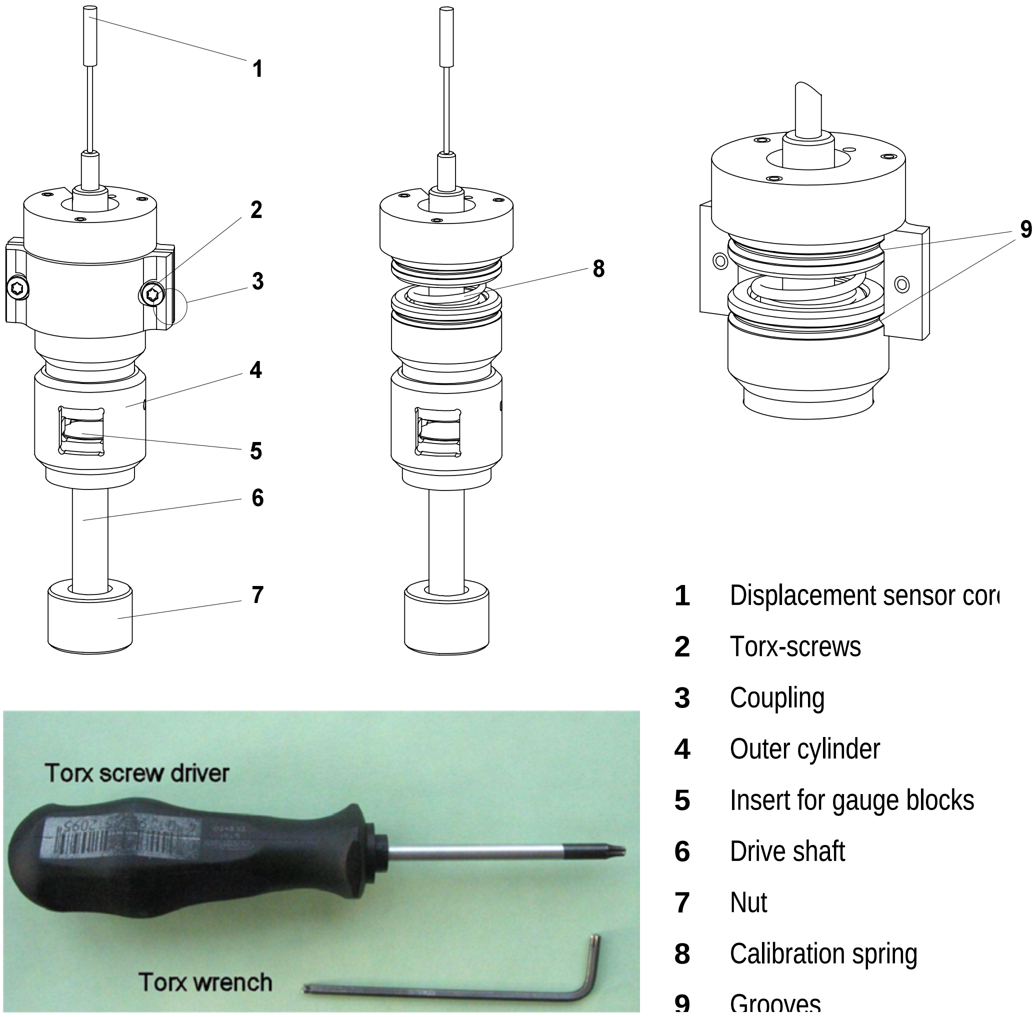


Fig. 8-1. Adjustment assembly and its parts

8.3.1 Mounting the coupling

The coupling **3** for the adjustment assembly has a function similar to that of the adjustment device in the clamping assembly. It forms a rigid connection between the movable and the fixed part of the adjustment assembly.

To mount the coupling on the adjustment assembly:

Start: Coupling **3** is ready

(1) Hold the two halves of the coupling **3** up to the adjustment assembly. Two Torx screws **2**, each with a washer, are mounted in the recessed holes of the one half. Make sure that the washers do not fall out.

(2) Make sure that both halves of the coupling **3** enclose the calibration spring **8** and that both rims fit in the grooves **9**.

(3) Tighten the coupling **3** firmly with the two Torx- screws **2**.

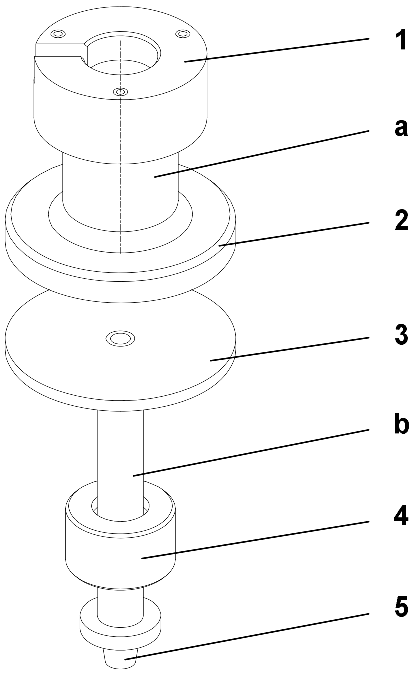
End:
- Coupling **3** mounted
- Adjustment assembly ready for installation

8.4 Pre-aligning the measuring sensors and installing the adjustment assembly

Before you install the adjustment assembly and adjust your DMA module the measuring sensors must be pre-aligned. This section explains how to pre-align the measuring sensors with the alignment aid to subsequently install the adjustment assembly.

8.4.1 Pre-aligning the measuring sensors with the alignment aid

The alignment aid shipped with the DMA/SDTA861e serves the purpose of pre-aligning the displacement and the force sensors with the clamping assembly. It is shown in Fig. 8-2.

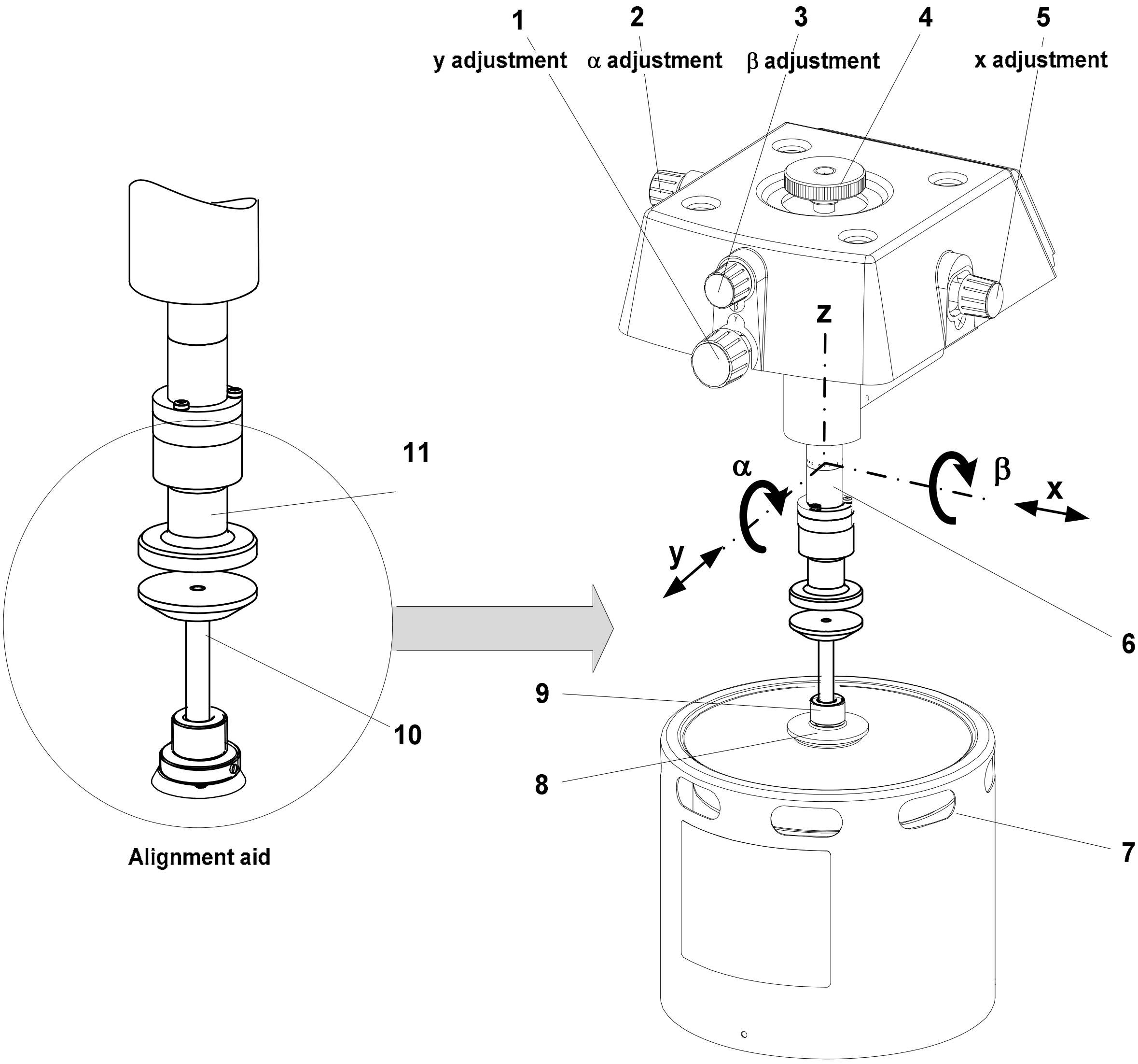


a	Alignment aid, upper part
1	Flange
2	Plate
b	Alignment aid, lower part
3	Plate
5	Nut
6	Conical end

Fig. 8-2. Alignment aid

♣ The fine alignment of the measuring sensors is carried out after the manual pre-alignment via a dialog in the DMA module software (see section *Fine alignment of the measuring sensors (four axis alignment)*.

Fig. 8-3 shows the four axes alignment device, the measuring sensors, the alignment aid and the positions and angles to adjust. With the help of the alignment aid the positions and angles of the measuring sensors can be adjusted quickly so that the sensors are in the right position when you install the adjustment assembly.



1	Knob for adjusting the y -position	**7**	Drive motor
2	Knob for adjusting the α angle	**8**	Threaded fitting
3	Knob for adjusting the β angle	**9**	Nut
4	Locking screw	**10**	Alignment aid, lower part
5	Knob for adjusting the x -position	**11**	Alignment aid, upper part
6	Displacement sensor housing		

Fig. 8-3. Pre-alignment with the alignment aid with the alignment aid

ALA

To pre-align the measurement sensors with the alignment aid:

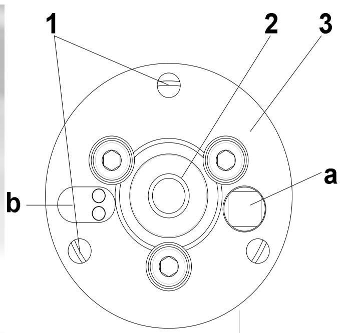
⇨ *See page 8-7, Fig. 8-3*

Start: - Alignment aid is ready

♣ The mechanism of the 4-axis alignment device operates best when it is pressed together slightly by the locking screw.

Install alignment aid

(1) Fasten the upper part of the alignment aid as follows to the flange of the displacement sensor, ❶:



1 Torx screws
2 Displacement sensor core
3 Displacement sensor flange
a CA Thermocouple
b Hole for SDTA Thermocouple

Fig. 8-4. Displacement sensor flange (view from below)

- Hold the upper part of the alignment aid under the flange of the displacement sensor. Ensure that the thermocouple projecting from notch is positioned freely in the notch (cp. Fig. 8-4). Make sure not to wedge the thermocouple while fastening the upper part of the alignment aid.

♣ The Torx screws must be aligned with the tapped holes in the flange of the alignment aid.

- Screw in the three Torx screws and tighten them firmly with as equal torque as possible using the Torx Allen wrench supplied.

The upper part of the alignment aid is now fixed to the flange of the displacement sensor.

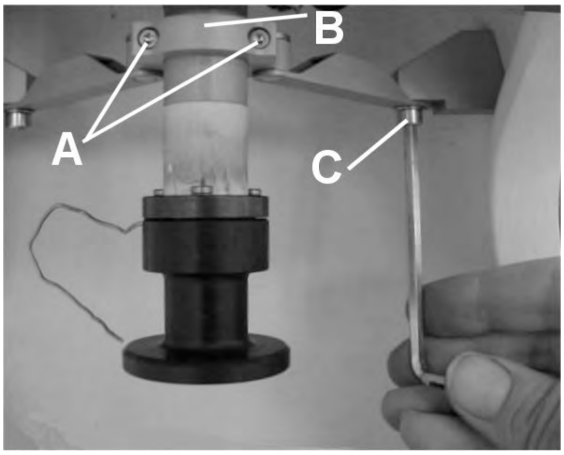
(2) Set the lower part of the alignment aid into the seat in the threaded fitting of the drive motor. The conical end fits snugly into the conical bore. Tighten the nut on the fitting, ❷.

(3) Loosen the support structure of the measuring sensors as follows:

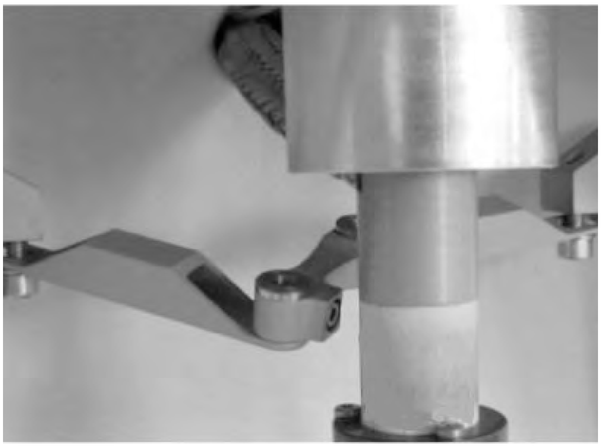
- Remove the Torx screws (A) with the Torx wrench and remove the front clamp (B). Loosen the Alllen screws (C) with a 4 mm Allen wrench, ❸, but do not completely remove them so that the support structure is still attached.
- Fold the support structure back so that it is no longer in contact with length sensor flange, ❹.

❶

❷



❸



❹

(4) Loosen the locking screw of the four axes alignment device, ❺, then slightly tighten it again to the point where you feel a slight resistance.

Do not drive the lower part of the alignment aid mounted on the z-axis table too far up toward its upper part.

The drive motor could suffer damage.

---

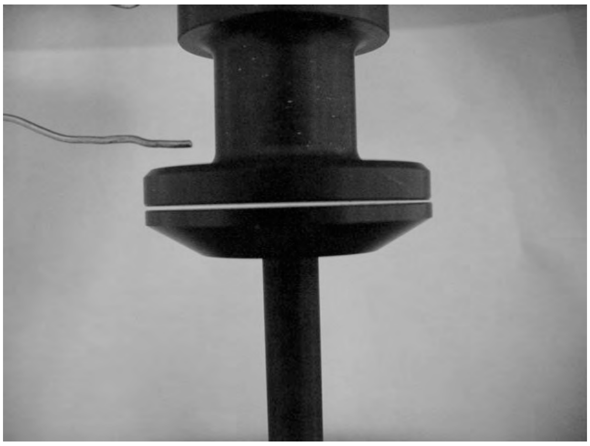
(5) Drive the z-axis table upward as follows.

- Press the MOTOR UP key once. The z-axis table moves upward and stops at a pre-set position. The two plates of the alignment aid are still well apart in this position, **❻-a** and **❻-b**.
- Press the MOTOR UP-key repeatedly to slowly move the lower plate of the alignment aid toward the upper plate until the gap between the two plates is about 0.5 mm, **❻-c**.

❺

❻-a

❻-b



❻-c

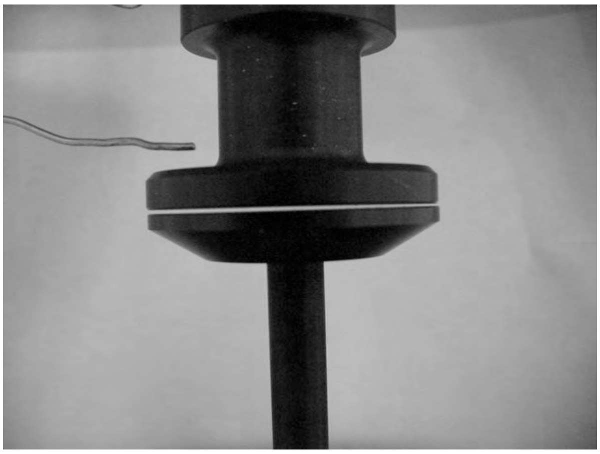
ALA

(6) Check visually in the x and y directions whether the upper and the lower plate are flush with each other, ❼**-a** and ❼**-b**. Using the knobs **1** and **5** adjust the x and y positions so that the plates are flush, ❽**-a** and ❽**-b**.

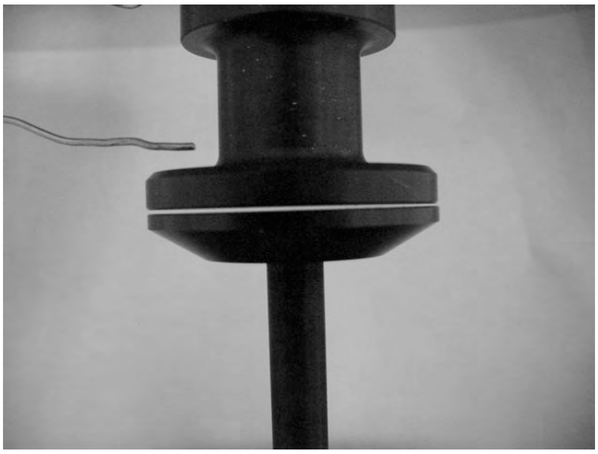
♣ The gap is better visible if you insert a sheet of white paper behind the adjustment aid.

(7) Check visually whether the upper plate is parallel to the lower plate by looking at the gap in the x and y directions ❼**-a** and ❼**-b**. Using the knobs **2** and **3**, adjust the α and β angles so that the two plates are parallel, ❽**-c** and ❽**-d**.

♣ Knobs **2** and **3** are not mounted when you receive the DMA/SDTA861e from the factory. You have to mount the knobs first to adjust the angles.



