

Instruction Manual

LSR - 3

Seebeck Effect & Electric Resistivity

**Hardware
Software
Evaluation
Maintenance & Service
Installation**

Linseis Messgeräte GmbH
Vielitzer Str. 43
D-95100 Selb
Phone: 0049-9287-8800
Fax: 0049-9287-70488
Email: info@linseis.com
Web site: <http://www.linseis.com>

Instruction Manual
LSR - 3
Seebeck Effect &
Electric Resistivity
Hardware

GENERAL INFORMATION

In the design of your Linseis instrument, we try to take individual solutions into account and to include these in the documentation.

Nevertheless, to keep the range of the instrument documentation at a level which can be handled, the instrument manual/description has to be limited to common or standard models.

Electronic or mechanical distribution or duplication of this/these instructions requires written permission from LINSEIS MESSGERÄTE GmbH.

All instrument features, technical data and other information described in these system operating instructions are provided to the best of our knowledge and in agreement with the technical standards at the time of creation of this document.

Linseis Messgeraete GmbH welcomes any remarks, suggestions or new ideas regarding the instrument and these operating instructions. Please send any comments to the following address:

LINSEIS Messgeraete GmbH
Vielitzer Str.43
D-95100SELB

Telephone: (+49) 9287 880-0
Fax: (+49) 9287/70488
E-Mail: info@linseis.de
Internet: http://www.linseis.com

Any type of maintenance or service of the instrument must be carried out by LINSEIS trained customer service personnel.

A service contract for your thermal analyzer is available on request.

This instrument instruction manual is designed to allow proper operation of the instrument by the customer. LINSEIS Messgeraete GmbH will accept no liability for damage following-on improper use.

1. BASIC SYSTEM SAFETY INFORMATION

Allowed instrument operation

- Any operation or misuse of the instrument other than explicitly certified requires written approval from LINSEIS.
- Any use exceeding the (expanded) authorized system operation is considered unauthorized. The producer cannot be held accountable for any damage follow-on such unauthorized use of the instrument.

Instrument Design

- Your Thermal Analyzer has been designed and manufactured with state-of-the-art expertise and is secure to operate.

General Requirements

- Manufacturer approved operation of the instrument includes training of personnel and compliance with manufacturer's requirements regarding installation, commissioning, operation and maintenance.

Training of User Personnel

- Your Linseis instrument may only be operated and maintained by authorized, trained and individually instructed personnel.

Responsibility for Commissioning, Operation and Training

- Responsibility for commissioning, operation and maintenance must be clearly defined and compliance must be ensured. The resulting responsibilities can be clearly resolved under the aspect of safety.

Instrument Operation Guideline

- The operator must make sure that only trained personnel work on the instrument.
- Any type of operation of the instrument which reduces the safety of the instrument user and the operability of the analyzer should be avoided.

Unauthorized changes to the system

- Unauthorized modifications and changes which affect the safety of the thermal analyzer are not permitted.
- The person using the analyzer is obligated to immediately report to the operator in charge any changes to the instrument which affect the safety of the system.

Instrument Maintenance Obligation

- The thermal analyzer operator must ensure that the instrument is operated in perfect condition at all times.
- During Maintenance and inspection the instrument must be switched off and unplugged.
- Instruments or parts sent in for maintenance or repair should, to the extent possible, be free of dangerous or harmful substances (e.g. toxic, radioactive, etc.). Otherwise, the type of contamination must be declared. Instruments or parts not explicitly stated to be "not dangerous to handle" will be decontaminated at the expense of the owner.

Checks following Maintenance or Repair Work

- After maintenance or repair work, a check should be made to ensure that all protective devices are in place and operate properly. Only then should the instrument be started.

Accident Prevention Regulations: Industry-specific

- The relevant regulations and protective measures must be observed by the user when handling the required gases. Furthermore, possible dangerous reactions with the materials used must be considered. At operating temperatures above 50°C, protective gloves must be worn. Any industry specific regulations for the analyzer are always relevant.

Consider possible reactions with the materials.

- Warning signs should be hung in the appropriate places in accordance with national and regional regulations.

Using Gases

- Obey the appropriate national or international rules and protective measures when using gases for a measurement.

Removals of Production Materials

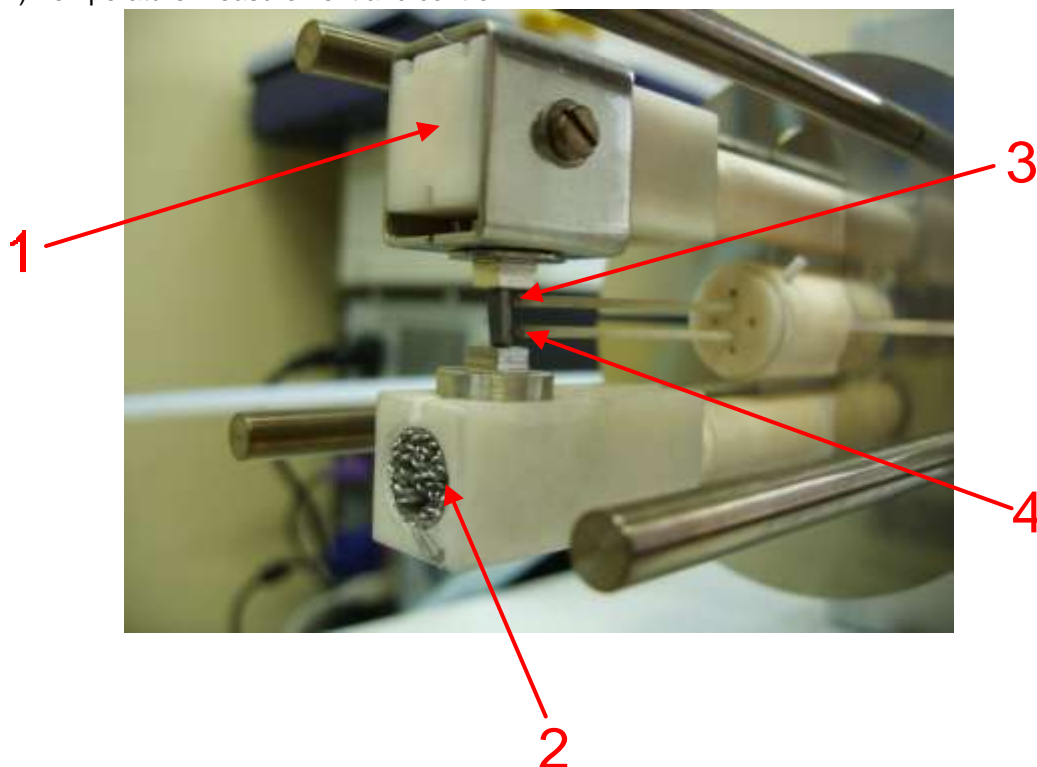
- Any type of waste occurring out of the use of the analyzer is to be disposed in accordance with local regulation.

Use of the Operating Instructions

- Using guidelines from the system Operation Manuals, the operator should arrange additional internal operating instructions which specify the actions and tasks required for additional safe operation.
- The operating instructions should be easily accessible at the work place of the instrument.

Important information for LSR-3 measurements

1) Temperature measurement and control:



The LSR-3 uses 5 thermocouples to measure temperatures: 1=Differential TC upper side (DTCU), 2=Differential TC lower side (DTCL), 3=Probe TC High side (PTCH), 4=Probe TC lower side (PTCL) and the control TC in the susceptor (CTC). The real temperature of the sample calculated, is the average temperature measured by the probe TCs (PTCH and PTCL). Because the real sample temperature can't be measured during resistance measurement, the temperature reading of the differential TC of the upper side is used for temperature closed-loop control: From the calculated set-temperature and the reading of the upper differential TC a set-temperature for the susceptor is calculated (Controller 1). From the set-temperature of the susceptor and the actual temperature of the susceptor the power output for the furnace is calculated (Controller 2). Since the gradient heater in the lower sample support (2) introduces extra heat into the sample, the real temperature of the sample will be higher than the temperature setup in the profile. So, if the maximum temperature in the profile is set close to the melting temperature or a temperature where the sample material starts to react with the thermocouples or the electrodes, care must be taken not to exceed this critical temperature. So the maximum temperature in the profile must be set lower, at least by the gradient temperature entered.

Example:

Melting temperature of the sample is 660°C

Gradient setup is 50K

Maximum temperature allowed in the profile is app. 600°C to be on the safe side.

In the new LSR.EXE program a safety temperature can be specified in the Menu Temperature Profile It is named SAFTY TEMPERATURE (Average temperature measured by the two probe thermocouples)

If in this case the sample temperature (Average temperature measured by the two probe thermocouples) is used, if temperature exceeds the value the controller and the measurement will be stopped and a related message will be displayed in the status line.

2) Problems of sample materials:

Some sample materials may react with the materials used for the probe-thermocouples and the upper and lower contacts, made of Platinum. Since Platinum is known as a very stable, high melting material, most people don't think that a problem with some materials could occur at relative low temperatures. The problem is that some elements are building low melting alloys when they are in contact to Platinum. In this case, the alloy grows from the junction of the sample and the Platinum until sample and thermocouple or electrodes are DESTROYED entirely:



Remainder of a sample, containing 50% lead and the lower platinum electrode
Critical substances are low melting metals (e.g. Indium, Tin, Lead, Bismuth ...), Silicon (even as SiO₂ or SiC), Arsenic, Phosphorus, Sulfur and reducing substances like Carbon.

Warranty

The standard warranty period for this instrument is (12) twelve months. If requested prolonged warranty contracts are available.

If any problem with the system occurs, due to defective components or false assembly, Linseis will immediately correct / repair the defective parts without any charge.

Not covered by this warranty are among others.

- Inappropriate operation of the Instrument, thus not covered in this manual.
- Any type of modification or repair of the instrument, not authorized by Linseis

This warranty does not apply to damages as a result of an act of God, acts or omissions of third parties, misuse, or improper installation and will be deemed null and void if our products are in any way altered or modified after delivery.

Limitations of Liability

In no event shall Linseis be liable for special, incidental, or consequential damages, including but not limited to loss of profit or opportunity.

Applications of the Instrument

The Thermal power, thermoelectric power, or Seebeck coefficient of a material measures the magnitude of an induced thermoelectric voltage in response to a temperature difference across that material. The thermal power has units of (V/K).

The thermoelectric effect is sometimes used to generate electrical power, starting from a source of a temperature gradient. For example, some spacecraft are powered in this way, exploiting the temperature difference between a radioactively-heated plate and the cold empty space surrounding the craft. Some researchers hope that, in the future, much wider use could be made of thermoelectric power generation, including using waste heat from automobiles and power plants.

The efficiency with which a thermoelectric material can generate electrical power depends on several material properties, of which perhaps the most important is the thermopower. A larger induced thermoelectric voltage for a given temperature gradient will lead to a higher efficiency. Ideally one would want very large thermopower values since only a small amount of heat is then necessary to create a large voltage. This voltage can then be used to provide power.

In recent years much interest has been shown in various methods of direct conversion of heat into electricity. Waste heat from hot engines and combustion systems could save billions of dollars if it could be captured and converted into electricity via thermoelectric devices

What is Seebeck

An electric potential (voltage) is generated within any isolated conducting material that is subject to a temperature gradient; this is the absolute Seebeck effect "ASE". The absolute Seebeck coefficient "ASC" is defined as the instantaneous rate of change of the ASE with respect to the temperature at a given temperature: $ASC = [d(ASE)/dT]T$.

The relative Seebeck coefficient "RSC", is the instantaneous rate of change of the relative Seebeck emf "RSE" with temperature at a given temperature: $RSC = [d(RSE)/dT]T$.

The measurement principle

A prism or cylindrical sample is set in a vertical position between the upper and lower blocks in the heating furnace. While the sample is heated to, and held, at a specified temperature, it is heated by the heater in the lower block to provide a temperature gradient.

Seebeck coefficient is measured by measuring the upper and lower temperatures T_1 and T_2 with the thermocouples pressed against the side of the sample, followed by measurement of thermal electromotive force dE between the same wires on one side of the thermocouple.

Electric resistance is measured by the dc four terminal method, in which a constant current I is applied to both ends of the sample to measure and determine voltage drop dV between the same wires of the thermocouple by subtracting the thermo-electromotive force between leads.

Schematics of the measurement setup

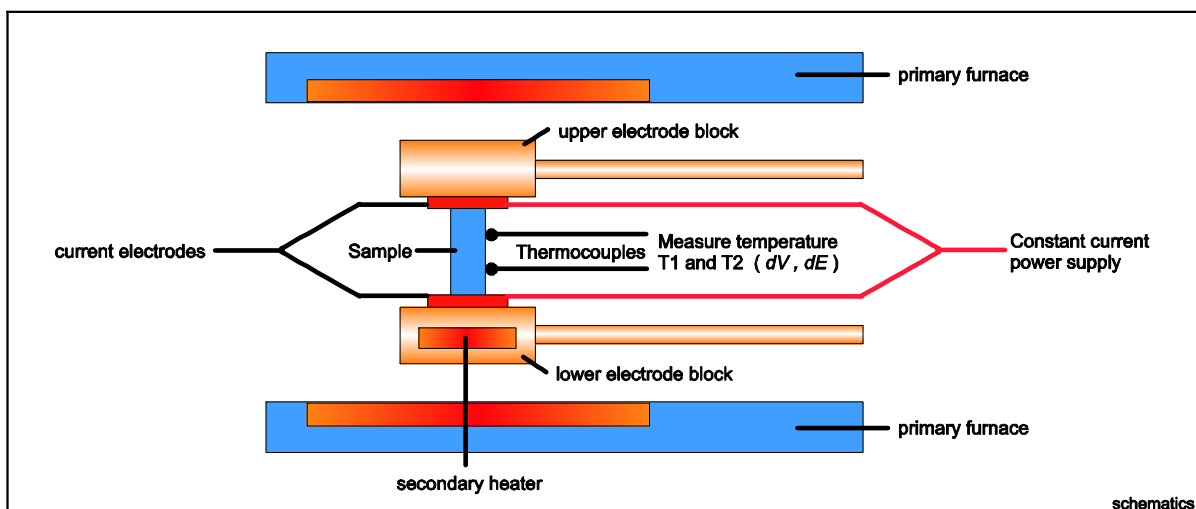
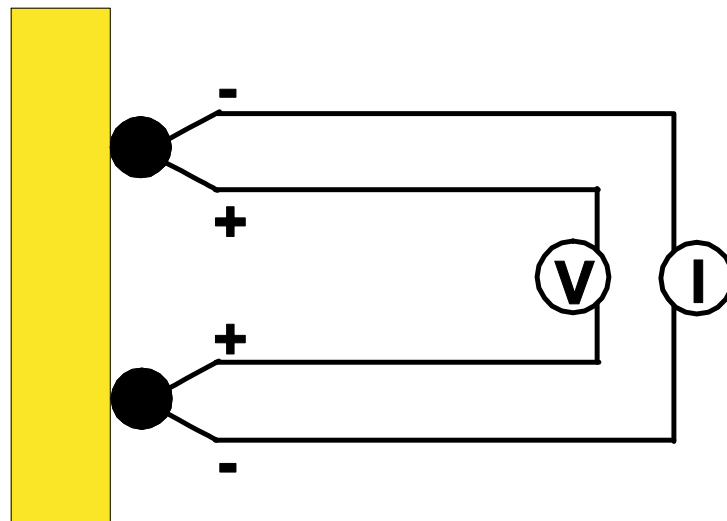
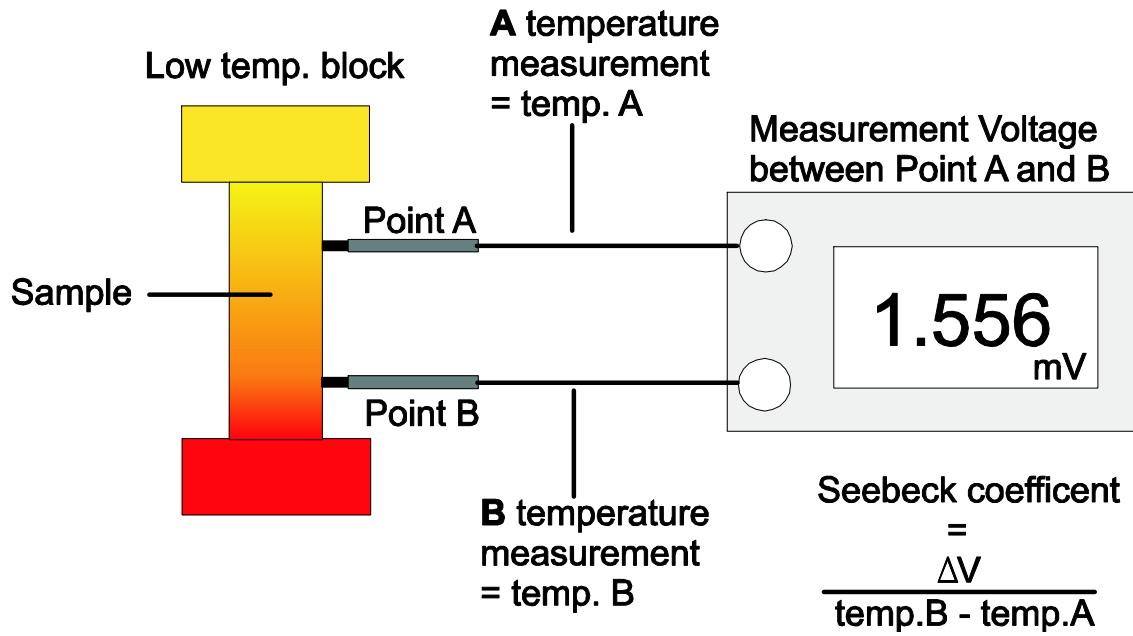
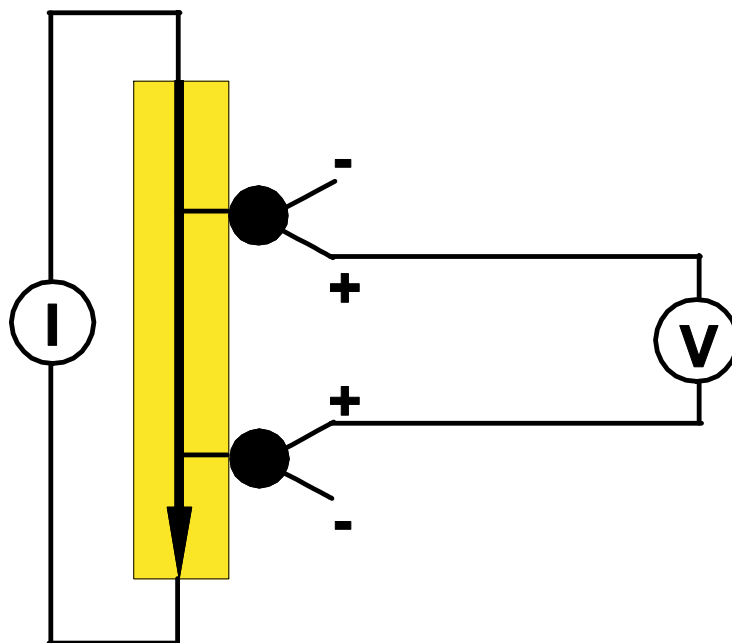
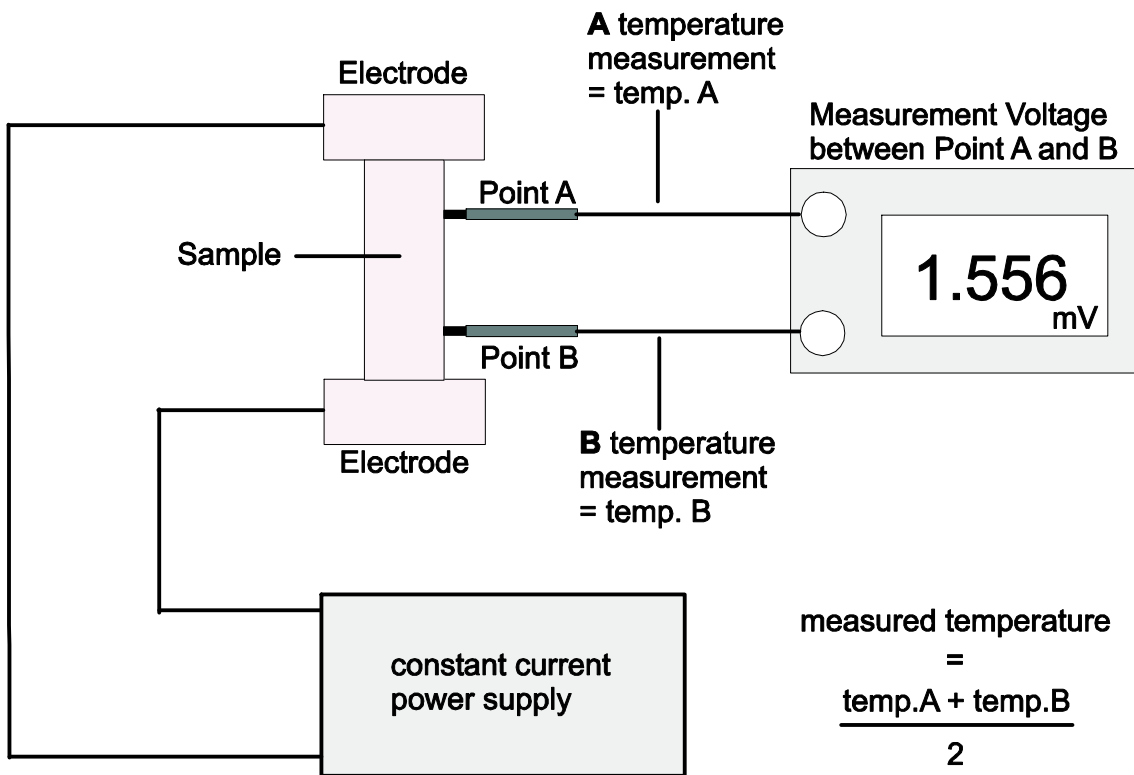


Diagram describing concept of measurement of thermal emf (Seebeck coefficient)



2 terminal method - check contact about 2 points

Temperature and electromotive force are calculated via Probe A and Probe B.



4 terminal method - accurate value when measured about 4 points

Installation

- All installation and training is done by a trained Linseis service engineer. Linseis strongly recommends this procedure to ensure a correct setup of the instrument and training of the operator from the very beginning
- It is not recommended to install the system yourself as this can affect the warranty or damage the system.

The Packing and Delivery

Linseis delivers all instrument parts in a separate carton. All components, such as instrument, measuring unit, PC and printer are packed in form molded foam or an equivalent, to protect against transport damage.

- Please keep the original cartons stored, in case of repair or upgrade, the original cartons ensure a relative safe return to our production facility.
- All Linseis instruments are carefully tested prior to shipping.
- Please do not unpack the components yourself (unless stated otherwise). The Linseis service engineer will unpack the parts and check if any transport damage is visible.

Location requirements

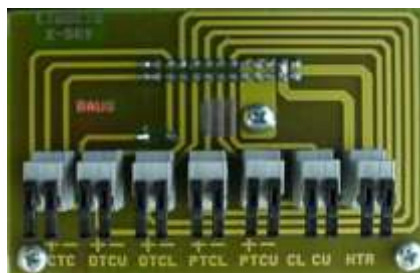
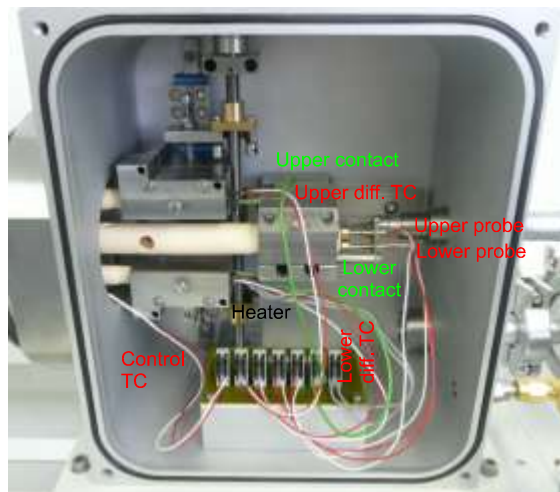
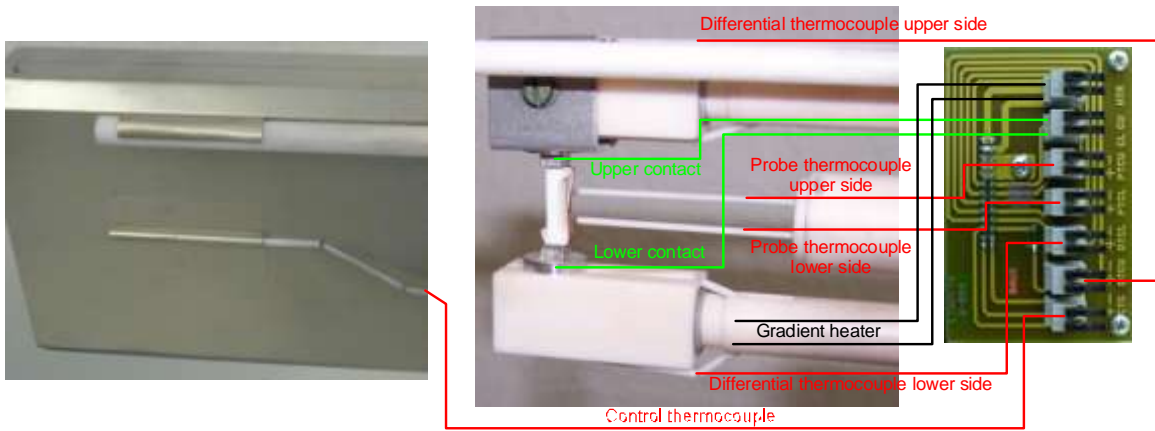
- Select an optimum component arrangement for the space available

The following location requirements for the instrument are imperative

- Stable temperature conditions
- No direct sunlight on the instrument
- A dust free environment (to the extent possible)

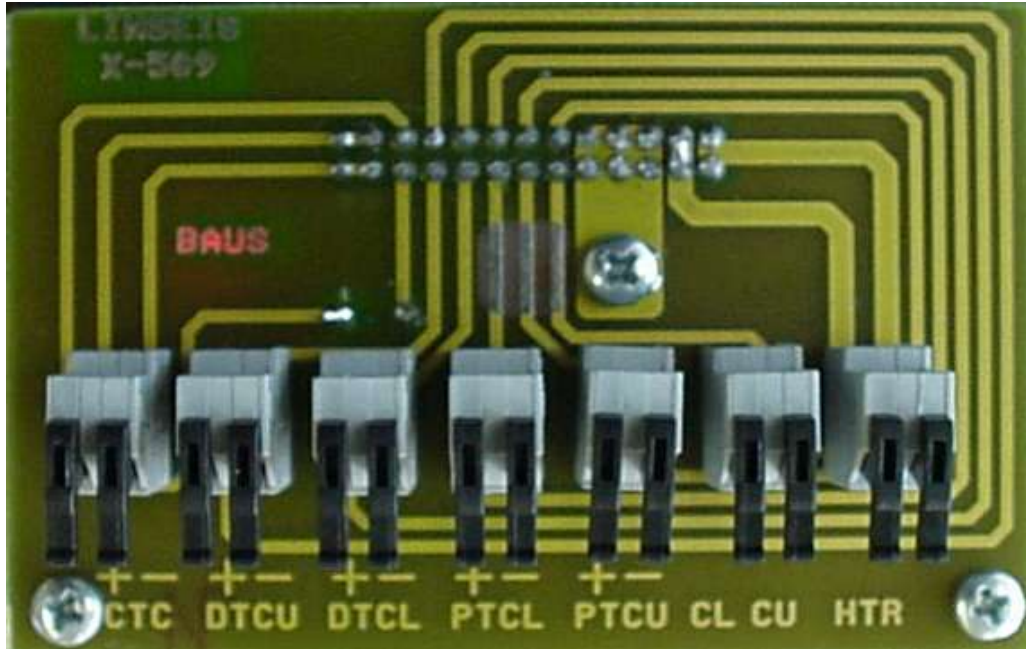
For detailed Installation requirements see additional Manual INSTALLATION at different position in this manual

Thermocouple connections LSR-3



- Control TC +
- Control TC -
- Upper Diff +
- Upper Diff -
- Lower Diff +
- Lower Diff -
- Lower Probe +
- Lower Probe -
- Upper Probe +
- Upper Probe -
- Lower contact
- Upper contact
- Heater

