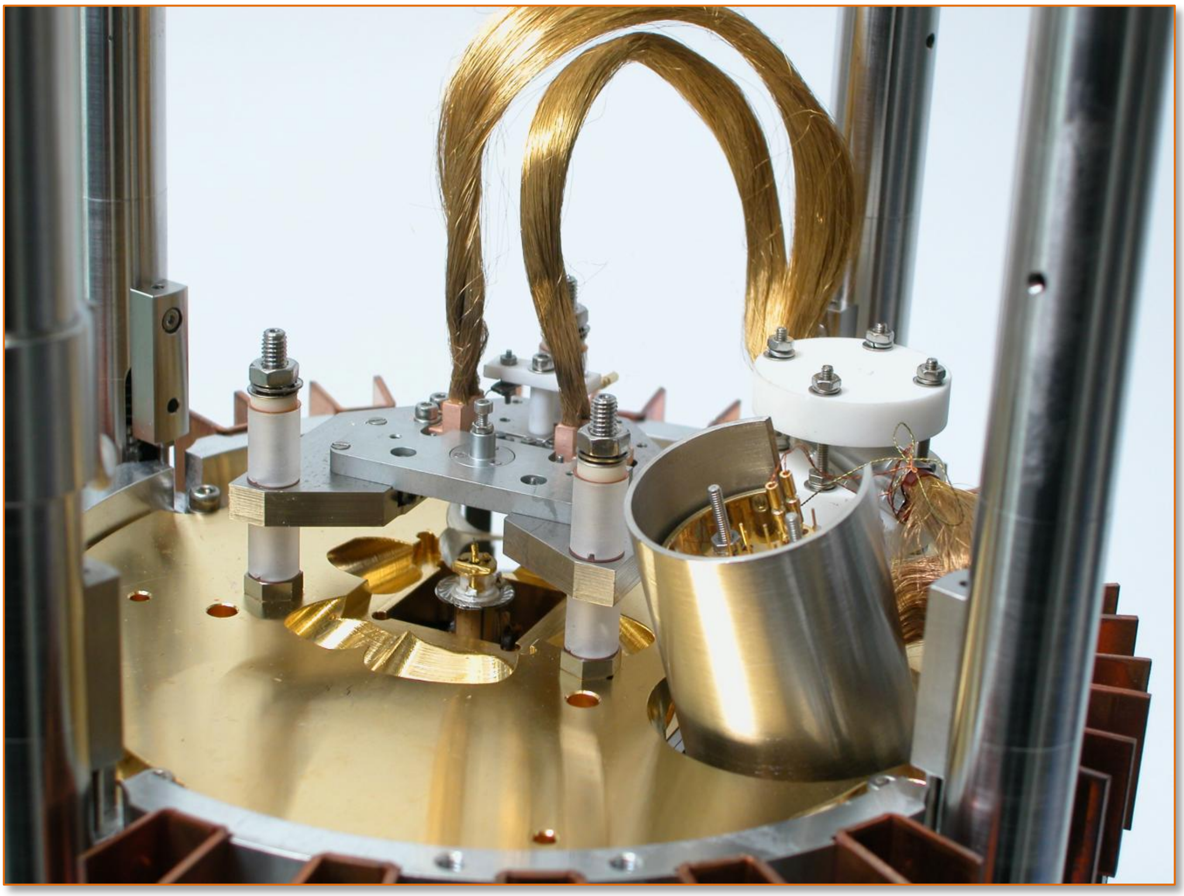
VT STM XA User's Guide

Version 3.2.1 • 21 August 2015



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Preface

This document has been compiled with great care and is believed to be correct at the date of print. The information in this document is subject to change without notice and does not represent a commitment on the part of Scienta Omicron GmbH.

Notice

Some components described in this manual may be optional. The delivery volume depends on the ordered configuration.

Notice

This documentation is available in English only.

Notice

Please read the safety information on pages 11 to 15 before using the instrument.

® Notice

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Warranty

Scienta Omicron acknowledges a warranty period of 12 months from the date of delivery (if not otherwise stated) on parts and labour, excluding consumables such as filaments, sensors, etc.

No liability or warranty claims shall be accepted for any damages resulting from non-observance of operational and safety instructions, natural wear of the components or unauthorised repair attempts.