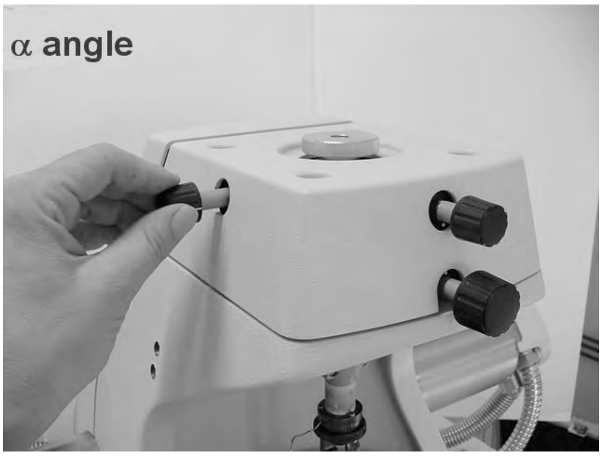
❼**-a**



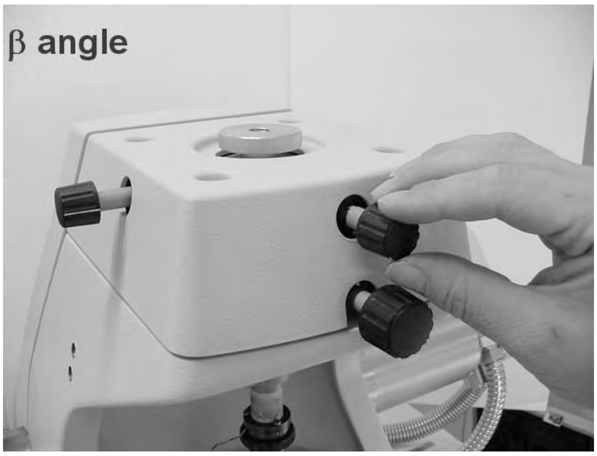
❼**-b**

❽**-a**

❽**-b**



❽-c



❽-d

❾

❿

(8) Drive the z-axis table fully downward. Depress the MOTOR DOWN key until the z-axis table stops in the intermediate position. Briefly depress the MOTOR DOWN key once more to move the z-axis table into the lower position. The z-axis table stops automatically in the lower position, ❾.

♣ The travel speed of the z-axis table changes from low to high at the intermediate position.

(9) Fasten the locking screw **4**, ❿.

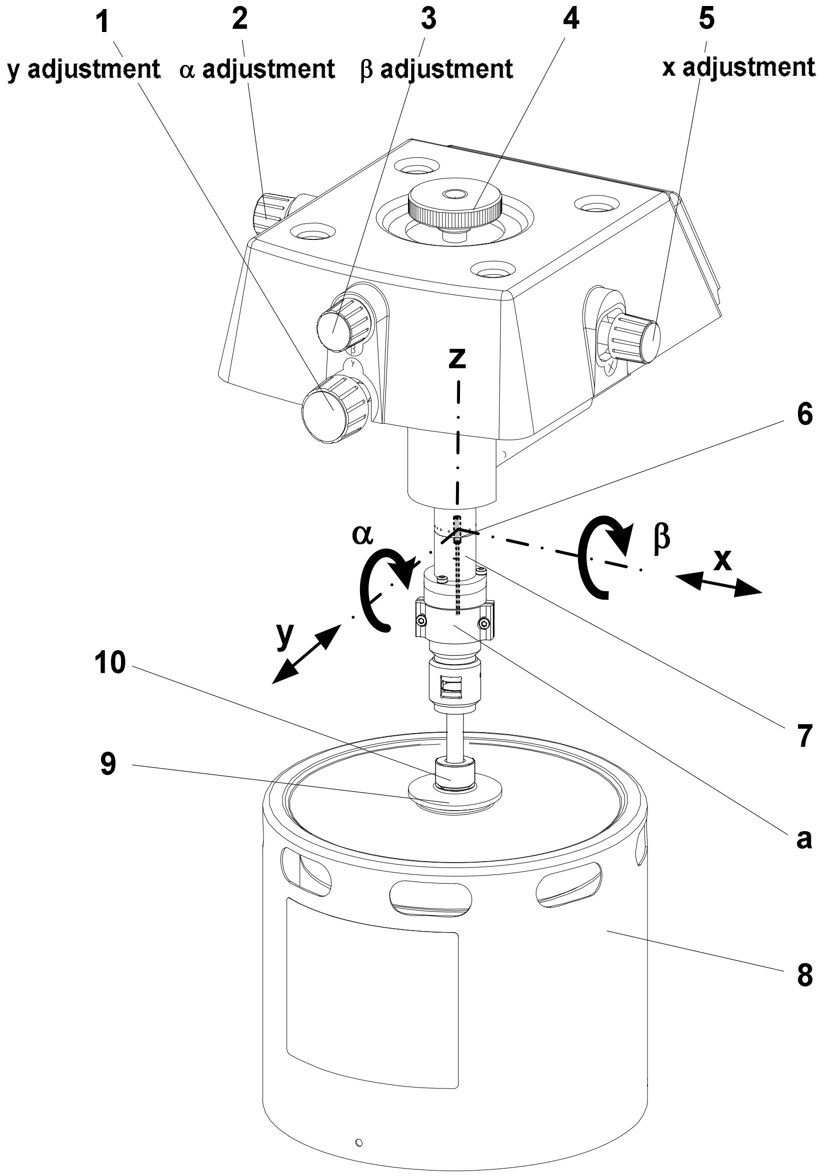
(10) Remove both parts of the alignment aid. To remove the upper half, loosen the three Torx screws on the flange of the displacement sensor core. Loosen the nut and take the lower half out of the seat of the drive motor.

End:
- Pre-alignment completed
- Alignment aid removed

8.4.2 Installing and removing the adjustment assembly

The adjustment assembly is installed in the same way as a clamping assembly. After the manual alignment with the alignment aid you have aligned the measuring sensors by adjusting the x and y positions and the angles α and β on the four axes alignment device. You may have to readjust the x and y positions while installing the adjustment assembly.

Fig. 8-5 shows the adjustment assembly installed:



1	Knob for adjusting the y position
2	Knob for adjusting the α angle
3	Knob for adjusting the β angle
4	Locking screw
5	Knob for adjusting the x position
6	Displacement sensor core
7	Displacement sensor housing
8	Drive motor
9	Threaded fitting
10	Nut
a	Coupling

Fig. 8-5. Adjustment assembly installed

AAS

To install the adjustment assembly:

➩ *See page 8-15, Fig. 8-5*

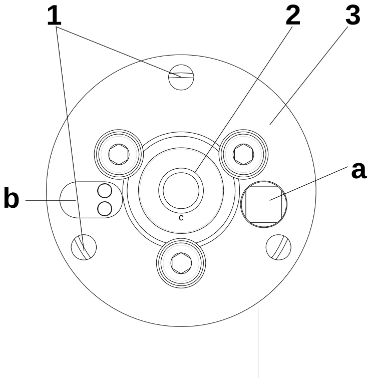
Start: Adjustment assembly is ready, coupling **3** in Fig. 8-1 is mounted

Always use the Torx screwdriver supplied to tighten and loosen the Torx screws. Using a different tool could damage the Torx screws.

(1) Hold the adjustment assembly up at an angle below the displacement sensor flange and guide the displacement sensor core **6** into the displacement sensor housing **7** by raising the entire adjustment assembly, ❶.

Installl adjustment assembly

(2) Fasten the adjustment assembly as follows to the flange of the displacement sensor, ❷:



1 Torx screws
2 Displacement sensor core
3 Displacement sensor flange
a CA Thermocouple
b Hole for SDTA Thermocouple

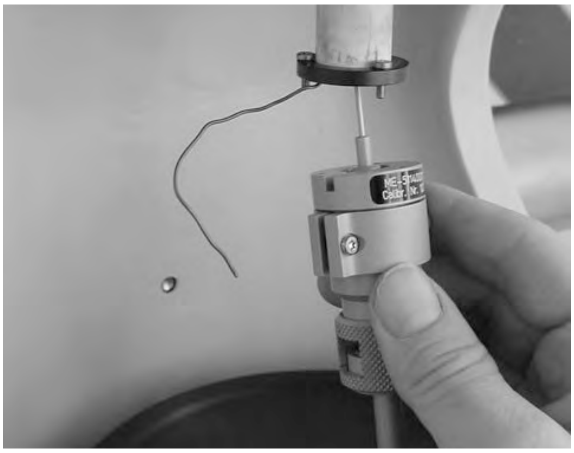
Fig. 8-6. Displacement sensor core (view from below)

- Hold the adjustment assembly under the flange of the displacement sensor. Ensure that the thermocouple projecting from hole b is positioned freely in the notch (cp. Fig. 8-6). Make sure not to wedge the thermocouple while fastening the adjustment assembly.

♣ The Torx screws must be aligned with the tapped holes in the flange of the adjustment assembly.

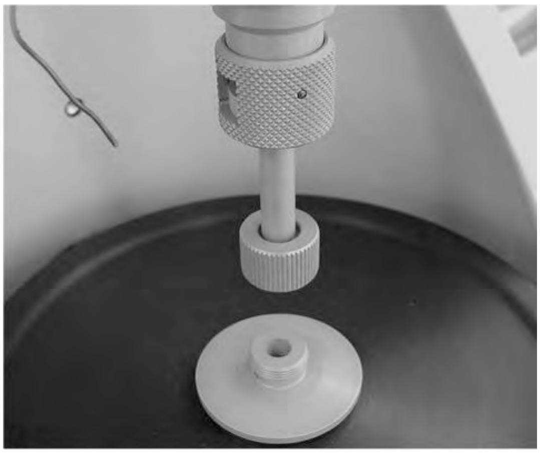
- Screw in the three Torx screws and tighten them firmly with as equal torque as possible using the Torx Allen wrench supplied.

The adjustment assembly is now fixed to the flange of the displacement sensor.



❶

❷



❸

❹

Do not drive the z-axis table too far toward the adjustment assembly

The drive motor could suffer damage.

---

(3) Drive the z-axis table upward by pressing the MOTOR UP-key once. The z-axis table stops automatically in a position in which the conical end of the drive shaft is just over the bore of the threaded fitting **9** of the drive motor, ❸ and ❹.

♣ The travel speed of the z-axis table changes from high to low in this intermediate position.

(4) If you have previously not done so, loosen the support structure of the measuring sensors as follows:

- Remove the Torx screws (A) with the Torx wrench and remove the front clamp (B). Loosen the Allen screws (C) with a 4 mm Allen wrench, ❺, but do not completely remove them so that the support structure is still attached.
- Fold the support structure back so that it is no longer in contact with length sensor flange, ❻.

(5) Loosen the locking screw of the four axes alignment device, ❼, then slightly tighten it again to the point where you feel a slight resistance.

Readjust x and y positions

(6) Readjust the x and y positions as follows:

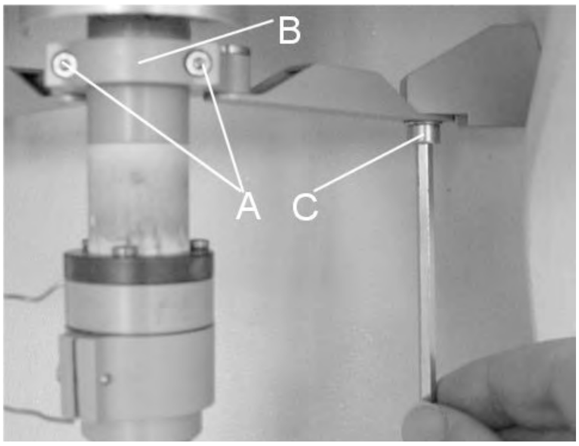
- Slide the nut **10** upward and fix it nut with the supplied paper clip, ❹, (see previous page).
- If the conical end of the drive shaft is not concentric above the bore in the threaded fitting **9** of the drive motor: Using knobs **1** and **5**, adjust the x and y positions until the conical end of the drive shaft is positioned concentrically above the bore, ❽**-a** and ❽**-b**.
- Drive the z-axis table upward carefully by pressing the MOTOR UP-key. Continuously check visually that the position of the conical end of the drive shaft in the bore is concentric. If necessary, adjust the x and y positions again.

(7) Check the play of the drive motor diaphragm before tightening the nut **10**, by pulling the diaphragm upward with the threaded fitting **9** using your fingers, ❾. Drive the z-axis table upward until the play is less than 0.5 mm. Do not pull the diaphragm while the z-axis table is moving.

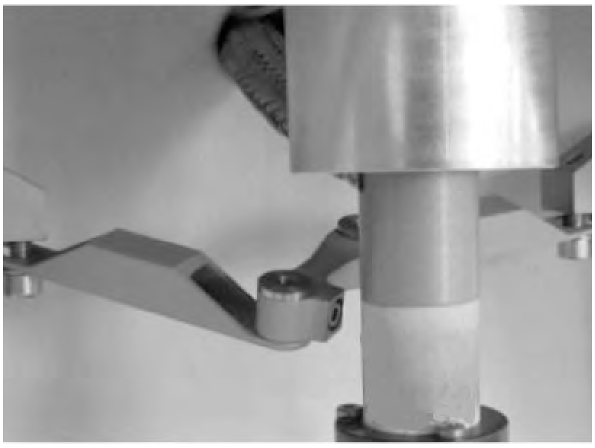
(8) Tighten the nut **10** firmly.

End: - Small clamping assembly installed
- Support structure loosened

The pre-alignment of the measuring sensors has been completed. You can now begin the fine alignment of the measuring sensors (see section *Fine alignment of the measuring sensors (four axes alignment)* on page 8-22).

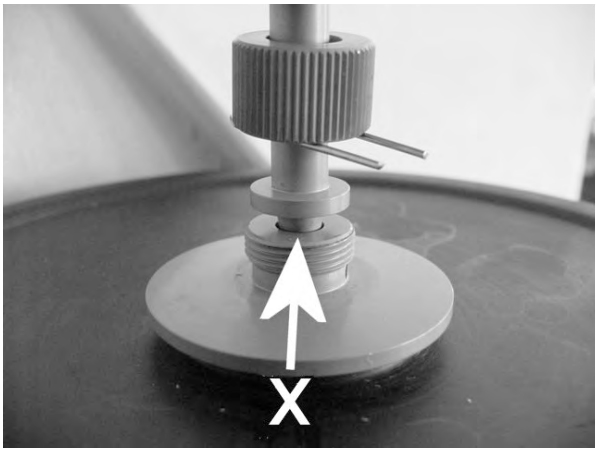


❺

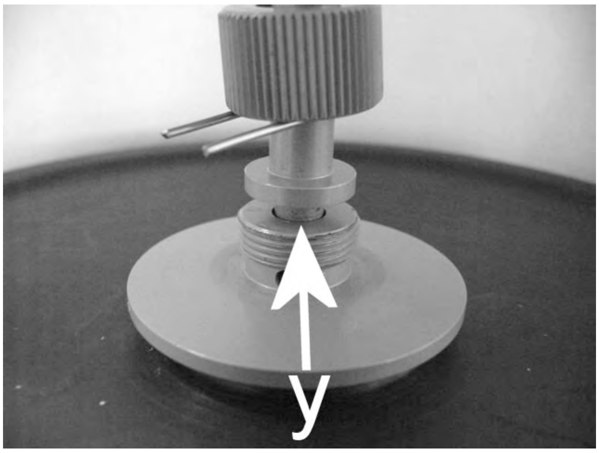


❻

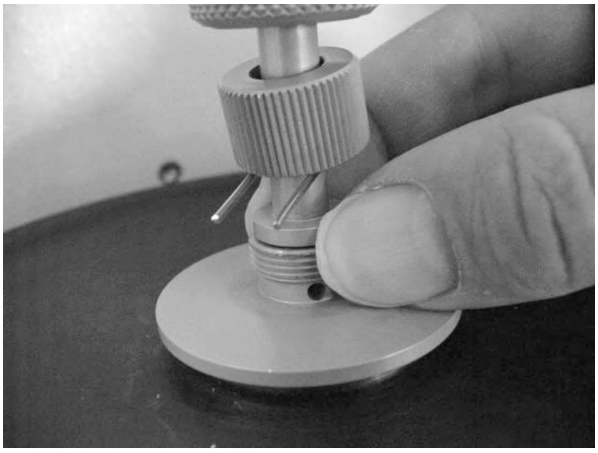
❼



❽-a



❽-b



❾

AAS

Removing the adjustment assembly

To remove the adjustment assembly from your DMA module, the coupling must be mounted.

To remove the adjustment assembly

⇨ *See page 8-15, Fig. 8-5*

Start:
- Coupling mounted
- Two halves of the furnace are fully open and tilted backward

(1) Fully loosen the three Torx screws (A) on the displacement sensor, ❶. Use the Torx Allen wrench supplied. Make sure that the adjustment assembly is completely loose and can move freely downward when you drive the z-axis table downward..

(2) Drive the z-axis table fully downward. Depress the MOTOR DOWN key until the z-axis table stops in the intermediate position. Briefly depress the MOTOR DOWN key once more to move the z-axis table to the lower position. The z-axis table stops automatically at the lower position.

♣ The travel speed of the z-axis table changes from low to high in the intermediate position.

The adjustment assembly has been driven right down with the z-axis table. There is now sufficient room above the displacement sensor core to remove the adjustment assembly.

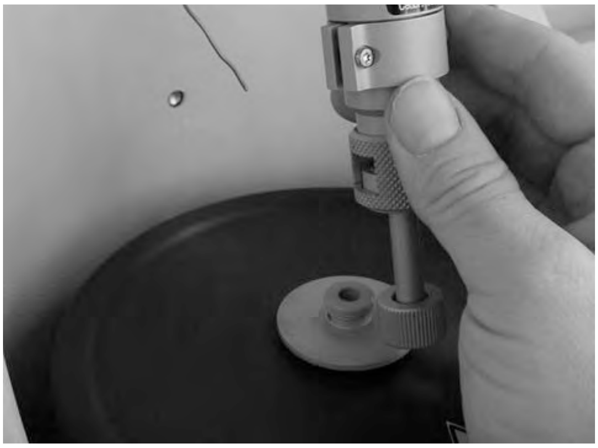
(3) Loosen the nut **10** of the drive shaft from the threaded fitting of the drive motor.

(4) Lift the adjustment assembly upward out of the drive motor fitting. Remove the adjustment assembly carefully in a downward direction. Make sure that you do not damage the displacement sensor coil and the displacement sensor core, , **❷-a** and **❷-b**.

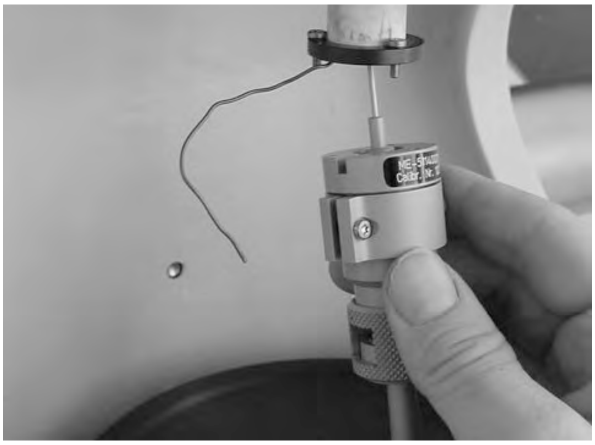
(5) Screw on the protective cover, ❸.