Connection LSR board



CTC	:	Thermocouple Control Susceptor	(Ni cover) not used in low temperature furnace
DTCU:		Upper Thermocouple	
DTCL:		Lower Thermocouple	
PTCL:		Thermocouple lower probe	
PTCU:		Thermocouple upper probe	
CL:		Lower Contact	usually blue wire
CU:		Upper Contact	usually blue wire
HTR:		Heater	usually gray wires

Thermocouple K (Ni/CrNi)	Low Temp -100°C +500°C	+: green wire	-: white wire
Thermocouple S (Pt/RhPt10)	High Temp RT .. 800°C or 1100°C	+: red wire	-: white wire

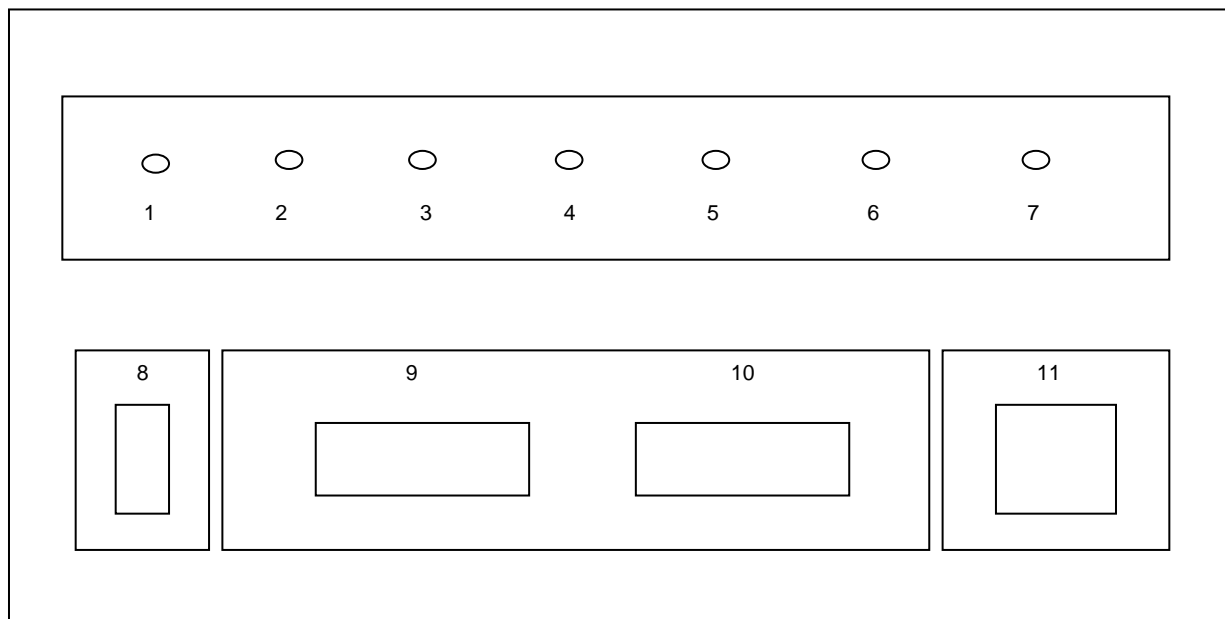
The Instrument

Models:

- LSR - 3

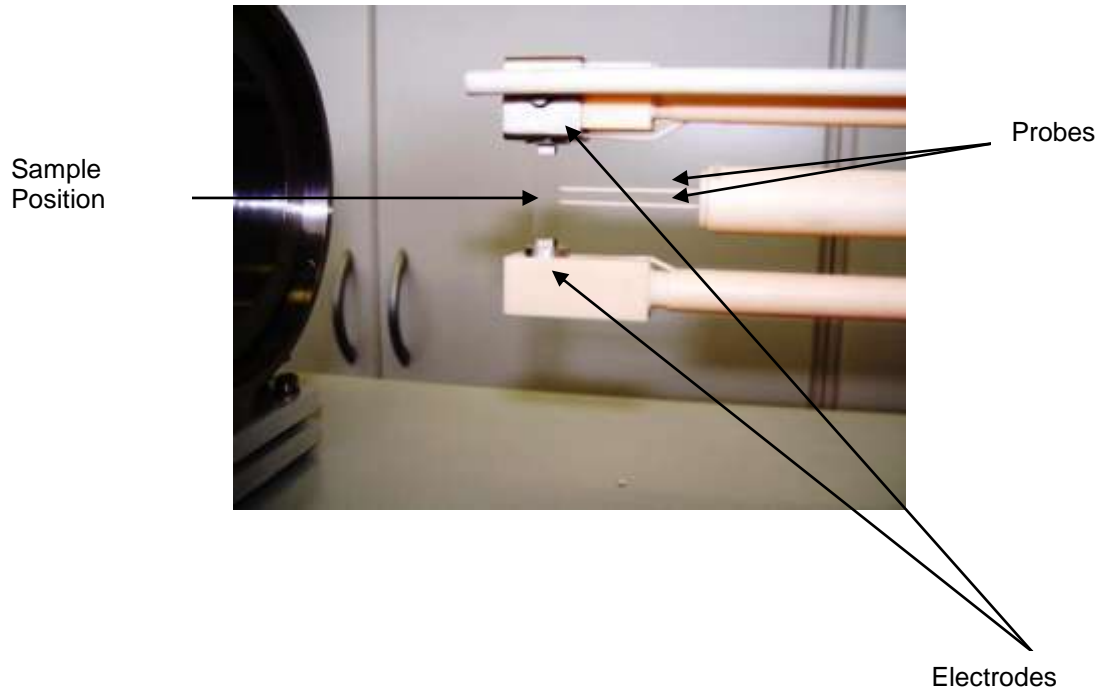


User panel



Label	function
1. Hot	Sample temperature is over 75°C
2. Furnace on	Furnace heater is active
3. Diff. Heater	Diff. Heater is active
4. Acquisition on	Data acquisition is active
5. Measuring Resistance	Displays the last measured value
6. Measuring Seebeck	Displays the last measured value
7. USB ready	The connection between the instrument and the PC is active
8. Power on / off	The power supply is switched on / off
9. Sample	Displays the sample temperature
10. Susceptor	Displays the susceptor temperature
11. Emergency Off	Switch for emergency off

Sample holder



Sample geometries

Shape	Sample size
Rectangular cylinder	5 to 22 mm long and 2 to 4 mm diameter
Circular Cylinder	5 to 22 mm long and 2 to 4 mm diameter

Furnaces:

The LSR can be equipped with the following furnaces

Low Temperature Furnace

LSR/LT low temperature furnace - 100 up to 500°C

Infrared Furnace

LSR/IR/800 IR furnace RT up to 800°C
LSR/IR/1100 IR furnace RT up to 1100°C

High Temperature resistance Furnace

LSR/RF/1500 high temperature furnace RT up to 1500°C

Operation under purge gas

The instrument is capable of performing measurements under dynamic atmospheres. Inert gases, Air and some reaction gases can be used.

Please follow the following instructions when operating the system under purge gas.

- The sample chamber is vacuum tight, thus it is possible to carry out experiments under dynamic/controlled environmental conditions
- Since the measuring compartment is made of glass and ceramic components, these may be exposed to stress when heated to elevated temperatures. The current cycling may cause cracks in the furnace or sample holder. These cracks cannot be completely prevented even when conducting a continuous maintenance (service and maintenance contract) of the instrument. A continuous exchange of parts may limit this risk but cannot prevent it entirely.
- When purging the sample chamber we recommend dry inert gases. A leakage test should be done prior to the insertion of a gas in order to guarantee the purity of the sample atmosphere. This test can be performed while pre-evacuating the instrument.
- Outgasing should be passed in an appropriate hood or equivalent device to prevent any danger for the operator or the environment. Please consider that certain measurement conditions may lead to creation of dangerous outgasing such as dioxins, HCL etc.
- For this the user must decide prior the experiment if any dangerous outgasings may occur, if so precautions are critical.
- Recommended gas for all measurements: helium (he)

Some guidelines

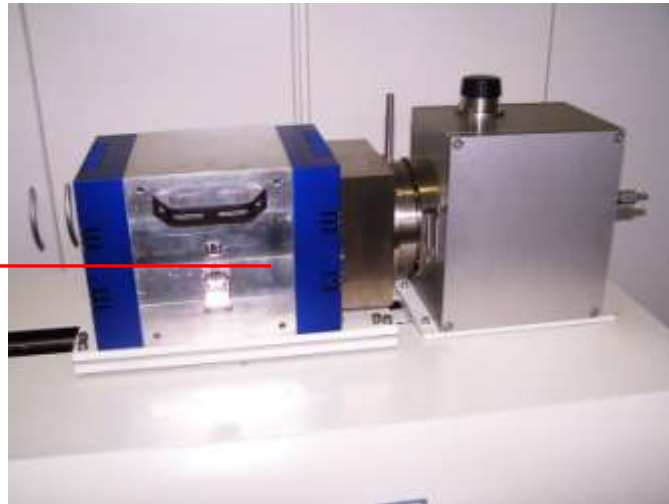
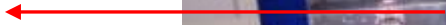
- Consider the possibility of corrosion. This may affect not just the instrument but any surrounding parts such as valves, the gas box etc.
- Does the experimental temperature lead to the creation of toxic or dangerous gas compounds?
- Can a reaction between sample and oxygen (air) lead to the creation of an explosive gas mixture?

Please follow the additional instructions when operating the system under air or oxidizing gas atmospheres:

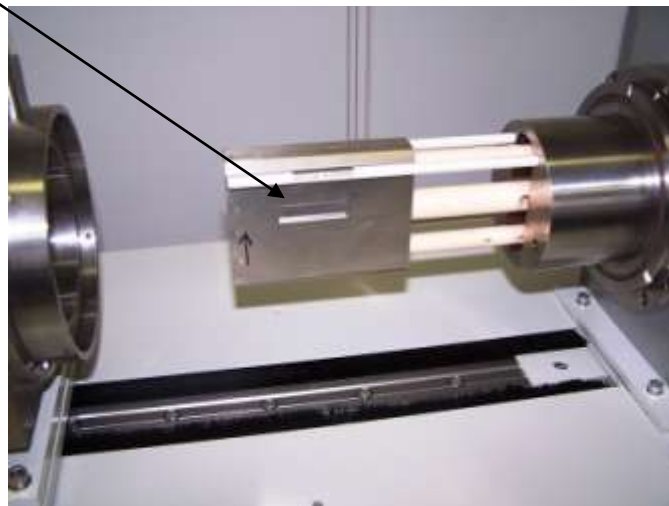
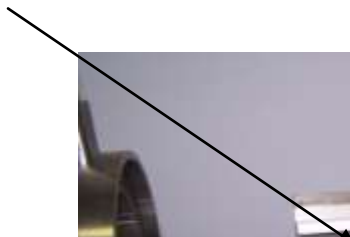
Oxidizing gas atmospheres destroy the suszeptor if the suszeptor temperature rises above 300°C (572°F).

Insert a sample

Move furnace to the left to open



Remove the susceptor



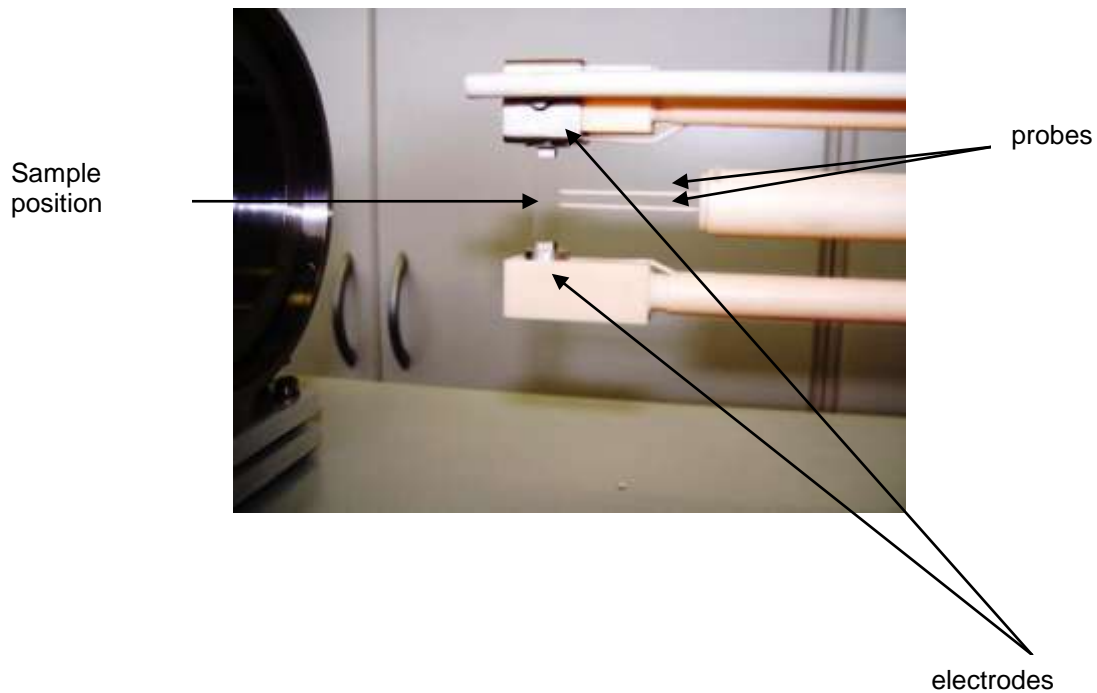
Turn the probe moving knob counterclockwise so that the probes are not going to touch the sample to be entered.



Turn the electrode elevating dial clockwise so that the sample can be positioned on the lower electrode.



Basic sample arrangement before inserting the sample

**Annotation**

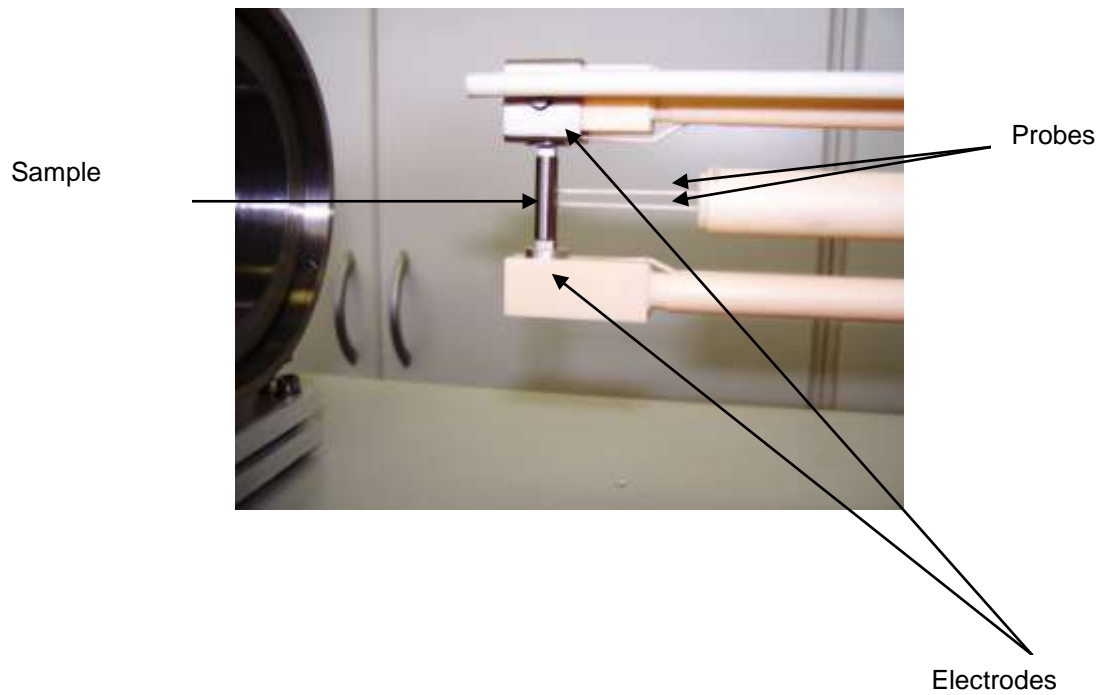
The following recommendations are supposed to ensure a safe system operation:

1. Previous to inserting the sample, lightly polish the sample surface in contact with the electrodes. Linseis recommends sandpaper of No. 1000 or similar.
- This recommendation is less critical for platinum electrodes.
2. If a sample with a low melting point or sublimating material is measured, or if the sample assembly and electrode are contaminated with outgassing during heating/cooling etc, polish and clean the electrodes.
3. If the electrode contact surface is oxidized or contaminated, polish it with sandpaper No. 1000. Polish the probe contact surface, too.
Oxidation is a problem of nickel electrodes, thus platinum electrodes are preferable.

Please note:

When cleaning the probe, handle it carefully and do not damage it.

Basic sample arrangement when loading the sample



Position the sample on the lower electrode. Then close the gap between the two electrodes by turning the "electrode positioning wheel" on top of the main compartment.

Note:

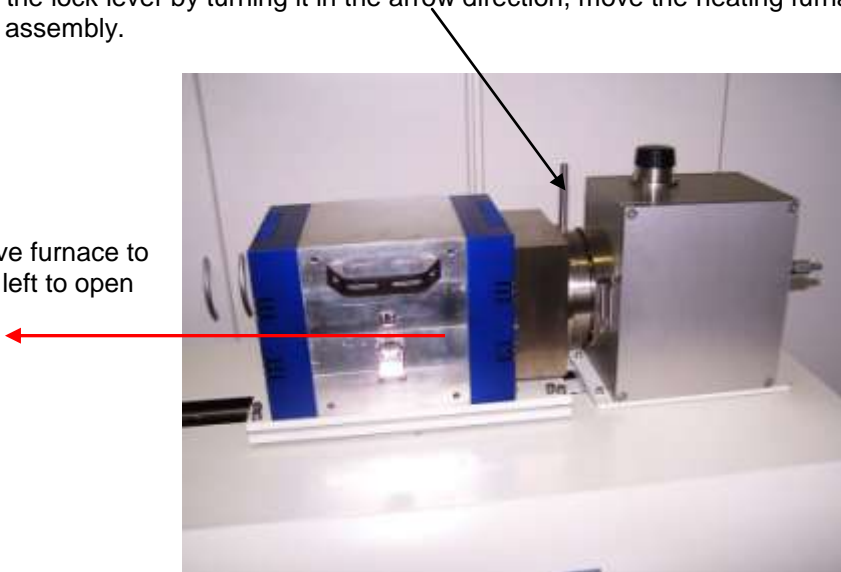
Please note, the Al₂O₃ holders with the electrodes have spring protection to prevent damage due to sample expansion, or misplacement.

Release a Sample

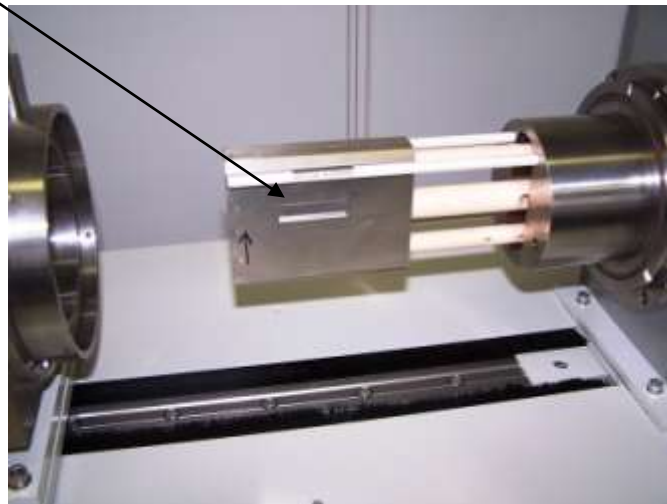
Usually, the atmosphere in the sample chamber is kept at a negative pressure after a measurement is stopped. Vent it to atmospheric pressure.

Loosen the lock lever by turning it in the arrow direction, move the heating furnace to left and open the sample assembly.

Move furnace to
the left to open



Remove the susceptor



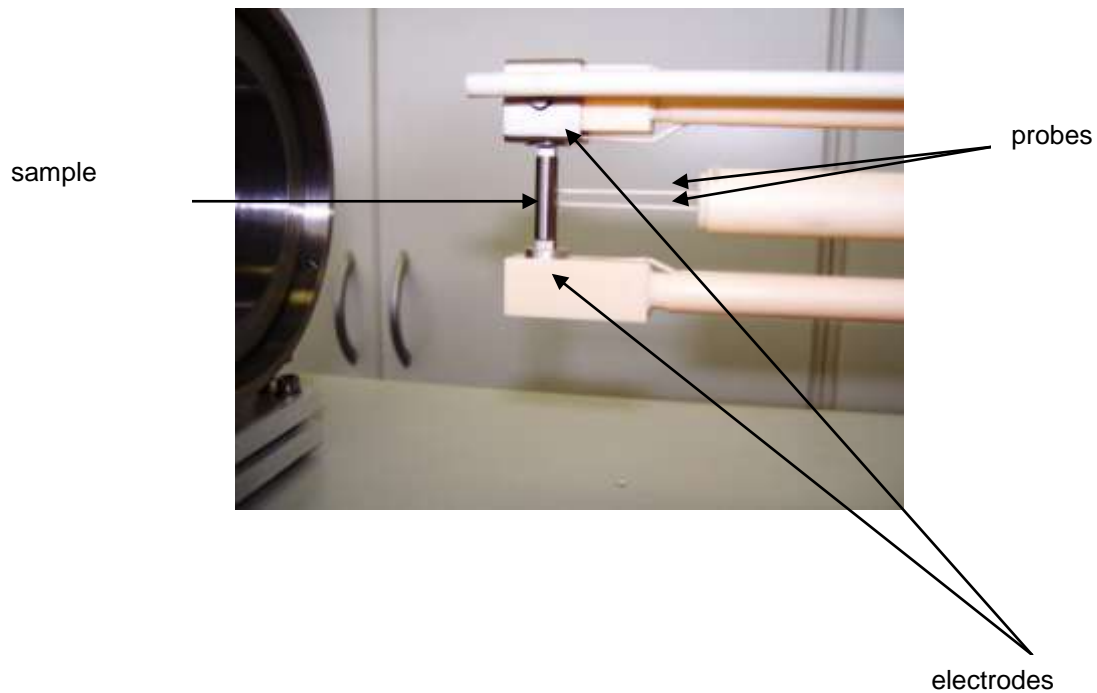
Turn the probe moving knob counterclockwise to move the thermocouples away from the probe.



Turn the electrode elevating dial clockwise to release the sample from the two electrodes fixing it.



Basic sample arrangement before releasing the sample



Annotation

The following recommendations are supposed to ensure a safe system operation:

1. Previous to inserting the sample, lightly polish the sample surface in contact with the electrodes. Linseis recommends sandpaper of No. 1000 or similar.
2. If a sample with a low melting point or sublimating material is measured, or if the sample assembly and electrode are contaminated with outgasings during heating/cooling etc, polish and clean the electrodes.
3. If the electrode contact surface is oxidized or contaminated, polish it with sandpaper No. 1000. Polish the probe contact surface, too.

Please note:

When cleaning the probe, handle it carefully and do not damage it.

Quick start for a sample measurement LSR 3

This is a guideline for a typical procedure to do a measurement

the LSR should be switched on approx. 24 h before use, amplifier has to stabilize.
In the case you want to do frequently measurements leave system switch on all the time

open cooling water supply (see green LED at power supply)

switch on the computer

loose the lock for the furnace and move the furnace to the left side

remove the thermocouple and the susceptor

retract probes to the right side (turn micrometer screen counter clockwise)

Attention !

turn approx. the same amount of turns and a little more back as you did when you moved the probes towards the sample. usually 1 full turn and another ¼ turn

If you don't see a gap between sample and probes they may stick so don't turn further

Open the electrodes with the knob on the top (turn clockwise)

Attention !

turn approx. the same amount of turns and a little more back as you did when you moved the electrodes towards the sample. usually 1 full turn and another ¼ turn

If you don't see a gap between sample and upper electrode sample may stick so don't turn further. In this case see also that lower electrode will be lifted up together with sample

if samples sticks try to loosen the probes first than try to loose contact at upper electrode than at lower. Remember the lower electrode is not fixed and should not be lifted up or twisted. Use proper tooling to do so

Prepare your samples:

- sample must be machined to have perfect parallel ends
- clean all surface with a fine sandpaper (1000 / 1200) and alcohol
- do not touch it with your hand anymore use tweezers or/and gloves
- look for a good contact
- the upper side and the lower side must be right angled to the contact side
- measure the diameter for a round sample or the side a and side b for a square sample before inserting

place the sample on lower electrode

close the electrodes until the sample is fixed. Upper and lower electrode contact the sample. than make approx 1 more full turn with the knob on the top to created some force to hold the sample properly and make good electrical and thermal contact

move probes towards the sample with the micrometer screw. When probes are in contact with sample make approx 1 more full turn with the micrometer screw to make good electrical contact

open the measurement software at the PC

select OPTIONS, ADDITIONAL PARAMETERS and select a proper current for contact test (between 1 and 160 mA)

select OPTIONS, TEST CONTACTS

“Test 1” is the check for the electrode contacts , “Test 2” is the check of the probe contacts.

Insert data for the sample

Setup DAQ

- the name of the sample
- the filename to store to Harddisk
- the distance “x” – you can measure the distance with the digital calliper for more exact results do as good as possible
- insert the diameter of a round sample or and b of a squared sample

Setup Params

- select current (between 1 to 160 mA)
- 2 or 4 terminals
- select the wire Seebeck (material of the probe wires)
- the name heat conductivity file (λ) if available (only necessary to calculate the figure of merit)

Controller --> Temperature profile

- the heating rate (K/min)
- the temperature of the first measuring (usually start from 25°C or 30°C, if start 20°C and tolerance is set to 10K, measurement may never start because the two electrodes always has temperature difference, if the upper one is at room temperature the bottom one cab be up to 50°C higher. if waiting time set long, the temperature of the upper electrode will increase up more than 30°C, this is already out of tolerance (default tolerance is 10K), measurement will not start
- the number of measurement
- Bipolar yes or no. for Seebeck coefficient only. measured bipolar results in higher accuracy
- the time for wait (min)

Attention: if temperature profiles goes up to more than 800°C, add a cooling segment with 30 K/min down to 500°C (0 measurements, gradient heater 0 K)

Install the susceptor and insert thermocouple into susceptor

Close furnace and lock it

Maybe perform another ELECTRODE CONTACT TEST

Open He gas supply at cylinder and adjust approx 5 to 10 psi
switch on the vacuum pump and open the large vacuum valve until the manometer shows -1 Bar

now lock the furnace a little bit more

close the valve at the pump

open the gas valve and fill gas until the manometer shows +0.2 Bar

close the gas valve

do another purging (pumping down and refill) for higher purity of gas

fill chamber again with He to approx +0.2 Bar

close gas input valve

close the valve of the gas bottle.

Stop power to vacuum pump and open venting valve for approx. 2 seconds close venting valve to avoid oil coming up from pump

- "Start" your measurement

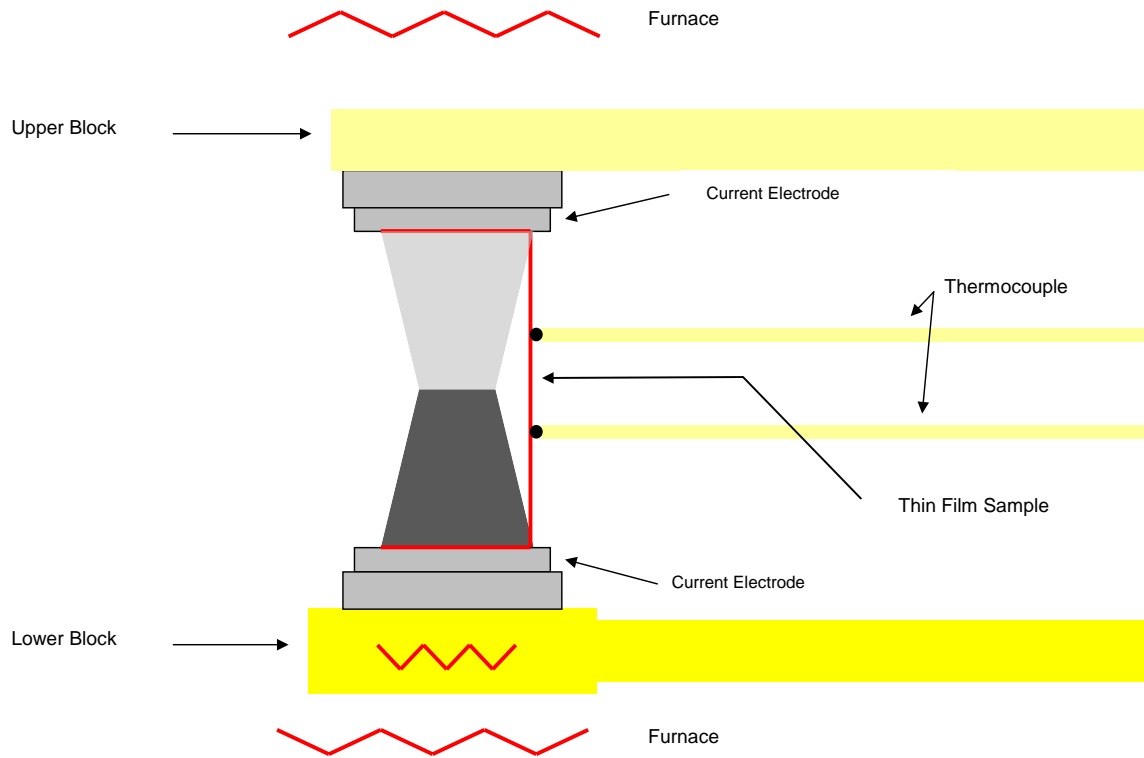
The measurement will be stopped automatically after the complete measurement is done, you can also stop it with "Stop" or "Emergency Stop" in the software or the button at the front panel of the LSR.