Waste Electric and Electronic Equipment

In compliance with the WEEE directive (2002/96/EC) Scienta Omicron ensures that all products supplied by Scienta Omicron which are de-commissioned and which are labelled with a WEEE Registration Number will be taken back by Scienta Omicron free of charge.

All costs of packing, transport, duty, etc. to the destination of the nearest Scienta Omicron Returned-WEEE-Centre shall be borne by the customer. The customer is required to:

- Declare the returned material is free from contamination or hazardous materials from usage (include Decontamination Declaration sheet),
- Request a valid Return Material Authorisation (RMA) available from the Scienta Omicron service department,
- Ship all returned goods to the advised destination "Scienta Omicron Returned-WEEE-Centre, DDP (INCOTERMS)".

Otherwise Scienta Omicron will not accept any shipment.

No Third Party Additions for Scienta Omicron Instruments

Attention

Third party equipment can destroy your Scienta Omicron instrument. If connecting other than Scienta Omicron supplied equipment please make sure this is compatible with your Scienta Omicron instrument, particularly with respect to power rating, maximum voltages, currents and impedances as well as fault handling and safety shutdown.

Scienta Omicron shall refuse any warranty claims in case of non-compliance.

Notice

Scienta Omicron cannot guarantee compliance with CE directives in case of changes to the instrument not explicitly agreed by Scienta Omicron, e.g. modifications, add-on's (including third party equipment), or the addition of circuit boards or interfaces to computers supplied by Scienta Omicron.

Normal Use

The Scienta Omicron VT STM XA is an ultra high vacuum (UHV) scanning probe microscope (SPM) for topographic and spectroscopic imaging of solid surfaces with sub-nanometre resolution only to be used

- in combination with the Scienta Omicron MATRIX SPM control system
- with original Scienta Omicron cable sets/accessories which are explicitly specified for this purpose
- within the originally supplied UHV chamber mounted to a suitable vacuum system
- under UHV conditions
- with all cabling connected and secured, if applicable.
- with all electronics equipment switched on
- in an indoor research laboratory environment with low vibration level
- by personnel qualified for operation of delicate scientific equipment and handling of cryogenic liquids
- in accordance with this manual and related manuals.

Caution

Adjustments and fault finding measurements as well as SPM experiments in environments other than UHV may only be carried out by authorised personnel qualified to handle lethal voltages.
Read the safety information in all related manual(s) before using the instrument.

Attention

The chamber must always be mounted on the vacuum system during operation due to mechanical instabilities.

Attention

Tip cleaning with field emission carries the risk of causing damage (ESD) to the input stage.

Until further notice we strongly advise not to supply any external voltages to the tip-sample-gap exceeding 25 V – not even with Scienta Omicron supplied field emission adapters. This warning relates to all SPMs equipped with the QPlus option and is true with simple STM tips as well as QPlus sensors mounted.

Conditions of CE Compliance

Scienta Omicron instruments are designed for use in an indoor laboratory environment. For further specification of environmental requirements and proper use please refer to your quotation and the product related documentation (i.e. **all** manuals, see individual packing list).

The VT STM XA complies with CE directives as stated in your individual delivery documentation if used unaltered and according to the guidelines in the relevant manuals.

Limits of CE Compliance

This compliance stays valid if repair work is performed according to the guidelines in the relevant manual and using original Scienta Omicron spare parts and replacements.

This compliance also stays valid if original Scienta Omicron upgrades or extensions are installed to original Scienta Omicron systems following the attached installation guidelines.

Exceptions

Scienta Omicron **cannot** guarantee compliance with CE directives for **components** in case of

- changes to the instrument **not authorised by Scienta Omicron**, e.g. modifications, add-on's, or the addition of circuit boards or interfaces to computers supplied by Scienta Omicron.

The customer is responsible for CE compliance of the entire **experimental setup** according to the relevant CE directives in case of

- installation of Scienta Omicron components to an on-site system or device (e.g. vacuum vessel),
- installation of Scienta Omicron supplied circuit boards to an on-site computer,
- alterations and additions to the experimental setup not explicitly approved by Scienta Omicron

even if performed by an Scienta Omicron service representative.