End: Adjustment assembly removed

❶



❷-a



❷-b

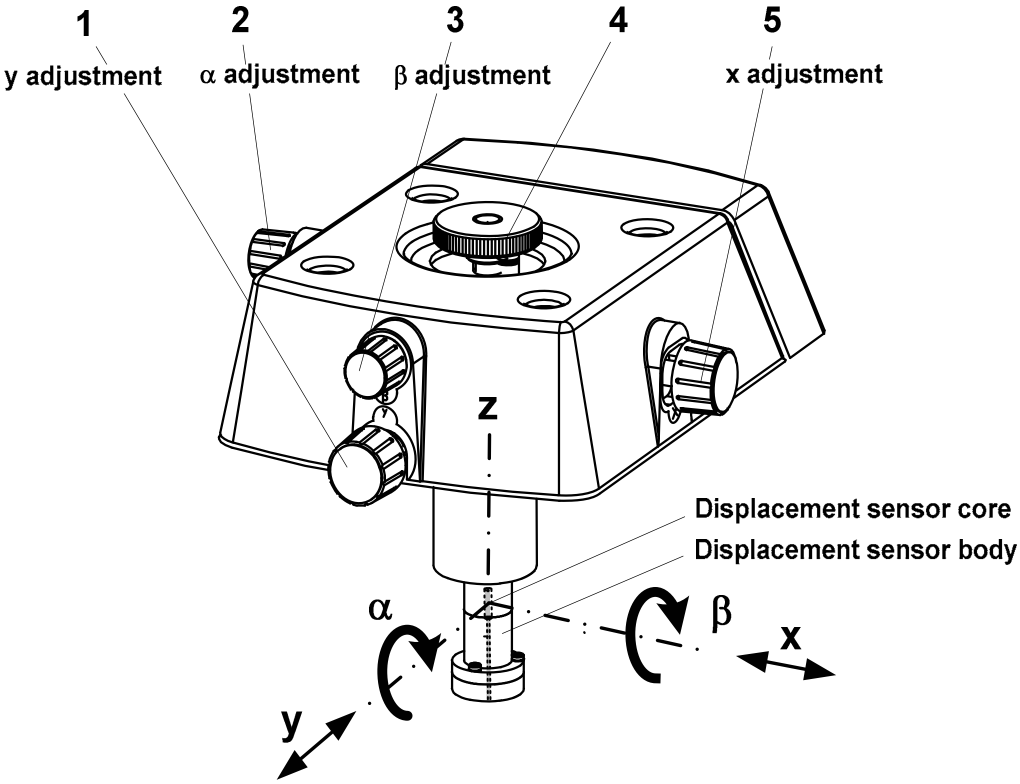
❸

8.5 Fine alignment of the measuring sensors (four axes alignment)

To measure properly, you must follow a special procedure to perform the fine alignment of the measuring system after the installation of the adjustment assembly.

By rotating the knobs on the four axes alignment device you can position the displacement sensor core within its coil.

Fig. 8-7 shows the function of the knobs on the four axes alignment device:



1 Knob for adjusting the y -position

2 Knob for adjusting the α angle

3 Knob for adjusting the β angle

4 Locking screw

5 Knob for adjusting the x position

Fig. 8-7. Knobs on the four axes alignment device

The following diagram shows the procedure for the fine alignment of the measuring sensors:

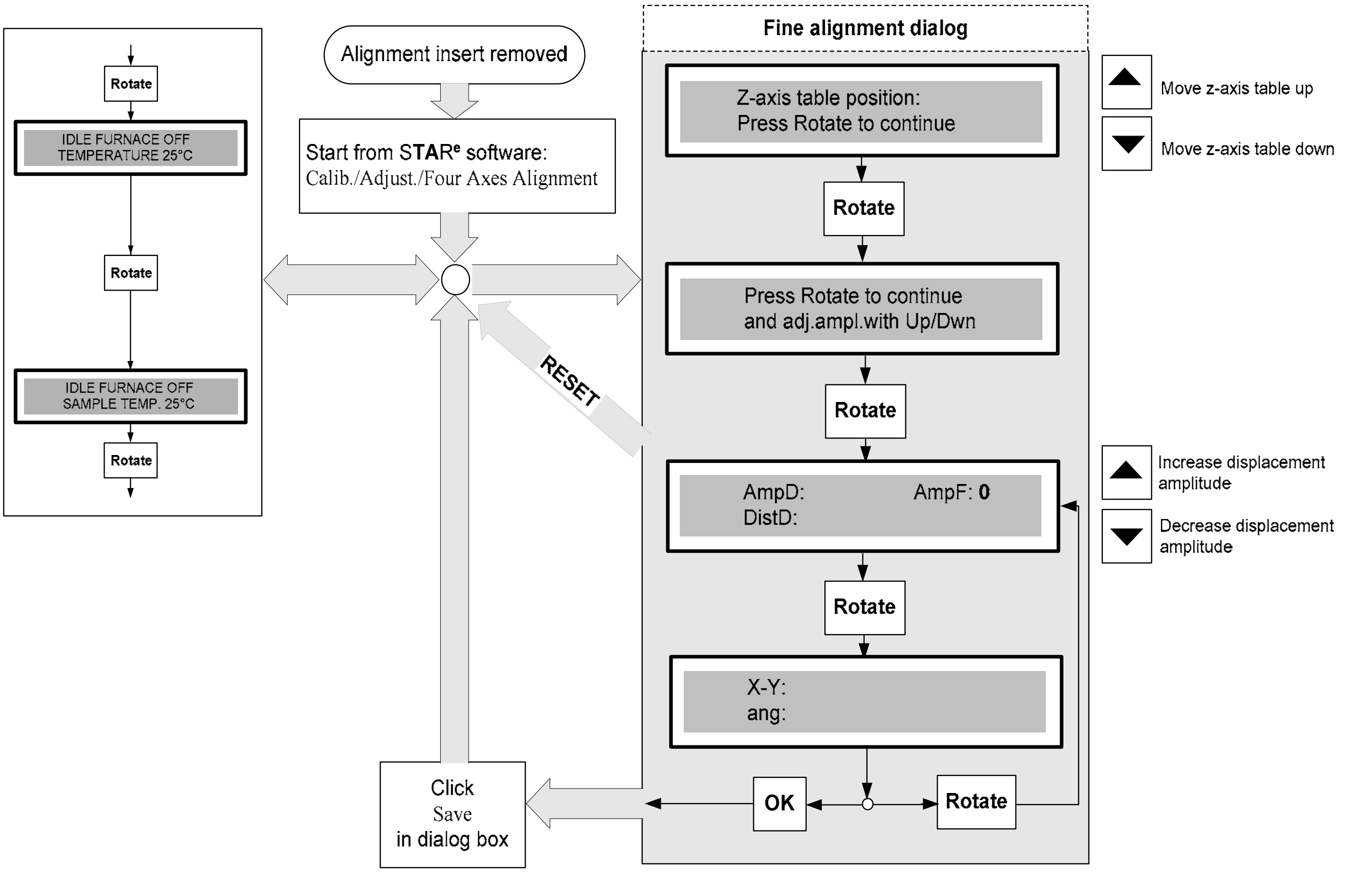


Fig. 8-8. Procedure for the fine alignment of the measuring sensors

First the x position is adjusted then, if necessary, the y position.

The α and β angles have already been adjusted during the pre-alignment with the alignment aid and need not be changed.

AAS

To perform the fine alignment of the measuring sensors:

➩ *See page 8-22, Fig. 8-7*

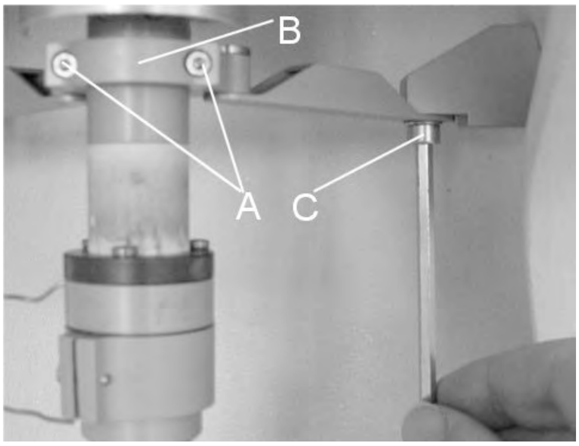
Start:
- **Furnace arms moved apart.**
- **Adjustment assembly is mounted**
- **Coupling is removed and 3.0 mm gauge block is inserted**

(1) If you have previously not done so, loosen the support structure of the measuring sensors as follows:

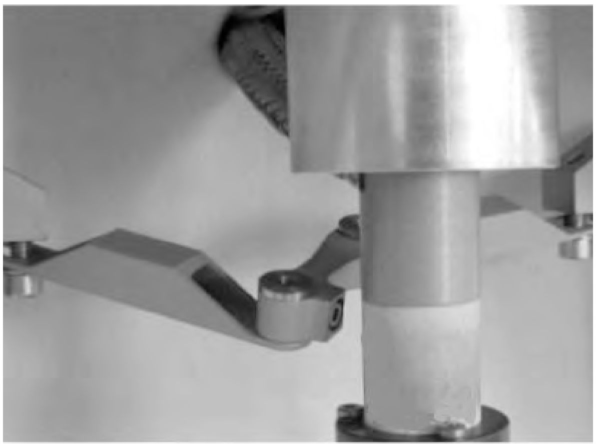
- Remove the Torx screws (A) with the Torx wrench and remove the front clamp (B). Loosen the Allen screws (C) with a 4 mm Allen wrench, ❶, but do not completely remove them so that the support structure is still attached.
- Fold the support structure back so that it is no longer in contact with length sensor flange, ❷.

(2) Loosen the locking screw of the four axes alignment device, ❸, then slightly tighten it again to the point where you feel a slight resistance.

♣ The mechanism of the 4-axis alignment device operates best when it is pressed together slightly by the locking screw.



❶



❷

❸

(3) In the STARe software Module Control Window, start the dialog for the four axes alignment procedure by choosing Calib./Adjust./Four Axes Alignment Device.

The current value of the Offset Position of the z-axis table (**z-axis table position**) is shown on the LCD display. This value represents the current position of the z-axis table and can now be changed with the arrow key.

(4) If the position of the z-axis table deviates more than 50 µm from the zero position: Move the z axis table into the zero position by pressing the MOTOR UP and MOTOR DOWN keys. The zero position is sufficiently accurate when a value between -50 µm and +50 µm is displayed.

The z-axis table is now in the zero position.

♣ The z-axis table automatically moves into the current zero position at the end of a displacement adjustment dialog.

(5) Press ROTATE to continue.

The following message appears:

Press Rotate to continue and adj.ampl.with Up/Dwn

or:

Press RESET, to return to exit the dialog. You can always exit the dialog by pressing the RESET key from this point onward without saving the alignment settings.

♣ The MOTOR UP and MOTOR DOWN arrow keys are multi-function keys. From this point onward in the dialog they serve to increase or decrease the displacement amplitude of the drive motor.

(6) Press ROTATE to proceed with the adjustment of the four axes alignment device.

The current settings of the measuring system appear on the LCD display. The drive motor produces a default oscillation with an amplitude of about 23 µm and 2 Hz.

Quality factor	Definition	Adjust to
AmpD	Current value of the displacement amplitude	400-600 µm
AmpF	Current value of the force amplitude	0 (zero) mN
DistD	Current value of the distortion factor	Minimum value

♣ If the mechanical pre-adjustment has been done well, the value of the force amplitude should be 0 (zero) mN.

AAS

⇨ See page 8-22, Fig. 8-7

(7) Increase the displacement amplitude in steps to about 400 to 600 µm by pressing the ▲ key. Take note of the force amplitude display. If the force amplitude becomes greater than 0 (zero) mN:

adjust x and y position)

- Adjust the x-position of the displacement sensor by rotating knob **5** until the force amplitude returns to 0 (zero) mN, ❺-a. Try rotating the knob in the other direction. If the force amplitude does not decrease, adjust the y-position by rotating knob **1** in the same way, ❺-b.
- In this way, increase the displacement amplitude in steps to about 400 to 600 µm, ❻.

❺-a

❺-b

❻

♣ By pressing the ▲ and ▼ keys, the value of the force amplitude changes each time by a factor of 1.3. It is therefore difficult to set an exact value. For the adjustment however it is sufficient to set a value close to the desired value.

(8) If the force amplitude is not 0 (zero) mN: press the RESET key to terminate the alignment procedure. Repeat the mechanical pre-adjustment. We recommend you to remove and reinstall the adjustment assembly for this (see section *Installing and removing the adjustment assembly*) and repeat the fine alignment procedure described in this section.

The force amplitude is 0 (zero) mN at a displacement of 400 to 600 µm.

(9) Press OK to quit the four axes alignment procedure.

(10) In the S**TA**R^e software Module Control Window, click Save to save the current adjustment data of the four axes alignment device. Then start the dialog for the four axes alignment procedure again by clicking Calib./Adjust./Four Axes Alignment Device.

(11) Press ROTATE twice on the DMA module keypad to display the values of DistD again.

The value of the distortion factor DistD should be as small as possible and always less than 30 dB.

(12) Then press OK to terminate the four axes alignment procedure.

The measurement sensors have now been aligned and the dialog returns to the starting point and displays **Idle furnace off**. A dialog box with the new adjustment data appears in the Module Control Window of the S**TA**R^e software. The data for the previous adjustment are given in brackets.

(13) In the S**TA**R^e software Module Control Window, click Save to save and activate the current adjustment data of the four axes alignment device.

(14) Fasten the locking screw **4** first, ❼, and then fasten the support structure as follows:

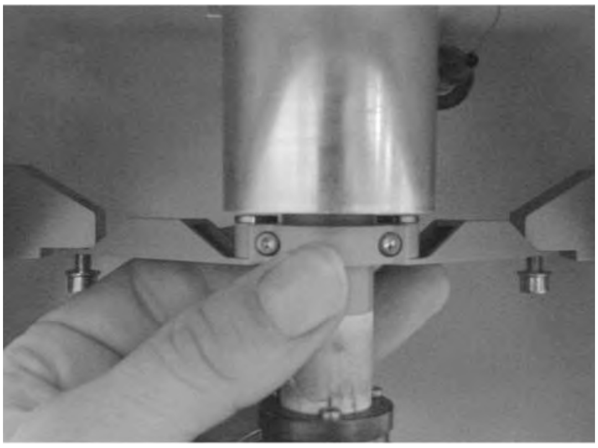
- Screw in the two Torx screws (A) and the attach the clamp (B) while holding the support structure at the same level as the two cantilevers on the stand, ❽ and ❾ (see next page).
- Fasten the Allen screws (C) with the 4 mm Allen wrench, ❾.

(15) Make a final check of the alignment as follows:

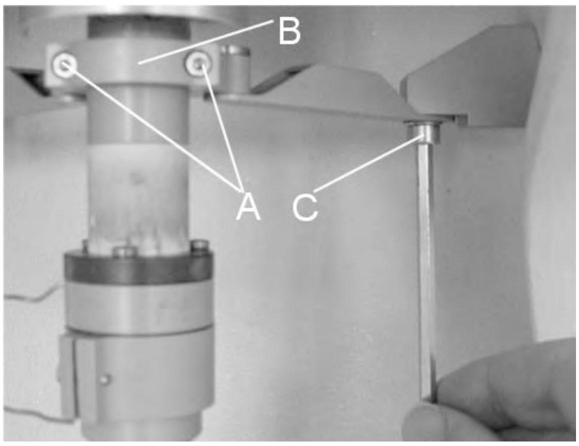
- In the STAR^e Software Module Control Window, start the dialog of the four axes alignment device again by choosing Calib./Adjust./Four Axes Alignment Device.
- Press ROTATE twice to display the settings of the measuring system and increase the displacement amplitude in steps to about 400 to 600 µm by pressing the ▲ key.
- Take note of the force amplitude display:
  - If the force amplitude is 0 (zero) mN, press RESET to exit the for axes alignment dialog without saving the adjustment data.
  - If the force amplitude becomes greater than 0 (zero) mN, repeat the alignment procedure with step (1).

End: Fine alignment completed. Measuring sensors aligned with the clamping or adjustment assembly.

❼



❽



❾

Problems that may occur while aligning the measuring sensors

Force value is not 0 (zero)

If you do not succeed in aligning the measuring sensors by the above procedure, one of the following reasons might be the cause:

- The pre-alignment on installing the adjustment assembly was not performed correctly at first.
- The X and Y positions or the α and β angles have been changed by accident.
- Some of the screws were not tightened enough during the installation of the clamping assembly.
- The adjustment assembly was not properly aligned within itself at first.

To solve the problem you and re-align the measuring sensors. To do this we recommend you to remove and reinstall the clamping assembly (see chapter 6, section *Installing and removing the small clamping assembly* or *Installing and removing the large clamping assembly*) and repeat the procedure for the fine alignment of the measuring sensors described above.

Message **Risk of damage!** appears on the LCD display

If the force sensor measures an impermissible value due to an external influence (shake or shock) the error message **Risk of damage!** may appear on the LCD display. When this message occurs the force measurement is interrupted to protect the force sensor. You can however acknowledge the message by pressing the OK key on the keypad of the module and continue with the four axes alignment. On its third occurrence however it is not possible to acknowledge the message. In this case you have to switch off the DMA module by the mains switch at the back of the instrument and restart it .

8.6 Mechanical adjustment

This section explains the procedures for the mechanical calibration and adjustment of the DMA/SDTA861^e.

There are two types of mechanical adjustment procedures for the DMA/SDTA861^e:

- Spindle and force adjustment
- Displacement adjustment

The adjustment assembly is required for the spindle and force adjustment. On installing the adjustment assembly you have to adjust the four axes alignment device. The displacement adjustment is performed with every new clamping assembly but not the with the adjustment assembly.

8.6.1 Adjusting the spindle and the force

spindle adjustment

The spindle, driven by a step motor, moves the z-axis table and the drive motor up and down. This movement of z-axis table changes the z position of the displacement sensor core in the displacement sensor coil and allows the core to be set in the optimum range for the measurement. The adjustment of the spindle must be performed so that the displacement sensor core can be positioned accurately in its coil in the subsequent displacement adjustment.

The force adjustment consists of two parts:

- Force adjustment of the drive motor
- Force adjustment of the force sensor

The following procedure allows you to calibrate and adjust the force created by the drive motor and also the force measured by the force sensor.