Attention !

do not stop the cooling water as long system temperature is higher than 200°C
this can do damage to the Al₂O₃ measuring system.

do not open the furnace as long as the LED "HOT" is on (approx. 65°C)
when you open the furnace be careful, the sample can still be very hot!

Optional available adapter for thin film measurements



Optional available adapter for substrate measurements

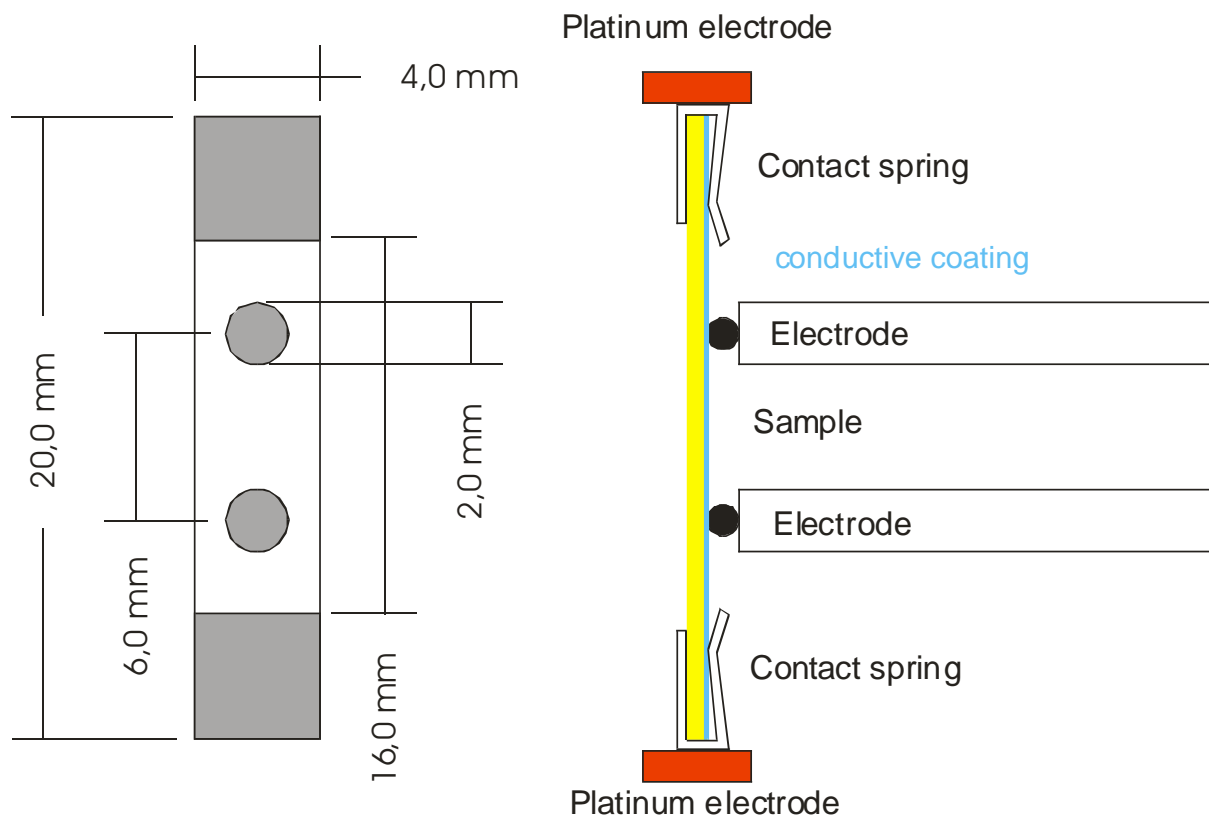


figure 1: Schema sample preparation

Hints:

- The contact springs are suitable for max. 1 mm thick specimens.
- Maximal temperature: 450 °C

Calibration of differences in the signals of Sample Thermocouples with LSR

When welding the thermocouple wires, at the melting spot, there are different materials being created with different material properties, this could be Ni, Cr, or Ni/Cr. This makes it particularly difficult for the Seebeck measurement at the LSR, to have two identical T/C beads, especially in the area, where the beat touches the sample.

The Problem

The fact, that the thermocouple beads are slightly different at the point where they are touching the sample lead to unwanted differing temperature measurements of the relative Seebeck Effect and this leads to wrong measurements of the Absolute Seebeck Effect. This becomes especially noticeable when it is performed with a measurement of a Constantan Standard and after evaluating the Absolute Seebeck Effect you will get widely differing results.

The Solution

There are two solutions of the problem, the most elegant solution is, that you produce thermocouple pairs which have the exact same material property, especially on the touching spot. Unfortunately this is not possible.

The other solution is to adjust the thermocouple temperature with respect to the reference cold junction. This way a fine tuning is possible which has no influence to the measurement and therefore does not produce wrong measuring results.

For example, after the first measurement with the Constantan Standard if the Absolute Seebeck is below the lower limit of the tolerance, you can change the calibration of the upper sample thermocouple for 0,5 to 1,5°C downwards.

On the other hand, in case of exceeding the upper tolerance limit, you have to change the cold junction of the upper sample thermocouple by 0,1 – 1.5°C upwards. Through this fine-tuning of the cold junction you can adjust the differences in the thermo couples until the Absolute Seebeck Effect fits exactly in the middle of the upper and lower tolerance limit.

As additional information we would like to mention that the Relative Seebeck Effect at 30°C is normally between 19 and 20 $\mu\text{V}/\text{K}$, based on the measurement of a Constantan Standard. With this fine-tuning it is relatively simple to calibrate the different thermocouples to show no different signals.

All calibrations are done through the software menu: Options: Adjust Cold Junction Reference

Linseis Standard reference Material

Certificate

Standard reference Material

Constantan

Temp. [°C]	Resitivity [$\mu\Omega$]	ASC [$\mu\text{V/K}$]
20	0.490	-36.40
50	0.491	-38.80
100	0.493	-42.42
150	0.494	-45.58
200	0.496	-48.33
250	0.498	-50.71
300	0.500	-52.75
350	0.501	-54.49
400	0.503	-55.98
450	0.505	-57.24
500	0.507	-58.33
550	0.508	-59.27
600	0.510	-60.12
650	0.512	-60.90
700	0.513	-61.66
750	0.515	-62.43
800	0.517	-63.25

Procedure of Calibration:

The provided Reference Material "Constantan" was heated up to 800°C with 10K/min.

Origin of reference Material:

The provided Constantan has a composition of Cu55/Ni45. For this please refer to manufacturer certificate.

Selb – 95100 / Germany
October 23, 2009
Standard reference program



Instruction Manual
LSR - 3
Seebeck Effect &
Electric Resistivity

Software

This License Agreement is your proof of license. Please treat it as valuable property. This is a legal agreement between you (either an individual or an entity) and Linseis. By breaking the seal on your disk package, you agree to abide by the terms and conditions of this agreement. Failure to comply with the terms of this license terminates the agreement and your rights to use this software.

LINSEIS Software License

1. GRANT OF LICENSE. This Linseis License Agreement ("License") permits you to use this copy of the specified version of the Linseis software product identified above ("SOFTWARE"), provided the SOFTWARE is in use on only one computer at any time. If you have multiple Licenses for the SOFTWARE, then at any time you may have as many copies of the SOFTWARE in use as you wish.

2. RESTRICTIONS. This Linseis License Agreement is your proof of license to exercise the rights granted herein and must be retained by you. You may not rent or lease the SOFTWARE, but you may transfer your rights under this License Agreement on a permanent basis provided you transfer this License Agreement, the SOFTWARE and all accompanying written materials and retain no copies, and the recipient agrees to the terms of this Agreement. You may not reverse engineer, decompile, or disassemble the SOFTWARE. Any transfer of the SOFTWARE must include the most recent update and all prior versions.

3. NO WARRANTY: The software and documentation are provided on an "as is" basis and all risk is with you. Linseis makes no warranties, express, implied, or statutory, as to any matter whatsoever. In particular, any and all warranties of merchantability, fitness for a particular purpose or non-infringement of third parties rights are expressly excluded. Further, Linseis makes no representations or warranties that the software and documentation provided are free of errors or viruses or that the software and documentation are suitable for your intended use.

4. LIMITATION OF LIABILITY: In no event shall Linseis or its suppliers be liable to you or any other party for any incidental, special, or consequential damages, loss of data, or data being rendered inaccurate, loss of profits or revenue, or interruption of business in any way arising out of or related to the use or inability to use the software and/or documentation, regardless of the form of action, whether in contract, tort (including negligence), strict product liability or otherwise, even if any representative of Linseis or its suppliers has been advised of the possibility of such damages.

Software

Introduction

This Software is designed solely for use in combination with the LINSEIS Seebeck and Electric Resistivity measuring unit "LSR". It has been developed exclusively for measurements using the LINSEIS power conversion characteristics evaluation system. The hardware to be used for measurements is limited to the LINSEIS "LSR" instrument; LINSEIS is not liable for any problems and troubles that may arise from the use of this program in an environment other than the current system.

Overview

Before you begin this section, we recommend that you familiarize yourself with the basic operation of the Windows system. Refer to the documentation for the operating system. This chapter provides you with a brief explanation of Instrument Control and general guidelines for setting up an experiment. For further details refer to the online help that accompanies the *Linseis TA-WIN* software.

Definition of Thermal Analysis

Thermal Analysis is defined as the measurement of changes in chemical or physical properties of a sample as a function of temperature or time in a controlled atmosphere. In 1887 thermal analysis was introduced by the French scientist H. Le Chatelier during his study of the decomposition of clays using heat. The early attempts to characterize materials by Le Chatelier involved making photographic records of the heat emitted by the clays, then classifying the clays according to their heating patterns. It was thus that thermal analysis came to be used to identify materials such as minerals, clays, and metallic alloys.

LINSEIS TA-WIN & WIN - LSR SOFTWARE

Standard Software

Easy to use in daily routine, a maximum of intelligent analytical support, highly flexible even for most demanding applications: High performance Thermal Analysis is made easy with TA-WIN & WIN-LSR. A comprehensive software solution, which contains all of the instrument-specific settings, control operations, data storage and evaluation routines in one single package was introduced by LINSEIS Analyzing & Testing with the TA-WIN & WIN-LSR Software.

TA-WIN & WIN - LSR Basic Software

All *LINSEIS* thermo analytical instruments are PC controlled. The individual software modules exclusively run under Microsoft Windows™ operating systems. The complete software consists of 2 modules: data acquisition and data evaluation. The 32 bit software incorporates all essential features for measurement preparation, execution, and evaluation. Thanks to our specialists and application experts, LINSEIS was able to develop comprehensive easy to understand user friendly application software.

2. Installing the TA-WIN & WIN-LSR Software

For USB Connection

!!!! Before connecting Thermo analytical Devices with your PC, make sure that the Software installation has been completed!!!!

To perform a correct software installation, follow the points outlined in the appropriate order, do not leave out any point.

Installation procedure:

1. Place the CD-Rom with the Linseis TAWIN software in the CD-drive.

Normally the installation will now start automatically. In case this does not happen, start the file "setup.exe" located on the CD.

2. Follow the instructions displayed on your monitor.
3. After successfully installing the appropriate software for your ordered thermo analytical device, you should now find the LINSEIS TAWIN software ICONS on your desktop.
4. Now please switch on your Thermal Analyzer and connect it with the computer via the USB cable provided from LINSEIS.
5. WINDOWS will tell you that it has found a new hardware device. The displayed message will indicate that the found device is a Thermal Analyzer.
(The exact Model will be indicated).
6. Windows will now ask how the search for the appropriate driver shall be performed: „automatically or manually“. Confirm that the automatic search shall be performed. Normally Windows will automatically detect and install the driver.

In the event that Windows does not automatically find the correct driver, select the driver manually. The appropriate driver location is as follows: **C:\tawin\driver**

7. After successfully completing the installation, you can now start to perform measurements with your Thermal Analyzer.

Installing the USB-Driver under the 32-Bit Version of WINDOWS 7

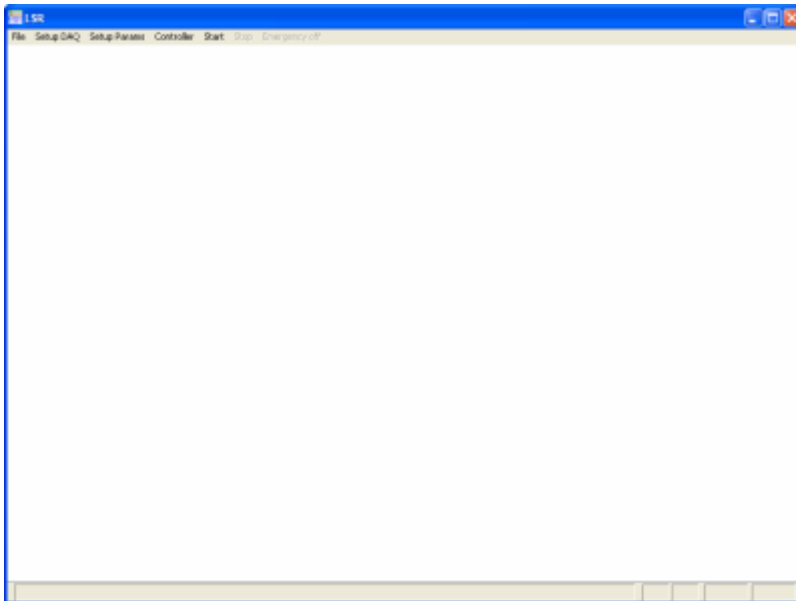
Since the USB-Drivers are not signed for WINDOWS 7, they only can be installed in Test-mode of WINDOWS 7. To do this, either press 'F8' during booting and select "Disable *Driver* Signature Enforcement" to enable Test-mode. Then the drivers can be installed and used normal. Hint: Pressing 'F8' does not make Test-mode permanent – it's only setup until next boot. So, either you have to press 'F8' every time the computer is started, or you must make the Test-mode permanent. To do this, press the 'CTRL' and the 'SHIFT' while opening the MS-Dos command prompt. In the appearing dialog select 'Administrator'. When the command-prompt appears, enter the following command:

```
Bcdedit /set testsigning on
```

To deactivate the Test-mode type:

```
bcdedit /set testsigning off
```

The Measurement Setup



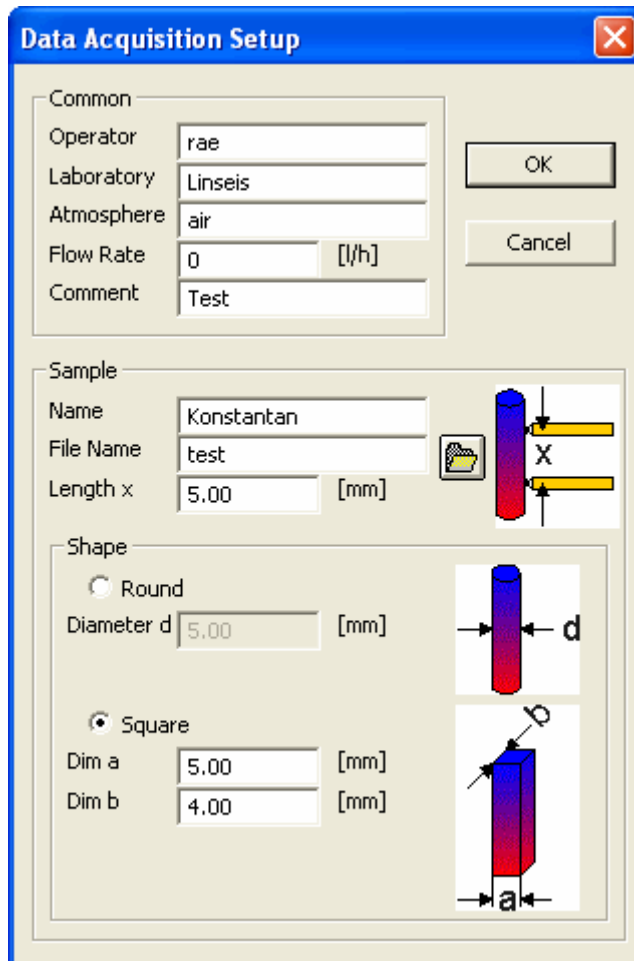
The measurement setup consists of seven “7” action buttons.

The buttons are as follows:

File	Open file Close file Exit program
Setup DAQ	Open data acquisition window
Setup parameter	Enter measurement parameter
Controller	Temperature profile Actual Values PID parameter Cold reference junction
Start	Start measurement
Stop	Stop measurement
Emergency Stop	Stop measurement in the event of an emergency

Data Acquisition

This window encounters all parameters required for the data acquisition setup.



The “Common” parameters:

Operator	Here the name of the user/operator can be entered
Laboratory	This field is for the name of the laboratory, which carried out the measurement. The name of the laboratory is also displayed in the comment field of the data evaluation program.
Atmosphere	This field is for the used gas atmosphere which was used in the measurement. The name of the gas is also displayed in the comment field of the data evaluation program.
Flowrate	This field is for the flowrate of the gas, which is displayed together with the name of the gas in the comment field of the data evaluation program.
Comment	This field is for a short comment about the measurement, which will be also displayed in the comment field of the data evaluation program.

The “Sample” parameters:

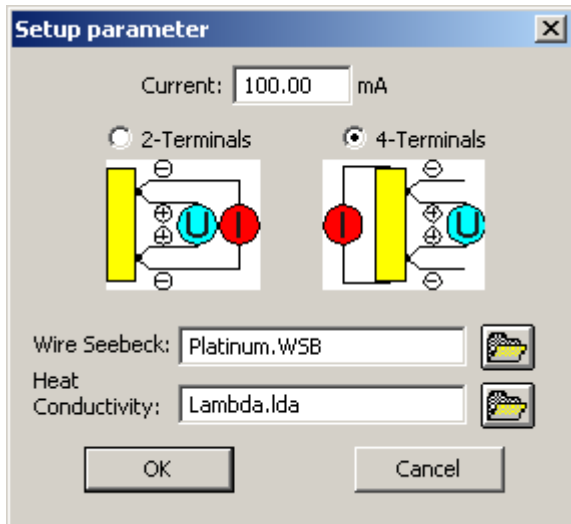
Name	Here the name the sample substance to be evaluated has to be entered
File name	This field is for the name of the sample file, which will hold the measured data. As an alternative it is possible to choose a file in a dialog window with help of the icon on the right side this field. The sample files have (in dilatometer measurements) always the name extension ".IPR". In case this extension is missing, the program will add it automatically. This field is deactivated in a zero measurement; no filename can be entered.
Length	This field is for the length / distance between the two thermocouples attached to the sample

The “Shape” parameters:

Round	The round button indicates that the shape of the sample is round
Diameter d	When the round button is activated the user has to enter the diameter of the sample into this field. Please be as precise as possible when measuring the diameter of the sample. It is best to use a caliper. (in mm)
Square	The square button indicates that the shape of the sample is square
Diam a	When measuring a square sample, the user has to enter the length of side a of the sample here. (in mm)
Diam b	When measuring a square sample, the user has to enter the length of side a of the sample here. (in mm)

Setup parameter

This window requires entering the parameters required for the measurement



The “Setup” parameters:

Current	Here the current applied has to be entered Values between 1 - 160 mA
2 Terminal Method	<p>2 terminal method - check contact about 2 points</p>
4 Terminal Method	<p>4 terminal method - accurate value when measured about 4 points</p>
Wire Seebeck	Here the file name for the measured Wire Seebeck has to be entered
Heat Conductivity	Here the file name for the measured Heat Conductivity has to be entered

Program Controller

The programming of the controller is responsible for the temperature stages of a measurement. Here the heating and cool-down periods of a measurement, as well as the regulator behavior are defined.

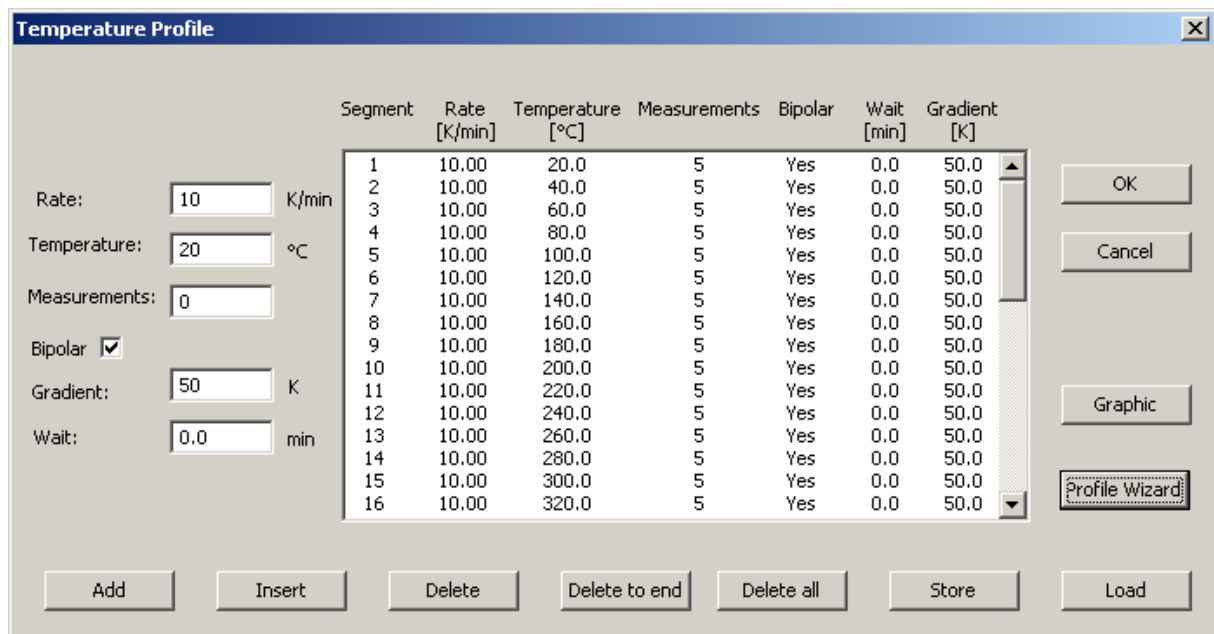
Temperature Profile

With the programming of the controller set values a temperature profile is defined, after which the measurement should run. Hereby the temperature readings are the temperature of the sample and not of the furnace.

Infinite different segments can be programmed, which will be processed one after each other. For every segment five parameters have to be programmed:

Parameters

- Heating Rate: Heating rate to the next segment
- Temperature: Target temperature (°C) for the individual segment
- Measurements: Number of repeating measurements
- Bipolar: The Seebeck Voltage will be measured bipolar and the average will be stored
- Gradient: Here the difference temperature between the primary heat source (the surrounding furnace) and the secondary heat source (heater integrated in the lower electrode body) has to be entered (0 to 50°C)
- Wait: Waiting time before measurement



The dialog box 'Temperature Profile' contains a table with 8 columns: Segment, Rate [K/min], Temperature [°C], Measurements, Bipolar, Wait [min], and Gradient [K]. The table lists 16 segments with a constant heating rate of 10.00 K/min and 5 measurements per segment. The temperature increases from 20.0°C to 320.0°C in 20°C increments. All segments have a wait time of 0.0 min and a gradient of 50.0 K. The 'Bipolar' checkbox is checked. On the left, there are input fields for Rate (10 K/min), Temperature (20 °C), Measurements (0), Gradient (50 K), and Wait (0.0 min). On the right, there are buttons for OK, Cancel, Graphic, and Profile Wizard. At the bottom, there are buttons for Add, Insert, Delete, Delete to end, Delete all, Store, and Load.

Segment	Rate [K/min]	Temperature [°C]	Measurements	Bipolar	Wait [min]	Gradient [K]
1	10.00	20.0	5	Yes	0.0	50.0
2	10.00	40.0	5	Yes	0.0	50.0
3	10.00	60.0	5	Yes	0.0	50.0
4	10.00	80.0	5	Yes	0.0	50.0
5	10.00	100.0	5	Yes	0.0	50.0
6	10.00	120.0	5	Yes	0.0	50.0
7	10.00	140.0	5	Yes	0.0	50.0
8	10.00	160.0	5	Yes	0.0	50.0
9	10.00	180.0	5	Yes	0.0	50.0
10	10.00	200.0	5	Yes	0.0	50.0
11	10.00	220.0	5	Yes	0.0	50.0
12	10.00	240.0	5	Yes	0.0	50.0
13	10.00	260.0	5	Yes	0.0	50.0
14	10.00	280.0	5	Yes	0.0	50.0
15	10.00	300.0	5	Yes	0.0	50.0
16	10.00	320.0	5	Yes	0.0	50.0

Store Controller Set Values

If a temperature profile should be available at a later time for other measurements, then it can be stored after selecting "STORE" on the screen and entering a name.

Load Controller Set Values

To load a temperature profile the field "LOAD" has to be selected. After that another dialog window will appear on the screen, where already stored profiles are listed. From this selection you can pick and activate the desired profile.

Delete Controller Set Values

If, by accident incorrect controller set values were entered in the dialog window, then the whole input field can be deleted by selecting "DELETE" on the screen.

Delete to end Controller Set Values

The entire temperature window can be cleared when pressing this button

Store Values

When pressing this button the generated temperature profile can be stored

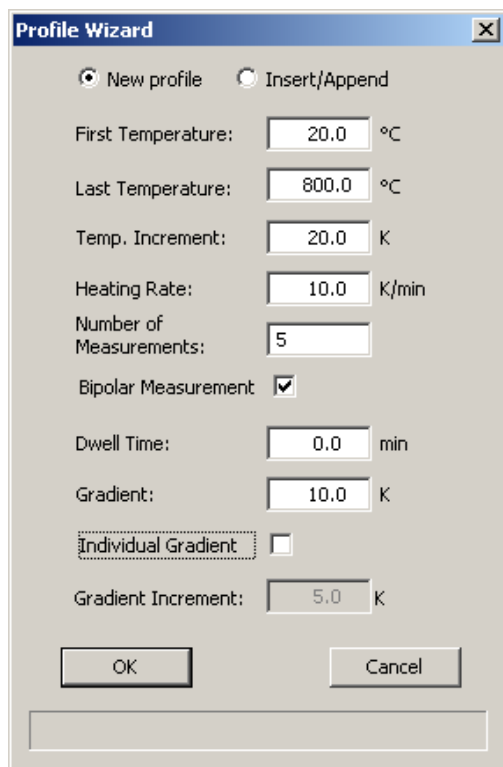
Load Values

When pressing this button a previously generated and stored temperature profile can be loaded to perform a now measurement.

Graphic

With graphic a preview of the temperature profile from the actual or any set Temperature can be displayed. The total running time can be longer than displayed depending on stabilization time at each Temperature segment

Profile Wizzard



with the Profile Wizzard the measurement segments can be created automatically

- **First Temperature:** This is the first Temperature for Measurements. This Temperature must be within the Temperature Tolerance (Options, Additional Parameters).
Attention: The gradient heater can cause a higher Temperature (it is the Real Temp. Sample, or the Temp. in the Upper electrode) If this Temp. to high (outside Tolerance). Measurement can not start
- **Last Temperature:** This is the last Temperature of the heating profile
- **Temp. Increment:** This is the value for the Temperature increase between 2 segments
- **Heating Rate:** Heating rate to the next segment
- **Measurements:** Number of repeating measurements at a specific temperature and Gradient
- **Bipolar:** The Seebeck Voltage will be measured bipolar and the average will be stored
- **Dwell time:** Waiting time before measurement
- **Gradient** Here the difference temperature between the primary heat source (the surrounding furnace) and the secondary heat source (heater integrated in the lower electrode body) has to be entered (0 to 50°C)
- **Individual Gradient:** If Individual Gradient is signed \sqrt it is possible to perform multiple gradients for each Temperature platform.