Spare Parts

Scienta Omicron spare parts, accessories and replacements are not CE labelled individually since they can only be used in conjunction with other pieces of equipment.

Notice
CE compliance for a combination of certified products can only be guaranteed with respect to the lowest level of certification. Example: when combining a CE-compliant instrument with a CE 96-compliant set of electronics, the combination can only be guaranteed CE 96 compliance.

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1. Safety Information

Caution

Important:

- Please read this manual and the safety information in all related manuals before installing or using the instrument or electronics equipment.
- The safety notes and regulations given in this and related documentation have to be observed at all times.
- Check for correct mains voltage and grounding/earth before connecting any equipment.
- Do not cover any ventilation slits/holes so as to avoid overheating.
- The VT STM XA may only be handled by authorised personnel.

Caution

Always

- All connectors which were originally supplied with fixing screws must always be used with their fixing screws attached and tightly secured.
- All connectors which were originally supplied with a short circuit plug must either be connected to the electronics or fitted with their short circuit plug.
- Always disconnect the mains supplies of all electrically connected units before
  - ⇨ venting, pumping down or opening the vacuum chamber
  - ⇨ opening a control unit case,
  - ⇨ touching any cable cores or open connectors,
  - ⇨ touching any part of the in-vacuum components (except for tip and sample exchange as described in this manual).
- Leave for a few minutes after switching off for any stored energy to discharge.

Warning

Warning: Lethal Voltages!!

- Adjustments and fault finding measurements may only be carried out by authorised personnel qualified to handle lethal voltages.
- Lethal voltages may present at parts of the instrument during operation.
- Lethal voltages are present inside the control unit/computer (if applicable).

Caution

Never

- Never exceed a total pressure of 1.2 bar inside the vacuum chamber.
- Never have in-vacuum components connected to their electronics in the corona pressure region, i.e. between 10 mbar and 10^{-3} mbar, so as to avoid damage due to corona discharge.

Attention

No Field Emission for QPlus Instruments

- Tip cleaning with field emission carries the risk of causing damage (ESD) to the input stage.
- Until further notice we strongly advise not to supply any external voltages to the tip-sample-gap exceeding 25 V – not even with Scienta Omicron supplied field emission adapters. This warning relates to all SPMs equipped with the QPlus option and is true with simple STM tips, cantilever sensors as well as QPlus sensors mounted.

Caution

Chemical Hazard

- For all supplied or recommended chemicals such as silver paint glueing agents or solvents please read the manufacturer's safety information.

Caution

Venting

- Make sure all parts of the VT STM XA have gained room temperature and disconnect all cables from the base flange before venting the vacuum chamber. Fit short circuit plug if applicable.
- Do not exceed a total pressure of 1.2 bar inside the vacuum chamber. All gas cylinders which are connected to the UHV system must be equipped with a suitable over-pressure relief valve, particularly if used for venting.

Caution

Liquid Nitrogen and Liquid Helium:

- LHe and LN_2 are colourless, odourless and tasteless non-toxic substances.

1 litre of liquid will turn into 700 litres of gas which can cause a large pressure build up followed by a possible explosion. Always fit a burst disk to your vacuum vessel in order to allow rapidly evaporating liquid gases to escape in case of a cryostat failure.

Make sure dewars are fitted with a suitable over-pressure relief valve (maximum total chamber pressure is 1.2 bar) or have an open vent.

Make sure the safety valves are functioning at all times (must not be blocked by ice).

LHe/He and LN_2/N_2 can cause rapid suffocation without warning. Store and use in areas with adequate ventilation.

Note that evaporated N_2 accumulates on the ground. Take care particularly when picking up parts from the floor.

In case of symptoms like headache, drowsiness, dizziness, vomiting or unconsciousness remove victim to fresh air. Give oxygen or artificial respiration if necessary. Seek medical assistance immediately.

Caution

LHe and LN_2 can cause severe frostbite. Do not touch frosted pipes or valves. Always wear suitable long sleeved full length clothing, as well as splash goggles and cryogenic gloves when handling cold gases.

- In case of frostbite rapidly warm affected skin areas with warm water (< 40°C). Do not rub and protect injured tissue from further damage and infection. If eyes are involved flush thoroughly with warm water for at least 15 minutes. Seek medical assistance immediately.