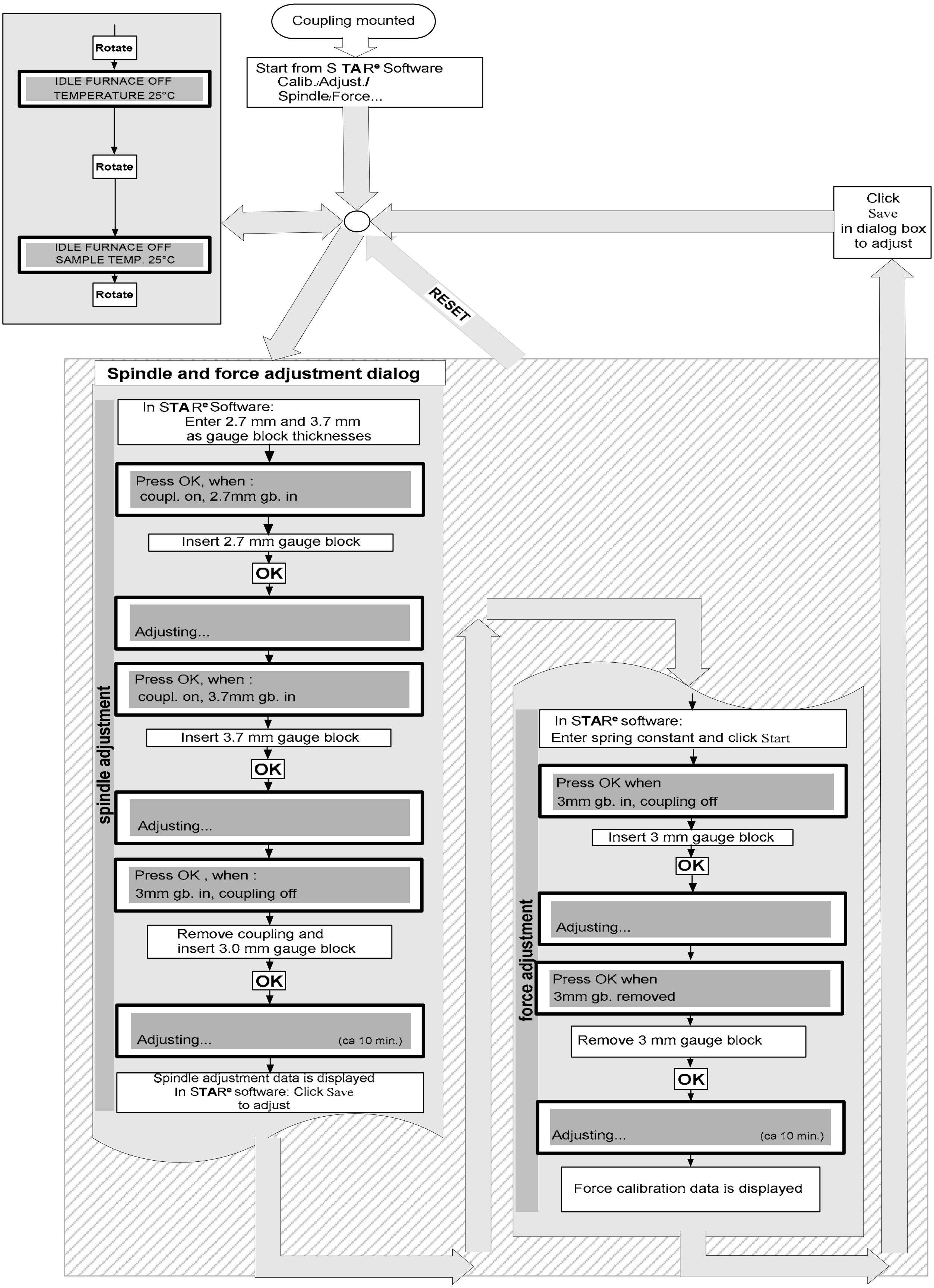


Fig. 8-9. Procedure for adjusting the spindle and the force

AAS

Notes on using the gauge blocks

gauge blocks

The 1 mm, 1,7 mm and 2 mm gauge blocks shipped with your DMA/SDTA861e are precision tools with a thickness tolerance of ± 0.0004 mm. They enable precise calculation of differences of displacement of the displacement sensor core.

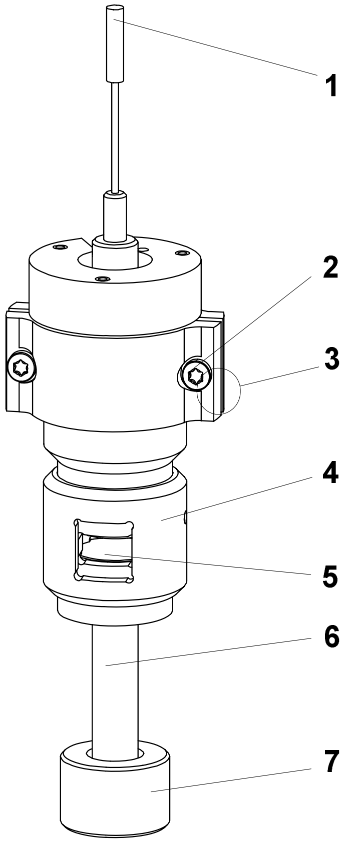
The gauge blocks must only be used in a clean and spotless state.

- Polish the gauge blocks with the deerskin cloth included in the gauge block set. Do not touch the polished surfaces after this.

♣ You can also use other precision gauge blocks with different dimensions.

- When using two or more gauge blocks: Place the gauge blocks in an angle of 90° on top of each other and bring them into good contact by rotating and pressing them together until their surfaces coincide.

The adjustment procedures are described in the following sections. The position numbers in Fig. 8-10 are used as reference.



1 Displacement sensor core
2 Torx screws
3 Coupling
4 Outer cylinder
5 Insert for gauge blocks
6 Drive shaft
7 Nut

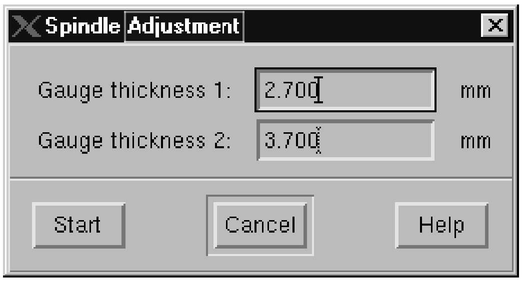
Fig. 8-10. adjustment assembly

To adjust the spindle and the force:

Start: - Adjustment assembly mounted, coupling **3** in Fig. 8-10 installed ➪ *See page 8-32, Fig. 8-10*
- Fine alignment of measuring sensors completed
- Gauge blocks ready

(1) Start the spindle adjustment dialog in the Module Control Window by choosing Calib./Adjust../Spindle/Force…

The spindle adjustment dialog begins. The following dialog box showing gauge thickness 1 and 2 appears.



♣ You can enter your own values for the gauge thickness. We recommend, however, that you use the default values (2.7 mm and 3.7 mm).

(2) Click Start.

The request **Press OK when: coupl. on, 2.7 g.b. in** appears on the liquid crystal display requesting you to insert the 2.7 mm gauge block to press OK.

(3) Mount the first gauge block with a thickness of 2.7 mm by pushing the outer cylinder **4** upward with your fingers and inserting the 2.7 mm gauge block.

(4) Press OK key.

A message appears on the LCD display that the adjustment procedure is in progress (**ADJUSTING...)**. When the adjustment procedure has been completed, you are requested to insert the 3.7 mm gauge block and to press OK (**Press OK when: coupl. on, 3.7 g.b. in**).

(5) Remove the 2.7 mm gauge block by pushing the outer cylinder **4** upward with your fingers and removing the gauge block. Insert the 3.7 mm gauge block.

(6) Press the OK key.

A message appears on the LCD display that the adjustment procedure is in progress (**ADJUSTING...**). When the adjustment procedure has been completed the request to remove the coupling, insert the 3.0 mm gauge block and to press OK appears (**Press OK when: 3mm gb. in, coupling off**).

(7) Remove the 3.7 mm gauge block by pushing the outer cylinder **4** upward with your fingers and removing the gauge block. Insert the 3.0 mm gauge block.

AAS

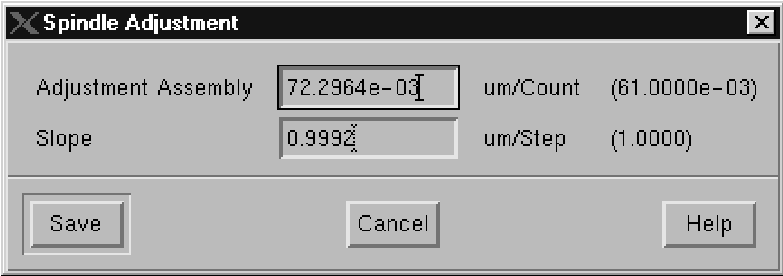
(8) Remove the coupling **3** by loosening the two Torx screws and removing both halves of the coupling.

(9) Press the OK key.

The spindle adjustment takes approx. 10 min.

The spindle is now adjusted. The z-axis table is driven to different adjustment positions. A message appears on the LCD display that the adjustment procedure is in progress (**ADJUSTING...**). When the adjustment procedure has been completed after approx. 10 minutes, a message appears on the LCD display telling you that the adjustment procedure has been completed. A dialog box showing the adjustment data appears in the Module Control Window of the S**TA**R**e** Software.

Example of adjustment data from the spindle adjustment:



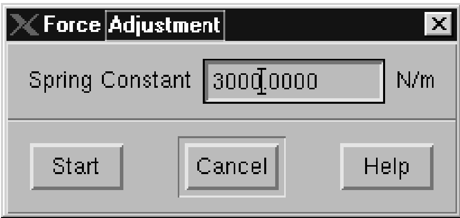
The values must be within the following ranges:

Adjustment Assembly:	0.042…0.090 µm/Count
Slope:	0.995…1.005 µm/Step

♣ If the Slope value is not in the range indicated above it is possible that the adjustment assembly is not correctly installed or the measuring sensors have not been properlya ligned. In this case we recommend that you repeat the corresponding sections in chapters 8.4 and 8.5.

(10) Click Save to adjust the spindle or Cancel to discard the adjustment data.

The spindle adjustment is completed and the adjustment of the force automatically begins by a dialog box in the Module Control Window of the S**TA**R**e** Software requesting you to enter the values for the spring constants.



(11) In the S**TA**R**e** Software, enter the correct certified value of the spring constant. The value is printed on the certificate supplied with the spring in your adjustment assembly.

♣ If nothing else is specified, we recommend that you enter a value of 3000 Nm for the spring constant.

(12) Click Start.

You are requested to insert the 3.0 mm gauge block and to press OK (**Press OK when 3mm gb. in, coupling off**).

AAS

(13) Press the OK key.

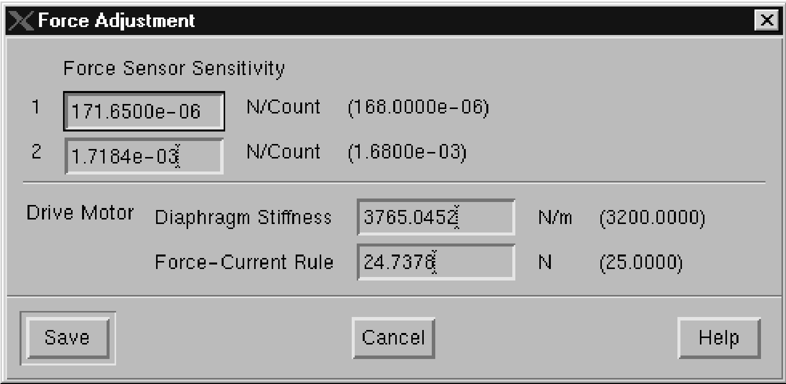
A message appears telling you that the adjustment procedure is in progress (**ADJUSTING...**). When the adjustment procedure has been completed, you are requested to remove the 3.0 mm gauge block and press OK (**Press OK when 3mm gb. removed**).

(14) Remove the 3.0 mm gauge block by pushing the outer cylinder **4** upward with your fingers and removing the 3.0 mm gauge block.

(15) Press the OK key.

The force adjustment is now performed. When the adjustment procedure has been completed after approx. 10 minutes, the adjustment data are displayed in the Module Control Window of the STARe Software.

The force adjustment takes approx. 10 min.



The values must be within the following ranges:

Force Sensor Sensitivity Range 1:	165e-06…185e-06 N/Count	x 9.85 … 10.15
Force Sensor Sensitivity Range 1:	1650e-06…1850e-06 N/Count	
Diaphragm Stiffness	2500 … 3700 N/m (20 N type)	6000 … 8000 N/m (40 N type)
Force-Current Rule	22 … 30 N (20 N type)	60 … 80 N (40 N type)

The values in the text boxes under Force Sensor Sensitivity are the sensitivity values of the force sensor in units of Newtons per AD converter count. The values correspond to two different measuring ranges. The text boxes under Drive Motor contain the characteristic values of the particular type of drive motor installed.

The Diaphragm Stiffness value is the stiffness of the drive motor diaphragm. The Force-Current Rule represents the full scale force value for the drive motor's power amplifier. **Please note that this value is slightly higher than the maximum force value specified for the drive motor to ensure that the maximum force can be realized**.

The values in brackets are the values previously saved in the database.

(16) Click Save to adjust the force or Cancel to discard the adjustment data.

End: Spindle and force adjustment completed

When you have adjusted the spindle and force you can mount the clamping assembly you require for your measurement and continue with the adjustment of the displacement (see section *Adjusting the displacement*).

CA

8.6.2 Adjusting the displacement

The displacement adjustment is the last part of the full mechanical adjustment procedure and is performed with the particular clamping assembly you require for your measurements. It also necessary to perform a displacement adjustment when you install a new clamping assembly. The adjustment data is saved in the S**TA**R**e** Software database and is always linked to a particular clamping assembly.

The displacement adjustment calibrates and adjusts the displacement sensor for the desired clamping assembly on the basis of the previous spindle adjustment. The travel of the z-axis table, which is driven by a high precision spindle, is used as reference for the displacement calibration. Additionally, the adjustment process compensates for any non-linear behavior (which is to be expected of the lvdt type sensor used) and for possible displacement offsets of the sensor's core. The internal offset compensation (IOC) values are displayed in a dialog box at the end of the adjustment procedure and have to be checked as described at the end of the following section.

Fig. 8-11 gives an overview of the displacement adjustment procedure:

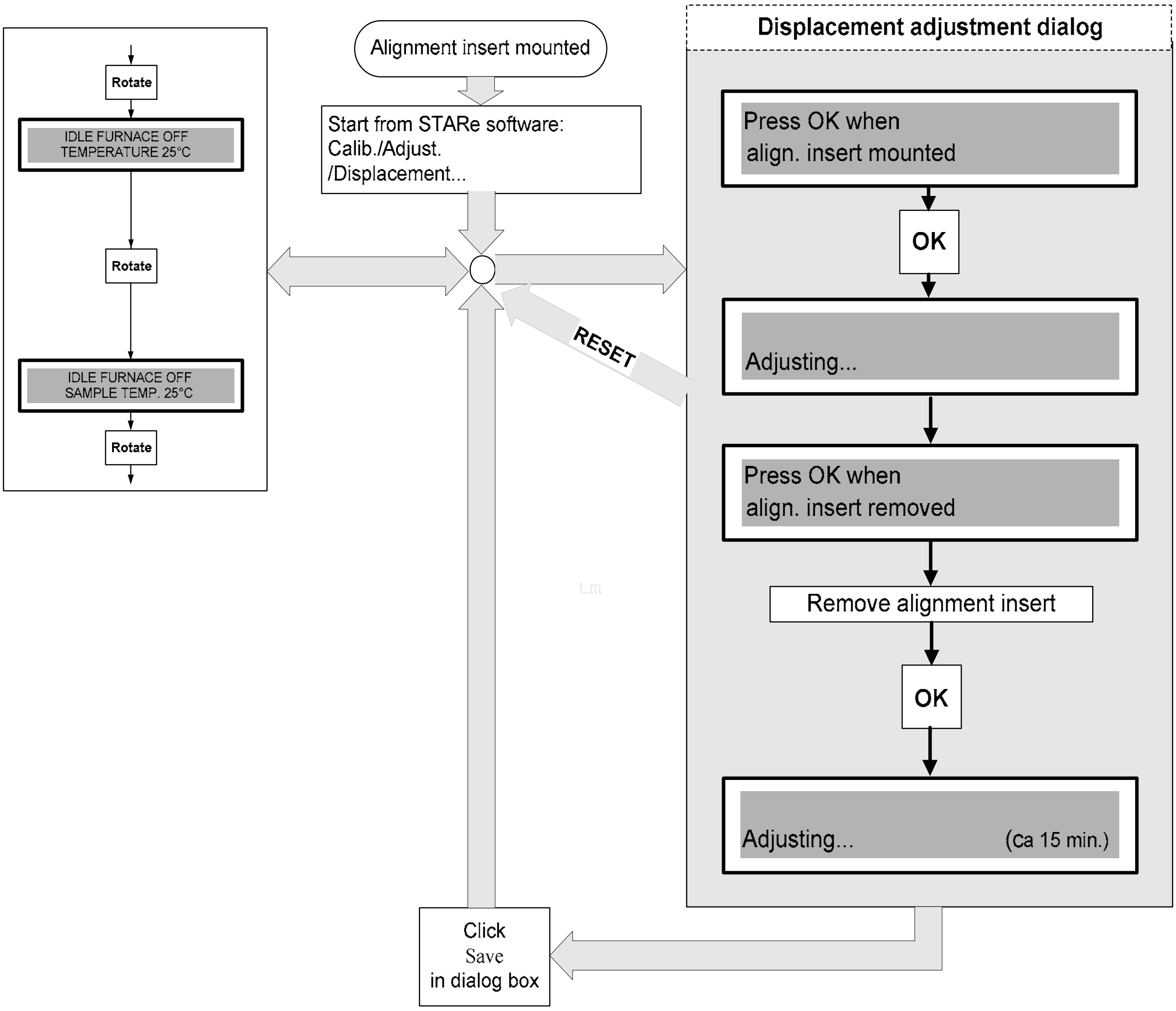


Fig. 8-11. Procedure for adjusting the displacement

To adjust the displacement:

Start:
- Clamping assembly mounted, alignment insert installed
- Fine alignment of measuring sensors completed
- Adjustment of spindle and force completed

(1) Start the displacement adjustment dialog in the Module Control Window by choosing Calib./Adjust./Displacement…

The displacement adjustment dialog begins. A message appears on the LCD display telling you to install the alignment insert (**Press OK when align. insert mounted**).

- Install the alignment insert as described in chapter 6, sections *Installing and removing the alignment insert of the small clamping assembly* or *Installing and removing the alignment insert of the large clamping assembly*.

(2) Press the OK key.