In this case the gradient value will be the first gradient value

- Gradient Increment:

this will be the step to the next gradient value for the same Temperature platform

the number of increments depends on the number of measurements this is also the limitation for the maximum gradient value. The overall maximum gradient value is 50 K

e.g. 5 measurements, first gradient 10K, increment 5K this will create measurements with 5, 10, 15, 20, 25 K gradient

if the value (first gradient+ number of measurements * increment) is higher than 50 there will be multiple measurements at 50 K gradient

Example

The Profile Wizard dialog box contains the following settings:

- Radio buttons: New profile, Insert/Append
- First Temperature: 20.0 °C
- Last Temperature: 800.0 °C
- Temp. Increment: 20.0 K
- Heating Rate: 10.0 K/min
- Number of Measurements: 5
- Bipolar Measurement:
- Dwell Time: 0.0 min
- Gradient: 10.0 K
- Individual Gradient:
- Gradient Increment: 5.0 K

The Temperature Profile dialog box displays a table of profile segments with the following data:

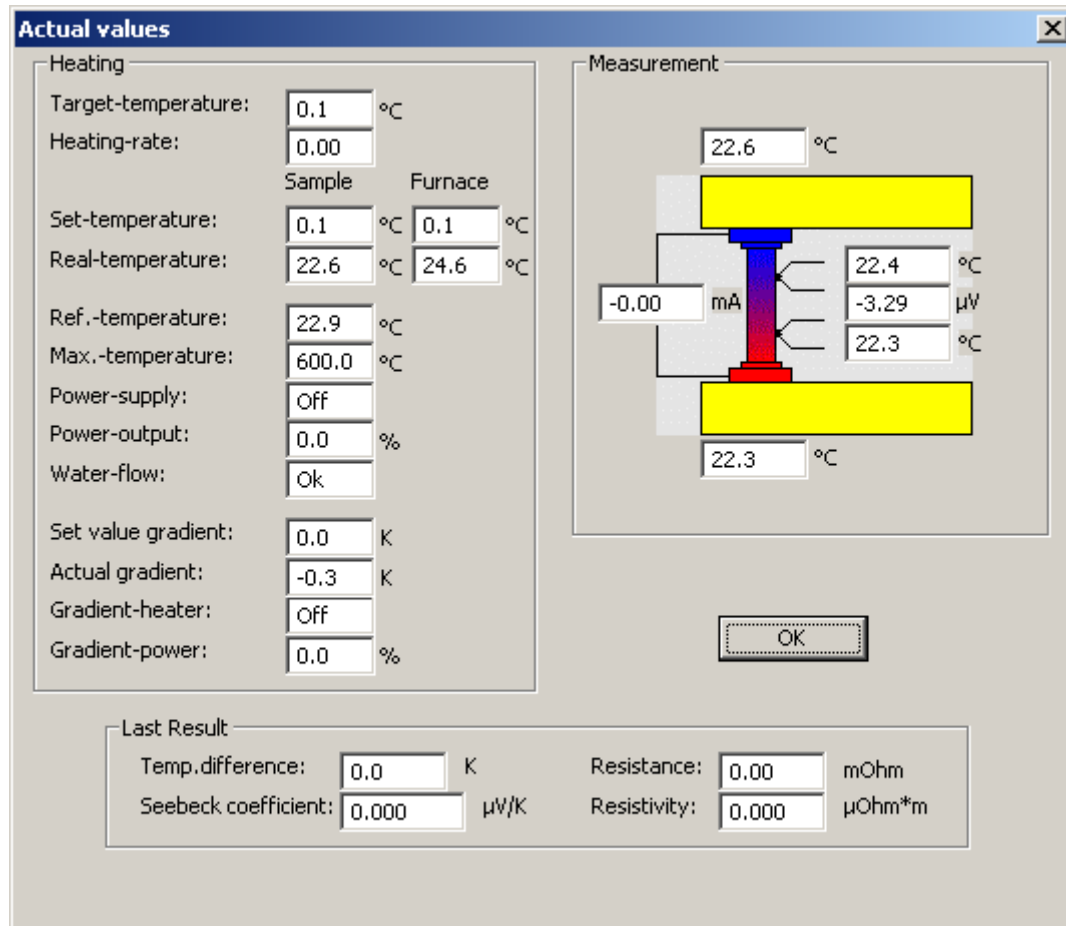
Segment	Rate [K/min]	Temperature [°C]	Measurements	Bipolar	Wait [min]	Gradient [K]
1	10.00	20.0	1	Yes	0.0	10.0
2	10.00	20.0	1	Yes	0.0	15.0
3	10.00	20.0	1	Yes	0.0	20.0
4	10.00	20.0	1	Yes	0.0	25.0
5	10.00	20.0	1	Yes	0.0	30.0
6	10.00	40.0	1	Yes	0.0	10.0
7	10.00	40.0	1	Yes	0.0	15.0
8	10.00	40.0	1	Yes	0.0	20.0
9	10.00	40.0	1	Yes	0.0	25.0
10	10.00	40.0	1	Yes	0.0	30.0
11	10.00	60.0	1	Yes	0.0	10.0
12	10.00	60.0	1	Yes	0.0	15.0
13	10.00	60.0	1	Yes	0.0	20.0
14	10.00	60.0	1	Yes	0.0	25.0
15	10.00	60.0	1	Yes	0.0	30.0
16	10.00	80.0	1	Yes	0.0	10.0

Additional controls in the dialog include input fields for Rate, Temperature, Measurements, Gradient, and Wait, along with buttons for Add, Insert, Delete, Delete to end, Delete all, Store, Load, OK, Cancel, Graphic, and Profile Wizard.

Display actual values

It might be interesting during a measurement to get a view of the actual temperature and control parameter of the system. This is made possible by choosing the menu selection "CONTROLLER - ACTUAL VALUES".

After that the following parameters will be displayed:



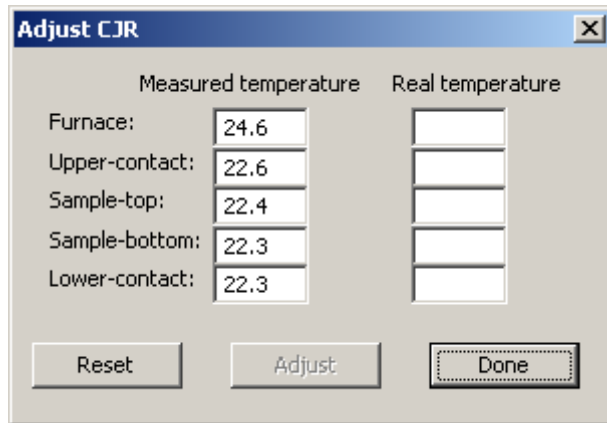
The screenshot shows a software window titled "Actual values" with a close button (X) in the top right corner. The window is divided into several sections:

- Heating:**
 - Target-temperature: 0.1 °C
 - Heating-rate: 0.00
 - Set-temperature: 0.1 °C (Sample) / 0.1 °C (Furnace)
 - Real-temperature: 22.6 °C (Sample) / 24.6 °C (Furnace)
 - Ref.-temperature: 22.9 °C
 - Max.-temperature: 600.0 °C
 - Power-supply: Off
 - Power-output: 0.0 %
 - Water-flow: Ok
 - Set value gradient: 0.0 K
 - Actual gradient: -0.3 K
 - Gradient-heater: Off
 - Gradient-power: 0.0 %
- Measurement:**
 - Top yellow bar: 22.6 °C
 - Bottom yellow bar: 22.3 °C
 - Left side: -0.00 mA
 - Right side: 22.4 °C, -3.29 μV, 22.3 °C
- Last Result:**
 - Temp. difference: 0.0 K
 - Resistance: 0.00 mOhm
 - Seebeck coefficient: 0.000 μV/K
 - Resistivity: 0.000 μOhm*m

An "OK" button is located at the bottom center of the window.

Adjust Reference Cold Junction

The controller board uses a built in reference cold junction to compensate the thermal voltages in the input connections. With this reference cold junction an adjustment of the sample thermocouple can be done. Hereby the thermocouple has to be brought up to a known temperature (e.g. 0°C in icewater). Or well known temperature of the environment. Then an adjustment dialog will be displayed on the screen ("CONTROLLER - ADJUST REFERENCE COLD JUNCTION").



The image shows a software dialog box titled "Adjust CJR" with a close button (X) in the top right corner. The dialog is divided into two columns: "Measured temperature" and "Real temperature".

	Measured temperature	Real temperature
Furnace:	24.6	
Upper-contact:	22.6	
Sample-top:	22.4	
Sample-bottom:	22.3	
Lower-contact:	22.3	

At the bottom of the dialog, there are three buttons: "Reset", "Adjust", and "Done". The "Done" button is highlighted with a dashed border.

Here, the actual temperature of the thermocouple is displayed, and the user can now enter the set temperature, which was assumed to be measured (e.g. 0°C). By using the field "ADJUST", the adjustment is made. The accuracy of the adjustment can be probably increased by repeating this step a few times.

If by accident the thermocouple had been adjusted to the incorrect temperature, then the adjustment can be reset by using the field "RESET" and then be started again.

Once the adjustment of the sample thermocouple was made, the according correction value will be stored and is used for future measurements, even if the program was turned off in the meantime.

This adjustment should be done only when the system is closed and stabilized for approx. 12 hours. In this case enter for all 5 Thermocouples the same Temperature (room Temperature)

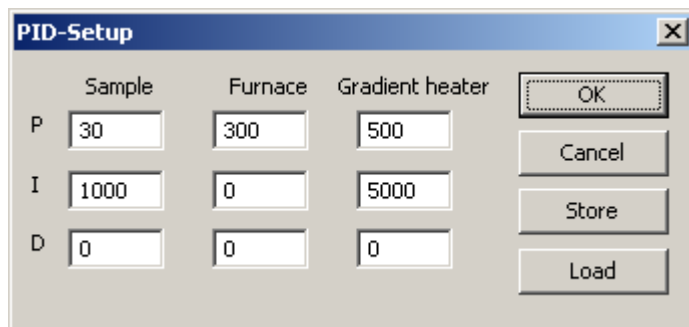
PID-Parameters

The PID-Parameters change directly the control behavior of the electronic temperature controller and have to be selected very carefully.

The controller works as a cascade controller, where it will process the sample as well as the furnace temperature as control parameters.

Parameter Definition

The PID-parameters have to be entered as separate P-, I- and D- fractions for controller 1 (sample temperature), as well as controller 2 (furnace temperature). The value range for each parameter can be between 0 and 32767. The presetting are made according to known values of Linseis.



The image shows a 'PID-Setup' dialog box with a table of parameters and control buttons. The table has three columns: 'Sample', 'Furnace', and 'Gradient heater'. The rows represent the P, I, and D parameters. The 'OK' button is highlighted with a dashed border.

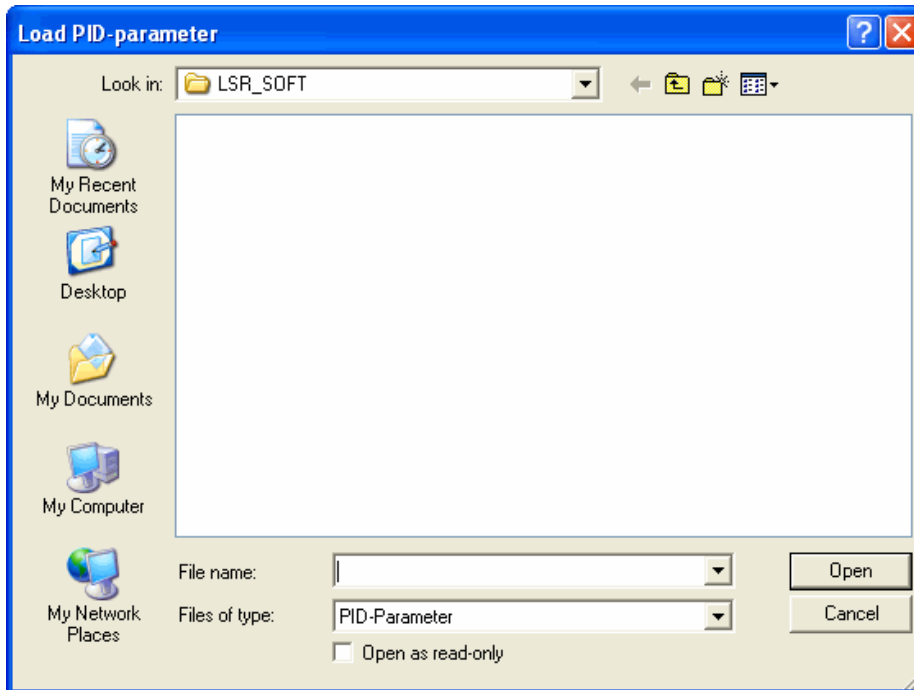
	Sample	Furnace	Gradient heater
P	30	300	500
I	1000	0	5000
D	0	0	0

Buttons: OK, Cancel, Store, Load

If the controller should be used in single state mode instead of a cascade mode, then all the parameter fractions of controller one have to be set to zero. The actual value used for the control loop uses then only the furnace temperature.

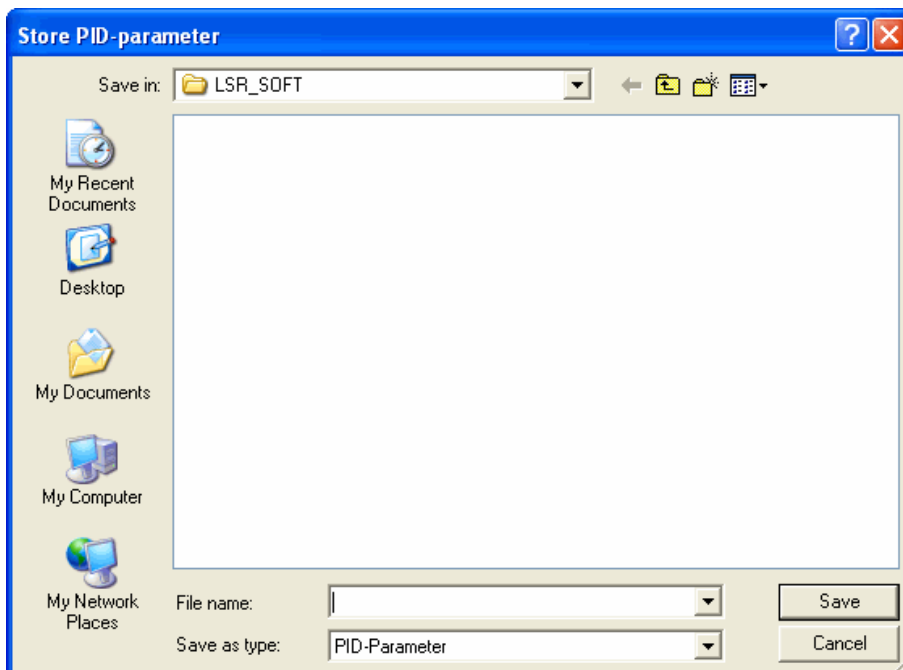
Store Parameters

If various sets of parameters should be available for different measurements, then these can be stored after selecting "STORE" and recalled for later measurements.



Load Parameters

To load an already stored set of parameters for a measurement the field "LOAD" has to be selected in the window for the setting of the PID-parameters.



Default Parameters

Sometimes it can happen that unfavorable parameters were set for the controller and that the proper parameters are lost. In this case we recommend to choose the defaults and change the parameters from there until the desired behavior is accomplished. The presetting can be entered through selecting the field "DEFAULT".

Start and stop measurement

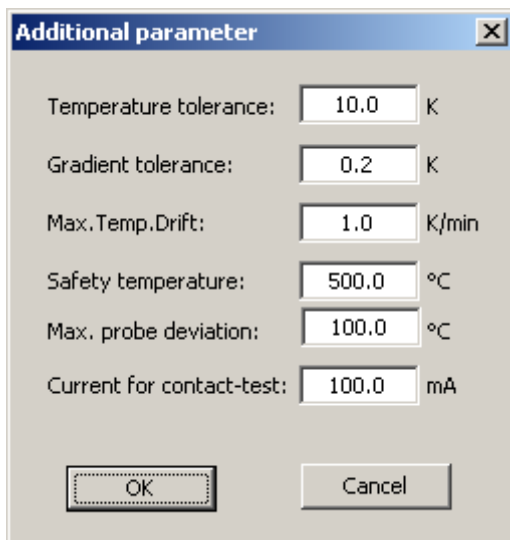
To start and stop a measurement are two fields marked "ON" and "OFF" in the main menu. After selecting one of those options a dialog window appears on the screen, which shows the user what choices he has.

In particular the data acquisition, and the controller program can be started and stopped individually. While a measurement is in progress, on the lower side of the screen the following important information about the measurement will be displayed:

- Number of stored data sets
- Duration of the measurement
- Controller program active
- Data Acquisition active

Options

Additional Parameters:



Parameter	Value	Unit
Temperature tolerance:	10.0	K
Gradient tolerance:	0.2	K
Max. Temp. Drift:	1.0	K/min
Safety temperature:	500.0	°C
Max. probe deviation:	100.0	°C
Current for contact-test:	100.0	mA

- **Temperature Tolerance:** When a set value Temperature of a heating profile is reached, the system will check if the real Temperature (real sample Temp, upper electrode Temp) is within the Tolerance margins \pm than measurements will be done.
- **Gradient Tolerance:** This is the Tolerance between set value and real value of the gradient Temperature
- **max. Temp Drift:** This is the maximum allowed Drift for Temperature and Gradient before a measurement will be done
- **Safety Temperature::** This is a limitation for the heating, this value can not be exceeded
- **Max Probe Deviation:** if the probe shows higher deviation than adjusted than the

system performs an emergency stop

- current for contact test: this is the current during contact test values are 1 - 160mA

Clear graphic

- clear graphic: this will clear the real time graphic screen after measurement is finished

Test Contacts

- Test contacts:: This performs a contact test for the sample. First test use the contact test current and checks if the sample has good electrical contact to the lower and upper electrode.

If it fails the following message will be displayed

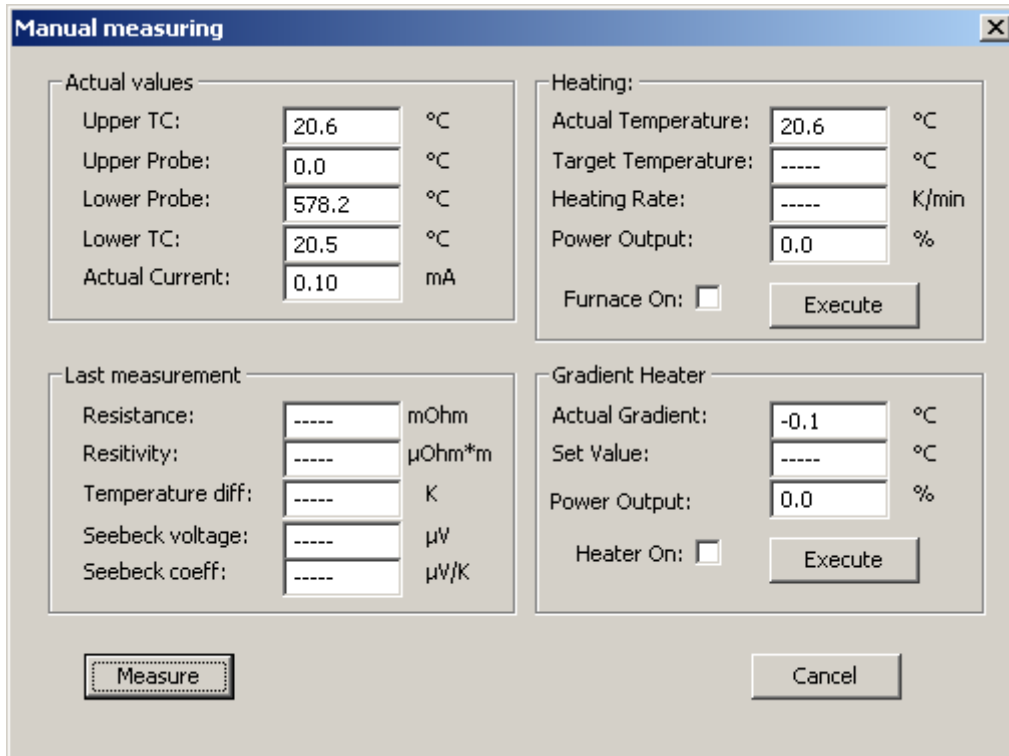


Second test will check if Probe Thermocouples has good electrical Contact with the sample

If it fails the following message will be displayed



Manual Measurement



Manual measuring

Actual values		Heating:	
Upper TC:	20.6 °C	Actual Temperature:	20.6 °C
Upper Probe:	0.0 °C	Target Temperature:	----- °C
Lower Probe:	578.2 °C	Heating Rate:	----- K/min
Lower TC:	20.5 °C	Power Output:	0.0 %
Actual Current:	0.10 mA	Furnace On:	<input type="checkbox"/>
			<input type="button" value="Execute"/>
Last measurement		Gradient Heater	
Resistance:	----- mOhm	Actual Gradient:	-0.1 °C
Resitivity:	----- μOhm*m	Set Value:	----- °C
Temperature diff:	----- K	Power Output:	0.0 %
Seebeck voltage:	----- μV	Heater On:	<input type="checkbox"/>
Seebeck coeff:	----- μV/K		<input type="button" value="Execute"/>
		<input type="button" value="Measure"/>	<input type="button" value="Cancel"/>

Heating:

When execute and furnace on field is signed \checkmark the system will heat up with specified heating rate to the specified target temperature. After reaching this temperature this will be hold until furnace on is signed off and execute or window is closed.

Gradient heater:

When execute and heater on is signed \checkmark the system will heat a gradient up to the value specified with set value. This value will be hold until heater is signed off and execute or window is closed

Measure:

A manual measurement will be done at the actual temperature and gradient (Display of Seebeck coefficient is only relative not corrected to absolute)

Instruction Manual
LSR - 3
Seebeck Effect &
Electric Resistivity
Data-Evaluation

Content:

1. Getting started	57
1.1 Opening an existing evaluation	57
1.2 Creating a new evaluation window	57
1.3 Loading traces	58
2 Evaluation	60
2.1 Calculation of the absolute seebeck coefficient	60
2.2 Calculation of the Power Factor	62
2.3 Figure of merit	64
2.4 Figure of merit Z	64
2.5 Figure of merit ZT	65
3 Other evaluation functions.....	66
3.1 Smoothing	66
3.2 Collection file.....	67

1. GETTING STARTED

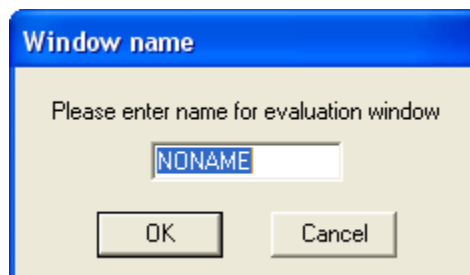
After starting the evaluation software, a new evaluation window must be created, or an existing evaluation must be opened.

1.1 Opening an existing evaluation

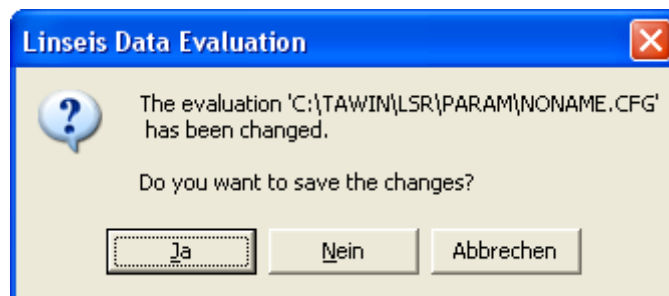
Select 'File' – 'Open' from main menu, select the desired configuration file and click 'Open'.

1.2 Creating a new evaluation window

Select 'File' – 'New' from main menu. Enter the desired name for the evaluation window in the appearing dialog:

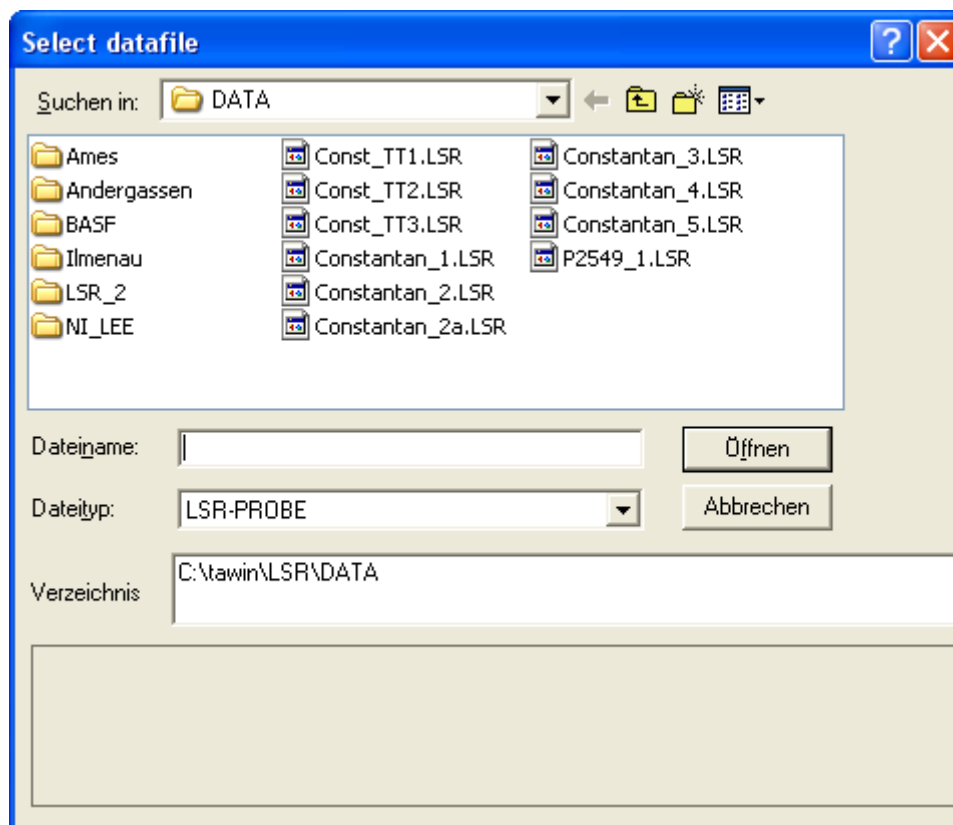


The name entered here is used for building the name of the configuration file (*.CFG) and the name of the table print file (*.TAB). Note: The name may have up to 8 characters, and must not include special characters – letters and numbers are ok. As default the name "NONAME" is used. If another evaluation with the same name exists it will be over-written when closing the evaluation window and 'Yes' is selected from the appearing Dialog:

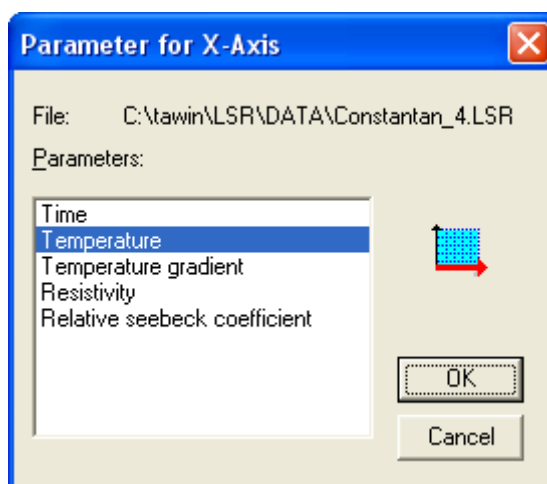


1.3 Loading traces

To load measured data into the evaluation window, select 'File' – 'Load Data' from main menu:

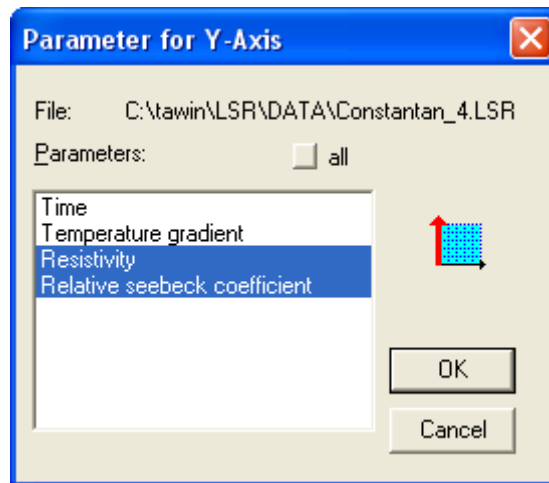


Select the desired data-file and click 'Open':