Caution

Pyroelectric Effect

- Temperature changes of the piezo materials used in scanner and coarse position drives can generate **charge build up** on the connectors. The discharge current is not dangerous (while painful) but **may seriously damage the electronics** or the piezos themselves. The charge build up must be avoided by terminating the electrical connectors:

  ⇨ During **bakeout** (including the heating up and cooling down times) fit the respective sockets with their short circuit plugs.

  ⇨ During cooling down and warming up of a **cryostat** leave the instrument connected to the live electronics or fit the respective sockets with their short circuit plugs.

Caution

Viewports

- Always handle viewports carefully. Improper installation and handling may cause implosion.
- Do not use viewports where the glass shows scratches or any other visible marks, abrasions or imperfections.
- Viewports must not be subjected to total pressures above 1.2 bar on the vacuum side and 1.0 bar on the atmospheric side.
- One accidental smack with a tool or the application of a strong laser beam may already damage the viewport sufficiently to cause cracking during pump-down or under UHV conditions later on.
- Protect viewports with a safety cover or vent the UHV system whenever handling tools or workpieces in the vicinity of a viewport.

Caution

- Protect viewports with aluminium foil on the outside during bakeout and do not exceed a temperature rise or fall of 5°C/min. Although the viewports withstand a bakeout temperature of 450°C, **the maximum bakeout temperature of the equipped system will be much lower due to other components connected.**
- Eye protection should be worn when near to a viewport, even if using protective covers.
- If viewports are used on instruments with X-ray generative sources, lead glass covers with sufficient X-ray absorption must be used.

Caution

This product is only to be used:

- within a dedicated UHV system
- under ultra-high-vacuum conditions
- indoors, in laboratories meeting the following requirements:
  - ⇨ altitude up to 2000 m,
  - ⇨ temperatures between 5°C / 41°F and 40°C / 104°F (specifications guaranteed between 20°C / 68°F and 25°C / 77°F)
  - ⇨ relative humidity less than 80% for temperatures up to 31°C / 88°F (decreasing linearly to 50% relative humidity at 40°C / 104°F)
  - ⇨ pollution degree 1 or better (according to IEC 664),
  - ⇨ overvoltage category II or better (according to IEC 664)
  - ⇨ mains supply voltage fluctuations not to exceed ±10% of the nominal voltage
- Condensation of humidity, particularly on water-cooled equipment, must be avoided.

2. Introduction

Scanning Tunnelling Microscopy

In Scanning Tunnelling Microscopy (STM) an electrically biased tip is scanned across a surface at a very close distance (about an atomic diameter). The current flow between the tip and the sample (due to the tunnelling effect) strongly depends on the tip-surface gap and can be measured with great accuracy. The changing current signal can in turn be used to generate a surface topography map. This method only works with conducting samples, e.g. metals, graphite, semiconductors.

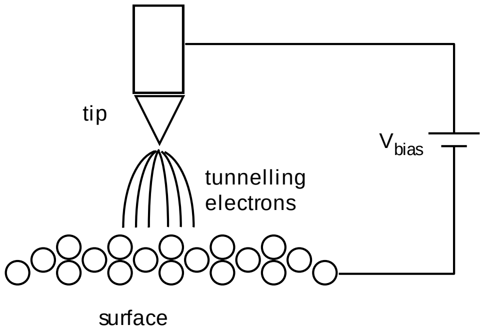


Figure 1. STM operation, schematic diagram.

For further information on scanning tunnelling microscopy see [1] to [5] on page 122.

QPlus AFM Function

In Atomic Force Microscopy (AFM) a tip is scanned across a surface at close distance tracing the surface contour. Inter-atomic, frictional, magnetic and electrostatic forces attract or repel the tip. In noncontact AFM these forces are detected as a shift in the resonance frequency of a vibrating probe.

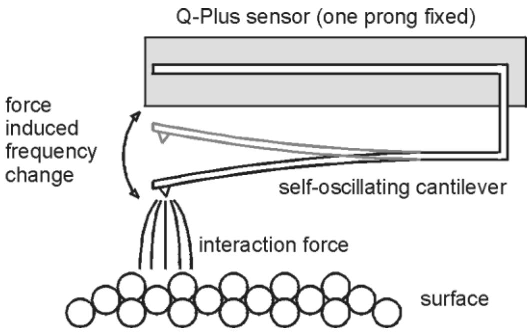


Figure 2. AFM operation, schematic diagram.