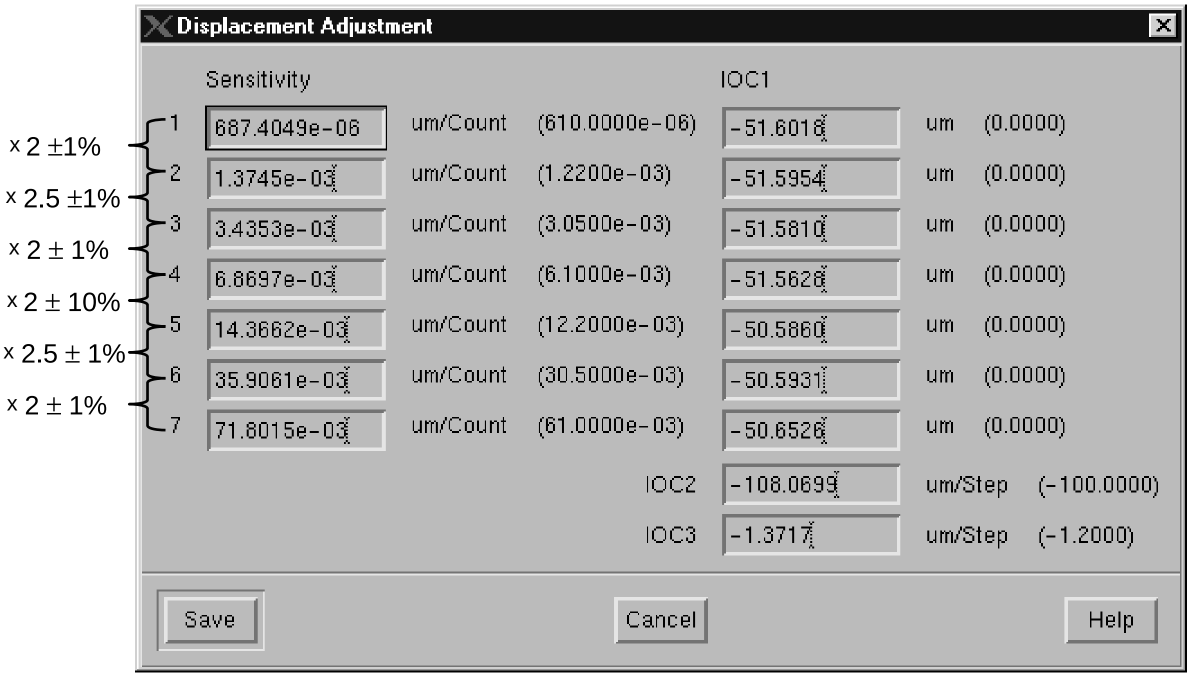
A message appears telling you that the adjustment procedure is in progress (**ADJUSTING...**). When the adjustment procedure has been completed, you are requested to remove the alignment insert (**Press OK when align. insert removed**).

(3) Remove the alignment insert as the alignment insert as described in chapter 6, sections *Installing and removing the alignment insert of the small clamping assembly* or *Installing and removing the alignment insert of the large clamping assembly*.

(4) Press the OK key.

The displacement adjustment takes approx. 15 min.

A message appears telling you that the adjustment procedure is in progress (**ADJUSTING...**). During the displacement adjustment, the z-axis table drives the displacement sensor core to different adjustment positions. When the adjustment procedure has been completed after approx. 15 min., the displacement adjustment data is displayed in the Module Control Window of the STARe Software.



The values in the text boxes under Sensitivity are the sensitivity values of the displacement sensor in units of micrometers per AD converter count. They correspond to seven different measuring ranges. if the adjustment result is good, the factors including the tolerances given on the left (2±1%, 2.5±1%, 2±1%, 2±10%, 2.5±1% and 2±1%) should apply for ratios of the sensitivity values.

The values in the text boxes under IOC1 (internal offset compensation) are the displacement offset values in units of µm with respect to the geometric zero position of the displacement sensor core which are used for internal mechanical offset compensation of the sensor core's position. There is an offset value for each measuring range to take account of different influences on the offset.

The values must be within the following ranges:

IOC 1: -200 µm … +200 µm, where deviation of values should be < 50 µm

IOC 2: -120 µm … -75 µm

IOC 3: -1.5 µm … -0.9 µm

In the boxes next to the text IOC2 and IOC3 are values that are used internally for the zero position adjustment of the offset compensation procedure. They are given in units of micrometers per offset compenstion value step.

The values in brackets are the values previously saved in the database.

(5) Click Save to adjust the displacement or Cancel to discard the adjustment data.

End: Displacement adjustment for desired clamping assembly completed

The z-axis table moves into the 0 (zero) position at the end of the adjustment procedure after you have confirmed the adjustment data with Save.

8.7 Temperature calibration and adjustment

Temperature calibration and adjustment can be performed with the DMA/SDTA861e to achieve accurate readings of sample temperature.

A temperature adjustment of your DMA/SDTA861e has already been performed in the METTLER TOLEDO factory for the clamping assembly you ordered. The sample temperature readings in measurements with this clamping assembly are accurate after this temperature adjustment and the temperature readings do normally not need to be readjusted.

If you have additionally ordered a different type of clamping assembly, however, we recommend you to perform a temperature adjustment yourself for this clamping assembly to be sure to obtain accurate temperature readings with both the small and the large clamping assembly.

The temperature adjustment consists of two stages:

1 Adjustment of the clamping assembly temperature

2 Adjustment of the sample temperature

Sample temperature adjustment is usually performed with the shear clamp in the small clamping assembly and the bending clamps (3-point bending, dual and single cantilever clamps) in the large clamping assembly. It is also possible to perform the sample temperature adjustment with other clamps but not recommended with the large tension clamp.

To carry out the temperature adjustment the Install Plus software option has to be installed with the S**TA**R**e** Software.

The following sections explain the procedures to adjust the sample temperature and the clamping assembly temperature.

Temperature adjustment of the liquid nitrogen Dewar.

For optimum dosing and accurate temperature control, the liquid nitrogen flow from the Dewar has to be adjusted. The adjustment procedure is explained in chapter 7 in section *Adjustment of liquid nitrogen flow*.

8.7.1 Temperature readings in the DMA module

The following temperatures are measured by the temperature sensors in the DMA module:

- SDTA temperature
- Clamping assembly temperature (CA temperature)
- Cooler temperature
- Furnace temperature

Fig. 8-12 shows the positions of sensors for the different temperature readings:

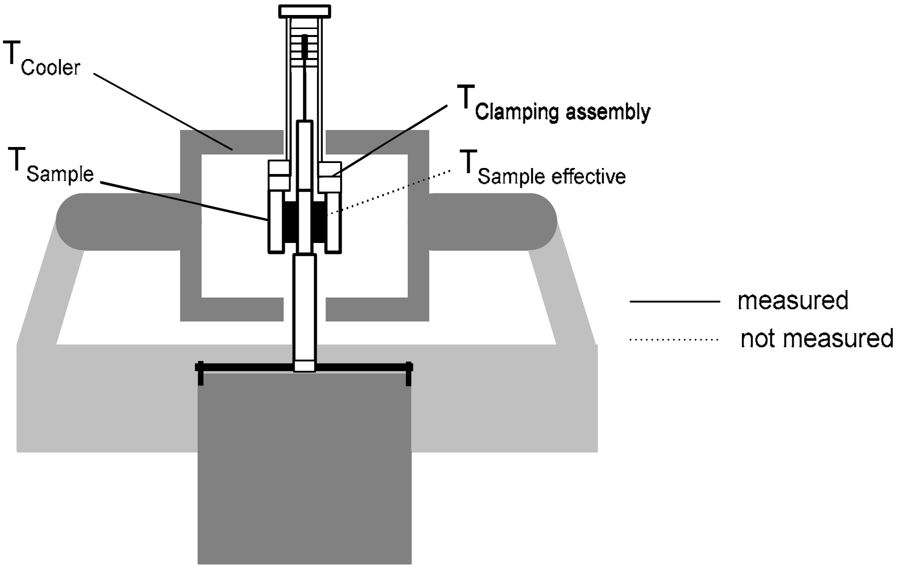


Fig. 8-12. Measured temperatures in the DMA/SDTA861e

The clamping assembly temperature is measured at the top of the clamping assembly in the flange of the displacement sensor with a K-type thermocouple. This temperature reading is used as the controlled variable in the temperature control loop.

The furnace temperature is measured on the surface of the heating element in the furnace by a K-type thermocouple. The cooler temperature is measured in heat exchanger in the furnace by Pt100 temperature sensor. The furnace temperature and cooler temperature readings are used as correcting variables in the temperature control loop.

♣ This is different than on the other TA modules: The DSC822e, the TGA/SDTA851e and the TMA/SDTA840 e use the furnace temperature as the controlled variable in the temperature control loop

8.7.2 Calibrating and adjusting the clamping assembly temperature

The adjustment of the clamping assembly temperature is performed before the sample temperature adjustment. To adjust the clamping assembly temperature, referred to as “CA temperature” in the following, sample temperature readings are used as reference values.

The CA temperature is used as the controlled variable in the temperature control loop. The purpose of the CA temperature adjustment is to ensure that the CA temperature is close as possible to the reference temperature given by the temperature program of the method. It will however not improve the accuracy of the sample temperature reading.

The clamping assembly temperature adjustment should be performed for each clamping assembly used.

A calibration run is performed and the clamping assembly temperature is adjusted in the STARe Software. The values of the CA temperature are entered in the Single Temperature Adjustment dialog box in the Module Control Window that can be called up on the Calib./Adjust. menu.

Running the calibration measurement for the adjustment of the clamping assembly temperature

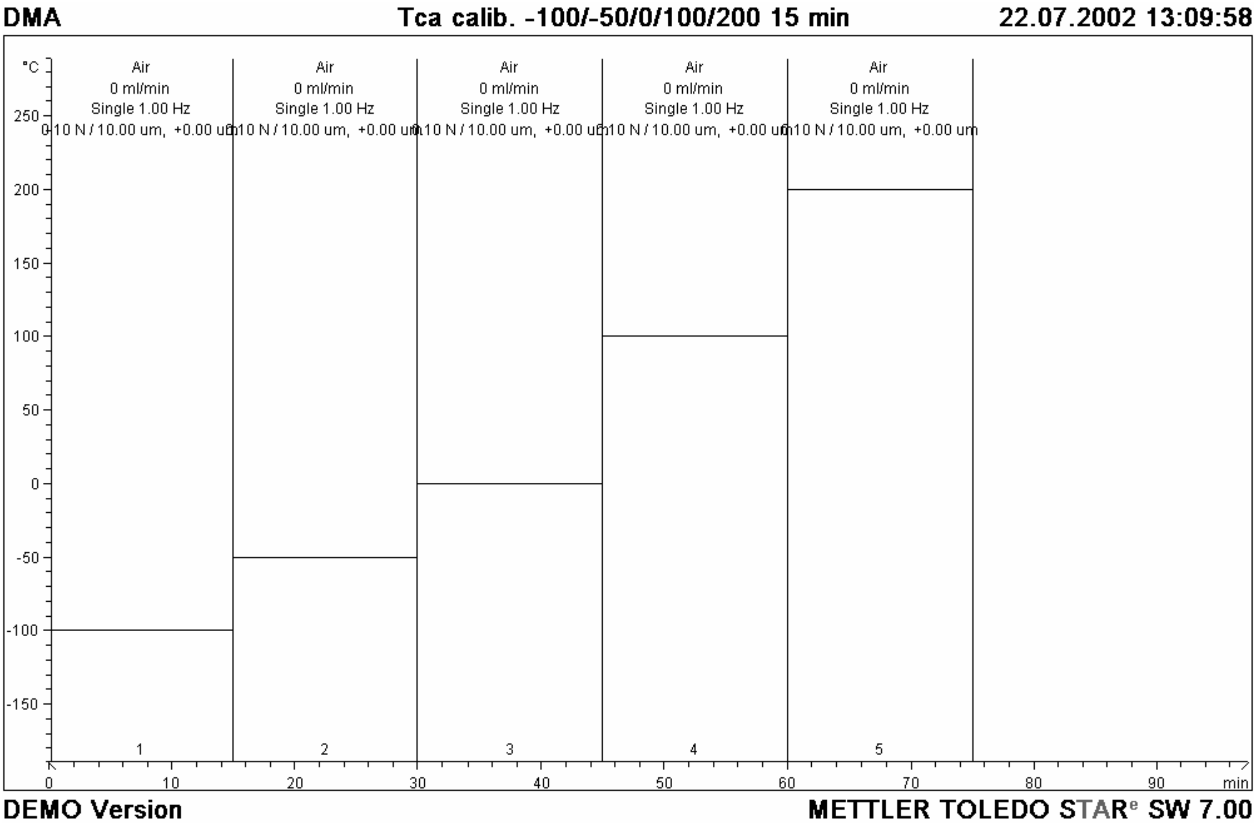
As in the sample temperature adjustment procedure, you have to run a calibration measurement before you can adjust the CA temperature. Isothermal segments that cover the desired measurement range are used in the temperature program of the method.

To run the calibration measurement:

- Create a method with isothermal segments that cover the desired measuring range. Define the segments as follows
  - Specify at least 15 min duration of each isothermal segment
  - Activate settling in every segment with the Time parameter set to 15 min.

Example:

– Temperature program in a method used for CA temperature adjustment within a typical measuring range from –100°C to 200°C.



- Run the calibration measurement with a typical sample for the desired measuring range. In the experiment, use a method with isothermal segments covering the desired measuring range as shown below. Set displacement and force amplitude values in the method that are typical for the sample.

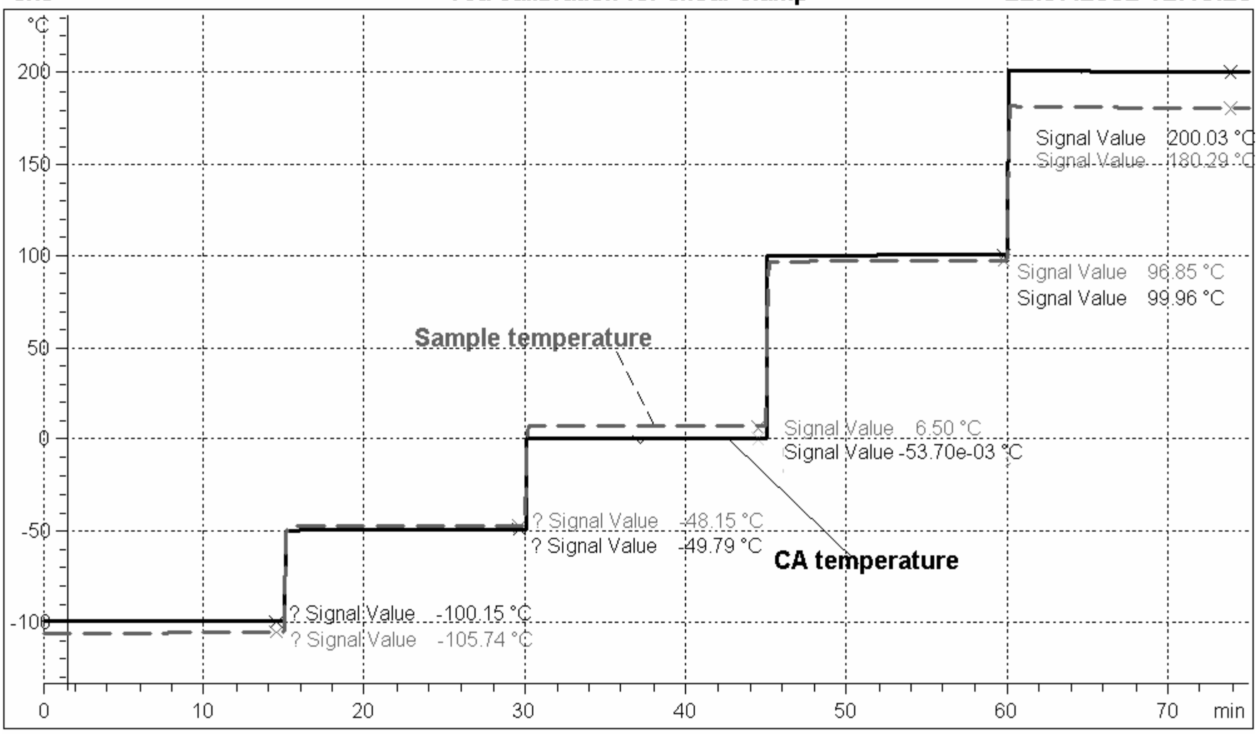
Adjusting the CA temperature

After you have run the calibration measurements with a typical sample you can adjust the CA temperature in the S**TA**R**e** Software. The Evaluation Window, the Install Window and the Module Control Window are used for the adjustment procedure.

To adjust the CA temperature:

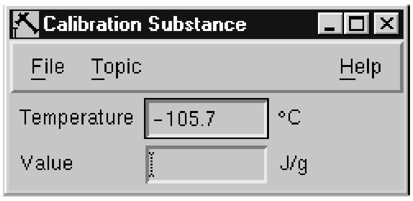
(1) In the Evaluation Window, display the sample temperature and the CA temperature versus time.

(2) With the Signal Value command on the TA menu, display the temperature values at the end of each segment.

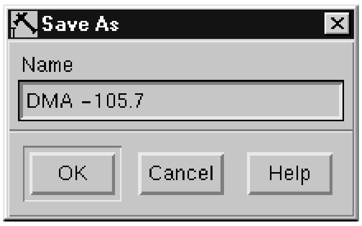


(3) In the Install Window, click the Calibration Substance tab and proceed as follows.

- Define a new calibration substance for the sample temperature of an isothermal segment and enter the sample temperature value from your previous evaluation in the Temperature box (rounded-off values to one-decimal place are sufficient).

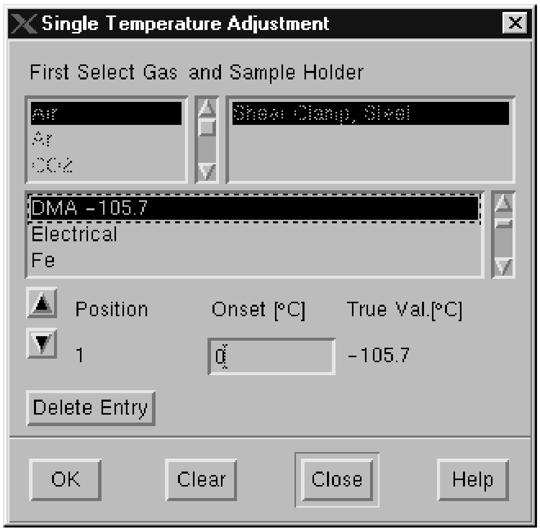


- Save the value as a “calibration substance” in the database. Include the value of the sample temperature in the name of the calibration substance.



- Repeat this for each isothermal segment.

(4) In the Module Control Window open the Single Temperature Adjustment dialog box from the Calib./Adjust menu.



♣ Further information about this dialog box can be found in the STARe Software User Handbook in chapter 8, *Calibration and Adjustment*.

(5) First select the gas type and clamp type, then the calibration substance in the corresponding boxes.

(6) Enter in the Onset box the values of the CA temperature determined in the evaluation in step (2) for each isothermal sample temperature (the entry in the above example would be 100.2).

(7) Click OK to adopt the values and to adjust the CA temperature.

The CA temperature has now been adjusted. The new adjustment parameters can be displayed in the Install Window under Topics/Module/Adjustment Overview.

Tips

- You can use the same CA temperature readings obtained from the calibration measurement for all the clamps of the small clamping assembly. With the large clamping assembly, CA temperature readings from the same calibration measurement can only be used for the clamps of the bending mode. Separate CA temperature calibration measurements must be carried out for the tension and compression clamps of the large clamping assembly. You can enter the data as explained above in steps (4) through (7) for the new clamp. The corresponding clamping assembly has to be installed and linked to a module dataset in the STARe Software Installation Window first.
- Because of the small heating rates and long time constants Tau lag adjustment is not necessary in DMA experiments.