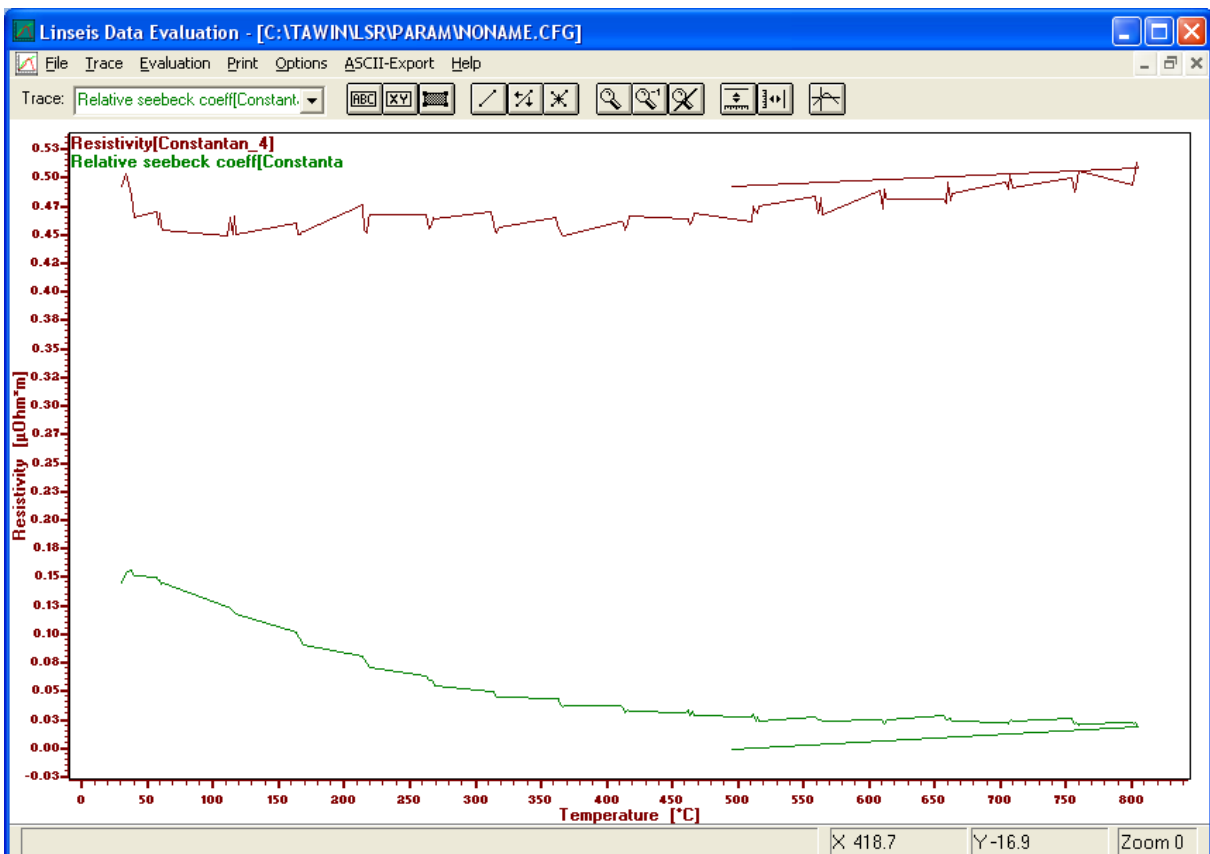


The data-files from LSR-3 measurements have five columns of data: Time[min], Temperature[°C], Temperature gradient[K], Resistivity [$\mu\Omega\text{m}$] and the Relative seebeck coefficient [$\mu\text{V/K}$]. The temperature stored is the average temperature measured by the upper and the lower probe, while the temperature gradient is the temperature difference of the temperatures measured by the upper and

lower probes. In the appearing dialog you must select, which of the columns shall be used as X-Axis in the diagram. Usually Temperature or Time is used. After clicking Ok, the dialog for Y-Axis selection appears:



Usually the Resistivity and the Relative seebeck coefficient are loaded. After clicking Ok, the selected columns are drawn into the diagram:



2 EVALUATION

After one or more traces have been loaded, a couple of evaluation steps can be performed:

2.1 Calculation of the absolute seebeck coefficient

The measured seebeck coefficient is not the absolute seebeck coefficient of the sample measured. The reason is, that the material of the probes used to measure the seebeck coefficient has an own absolute seebeck coefficient. So, the measured seebeck coefficient is the difference of the seebeck coefficient of the sample and the material of the probes. To correct this, the absolute seebeck coefficient of the probe's material must be added to the measured seebeck coefficient of the sample to get the absolute seebeck coefficient of the sample:

$$ASC_{sample} = RSC_{sample} + ASC_{probes}$$

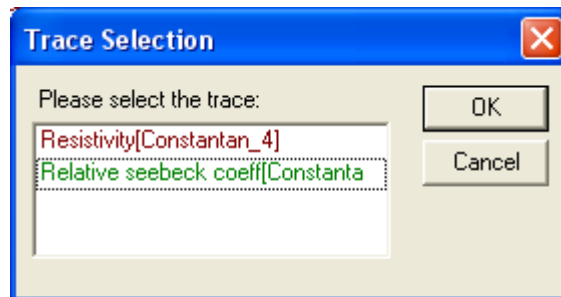
The values for the absolute seebeck coefficient of the probe's material are stored in a table-file. This is a text-file in the C:\TAWIN\LSR\REF directory, having the extension *.WSB (for wire seebeck). It consists out of two columns: 1st Temperature[°C], 2nd Absolute seebeck coefficient of the material used for the probes[μV]:

Example of the Platinum table:

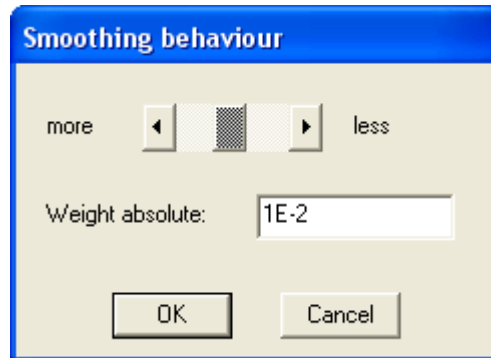
```
0      -4.45
27     -5.28
127    -7.83
227    -9.89
327   -11.66
427   -13.31
```

Values not included in the table are inter- or extrapolated from the existing entries.

To calculate the absolute seebeck coefficient at least one relative seebeck coefficient trace must be loaded. If more than one trace is loaded, a trace select dialog appears after selecting 'Evaluation – Absolute seebeck coefficient' from menu:

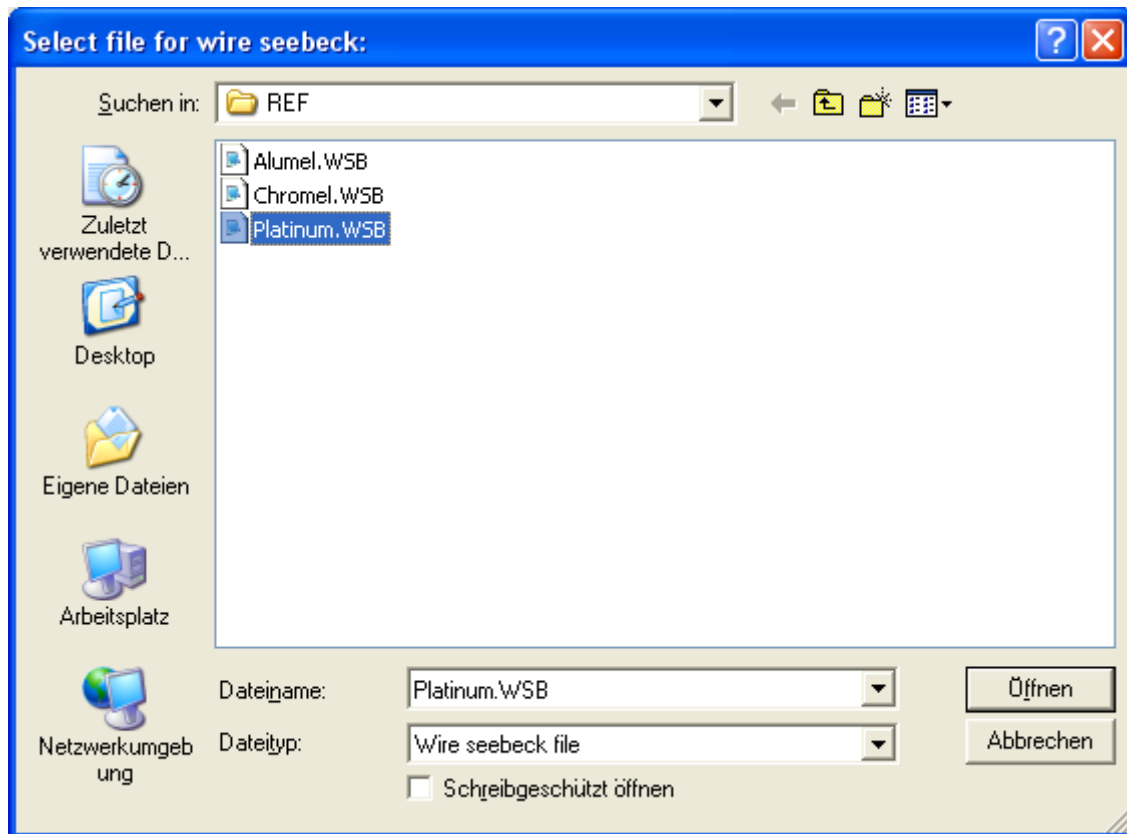


Select the desired trace and click OK. Next the dialog for smoothing of the X-Axis appears:

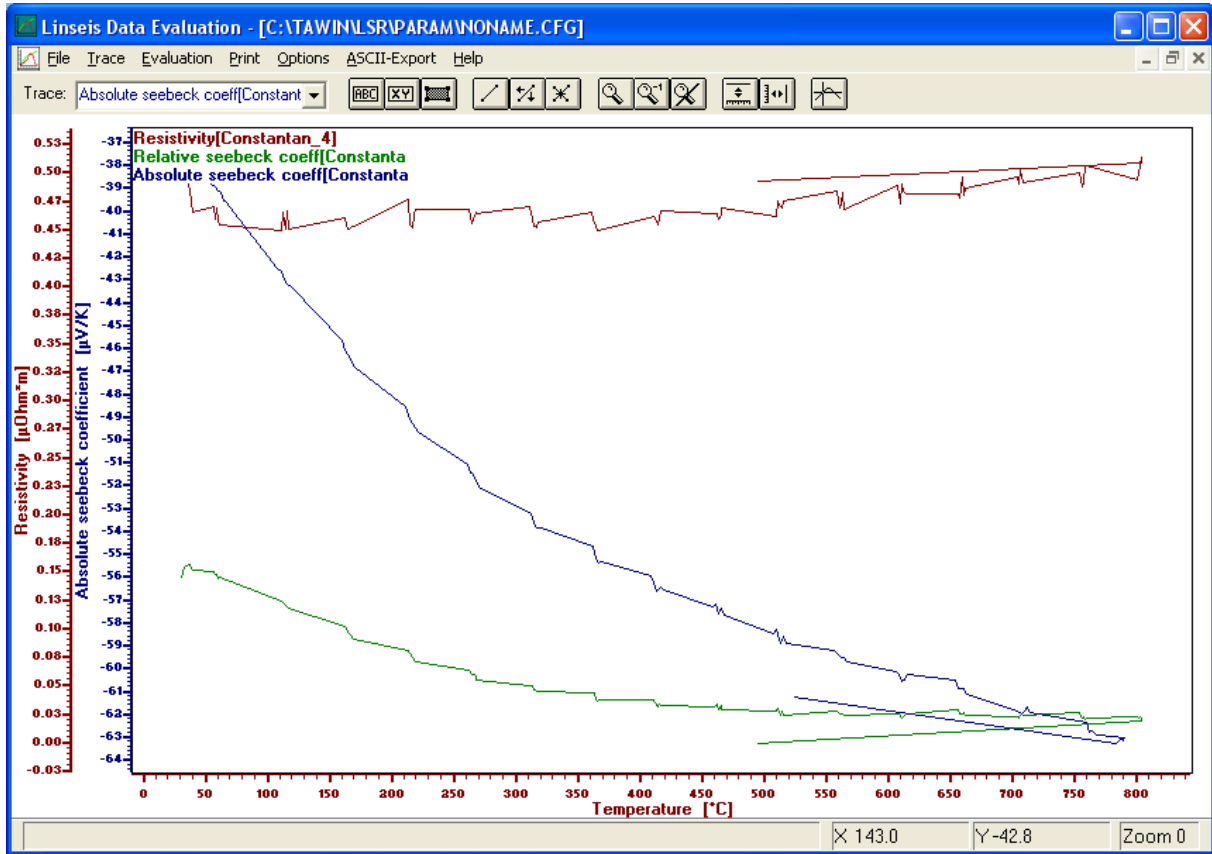


Simply keep the default setting and click OK. If there is a problem during the table print, that a part of the data is missing, perform more smoothing at this point.

Next, the dialog to select the wire seebeck table appears:



In this dialog the proper material for the probes used must be selected. It's either Platinum for Type-S probes, or Alumel for Type-K probes. After clicking 'Open' the absolute seebeck coefficient is calculated and displayed:



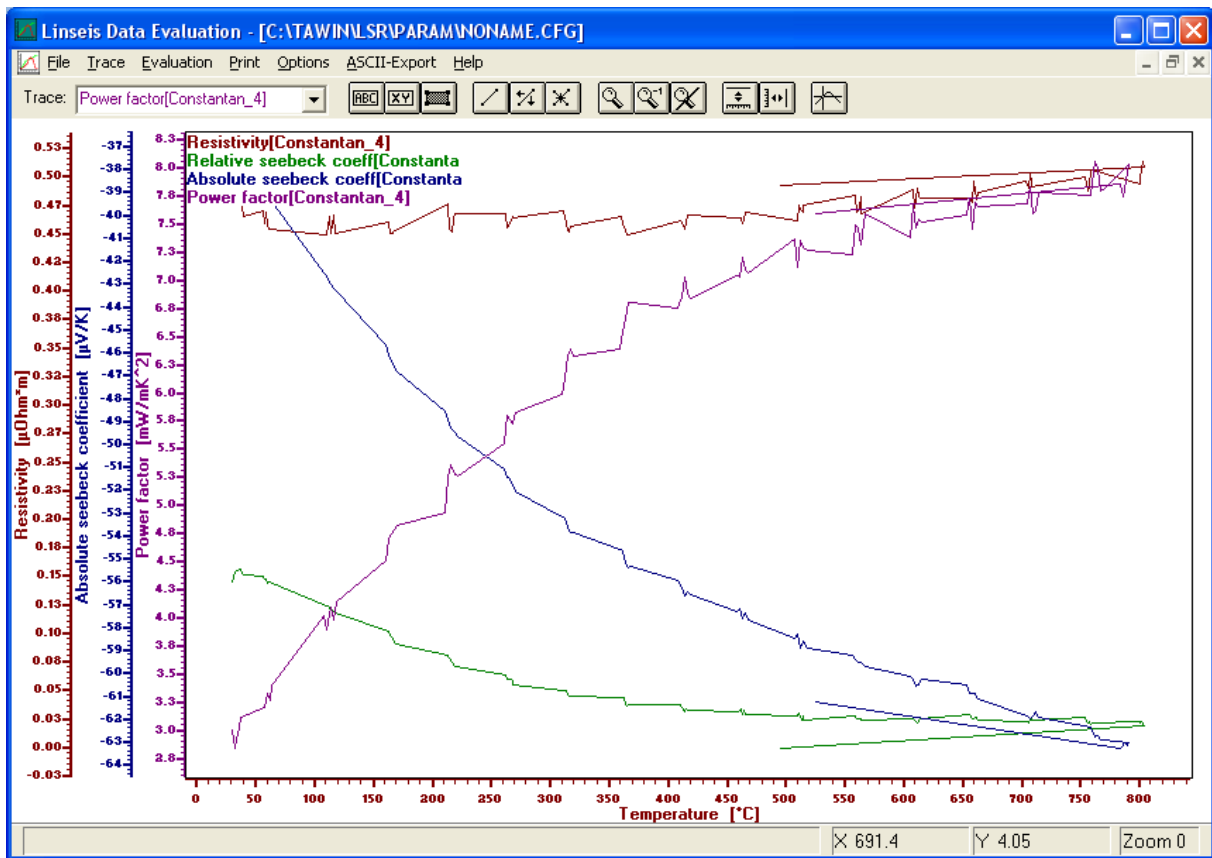
2.2 Calculation of the Power Factor

Since now the absolute seebeck coefficient and the resistivity are known, the Power Factor can be calculated:

$$PF = S^2 / \rho$$

PF=Power-factor [mW/mK²], S=Absolute seebeck coefficient [$\mu\text{V/K}$], ρ =Resistivity [$\mu\Omega\text{m}$]

After selecting 'Evaluation – Power factor', the power factor is calculated and the trace is added to the graphics:



2.3 Figure of merit

To calculate the figure of merit the thermal conductivity of the sample must be known. The values for the thermal conductivity of the probe's material are stored in a table-file. This is a text-file in the C:\TAWINLSR\REF directory, having the extension *.LDA (for Lambda). It consists out of two columns: 1st Temperature[°C], 2nd Thermal conductivity of the material used for the probes[W/mK]:

Example of the Constantan table:

```
20 49.0
```

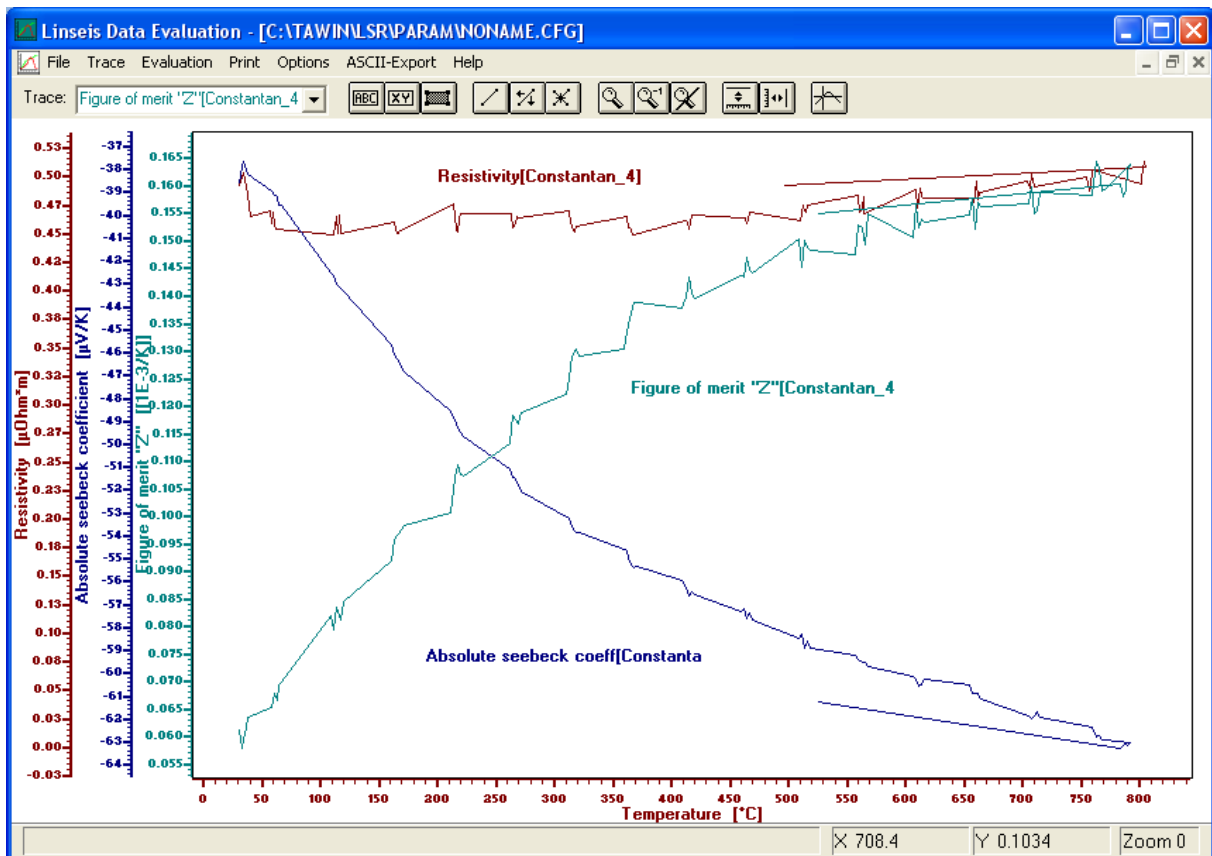
Values not included in the table are inter- or extrapolated from the existing entries. In this case only one entry is given, so it's used for all temperatures.

2.4 Figure of merit Z

After selecting 'Evaluation – Figure of merit Z' the figure of merit is calculated:

$$Z = S^2 / (\rho * \lambda)$$

Z = Figure of merit[1E-3/K], S=Absolute seebeck coefficient [µV/K], ρ=Resistivity [µΩm],
λ = Thermal conductivity [W/m*K]

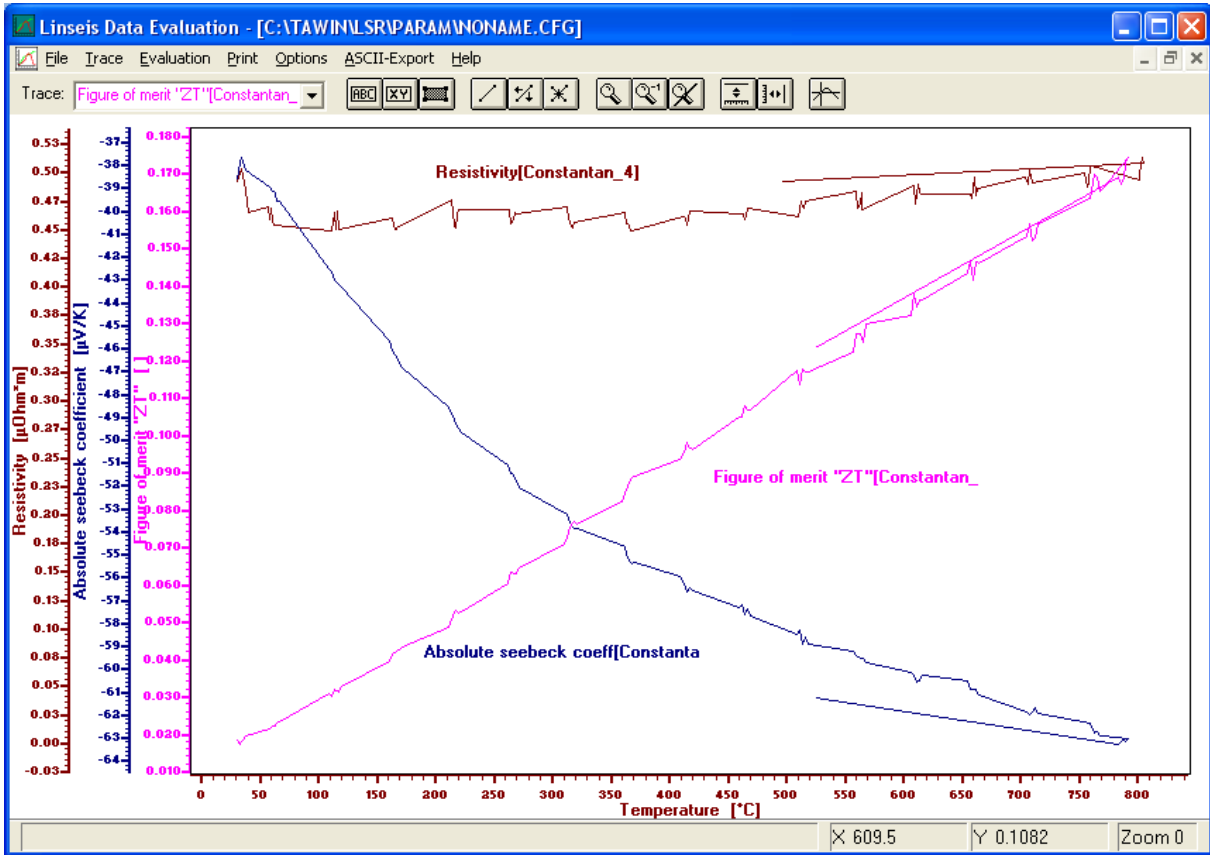


2.5 Figure of merit ZT

After selecting 'Evaluation – Figure of merit ZT' the figure of merit, related to the absolute temperature is calculated:

$$ZT = S^2 / (\rho * \lambda) * T$$

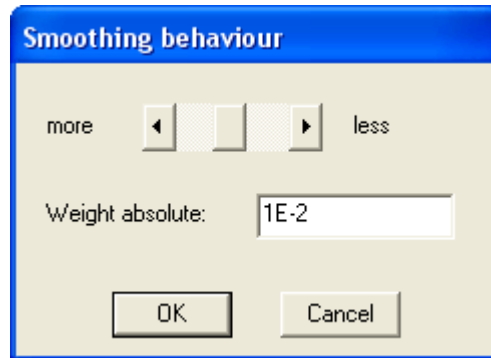
ZT = Figure of merit[], S=Absolute seebeck coefficient [$\mu\text{V}/\text{K}$], ρ =Resistivity [$\mu\Omega\text{m}$],
 λ = Thermal conductivity [$\text{W}/\text{m}^*\text{K}$], T absolute temperature [K]



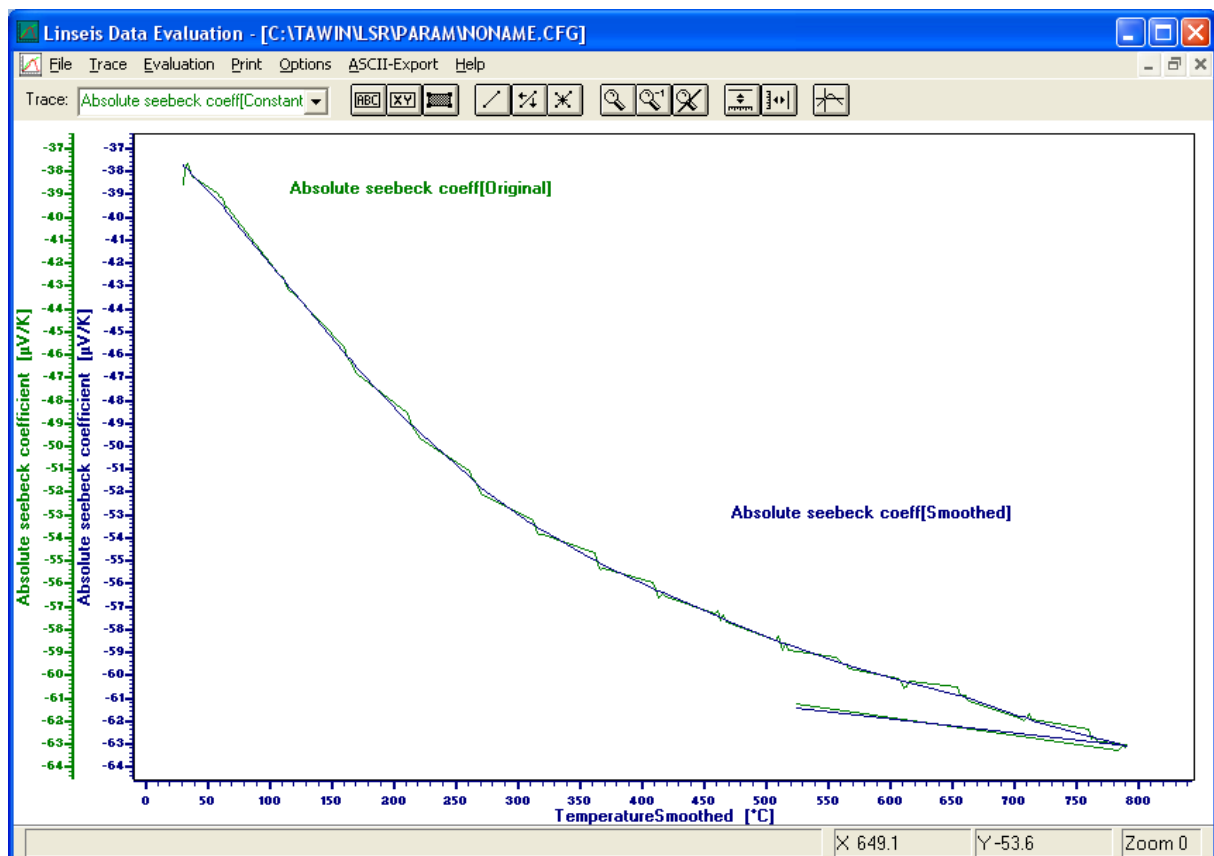
3 OTHER EVALUATION FUNCTIONS

3.1 Smoothing

By selecting 'Evaluation – Y-smoothing' a smoothing algorithm is applied to the Y-Axis of the selected trace. In the appearing dialog the amount of smoothing can be selected:

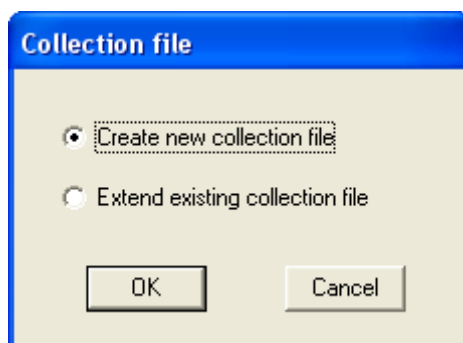


Result after smoothing:

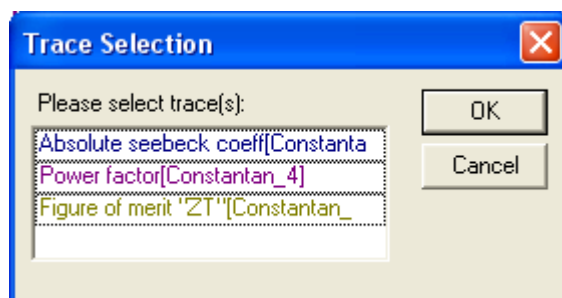


3.2 Collection file

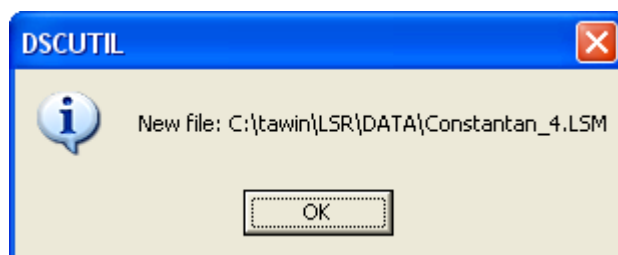
This is a helpful function for data export. If you have got several traces during evaluation, each one is an individual file. By creating a collection file, several traces can be put together in a single file with several columns: Column 1 and 2 always time and temperature, plus one extra column of Y-data for each trace included in the collection file. After selecting this function, an appearing dialog asks if a new collection file shall be created, or if an existing one shall be expanded:



After clicking OK, a list with all traces in the diagram is displayed:



Select all the traces which shall be included in the collection file and click OK. Now the collection file is generated. It has the same name like the original file and the extension *.ISM:



If now this file is exported, using 'ASCII-Export' from main menu, the result is a text-file, containing the data of all the traces specified:

```

NONAME: Collection file (Whole trace)
Source file:Constantan_4.L16 (X:Time, Y:Temperature gradient)
CONSTANTAN_4
Wed Oct 21 09:23:19 2009
Data Source: LSR
File Type: ORDER
Channels: 7
1: Time
Sensor Type: Clock
Sensor Range: -200.000 200.000 min
User Range: -200.000 200.000 min
Minimum: 0.718 min
Maximum: 112.802 min
2: Temperature Smoothed
Sensor Type: Type-S
Sensor Range: -1000.000 1000.000 °C
User Range: -1000.000 1000.000 °C
Minimum: 29.386 °C
Maximum: 790.113 °C
3: Temperature gradient
Sensor Type: Type-S
Sensor Range: -10.000 10.000 K
User Range: -10.000 10.000 K
Minimum: 4.865 K
Maximum: 9.644 K
4: Resistivity
Sensor Type: Unknown
Sensor Range: -1.000 1.000 µOhm*m
User Range: -1.000 1.000 µOhm*m
Minimum: 0.450 µOhm*m
Maximum: 0.514 µOhm*m
5: Absolute Seebeck coefficient
Sensor Type: Unknown
Sensor Range: -100.000 100.000 µV/K
User Range: -100.000 100.000 µV/K
Minimum: -63.040 µV/K
Maximum: -37.679 µV/K
6: Power factor
Sensor Type: Unknown
Sensor Range: -10.000 10.000 µV/K
User Range: -10.000 10.000 mW/mK^2
Minimum: 2.893 mW/mK^2
Maximum: 7.877 mW/mK^2
7: Figure of merit "ZT"
Sensor Type: Unknown
Sensor Range: -1.000 1.000 µV/K
User Range: -1.000 1.000
Minimum: 0.018
Maximum: 0.169
0.7183 29.3858 4.8647 0.4929 -37.6788 2.8930 0.0176
1.0317 31.2643 6.1664 0.5036 -37.7804 2.9234 0.0180

```

Instruction Manual
LSR - 3
Seebeck Effect &
Electric Resistivity

Maintenance & Service

Exchange of Sample-Thermocouple



Pic 1: Open Locking Screw for removal



Pic 2: Pull out tube with 2 TC



Pic 3: See above



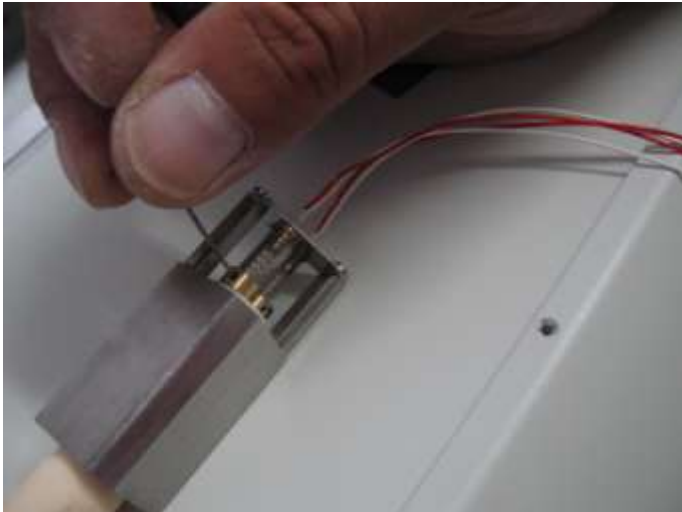
Pic 4: Pull out Al₂O₃ Locking tube



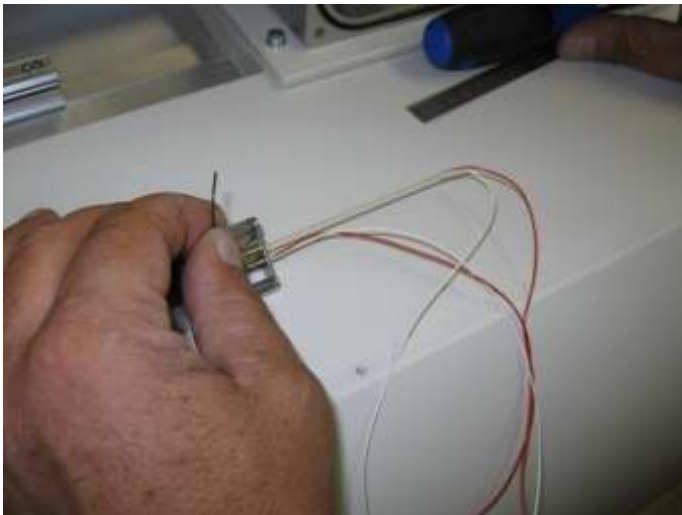
Pic 5: All parts removed on table



Pic 6: Measure length of overstanding TC, necessary for later assembly



Pic 7: loosen allenscrew



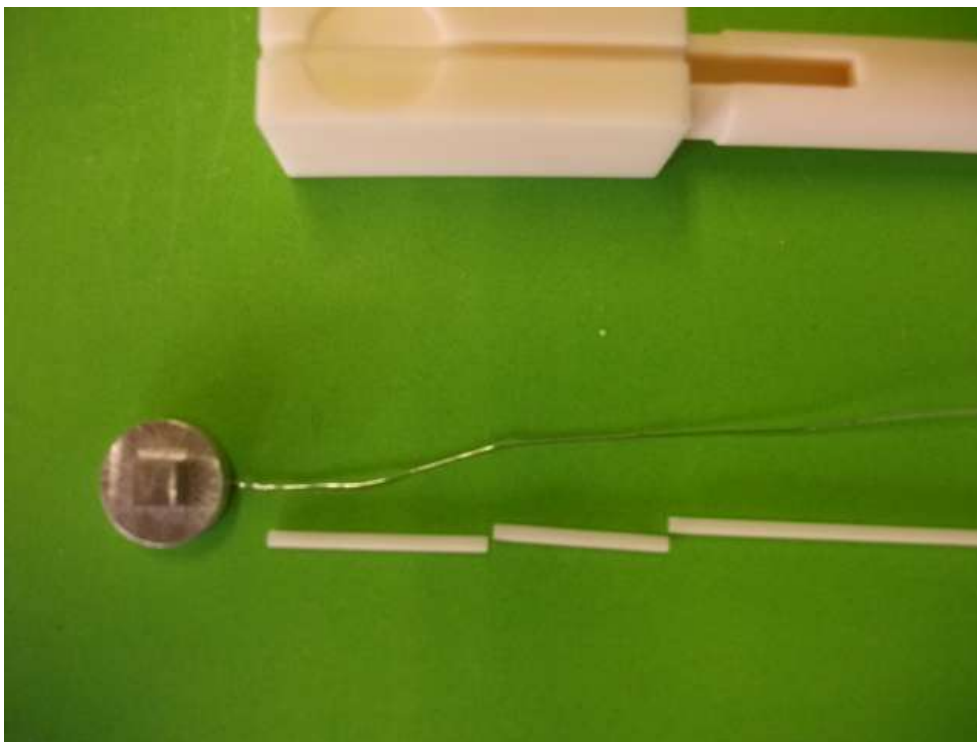
Pic 8: Pull out TC carefully



Pic 9: TC is removed

For replacement of TC do all steps backwards

Exchange of electrodes



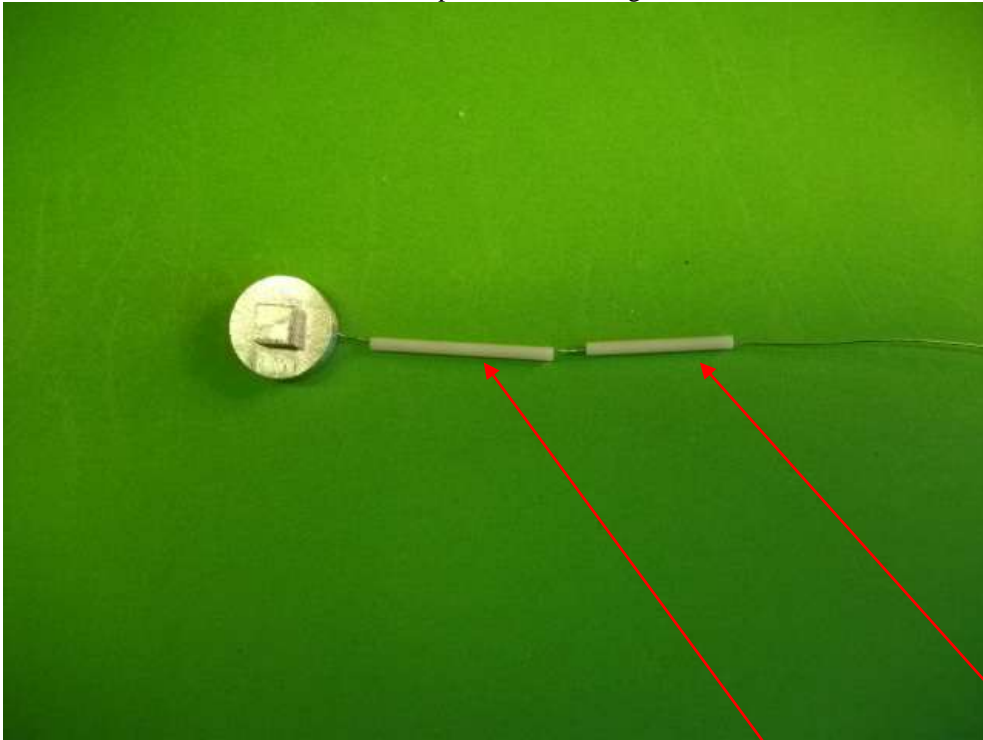
0410

The necessary parts for the exchange: New electrode, two short and one long Al_2O_3 capillaries. When taking the electrode out and back in please make sure to have the capillaries in right order.



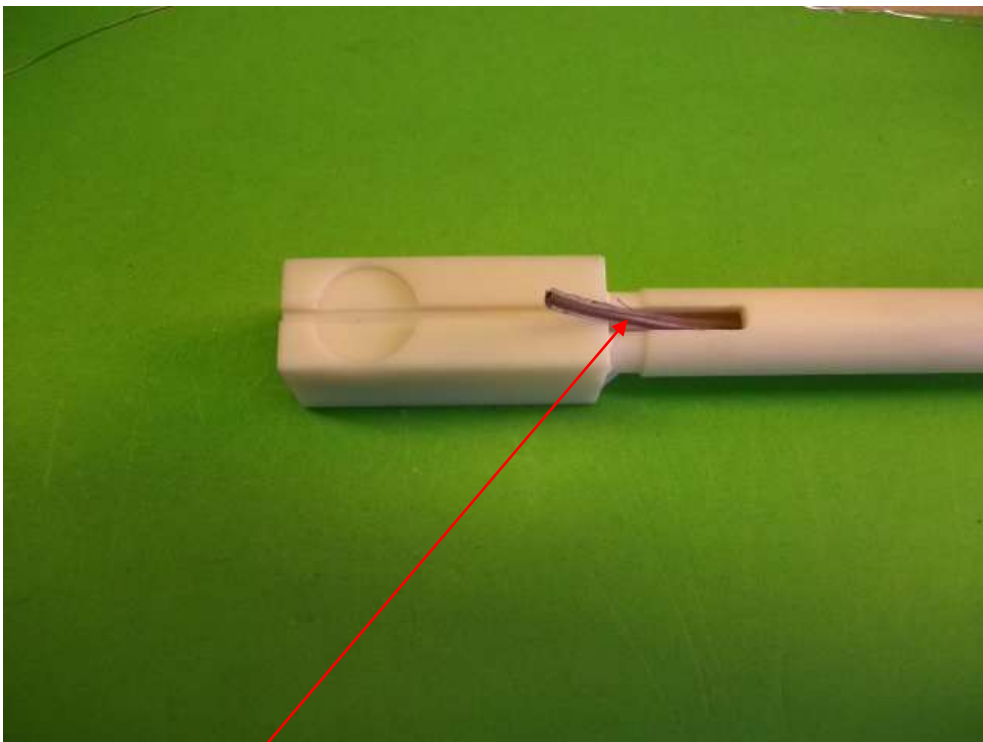
0444

Next we need the isolation tube and two pieces of shrinking isolation.



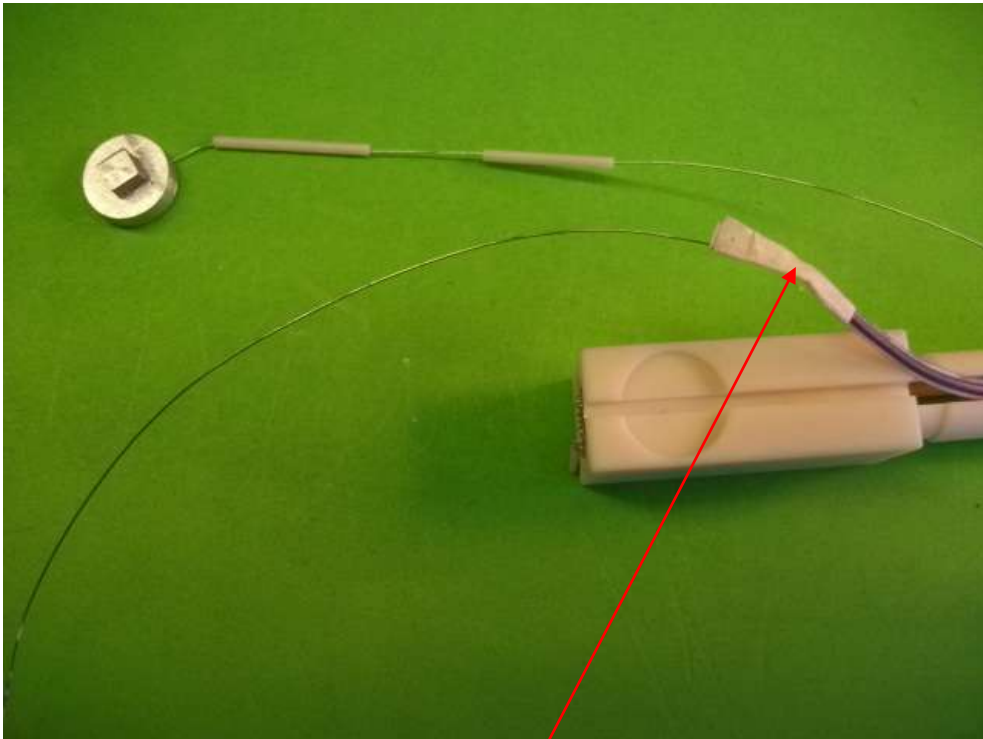
0445

Take new electrode and first put the long and then the short capillary over the lead.



0446

Take an extra thin piece of wire through the measurement rod, used to pull through the electrode connector.



0447

Now connect the electrode wire with the pulling wire, either with glue all with solder. Now pull the electrode wire with the pulling wire in to the measurement rod.

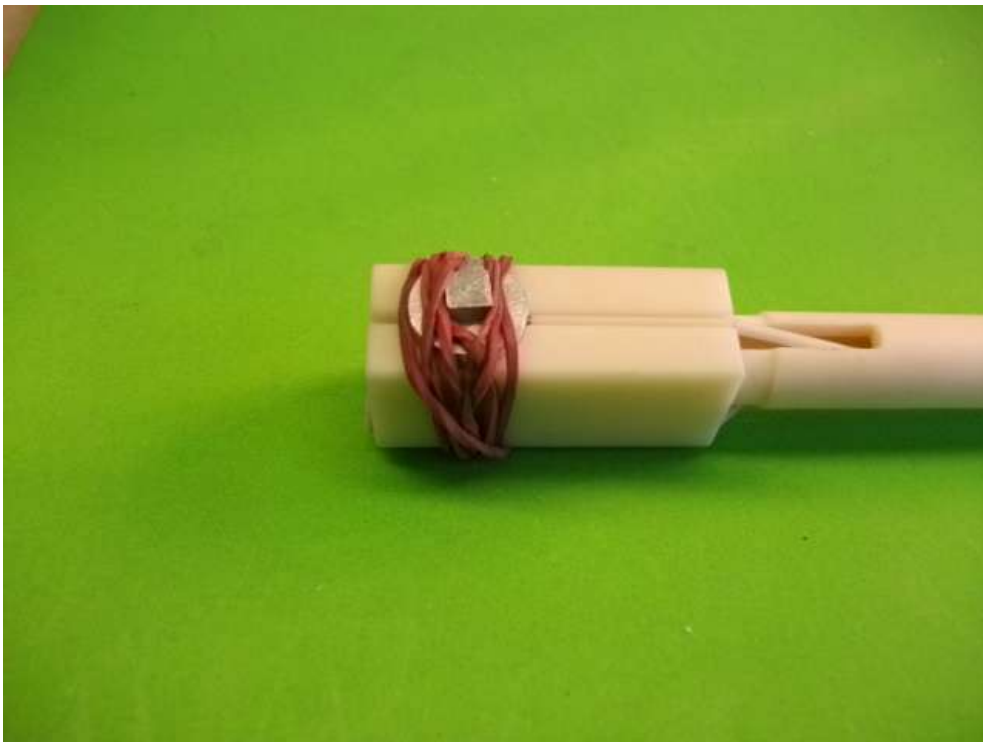


0448

Place electrode in to the position as above.



0449

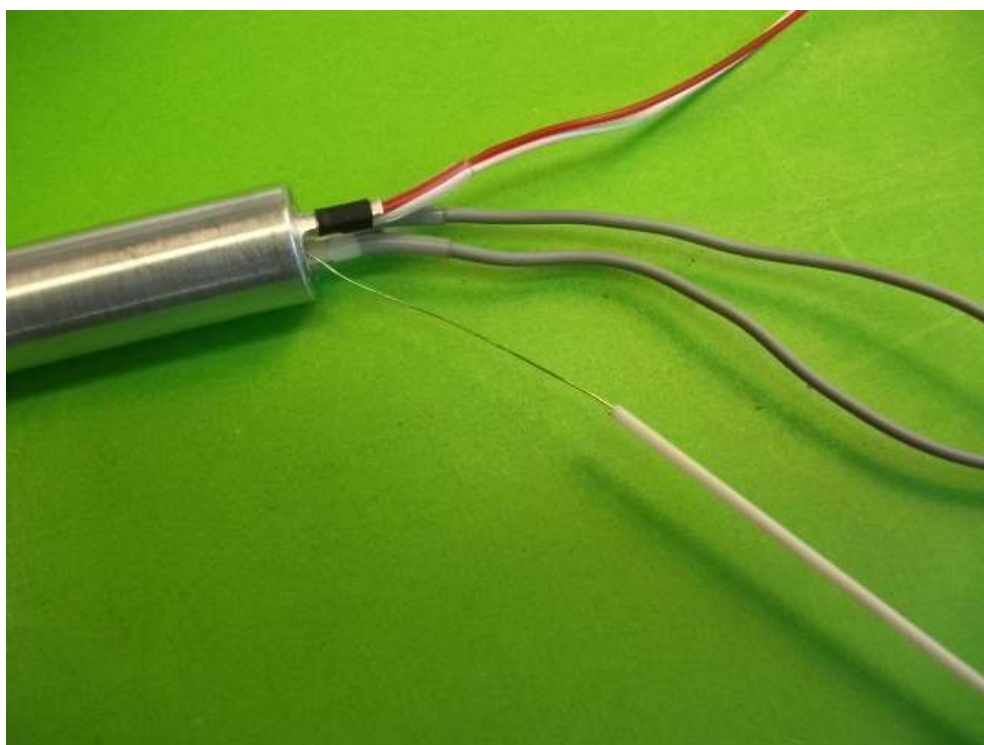


0450

Now take rubber and fix the electrode in its shown position as above.



0453



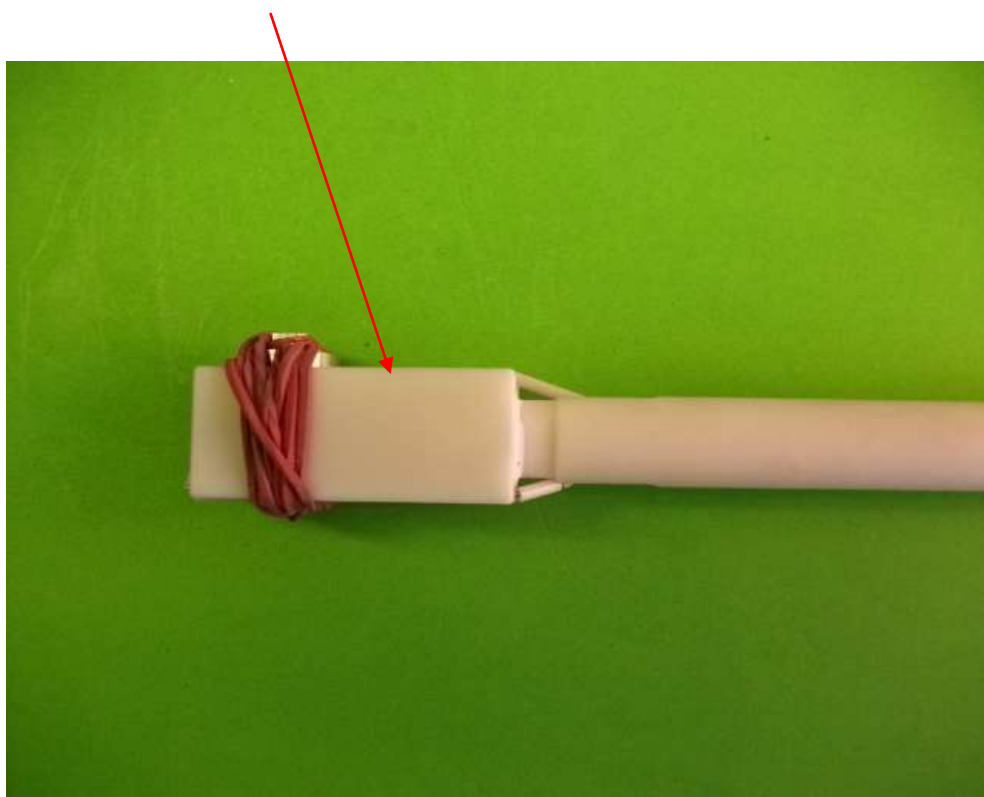
0454

This connect the pulling wire from the electrode wire and insert electrode wire in to long capillary.



0455

Now position the Al_2O_3 capillaries in the slot in a way that everything is nice and flat.

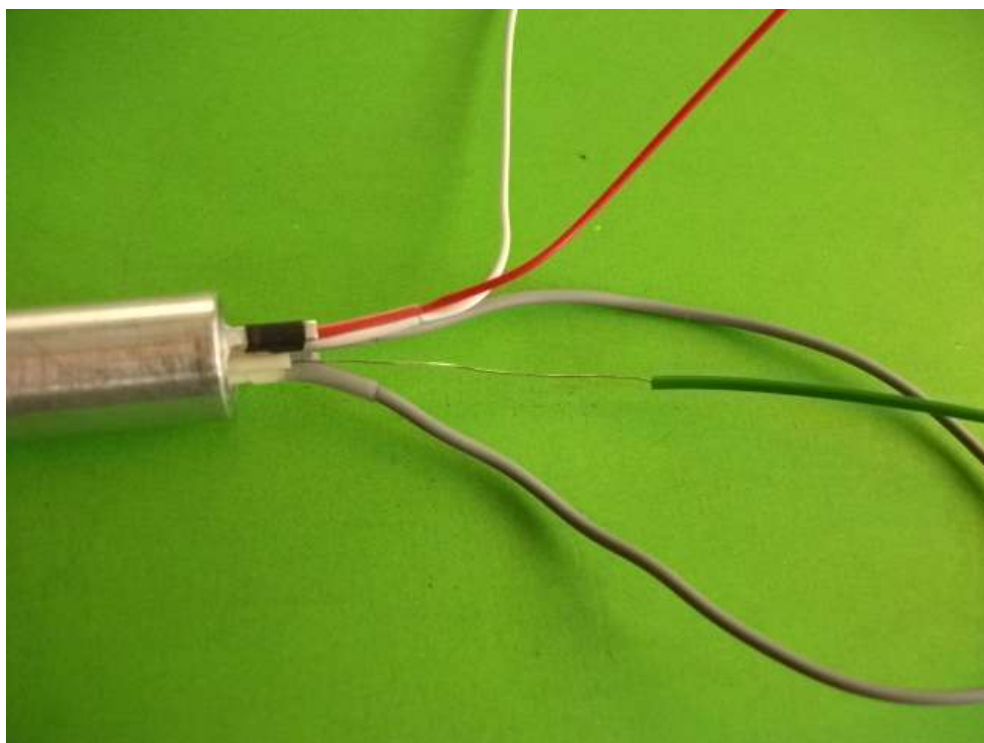


0456



0457

Now use a new or the old isolation and pull it over the electrode wire until it is covering all the wire until the capillary.