The QPlus sensor is an easy to handle force sensing probe in noncontact AFM microscopes. The sensor is based on a quartz tuning fork with one prong fixed and a tungsten tip or cantilever mounted on the other vibrating prong. It provides vibrational information as an electrical signal due to the piezoelectric effect in the fork.

In addition to the vibration signal the sensor offers a second independent detection channel for the tunnelling current.

QPlus AFM Modes

In most QPlus AFM applications the sensor tip touches the surface during scanning and the feedback signal is derived from normal force, i.e. the force component perpendicular to the surface.

These forces are also present when the sensor tip is a few angstroms away from the surface. They are, however, so small that they cannot directly be detected with the above method.

In QPlus non-contact mode AFM the feedback signal is derived from the force induced shift in resonance frequency of the vibrating QPlus sensor.

For further information on atomic force microscopy see [1], [4] and [5]; for details on non-contact operation see [6] and [11] on page 122.

Vibration Isolation and Spring Suspension

For successful high-resolution scanning probe microscopy a high quality vibration decoupling system is essential. The VT STM XA base plate is suspended by four soft springs which are protected by surrounding columns. The resonance frequency of the spring suspension system is about 2 Hz. Vibrations of the suspension system are intercepted using a nearly non-periodic eddy current damping mechanism. For this the VT STM XA base plate is surrounded by a ring of copper plates which come down between (permanent) magnets.

The spring suspension system can be blocked to allow tip or sample exchange, adjustments, etc. Blocking is achieved using a push-pull motion feedthrough (PPM), see figure 3.

The cryostat is fixed to the base flange with the heat exchanger of the LHe flow cryostat projecting through the STM base plate. The thermal connection between the sample and the cryostat is realised using a highly flexible copper braid. To minimise the vibration input through the copper braid this is mechanically decoupled at the copper braid decoupling stage.