8.7.3 Calibrating and adjusting the sample temperature

The sample temperature calibration and adjustment is performed with standard reference samples, for example, of water and indium.

The overall procedure is similar to the procedure used on other S**TA**R**e** TA modules. The reference samples are measured and the temperature readings of the fusion points are obtained by onset evaluations in the Evaluation Window of the S**TA**R**e** Software. The values of the fusion points are entered in the Single Sample Temperature dialog box in the Module Control Window.

Preparing the water and indium samples for the temperature adjustment

The reference samples water, H_2O, and indium, In, have to be prepared in different ways because of their different properties. The sample preparation procedure also depends on the clamping assembly you are using. The shear clamp is used for the small clamping assembly and the 3-point bending clamp is used for the large clamping assembly.

Small clamping assembly: The instructions to disassemble the shear clamp and remove the previous sample can be found in chapter 6 in section *Disassembling the shear clamp and mounting the sample* and section *Installing and Removing the small clamp*.

Large clamping assembly: The instructions to remove the previous sample and disassemble the bending clamp, can be found in chapter 6 in section *Removing the bending clamp and sample* and section *Installing the bending clamp with the sample in the large clamping assembly.*

The following sets of accessories required to prepare the samples for the temperature adjustment are included in the accessories case shipped with your DMA module (see also chapter 12, *Accessories*):

- Adjustment set for temperature adjustment with small clamping assembly and shear clamp
  Order number: ME 51 141 530
- Adjustment set for temperature adjustment with large clamping assembly and bending clamp.
  Order number ME 51 141 531

To prepare the water reference sample for the small clamping assembly

Start:
- Shear clamp in parts
- Sample removed
- Surfaces of sample supports and end disk clean
- Standard water samples, O-rings and syringe ready (included in the accessories set)

See chapter 6, *Disassembling the shear clamp and mounting the sample*

(1) Mount two O-rings like other samples between the disks of the shear clamp, ❶, ❷ and ❸.

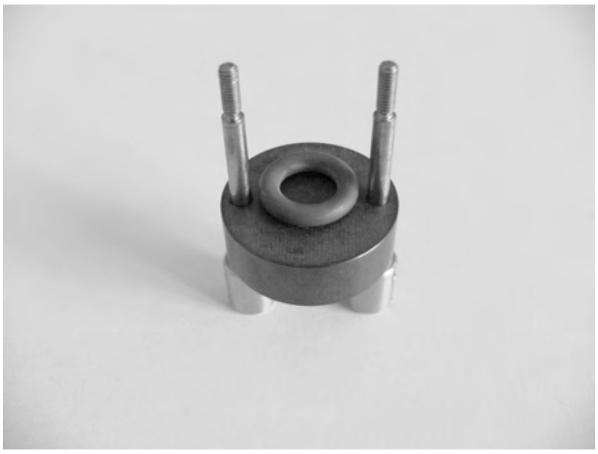
(2) With the syringe, pierce a hole in each of the two O-rings.

(3) Fill the syringe with water from one of the water samples included in the accessories set.

(4) Pierce the syringe into the side of the O-ring and inject the water into the inner space of the O-ring until the water penetrates through the first hole, ❹. Repeat this for the second O-ring.

♣ The O-ring has to be completely filled with water and all the air must be allowed to escape through the first hole.

End:
- Water sample mounted in shear clamp



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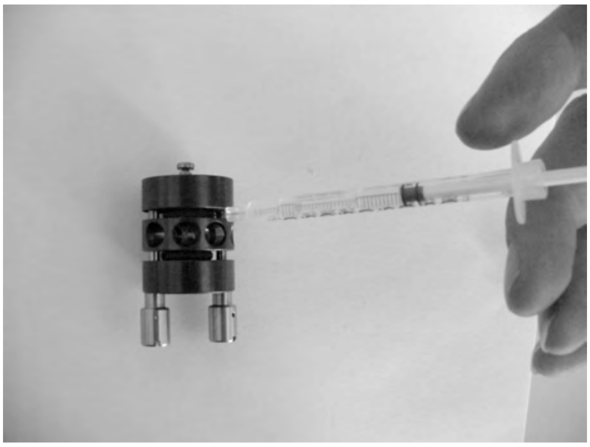

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To prepare the indium reference sample for the small clamping assembly

Start:
- Shear clamp in parts
- Sample removed
- Surfaces of sample supports and end disk clean
- Standard indium wire samples, 30 µl alumina pan ready (included in the accessories set)

See chapter 6, *Disassembling the shear clamp and mounting the sample*.

(1) Prepare the indium samples as follows:

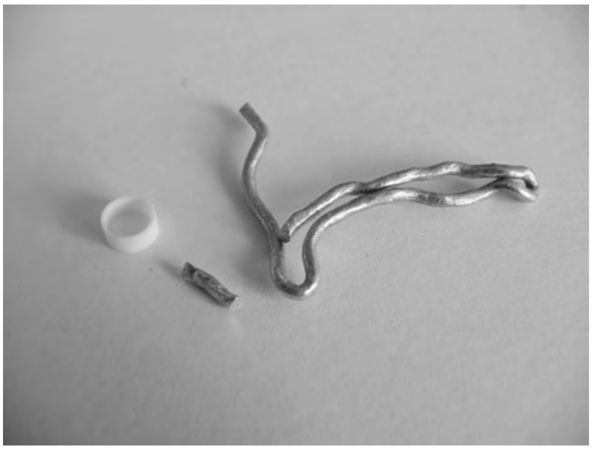
- Take a small piece of the supplied indium wire and press it well down into one of the supplied 30 µl alumina pans, ❶.
- Repeat this until the pan is filled to the brim with indium. Be sure to press the indium pieces well down so that there are no air gaps left, ❷.
- Make the surface of the sample as level as possible by applying some emery paper
- Fill the second pan in the same way.

(2) Mount two pans filled with indium like other samples between the disks of the shear clamp as follows:

- Place the pans on the sample support disks with the open side of the pan showing upward so that the indium sample is as close as possible to the SDTA thermocouple, ❸ and ❺.
- Insert a piece of alumina foil between each sample and the adjacent clamp disk to avoid direct contact between the sample and the surfaces of the clamp disks and to protect the high precison surfaces ❹.

♣ It is important to minimize possible temperature gradients between the samples in this way, in particular between the sample adjacent to the end disk and the SDTA thermocouple positioned in the end disk.

End:
- Indium sample mounted in shear clamp, ❻



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To prepare the water reference sample for the large clamping assembly

Start:
- Large clamping assembly installed
- 3-point bending clamp installed
- Standard water samples, PCB sample carrier and syringe ready (included in the accessories set)

See chapter 6, *Changing the sample in the bending clamp*

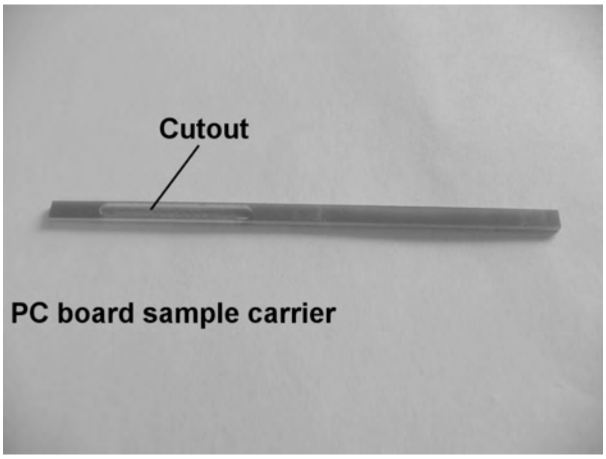
(1) Mount the PC board plate, ➊, in the 3-point bending clamp. The plate serves as a sample carrier for the reference samples, ➋.

(2) Fill the syringe with water from one of the water samples included in the accessories set.

(3) Carefully fill the cut out in the PC board plate with water from the syringe. Make the water surface level with the PC board plate's surface without letting the water overflow, ➌.

(4) Bend the thermocouple carefully to position it as close as possible to the sample without making contact.

End:
- Water sample mounted in 3-point bending clamp



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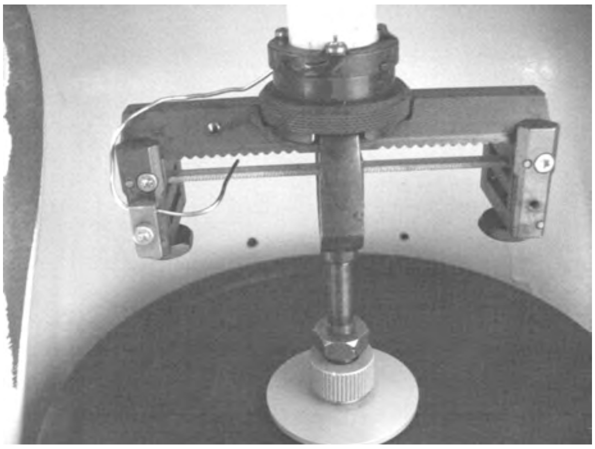

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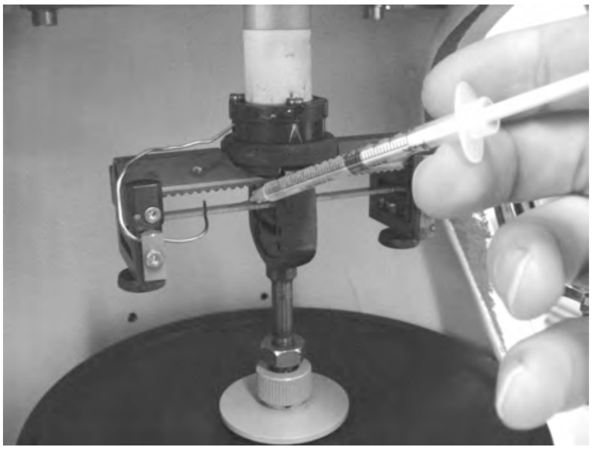

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CA

To prepare the indium reference sample for the large clamping assembly

Start:
- Large clamping assembly installed
- 3-point bending clamp installed
- Standard indium sample and PCB sample carrier ready (included in the accessories set)

See chapter 6, Changing the sample in the bending clamp

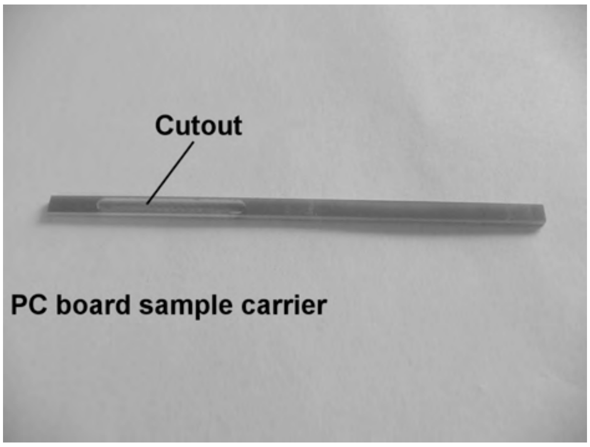
(1) Fill the cut out in the PC board plate, ❶, with indium. Make the indium surface as level as possible with the PC board plate's surface, ❷. The plate serves as a sample carrier for the reference samples

(2) Mount the PC board plate in the 3-point bending clamp, ❸.

(3) Bend the thermocouple carefully to position it as close as possible to the sample without making contact.

End:
- Indium sample mounted in 3-point bending clamp

♣ To ensure that the cut out is completely filled with indium, we recommend that you heat up the Indium sample so that it melts once before you perform the actual adjustment run.



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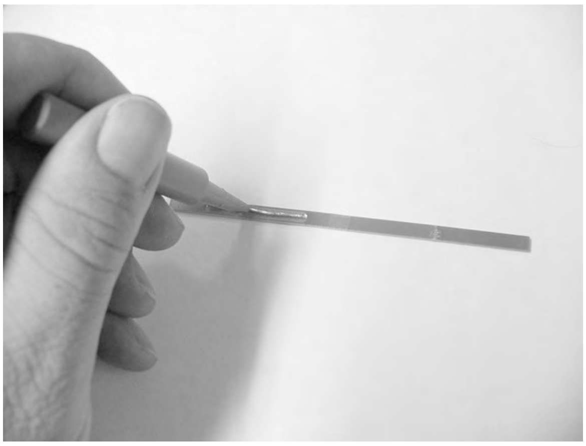

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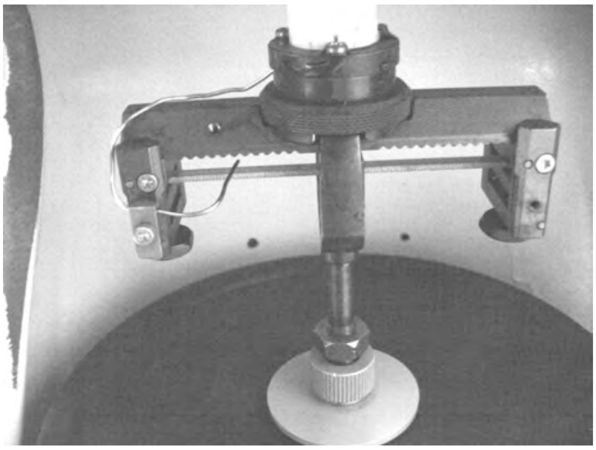

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Running the calibration measurements with the water and indium reference samples

heating rates for the calibration measurement

The fusion points of water and indium are used as reference points to calibrate and adjust the sample temperature measurement.

The temperature adjustment of the DMA module is similar to that of other modules in this respect. The method used includes a heating rate of 3 K/min which should be slow enough to make temperature gradients as small as possible.

To run the calibration measurement:

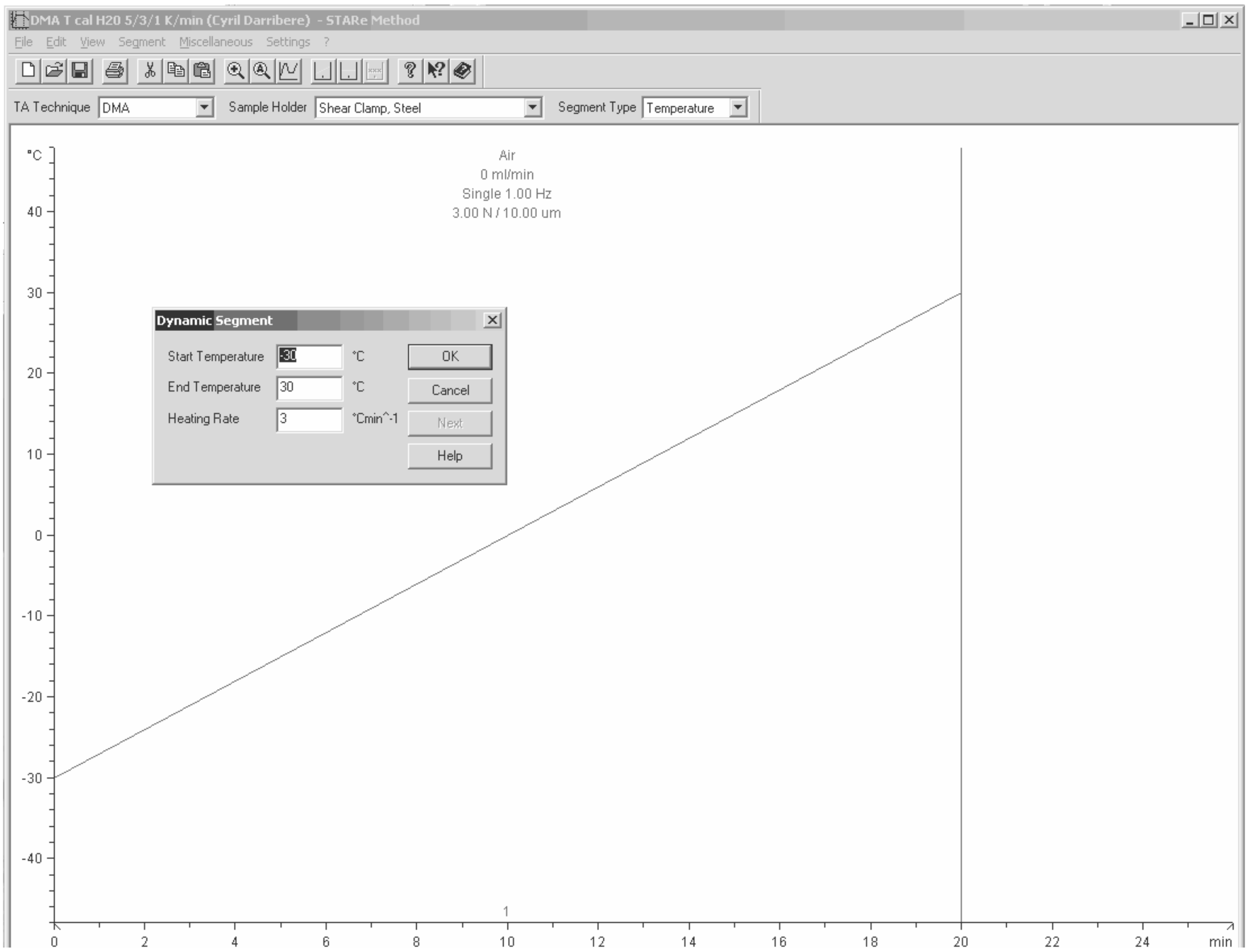
As a rule of thumb, we recommend to allow about fifteen minutes of the calibration measurement to elapse before and after reaching the corresponding fusion point. Given a heating rate,of 3 K/min, the start temperature of the temperature program can then be calculated.

method for the calibration measurement

The same method can be used for the small and the large clamping assembly to perform the calibration measurement for the sample temperature.

Example:

– Temperature program in a method used for the calibration measurement with water.



♣ If you run the indium sample for the large clamping assembly: To ensure that the cut out is completely filled with indium, we recommend that you heat up the indium sample so that it melts once before you perform the actual adjustment run.

(1) Create a method as described above. Set displacement and force amplitude values in the method that are typical for the sample.

(2) Set up an experiment in the Experiment Window using the method created in step (1).

(3) Run the calibration measurement with both the water and indium samples prepared as described in section *Preparing the water and indium samples for the temperature adjustment* for the desired clamping assembly.

Adjusting the sample temperature

After you have run the calibration measurements with the water and indium samples you can adjust the sample temperature in the S**TA**R**e** Software. The Evaluation Window and the Module Control Window are used for the adjustment procedure.