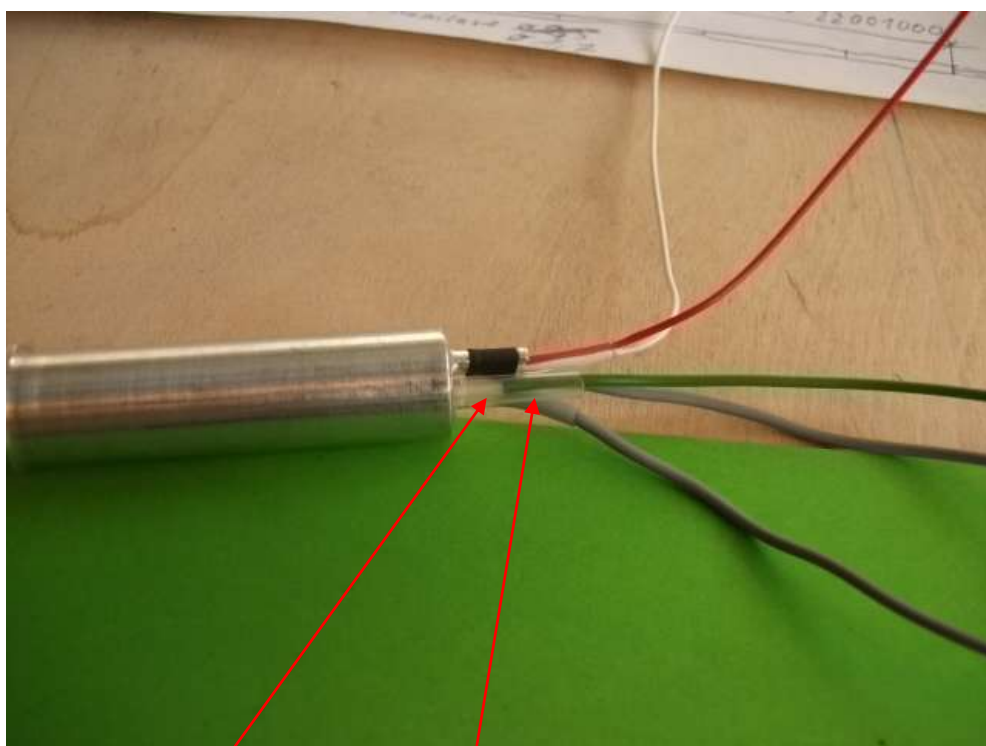


0458



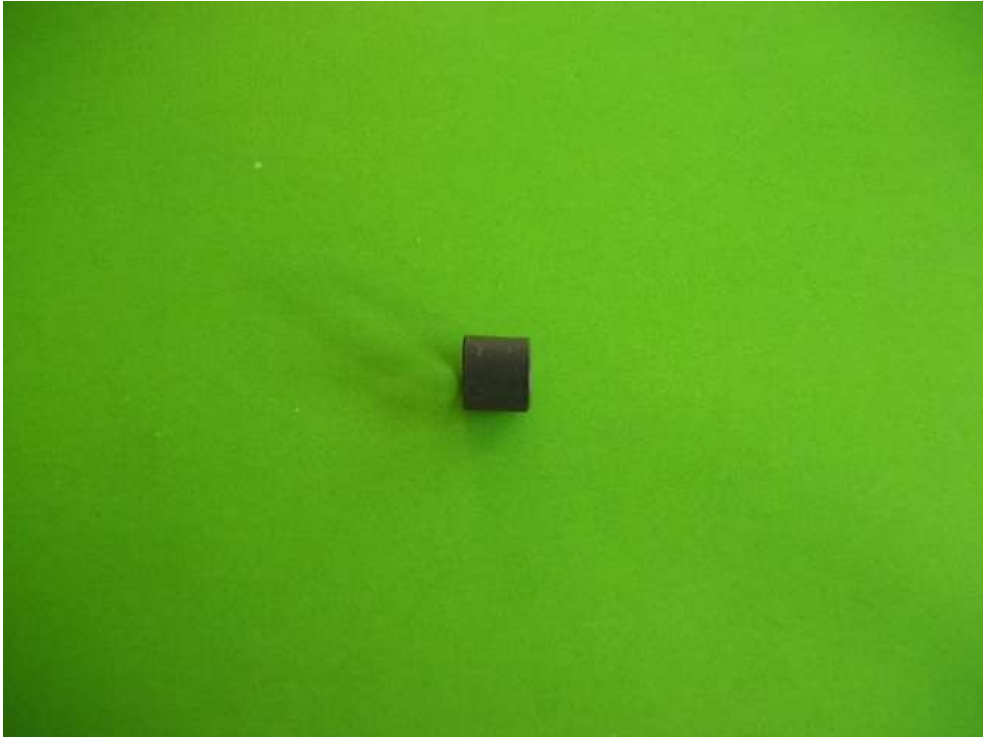
0459

Take a 1 cm long piece of shrinking isolation.



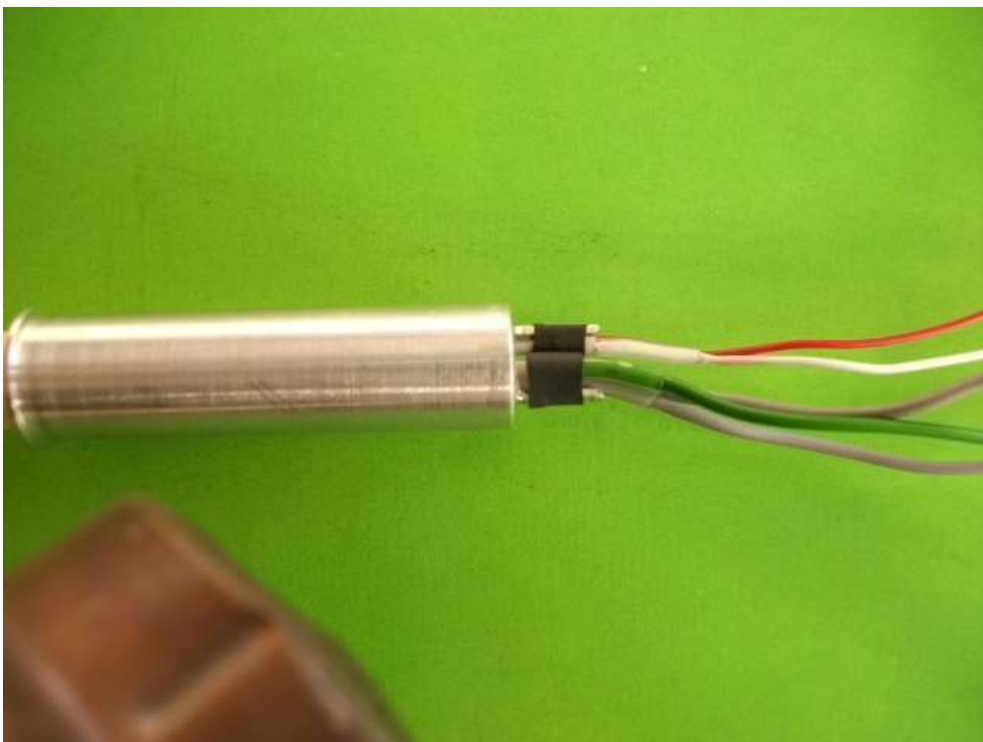
0460

And pull it over the connection between capillary and isolation.



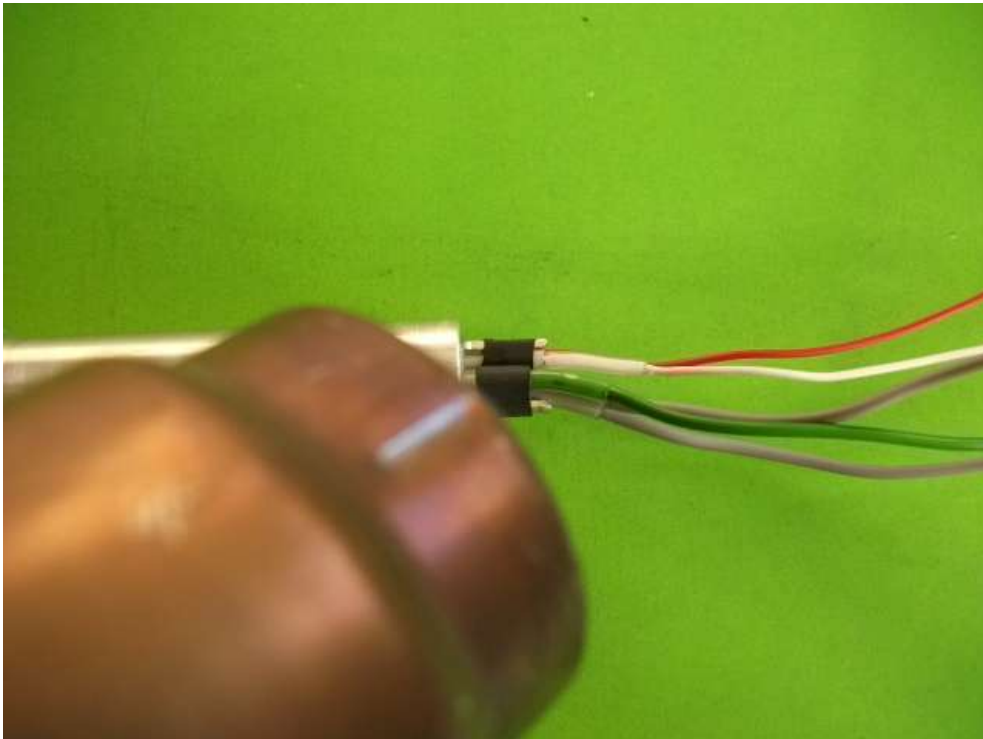
0461

Take a second piece of shrinking isolation, approx 5 mm and



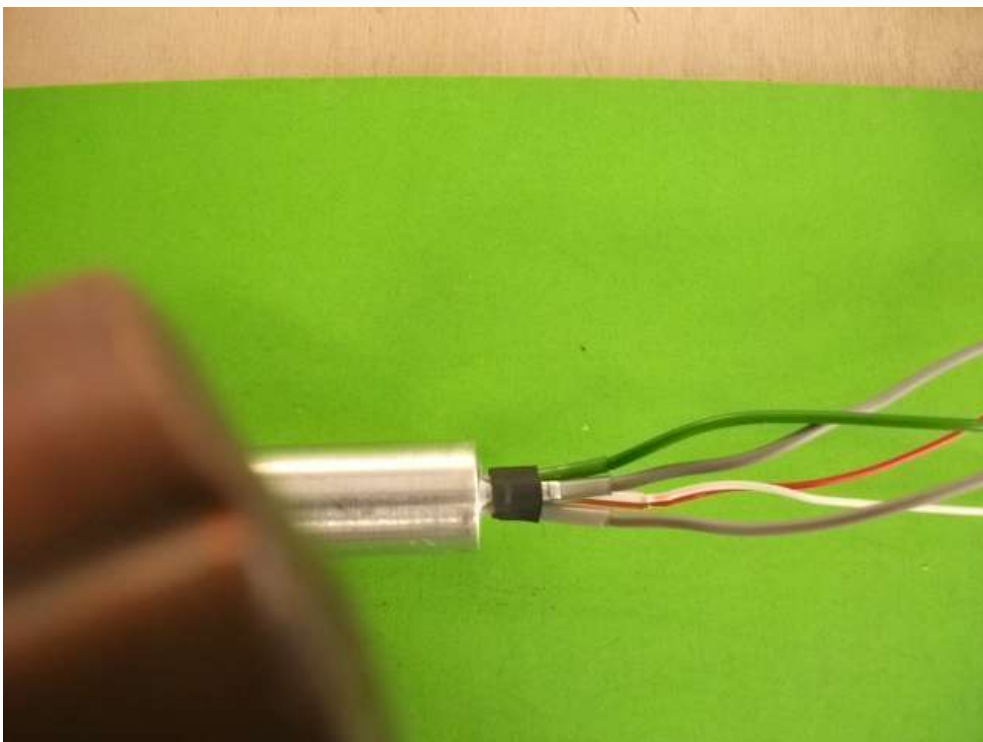
0462

Pull it over all the connecting wires of the electrodes of the heater. There is a total of three connecting wires.



0463

Now take a hot air fan and shrink the shrinking isolation. We can also use a lighter.



0464



0465

Ready measuring system / lower part with heater.

Furnace alignment

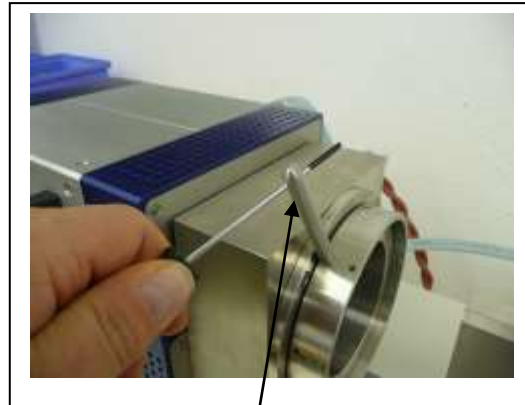
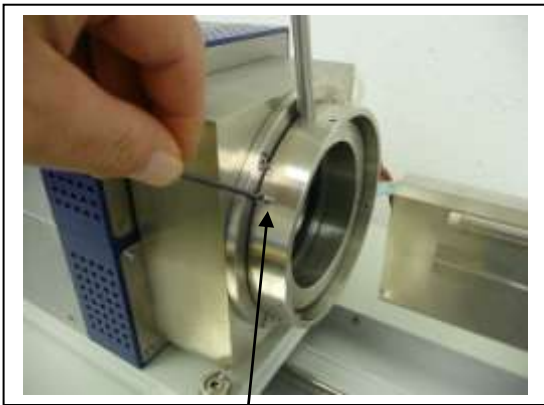
If system will not hold gas for long time there might be a leakage at the connection between furnace and chamber.

Therefore it is important that the furnace is properly aligned.

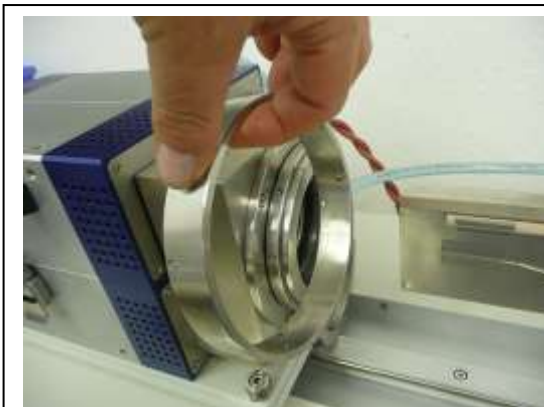
To align the furnace follow these steps:

Open furnace and move to left side

Remove the 4 screws with an allen key and the lever



After removing screws and lever remove the outside ring and the 2 half rings /





Remove the O-Ring at the chamber

Move the furnace to the right and check if the furnace and the chamber are perfect Parallel and there is no gap between the furnace and chamber

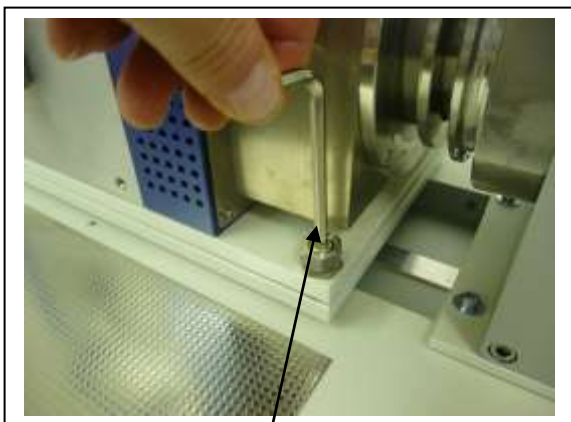


No gap all around

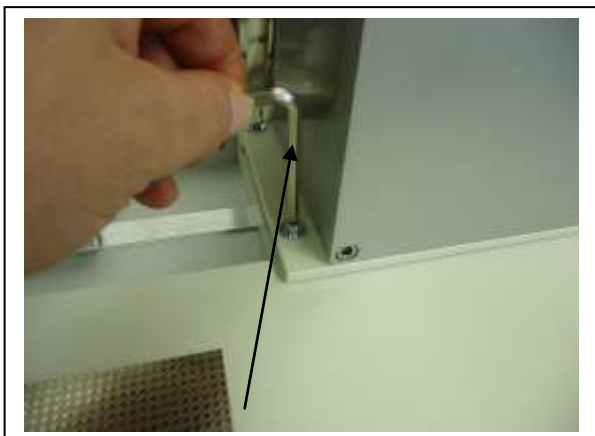


bad example gap at bottom

If alignment is necessary open the 4 locking screws which holds the furnace to the rail with an allen key than you can turn the hex screw to adjust the furnace
There are 4 screws in all 4 corners also the furnace can be rotated a very little bit.



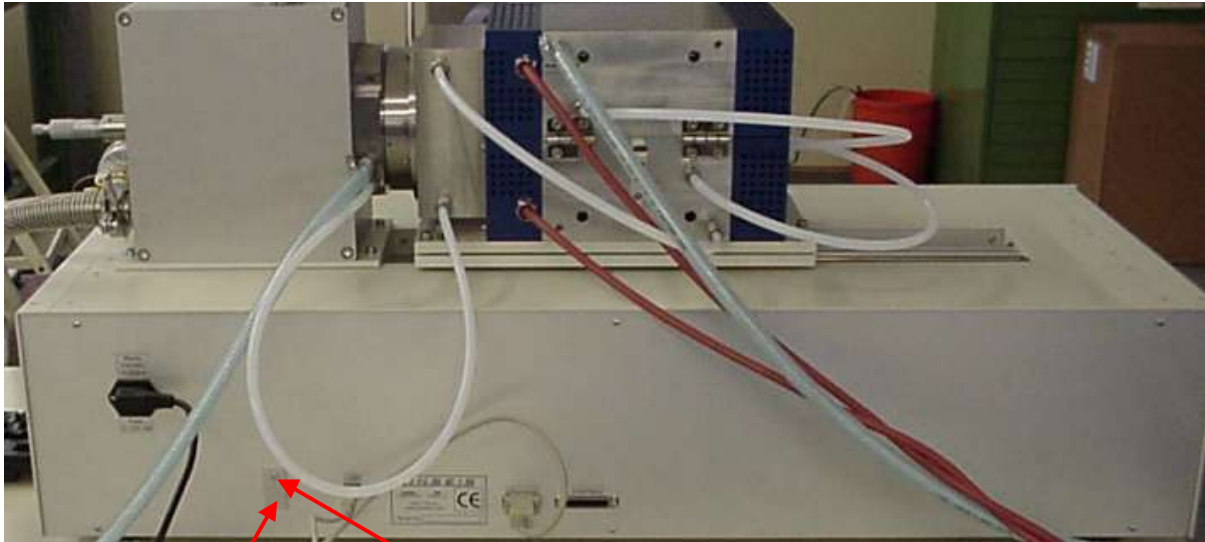
If it is still difficult to get a perfect parallelism also the chamber can be loosen and slightly adjusted therefore open the 4 allen screws which holds the chamber.



if alignment is perfect look the screws
and reassemble the ring and the two half rings
if than the furnace does not slide to the right side easy into the chamber the whole furnace must maybe adjusted
more higher or lower.
Therefore repeat the procedure.
After this install O Ring and check gas tightness
Fill with gas approx. 150 mBar and gas should stay inside at least 24 hours without
Significant drop.

Firmware update for LSR-3

To update the firmware inside of the LSR-controller, it must be put into the FLASH-mode. To do this, turn off the power switch of the LSR-3 and open the small cover on the rear-side of the LSR-3:



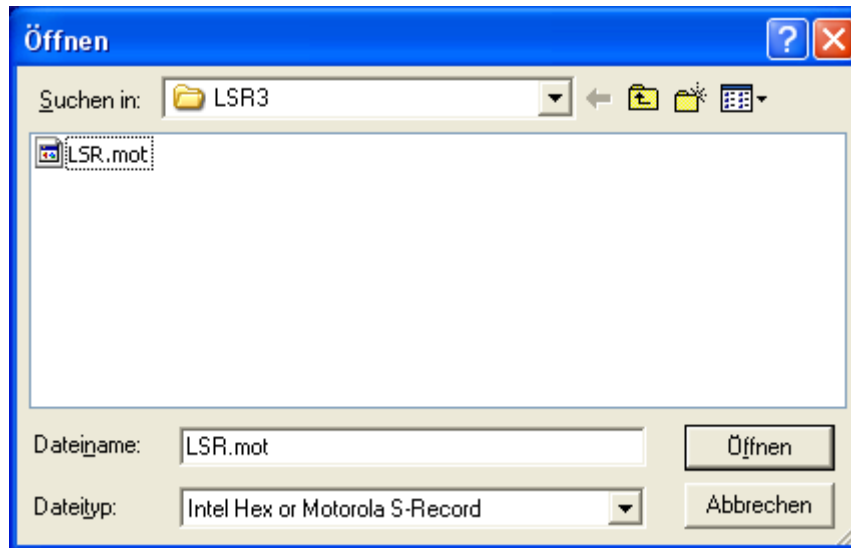


Cover for mode-switch
normal operation/FLASH

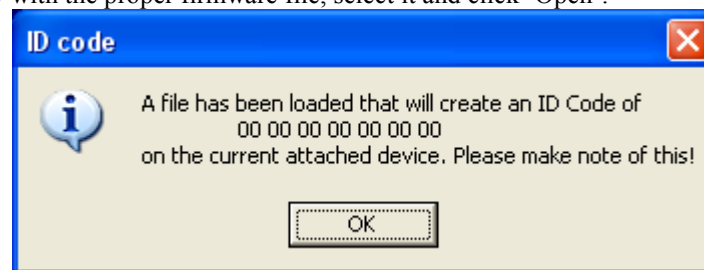
Loosen the screw and turn the cover 180° and put the lever key inside into it's opposite direction. Then turn the power switch on. If this is the first time that a firmware update is performed with computer used, an extra USB-driver must be installed. In this case you are prompted that a new hardware has been found, and a driver is needed. First select that no connect to the internet shall be done, next choose manual installation of the driver. Then browse to the C:\TAWIN\FIRMWARE\USB Drivers directory and click OK. Then the driver will be installed automatically. To flash the new firmware into the controller, launch the C:\TAWIN\FIRMWARE\FOUSB.EXE program:



Click OK to continue. Then select ,OPEN to select the new firmware file:



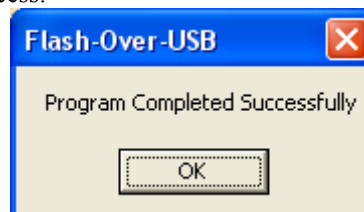
Browse to the directory with the proper firmware file, select it and click 'Open':



Click OK to confirm. Then click 'Program':



Click 'Program' to start the update process:



Now the firmware update is completed successfully and you can click OK and close the flash utility program. Then turn off the power switch, put the lever key into its original position and close the cover. Fasten the screw and turn power on. Now the new firmware is installed and should work properly.

Instruction Manual
LSR - 3
Seebeck Effect &
Electric Resistivity

Installation Guide

Technical data installation guide

LSR-3 low temperature

Main power: ~230 V / 16 A single phase

!! all devices on the same phase !!
!! all devices on the same grounded !!
!! computer on the same socket !!

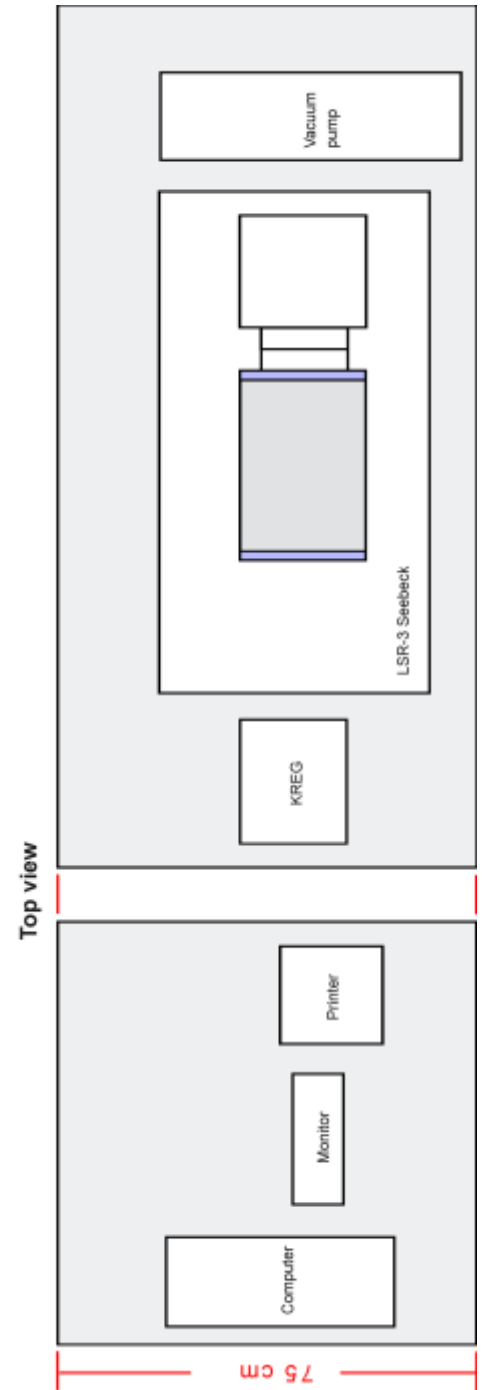
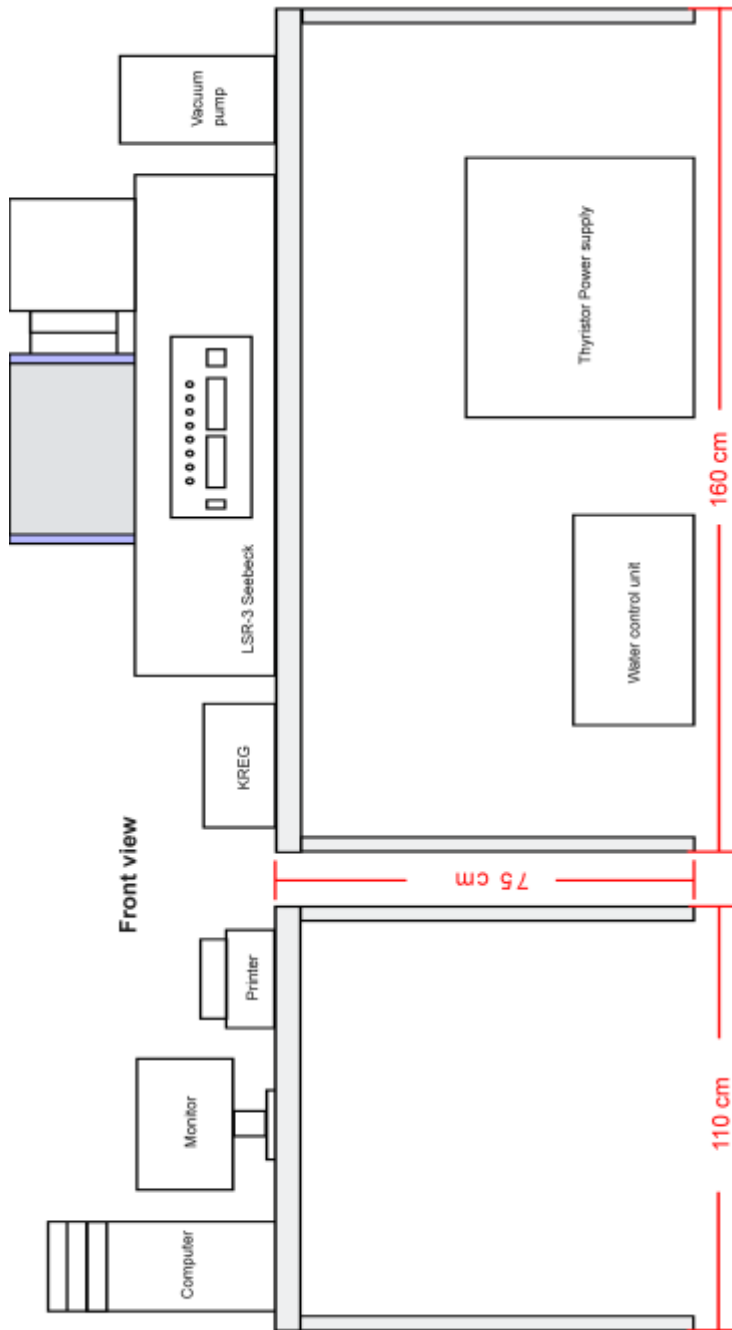
Water connection: tube 8mm inside diameter