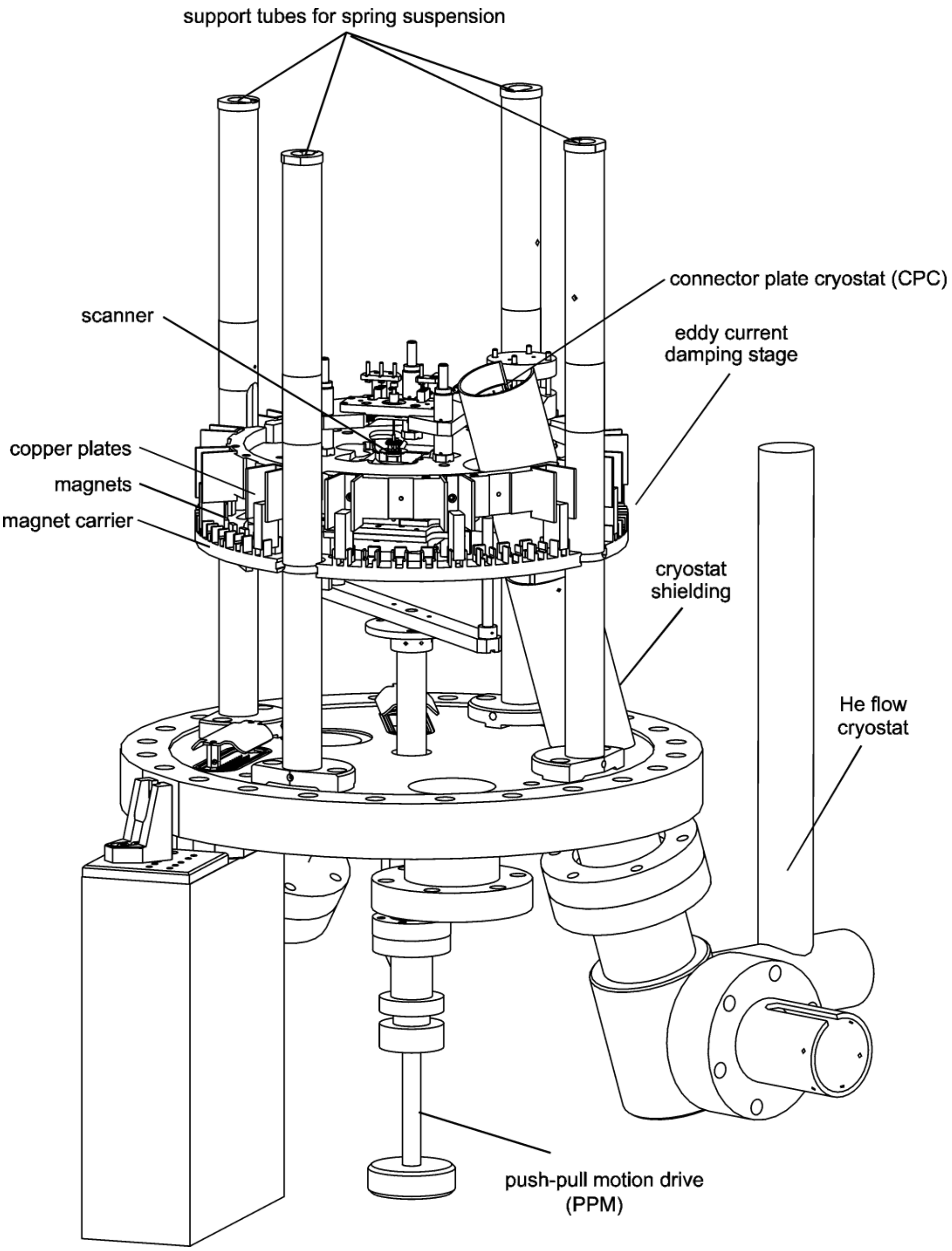


Figure 3. Side view of the VT STM XA.

3. Unpacking

Notice

Before unpacking please inspect the transport case for damage. If any damage can be seen or a rattling noise can be heard when moving the boxes **do not unpack** and contact your local Scienta Omicron agent **immediately!**

Attention

While unpacking be particularly **careful with the wobblestick and viewports.**

Upon delivery please **check for completeness** using the enclosed packing list. Please do a thorough **visual check on the mechanics and wiring** for any accidental transport damage.

Notice

All parts are clean to vacuum standards. Always use suitable polythene or cotton gloves for handling.

During transport the VT STM base plate is in its upper, locked position.

1. Open the outer box and remove the inner box.

2. Open the inner box and remove the top shock absorber.

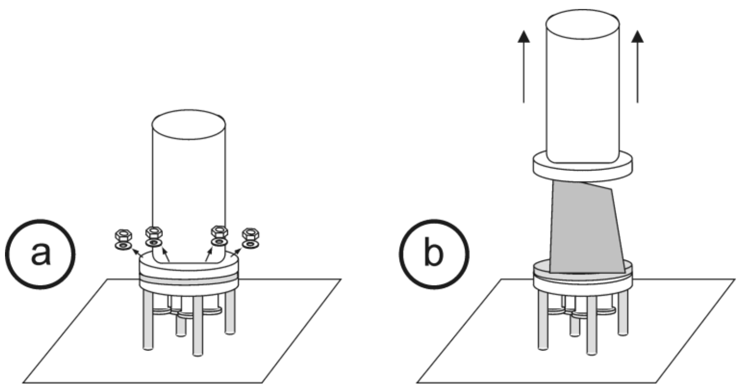
3. Remove the VT STM XA and wooden base from the bottom shock absorber.

4. Turn the VT STM XA upside-down and place it on a clean table.

5. Remove the 4 bolts which fix the wooden base to the feet and then turn back to stand the VT STM XA on its feet.

   Please wait until all parts have gained room temperature before removing the transport cylinder in order to avoid condensation of water on UHV parts.