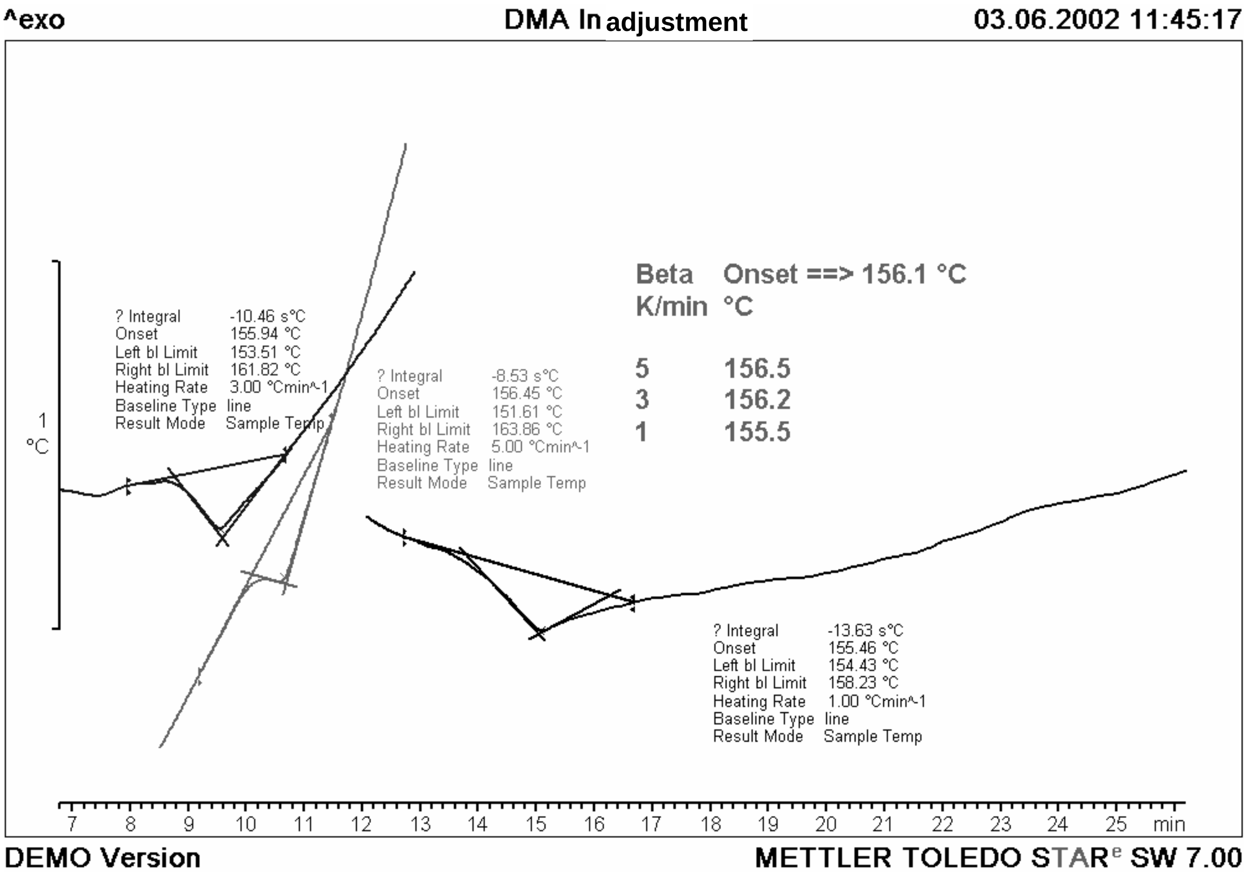
To adjust the sample temperature:

(1) In the Evaluation Window, perform onset evaluations on the SDTA curves from the water and indium measurements as follows:

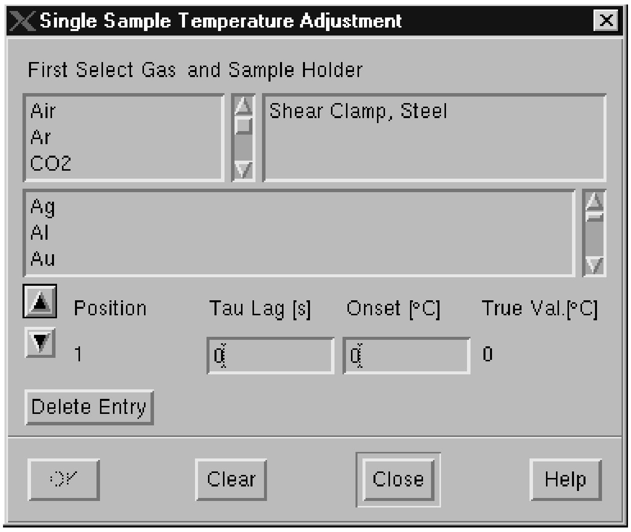
- Open the calibration measurement curve in the evaluation window.
- Display the SDTA curve versus time or reference temperature by using the commands TA/SDTA and TA/Curve vs. Time (or TA/Curve vs. Reference Temp).
- Perform an onset evaluation on the melting point peak using the command TA/Onset.

Example:

– Onset evaluations of melting point peaks of an indium measurement. Three measurement curves with different heating rates were evaluated and the average of the onset values used. Normally it is sufficient to evaluate only one curve corresponding to a heating rate of 3 K/min.



(2) In the Module Control Window call up the Single Sample Temperature Adjustment dialog box on the Calib./Adjust menu.



♣ Further information about this dialog box can be foung in the ST**A**R^e Software User Handbook in chapter 8, *Calibration and Adjustment*.

(3) Select the gas type, the clamp type and the calibration substance in the corresponding boxes.

(4) Enter in the Onset box the onset values determined from the water and indium measurements in step (1).

(5) Click OK to adopt the values and to adjust the sample temperature.

The sample temperature has now been adjusted on the basis of a 2-point calibration. The new adjustment parameters can be displayed in the Install Window under Topics/Module/Adjustment Overview.

9 Maintenance

Contents

9 Maintenance

To maintain the quality of the measurement results of your DMA/SDTA861^e and to keep the performance of the instrument within its specifications it is important that you carry out the maintenance work described in this chapter. The maintenance tasks are explained. Additionally, there are some notes on disposal of the parts.

Use only original spare parts from METTLER TOLEDO.

If you use non-original spare parts your DMA module can be damaged. METTLER TOLEDO cannot accept any liability for this.

9.1 Regular maintenance checks

The amount of maintenance work depends of course on how frequently the DMA module is used and the particular operating conditions. To keep the effort for maintenance at a minimum it is also imperative that you heed all operations procedures included in these operating instructions.

For the maintenance guidelines given here we assume that normal operating conditions are given and that the DMA module is used for its “intended use” as described in chapter 1.

Some of the maintenance tasks, such as the fine alignment procedure (four axes alignment), are included in the normal operation procedures and do not necessarily have to be performed additionally. Some of the tasks however have to be performed additionally on a regular basis.

The following table gives an overview of the maintenance tasks under normal operating conditions:

Maintenance check of	**Interval**	
Fine alignment	check once a week	
Position of furnace position	check once a week	
Reference measurement	check once a month	
Cleaning of clamps, clamping assemblies and furnace	after the measurement	
Displacement adjustment	once a month	
Spindle-force adjustment	once a year	
Replacement of fuses	when necessary	

9.1.1 Fine alignment of the measuring sensors (four axes alignment)

To produce sound measurement data the measuring sensors must be aligned properly with the clamping assembly. It is possible that the measurement sensors shift slightly from their initial position, even under normal measurement conditions.

It is important to check the alignment once a week and if necessary to readjust the position of the measuring sensor as described at the end of chapter 6 in section *Fine alignment of the measuring sensors (four axis alignment)*.

The four axes alignment procedure must be performed each time you change the clamping assembly.

When you run the fine alignment procedure we recommend that you also check the zero position of the z-axis table and, if necessary, adjust. it.

9.1.2 Control and adjustment of furnace position

For optimum signal quality, it is essential that the clamping assembly does not touch the sides of the furnace. The necessary adjustments are made when the instrument is first put into operation in the factory. In the correct position of the furnace height the marks on the rear side of the furnace arm (see photo) are flush, ❶. It is possible that the furnace arms sink slightly in the course of time so that the marks are no longer flush. If the furnace arms touch the clamping assembly it is necessary to adjust their position.

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To adjust the furnace arm position:

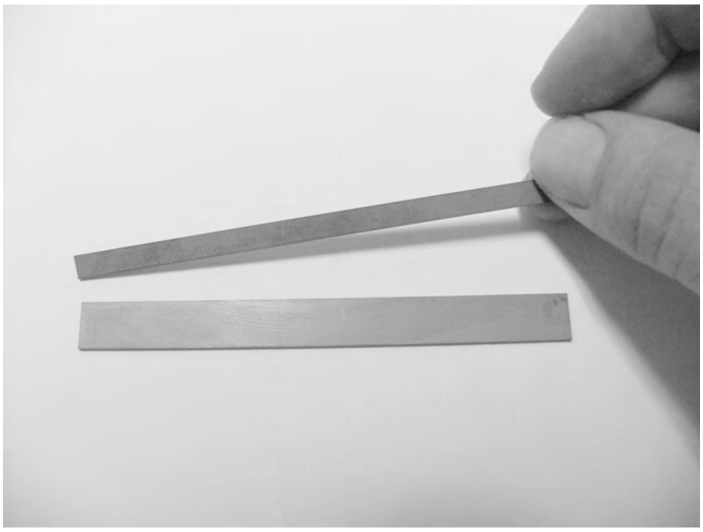
- Insert the Torx screwdriver in the hole next to the marks at the rear of the furnace arm, ❷. Rotate in the clockwise sense to lift the furnace arm, in the counter clockwise sense to lower the arm until the marks are flush.
- Check whether the furnace still touches the clamping assembly by closing the furnace halves.
- If one of the furnace halves still touches the clamping assembly when the two marks are flush, the furnace halve and the furnace arm are strongly misaligned, possibly due to mechanical impact. The two the marks then no longer indicate the correct position. In this case take the following actions:
  - Push the furnace halve manually toward the clamping assembly. The DMA module must be switched off for this.
  - Try to establish where the furnace halve is touching the clamping assembly and adjust its position as described above, ❷.

If the furnace still touches the clamping assembly after these measures or in case of uncertainty you should inform your service engineer.

9.1.3 Reference measurement

To check whether your DMA module is properly adjusted and to check the measurement accuracy it is important to perform a reference measurement with a suitable sample about once a month.

We recommend that you perform the reference measurement in the 3-point bending mode with the standard silicon samples (shown below) available from Mettler-Toledo. The modulus values for this material are well documented and are easy to reproduce.



9.1.4 Cleaning of clamps, clamping assemblies and furnace

After measurements of certain samples, for example some rubbers, it is possible that the surfaces of the clamps and clamping assembly are dirtied with sample residues. These parts should then be cleaned after the measurement with a suitable cleaning agent.

9.1.5 Displacement adjustment

A displacement adjustment should be performed once a month as described in chapter 8, section *Adjusting the displacement*. Additionally, you should always perform a displacement adjustment when you install a new clamping assembly.

9.1.6 Spindle-force adjustment

Under normal operating conditions a combined spindle and force adjustment should be performed once a year as described in chapter 8, section *Adjusting the spindle and the force*.

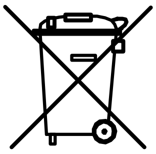
♣ If the Slope value in the spindle adjustment dialog box differs strongly from 1 it is possible that the spindle is dirtied or even damaged. In this case call your service engineer for advice.

9.1.7 Replacement of fuses

A failure of the power supply can be due to a blown fuse. In the event, the fuses must be replaced according to the specifications in chapter 11 in section *Electrical specifications*.

9.2 Disposal

At the end of its life cycle the DMA/SDTA861^e and its parts should be disposed of in an environment friendly manner. Some of the components and accessories of the instrument contain materials that must be treated as toxic waste on disposal. The following symbol at the back of the instrument reminds you that the instrument and its parts should not be treated as normal waste:



Please contact your METTLER TOLEDO service support for details and advice on how to dispose of your DMA/SDTA861^e and its accessories. The service manual to the DMA/SDTA861^e includes a list of the instrument's parts containing information about their toxic properties and the procedures for disposal. Please ask your service engineer for more details.

10 Error messages and warnings

The following error messages and warnings can appear on the LCD of the DMA module or the screen of your PC.

10.1 Error messages

Error	Cause	Measures
4	Temperature sensor failure detected (Error 23 or 25). Safety circuit has switched off the furnace power supply.	▸ Switch off power for at least 3 seconds. Safety circuit will reset when Error 23 or 25 is rectified.
5	Primary mains voltage below acceptable limit	▸ Call your service engineer.
7	The module software version does not correspond to the hardware version of the module	▸ Compare software version and version of the software indicated on the software cartridge. The software version appears on startup of the DMA module.
8	Gas controller is required and not connected	▸ Connect the cable of the gas controller to your DMA module. (See Chapter 4, Section *Setting up and making connections*)
9	LN2 cooling option is required and not connected	▸ Connect the control cable on your DMA module to the liquid nitrogen container.
17	Z-axis table step motor blocked. Occurs after two occurrences of warning 17	▸ Make sure that the clamping assembly or adjustment assembly is completely loosened from the displacement sensor flange. If necessary, remove all three screws from the displacement sensor flange. ▸ Switch DMA module off and on again.
18	Displacement sensor too hot. Can occur if the fast cooling is switched on during a temperature ramp to a high temperature	▸ Switch off the fast cooling.

Error	Cause	Measures
128…582	Internal module software errors	(1) Confirm with OK key and switch off your DMA module for at least 5 seconds. (2) Restart your DMA module. During the startup procedure the module performs an internal self test. The internal software error is automatically repaired. ▸ Please call your service engineer and report the error number.

If the following error messages occur call your service engineer and report the error number:

Error	Cause
20	DMA furnace drive control reports failure
21	Failure occurred on opening or closing the furnace halves
22	Furnace temperature out of permissible range
23…26	Error in the furnace or cooler temperature measurement
27	Power supply voltage failure
30	No Identification of your DMA module type stored in the module software
31, 32, 33, 34	Error in the memory of the module software
35	Error in the LCD display
38	SDTA sample temperature sensor error
39	SDTA sensor defective or error in DMA module electronics
40	SDTA sample temperature out of permissible range
42	Error in temperature measurement of a thermocouple
43	Error in temperature measurement of a thermocouple
51	Error in measurement of gas controller
60…81	Errors on the DMA board in the electronics. On occurrence of errors 60…71 and errors 74…78 you can try to switch off and switch on the DMA module again. If the error still occurs call your service engineer. Errors 79…80 are due to conflicts of the firmware running on the DMA module. In standby mode, error 81 may occur instead of error 18 (error 18 is usually due to a fault in the displacement sensor)
600…901	Internal module software or electronics board errors

10.2 Warnings

Warning	Cause	Measure
4	Communication error	(1) Check whether your DMA module and your PC are switched on. (2) Check RS232 cable. (3) Check ID of your DMA module on startup.
13	DMA module keypad fault	‣ Call your service engineer.
14	Start, Insert or Remove temperature was not reached within one hour or could not be maintained.	‣ Confirm the warning with the OK or the RESET key. The DMA module goes to the next stage in the experiment procedure and the message Insert Sample appears on the LCD display.
17 *	Z-table motor blocked. Movement of z-axis table motor is disabled due an impermissible force. Possibly the z-axis table is still mechanically connected to the displacement sensor via the clamping or adjustment assembly. External mechanical impact may also be the cause.	‣ Confirm with the OK key. ‣ Make sure that the clamping assembly or adjustment assembly is completely loosened from the displacement sensor flange. If necessary, remove all three screws from the displacement sensor flange.
20…39 41…54	Internal software error occurred and was repaired.	‣ Confirm with the OK key. ‣ On occurrence of errors 30...39: Try to install a new DMA module in the STAR^{e} Software Install Window
40	The four axes alignment has not been performed.	‣ Confirm with the OK key and perform the four axes alignment as explained in chapter 6 or chapter 8

* This warning occurs when an impermissible force sensor value is reached. The error message **Risk of damage!** appears on the LCD. You can acknowledge this message two times by pressing the OK key on the keypad of the module and continue with the four axes alignment. On its third occurrence however it is not possible to acknowledge the message. In this case you have to switch off the DMA module by the mains switch at the back of the instrument and restart it .

Warning	Cause	Measure
55	Module identification number ID mismatch.	▸ Check the ID number of your DMA module.
56....96	Internal software errors.	(1) Confirm with OK. The internal software error is automatically repaired. (2) On occurrence of Warnings 91...96: Try to create a new method in the STAR^e Software Method Window and install a new DMA module in the STAR^e Software Install Window (see chapter 4). (3) Please call your service engineer and report warning number.

If the communication between your DMA module and the STAR^e software is interrupted the message COMMUNICATION TIMEOUT appears on the LCD.

- Check that the RS 232 cable is connected properly from your PC to your DMA module your. Ensure that the two fastening screws on both ends of the cable are tightened properly.
- In the STAR^e software Install Window under Topics/Ports and Devices, check whether the correct port is assigned to your DMA module.
- Visually check of the plugs and sockets for oxidation and bent or broken pins.
- If necessary, replace RS 232 connection cable.

11 Specifications

Temperature		
	Range	-150 ... 500 °C
	Technical Resolution	0.003 K
	Accuracy	0.5 K
Force		
	Range	0.005 ... 40 N
		(12, 18 or 40 N depending on the drive motor option)
	Technical Resolution	0.15 mN (0 ... 5 N)
		1.5 mN (0 …50 N)
	Sensitivity	1 mN
Displacement		
	Range	+/- 1.6 mm
	Technical Resolution	0.6 nm
	Sensitivity	5 nm
Stiffness		
	Range	10 ... 10^8 N/m
	Precision	0.2 %
Tan delta		
	Range	0.0001 ... 100
	Technical Resolution	0.00001
	Sensitivity	0.0001

Frequency

	Range	0.001 ... 1000 Hz (*)
	Technical Resolution	0.00001
	Frequency steps (Δf)	0.0001
	Modes	• Logarithmic and linear scans • Multi frequency (sequentially) • Multi frequency (simultaneously)

Measurement modes

D: Diameter	**Bending:** - 3-point	L: 30 ... 90 mm
L: length	- Dual cantilever	L: 20 ... 80 mm
W: Width		W: < 15 mm T: < 5 mm
T: Thickness		Max. sample length: 100 mm
	Stiffness range in bending mode	10 … 10^6 N/m
	Shear	D: < 15 mm T: < 6.5 mm
	Stiffness range in shear mode	10 … 10^8 N/m
	Tension	L: 19.5 mm (19.5, 10.5, 5.5) W: < 7 mm T: < 3 mm
	Stiffness range in tension mode	10 … 10^7 N/m
	Compression	D: < 20 mm T: < 9 mm
	Stiffness range in compression mode	10 … 10^7 N/m

(*) deformation mode and/or sample itself can lower the upper frequency limit: shear (1000 Hz), bending (300 Hz), tension (300 Hz), compression (300 Hz)

Dimensions and Weight

Width x depth x height: 80cm x 65cm x 75cm

Weight: approx. 120 kg

Electrical specifications

Approvals

Electric Safety	S+, CSA, EN61010-1:2001 CAN/CSA - C22.2 No. 1010.1/-92
	UL Std: No.3101-1:1993
Electromagnetic compatibility	EN61326-1 :1997+A1 :98 (class B)
	EN61326-1 :1997+A1 :98 (Industrial environments)
Mark of conformity	CE

Mains Connection

Voltage	During factory assembly the module is built for 115V or 230V alternating voltage (cannot be changed!)
Frequency	50 or 60 Hz
Tolerance	+ 6% / -10%
Current consumption	115V / 12A or 230V / 6A
Maximum Charge	840 VA

Environmental conditions

Suitable location:	Only indoors at altitudes below 2000 m above sea level
Room temperature	10 to 31°C
Relative humidity	Below dewpoint
Degree of soiling	2
Overvoltage category	II

Three power line fuses are located in the line module at the rear of the DMA:

Fuse	for 115 V	for 230 V	comments
SI 1	3.15 AT	1.6 AT	Fuse for line output via power switch
SI 2	6.3 AT	3.15 AT	Fuse for line output via switched line socket
Switch	16 A	10 A	Over current protector (in switch)

In addition to the power line fuses, three fuses for the AC/DC converter are located on the front module.