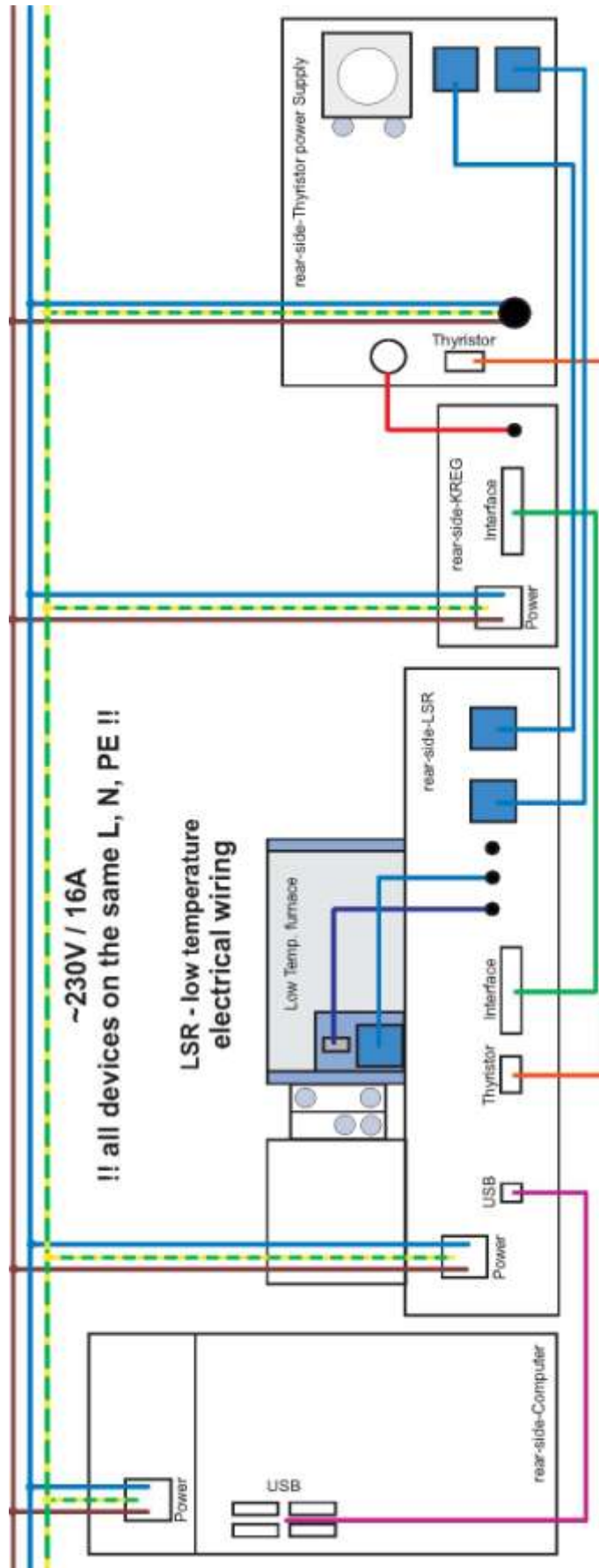
Gas connection: tube 6mm outside diameter

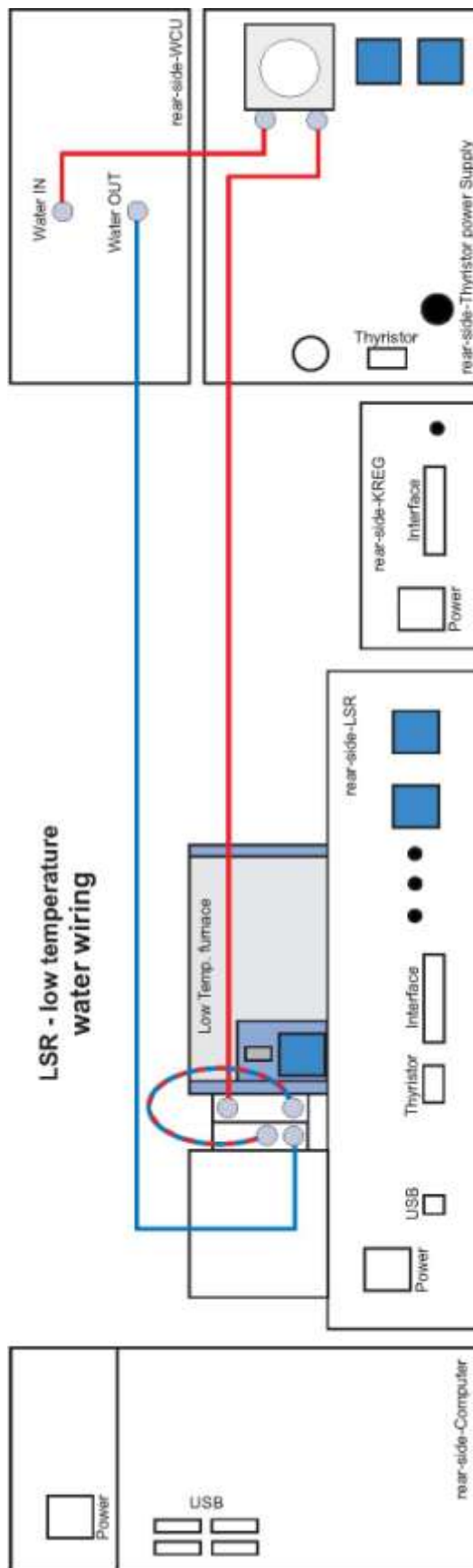
GAS: Helium (He) for measurement pressure 1 bar

Vacuum connection: NW 25

Cooling water: closed loop cooling
2 litre distilled water
flow rate 0.5l/min. minimum
20g corrosion inhibitor







Technical data installation guide

LSR-3 800

Mains power: ~230 V / 16 A single phase

!! all devices on the same phase !!
!! all devices on the same grounded !!
!! computer on the same socket !!

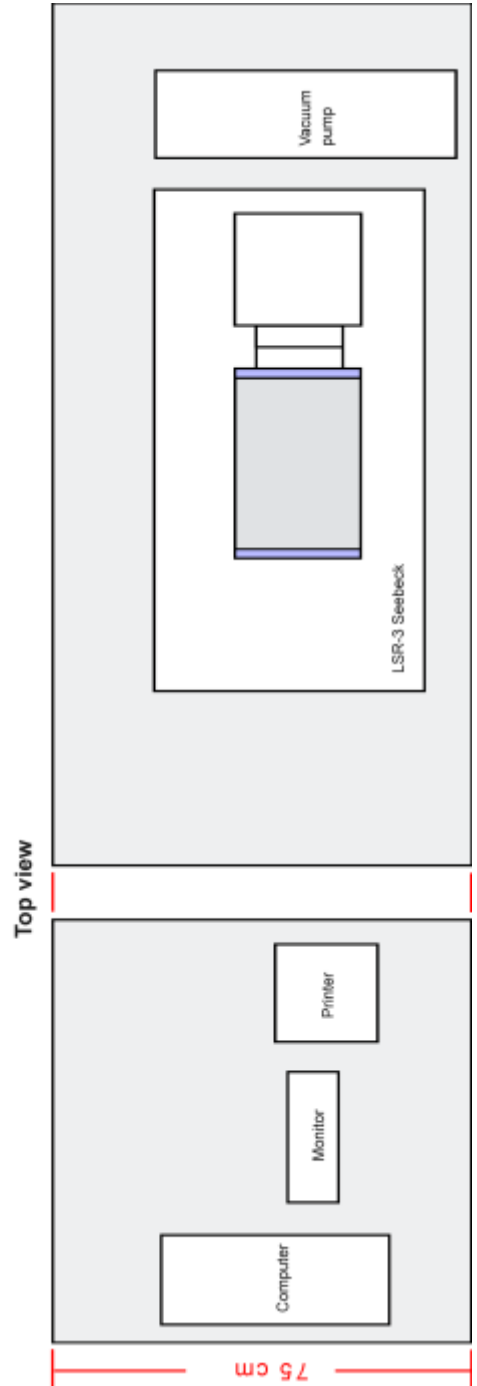
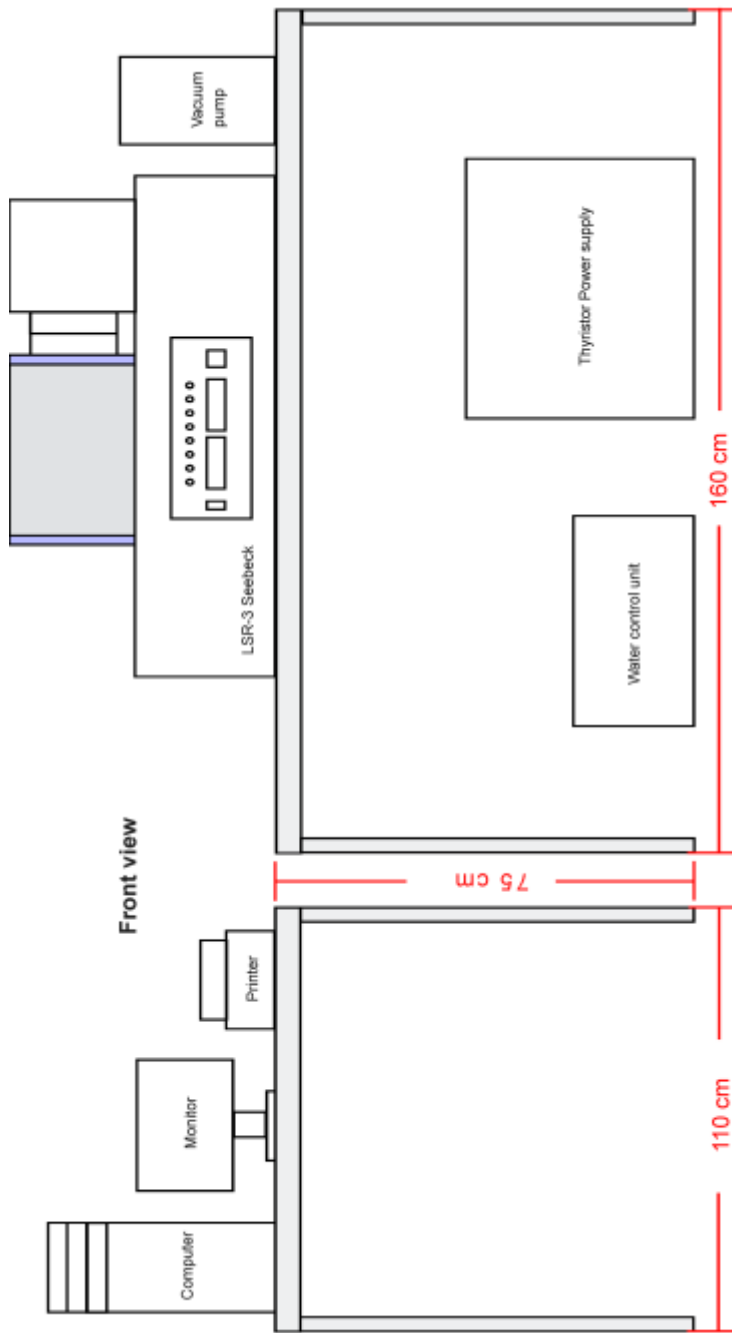
Water connection: tube 8mm inside diameter

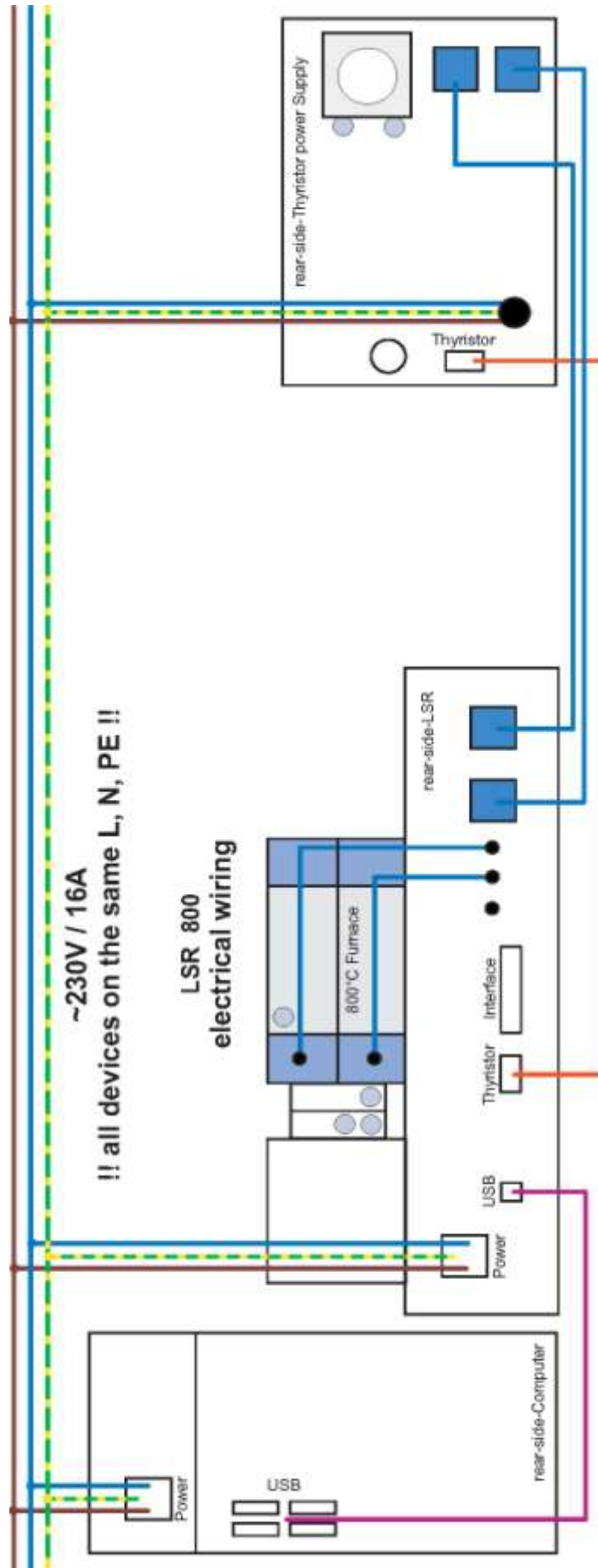
Gas connection: tube 6mm outside diameter

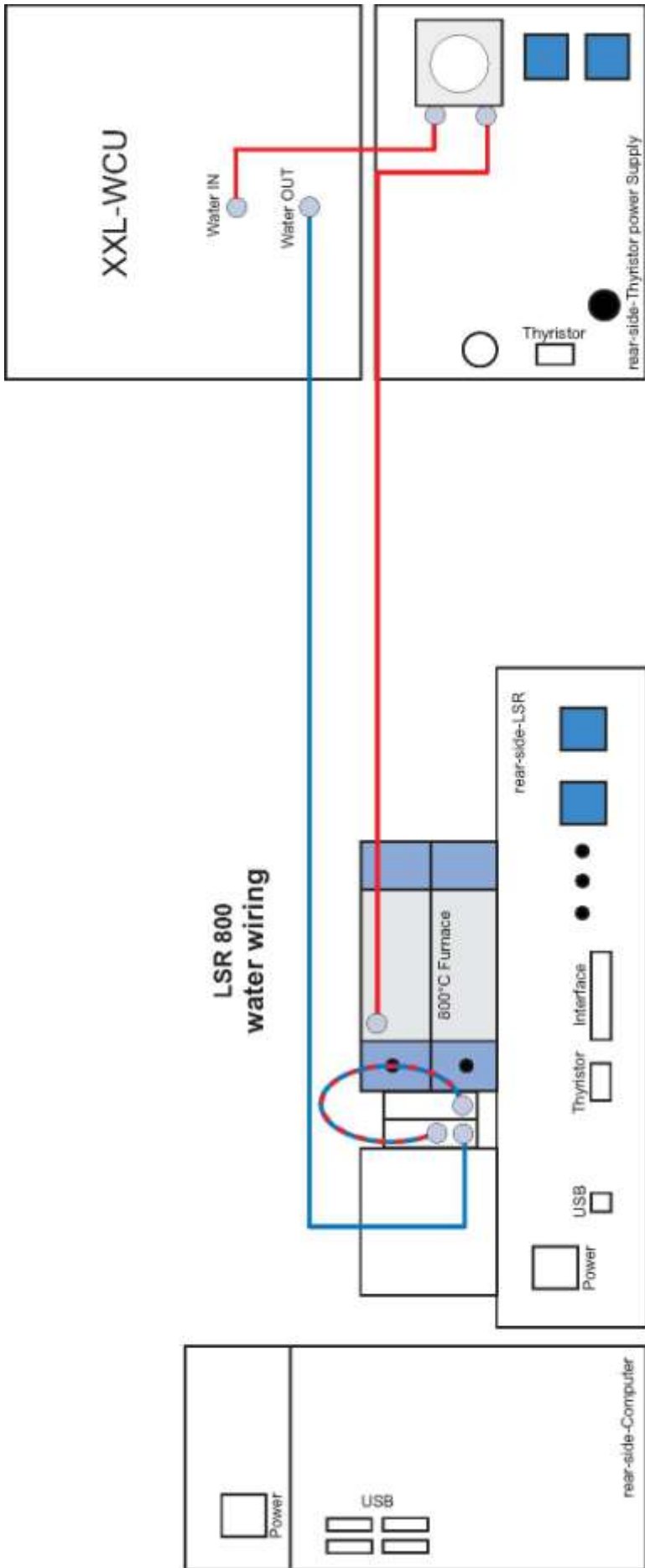
GAS: Helium (He) for measurement pressure 1 bar

Vacuum connection: NW 25

Cooling water: XXL - closed loop cooling
20 litre distilled water
flow rate 2l/min. minimum
200g corrosion inhibitor





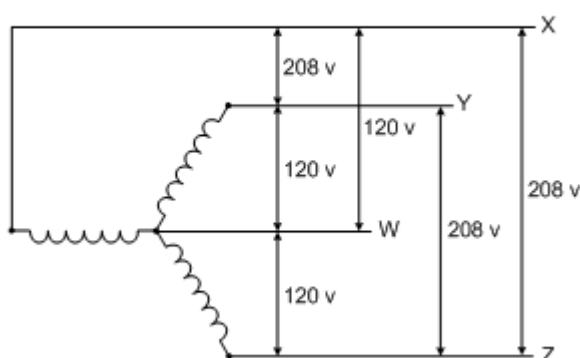


Technical data installation guide

LSR-3 1100

Mains power: 3*230 V / 3*16 A (3 phase)

For 115V or 120 V mains supply is a front end transformer available.
Power rating for the front end transformer: 208Y/120 volt Wye (four pole, five wire, grounding (Ground, Neutral and 3 service poles)), 30 amp.



Please note: The front end transformer supply cable is provided **without** plug. A permanent connection to an electrical distribution box is possible. Linseis recommends the provision of a suitable socket **and** properly plug by the client.

A suitable connector type is e.g. NEMA L21-30.

NEMA L21-30
120/208 volt, 3ØY,
four pole, 5 wire,
grounding
30 amp



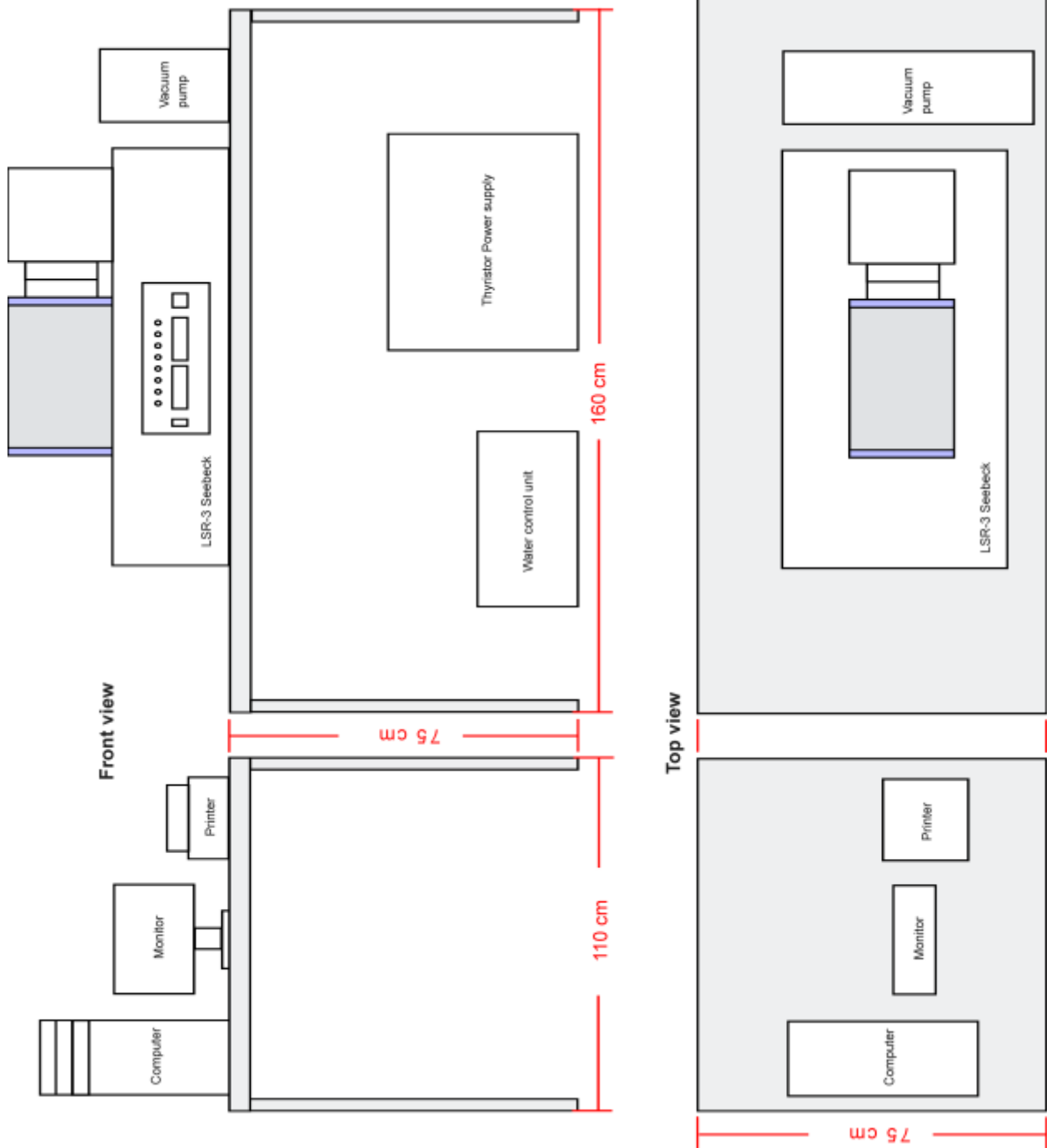
G - Ground, W - Neutral, X,Y,Z - Service Poles

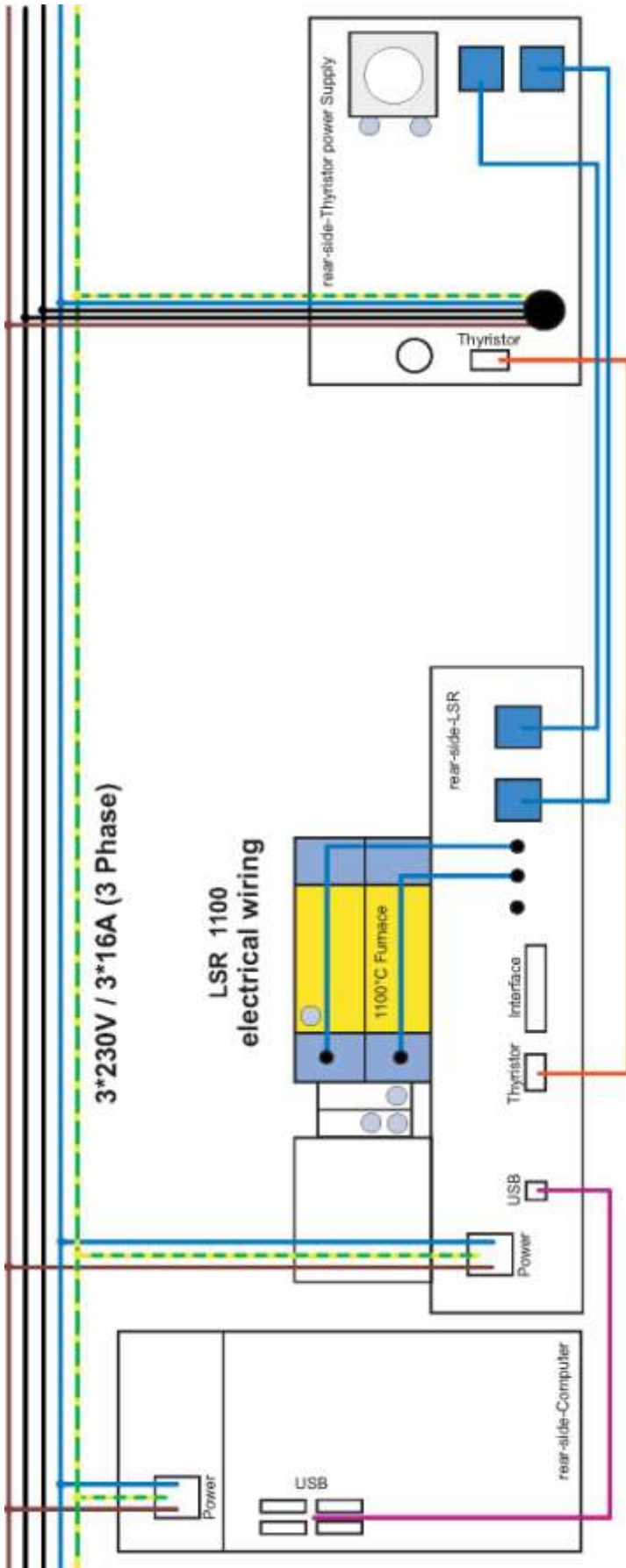
Water connection: tube 8mm inside diameter

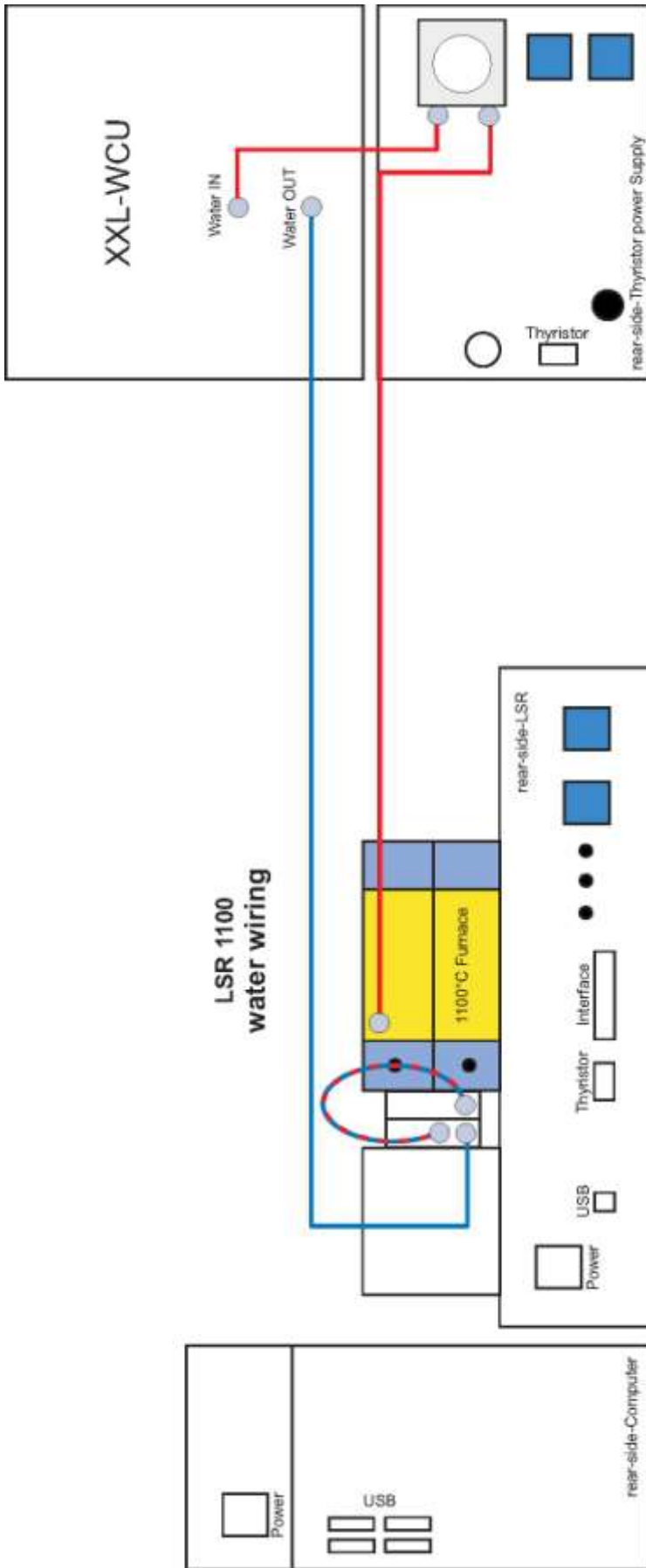
Gas connection: tube 6mm outside diameter GAS: Helium (He) for measurement
pressure 1 bar

Vacuum connection: NW 25

Cooling water: XXL - closed loop cooling:
20 litre distilled water
flow rate 2l/min. minimum
200g corrosion inhibitor







Example:

Interconnection for cooling-water LSR-3



Example for Cooler

Thyristor / Power supply