Fuse		comments
SI 1	6.3 AT	Fuse for DC/DC converter
SI 2	3.15 AT	Fuse for z-axis table board
SI 3	2 AT	Fuse for de-icer

After removing the rear housing of the DMA electronics five fuses are visible (on top of line module 2):

Fuse	Fuse rating	comments
SI 3	4.0 AT	fuse for +24 V and +5 V power supplies
SI 4, SI 5	1.25 AT	fuse for ±15 V power supply
SI 6	1.25 AT	fuse for +6.5 V power supply
SI 7	0.25 AT	fuse for -10.8 V power supply

12 Accessories

12.1 Accessories list with METTLER TOLEDO order numbers

The following list contains all the accessories that are available for your DMA/SDTA861e. Items marked with an “S” in the Standard / Optional column are included in every shipment. Items marked with an “O” are optional for all configurations of the DMA/SDTA861e. All other items are specific to the particular configuration of your DMA module and are listed separately in your order. You can also order all of the listed items separately by specifying the name and the ME number in your order.

Use only original spare parts from METTLER TOLEDO.

If you use non-original spare parts your DMA module can be damaged. METTLER TOLEDO cannot accept any liability for this.

Name	Standard / Optional	ME Number
General accessories for the DMA/SDTA861e		
Operating instructions	S	51 710 068
TA Logbook	O	51 709 754
Adjustment assembly, complete (incl. Spring and certificate)	S	51 140 001
Adjustment assembly	S	51 141 055
Gauge blocks (displacement adjustment set)	S	29 062
Adjustment set for temperature adjustment with SCA and shear clamp	S	51 141 530
Adjustment set for temperature adjustment with LCA and bending clamp	S	51 141 531
Indium wire sample for temperature adjustment		51 141 528
H_20 sample for temperature adjustment		51 338 006
Rubber rings for temperature adjustment with SCA		71 529
PCB sample carrier for temperature adjustment with LCA		51 141 532
30 µl alumina crucibles, 20 pieces		51 140 843

Name	ME Number
Large clamping assembly (LCA) with protective cover and alignment insert	**51 140 003**
Hexagonal torque wrench	51 141 255
Bending beam	51 140 390
Bending clamp set for 3 point bending	51 140 026
Bending clamp set for dual cantilever- bending	51 140 025
Tension clamp, complete fixed part for LCA	51 141 270
Tension clamp for LCA, movable part for 5.5 mm sample length	51 141 263
Tension clamp for LCA, movable part for 10.5 mm sample length (with sample mounting plate)	51 141 419
Sample mounting plate for 5.5 mm sample length	51 141 272
Tension clamp for LCA, movable part for 19.5 mm sample length (with sample mounting plate)	51 141 420
Sample mounting plate 19.5 mm	51 141 273
Compression clamp for LCA	51 140 024
Tweezers	70 661
Torx Allen wrench T9	51 190 835
Torx screw driver T9	51 190 834
Allen wrench, 4 mm	70 718
Torx-screw set M 2.5	51 141 184
Peripherial Option Board	119 350
Cryofab special dewar vessel	190 129
TSO800GC gas controller (with operating instructions)*	00 119 035
TSO800GC1 gas controller (with operating instructions)*	51 119 597
Tube set for gas controller	650 064?
Tube adapter part	71 356
RS232 connection cable 25M/9W	59 759
RS232 connection cable 25M/25W	59 342

* The gas controller ME-119035 or ME 51119597 can only be operated with the Peripherial Option Board (ME-119350)

Name	ME Number
Small clamping assembly (SCA) with protective cover and alignment insert	**51 140 002**
Torx/slot screw torque wrench	51 191 122
Blade for Torx torque wrench T9	51 191 123
Blade for slot screw torque wrench size. 6	51 191 124
Standard shear clamp (for highly viscous samples)	51 141 280
Standard shear clamp (for highly viscous samples) with smooth sample support surfaces	51 141 093
Special shear clamp (for low viscosity liquids)	51 141 092
Guide pins for standard shear clamp: • Short guide pin pair (23 mm) • Medium guide pin pair (27.5 mm) • Long guide pin pair (32 mm)	 51 140 042 51 140 044 51 140 037
Tension clamp for SCA	51 140 090

Abbreviations used in the accessory parts list:

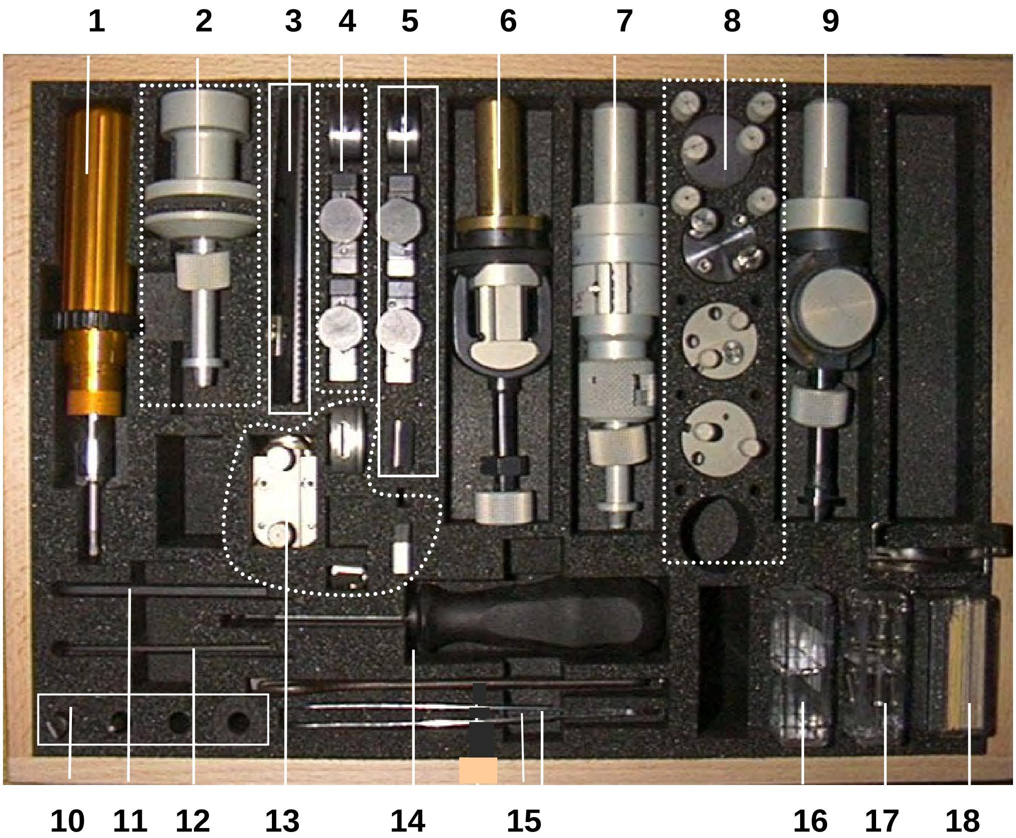
LCA : Large clamping assembly

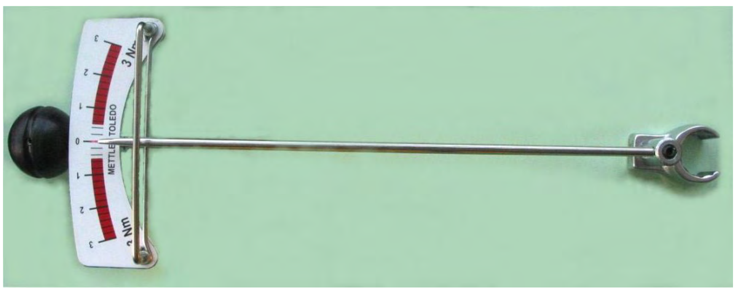
SCA : Small clamping assembly

PCB : PC board

12.2 Example of an accessories box for the DMA/SDTA861e

The accessories for the clamping assemblies you ordered with you DMA module are supplied in a wooden box that is shipped with the instrument. The contents of the box depends on the configuration of your DMA/SDTA861e. For example if you have ordered only the small clamping assembly the box will not contain any parts for the large clamping assembly and vice versa. The box can accommodate one complete set of accessories for both clamping assemblies.





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Accessories in the box (example):

1 Torque wrenches (Torx- and slot screw type)

2 Adjustment aid

3 Bending beam

4 Bending clamp for dual cantilever mode

5 Bending clamp 3-point bending mode

6 Large clamping assembly

7 Adjustment assembly

8 Clamps for the small clamping assembly

9 Small clamping assembly

10 Blades for the torque wrenches **1**

11 4 mm Hexagonal Allen wrench

12 Torx Allen wrench T9

13 Large tension clamp

14 Torx screw driver

15 Tweezers

16 Sample set for temperature adjustment

17 Spare screw set

18 Gauge block set

19 Torque wrench for the large clamping assembly (not contained in the box)

TSO800GC / TSO800GC1 Gas Controller
TSO800GC / TSO800GC1 Gaskontroller
Unité de contrôle de gaz TSO800GC / TSO800GC1

Option for TGA/SDTA851^e and DSC822^e Modules
Option für TGA/SDTA851^e und DSC822^e Module
Option pour les modules TGA/SDTA851^e et DSC822^e

Accessories / Zubehör / Accessoires

		Order Number / Bestellnummer / Numéro de commande
Operating Instructions	English	709 306
Bedienungsanleitung	Deutsch	709 305
Mode d'emploi	Français	709 307

14 Glossary

3-point bending	Mode of a DMA bending experiment in which the ends of the sample lie freely on the supports. The bending moment is zero at the supports.
3-point bending clamp	Clamp for a DMA bending experiment in which the ends of the sample lie freely on the supports (the bending moment is zero at the supports) see also „bending clamp“
adjustment assembly	Device for the adjustment of the DMA module. With the help of the adjustment assembly you adjust the movement of the z-axis table and the force exerted on the sample by performing the spindle or the force adjustment, respectively.
alignment	"Alignment" is the alignment of the displacement sensor and the clamping assembly on the DMA module.
alignment aid	Device for the manual alignment of the clamping assembly or the adjustment assembly. The accuracy of alignment you achieve with the alignment aid is usually already good and you only have to make small corrections during the subsequent fine alignment procedure.
alignment insert	Insert to align the clamping assembly when it is mounted on the DMA
amplitude	Value of the sinusoidal force or displacement oscillation generated by the drive motor
amplitude control	Force or displacement amplitude control of the sinusoidal oscillation of the drive motor
amplitude segment	An amplitude segment is a DMA segment in which the force or the amplitude can be varied. "Amplitude" segment is the general term for force or displacement amplitude (see also "force segment" and "displacement segment").
bending clamp	Clamp of the large clamping assembly to conduct 3-point bending or dual cantilever bending experiments
adjustment assembly	Device for adjusting the DMA module. The adjustment assembly is used to perform the spindle and force adjustment
clamp	The clamp is a mechanical clamp or supporting device for the sample and is part of the clamping assembly. The clamp is mounted on the clamping assembly together with the sample.
clamp holder	The clamp holder is a part of the clamping assembly on which the clamp is directly mounted together with the sample. The clamp holder comprises a fixed and a movable part. The movable part is connected to the drive shaft and embraces the middle part of the clamp.

clamp holder, fitting	Half of the fixed part of the clamp holder. The clamp is fixed in the clamp holder by tightening four Torx screws.
clamp holder, fixed/movable	See "clamp holder".
clamping assembly	The clamping assembly is a device consisting of several components in which the sample is mounted and clamped. The fixed part of the clamping assembly transfers the force exerted on the sample to the force sensor. The movable part of the clamping assembly transfers the oscillation generated by the drive motor to the sample. The clamping assembly is located between the drive motor and the displacement sensor and includes the drive shaft, the sample holder, the clamp and the displacement sensor core. Depending on the mode of measurement a different type of these component is used.
compliance	Shear compliance: reciprocal value of shear modulus Tension compliance: reciprocal value of Young's modulus
compression clamp	Insert used to fix the sample for compression experiments
displacement amplitude	Dimensional change that the sample suffers during the sinusoidal oscillation in a DMA experiment See also "oscillation"
displacement offset	Set value for the offset of displacement in a DMA experiment
displacement segment	A displacement segment is a DMA-specific segment type. The amplitude of displacement can be varied either linearly or logarithmically at a given frequency and temperature.
displacement sensor	Sensor for measuring the displacement of a sample. The displacement sensor consists of a core and a coil. The displacement signal is detected by means of induction.
displacement sensor core	Movable part of the displacement sensor the end of which moves inside the displacement sensor coil thereby generating the signal for displacement measurement. The displacement sensor core is part of the clamping assembly.
distortion factor	Harmonic content of a DMA signal
DMA	Abbreviation for **d**ynamic **m**echanical **a**nalysis
DMA-segment	Section of a DMA method. A DMA segment can either be an amplitude segment (force or displacement amplitude) or a frequency segment. It is also possible to define a DMA oscillation during a temperature segment.
drive motor	The drive motor generates the mechanical oscillation of the sample based on the principle of an electric linear motor. The drive motor is mounted on the z-axis table and can move upward and downward along the z-axis.
drive shaft	Connecting piece between drive motor and clamp holder that transfers the mechanical oscillation from the drive motor to the sample. The movable part of the clamp holder is connected to the upper end of the drive shaft.

dual cantilever clamp	Clamp for a DMA bending experiment in which the ends of the sample are firmly clamped at the supports (the bending moment is transferred to the supports) see also „bending clamp“
dynamic displacement	Maximum displacement in a DMA experiment
force amplitude	Amplitude of force in a sinusoidal oscillation of a DMA experiment (see also "oscillation")
force offset	Offset value of force specified in a DMA experiment.
force segment	A force segment is a DMA-specific segment type. The amplitude of force can be varied either linearly or logarithmically at a given frequency and temperature.
force sensor	Sensor for measurement of force generated by the drive motor and transferred to the sample. The force sensor is based on the piezo-quartz principle.
four axes alignment device	Adjustment mechanism to align the clamping assembly and the measurement sensors. The four axes alignment device is mounted on top of the stand. The position of the displacement sensors can be adjusted by rotating the knobs on the device.
frequency segment	A frequency segment is a section of a DMA method in which the user can vary the oscillation frequency of a sample. The frequency can be varied linearly or logarithmically and several frequencies can be overlayed simultaneously in given ratios. A frequency series is always isothermal.
frequency series	A frequency series consists of up to 10 consecutive individual frequencies. A frequency series can be defined additionally to a temperature program in a temperature segment of a DMA method. It is thereby possible to simultaneously vary frequency and temperature. (Compare multifrequency mode.)
furnace (DMA)	The term "furnace" denotes a chamber with a heating and cooling device that is used to produce the desired temperature of the sample in an experiment. The furnace consists of two halves that each have a heating and cooling element. To mount the sample the two furnace halves can be moved apart.
furnace arm	Supporting part of the furnace drive to which a furnace is fixed
furnace drive	Mechanism for opening and closing the furnace. The furnace is opened by moving the two furnace halves outward. The furnace drive consists essentially of the two furnace arms, the guide rods and the furnace drive motor.
furnace drive motor	Step motor that generates the force to move the furnace halves outward and inward. The driving force is transmitted via a rack to the furnace arms.
initial transient phase	Transition time of the oscillation in a DMA experiment during which a steady state oscillation is reached
loss factor	see also “tangent delta”
multi-frequency mode	Mode of multi-frequency oscillation on the DMA
multi-frequency oscillation	With the Multi-Frequency feature three frequencies can be defined to overlay the base frequency in fixed ratios. The frequency ratios are 1 : 2 : 5 : 10.

oscillation	In the context of DMA "oscillation" is understood as an oscillatory variation of force or displacement that is generated by the drive motor and transferred to the sample.
oscillation amplitude	Amplitude of the sinusoidal oscillation generated by the drive motor. The oscillation amplitude can be a force amplitude or a displacement amplitude.
platform (z-axis table)	Platform of the z-axis table that bears the drive motor
resonance detection insert	Special insert for the small clamping assembly with which the resonance frequencies of the DMA can be measured. The resonance detection insert establishes a firm connection between the fixed and the movable part of the clamp holder. The resonance measurement therefore resembles a measurement with a very stiff sample.
sample holder	General term for a device or vessel that holds the sample. Depending on the type of the TA module the sample holder can be the pan (DSC, TGA), the sample support (TMA) or the clamp (DMA).
sample support	Part of the clamp that bears the sample.
segment	Distribution of a set value in a time period. In thermal analysis the set value is typically the temperature (reference temperature). Adjoined segments make up a temperature program. In DMA experiments also frequency and amplitude segments can be created in which the measuring points are defined in terms of frequency or amplitude.
settling	Transient phase in which the sample temperature reaches the start value for the experiment
shear clamp	Clamp for mounting the sample for a shear stress experiment.
shear compliance	Reciprocal value of the shear modulus
shear modulus	Mechanical property that characterizes the sample of a substance when it is subjected to shear stress.
tangent delta	Tangent of the phase shift between the force exerted on the sample and its deformation. Also the ratio of the loss and the storage components of the modulus (therefore also termed “loss factor”)
temperature segment	Section of a temperature program in the method in which the temperature is varied with respect to time
tension clamp	Insert that is used to fix the sample for tension experiments. Depending on the clamping assembly used, the large or the small tension clamp is mounted.
tension compliance	Reciprocal value of Young's modulus
threaded roller spindle	Spindle that enables the precise upward and downward movement of the z-axis table
Young's modulus	Mechanical property that characterizes the sample of a substance when it is subjected to tensile or compressive stress.
z-axis table	The z-axis table is a platform on which the drive motor is mounted that can be moved up and down in the z direction. The z-axis table is powered by a step motor.

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STAR^e thermal analysis system, operating instructions to the DMA/SDTA861^e module

P 5 1 7 1 0 0 6 9

Subject to technical changes and to the availability of the accessories supplied with the instruments.

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