   Now continue with the instructions pictured below.



continued overleaf

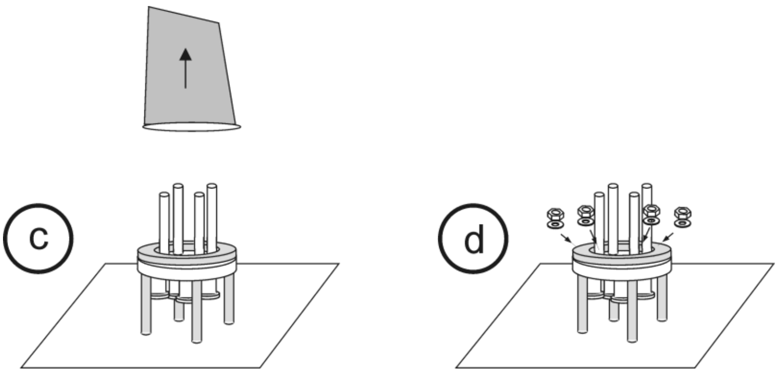


Figure 4. Unpacking instructions.

Transport Locks

Upon delivery the X/Y coarse motion drive is fixed with three screws at the bottom, see figure 5 on page 21. The whole STM stage is additionally protected with a head plate, see figure 6 on page 21.

Removing Transport Locks

To remove the transport locks please wear suitable **gloves** and use clean, non-magnetic tools.

- A set of **clean specialist tools** is provided together with the instrument.
- Place the VT STM XA head-down on a clean work bench / table. **Attention:** handle only at the legs and do not push the instrument across the table in order to avoid plastic abrasion on the UHV parts.

Notice
Remove transport locks in the following order: 1. Remove X/Y table transport locks 2. Remove STM stage transport locks.

Removing the X/Y Table Transport Locks

- Make sure the VT STM XA is sitting upside down on a clean work bench or table.
- In order to have more room turn the handle of the push pull motion drive (PPM) clockwise about half a revolution. Pull the handle up. Lock the PPM in this position by turning anticlockwise to the limit.
- The three transport lock screws of the X/Y-table can be seen in figure 5 on page 21.
- With the supplied spanner slacken the counter nuts and evenly turn the three screws counter clockwise. These lower the X/Y table plates back into their original position.
- Remove the transport lock screws with nuts spacers and washers and keep them with the other transport lock bits and pieces.

- Gently push the X/Y table until it touches a stopper in such a way that it does not slip when turning the instrument upside-down.
- Push the PPM to the locking position. It must lock the VT STM XA stage.
- Turn the VT STM XA over and carefully place it onto its feet.

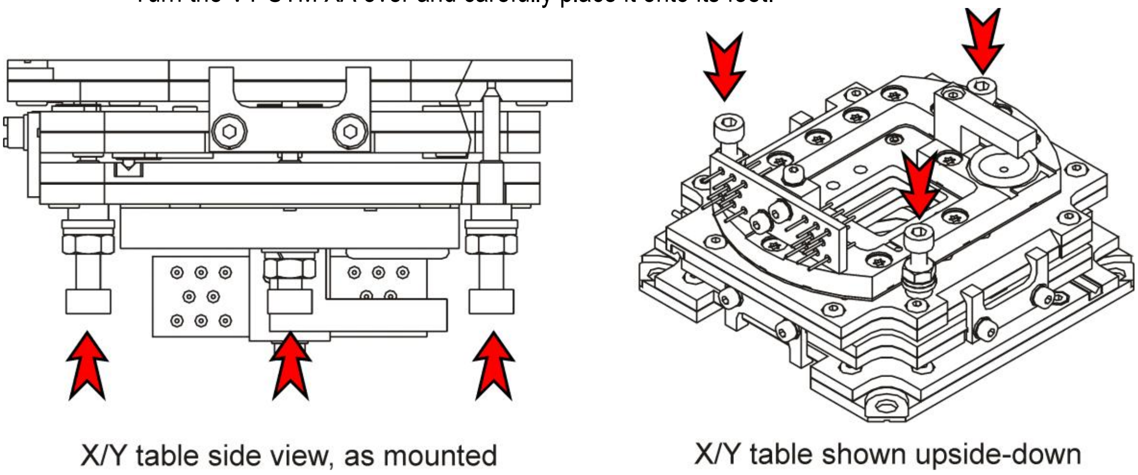


Figure 5. X/Y-table transport locks (3 screws and nuts.

Removing the STM Stage Transport Locks

- Make sure the VT STM XA is upright and standing on its feet.
- For cooling versions only: remove the three M3 screws and spring washers which hold the security tube and lift the tube out, see figure 6 (1).

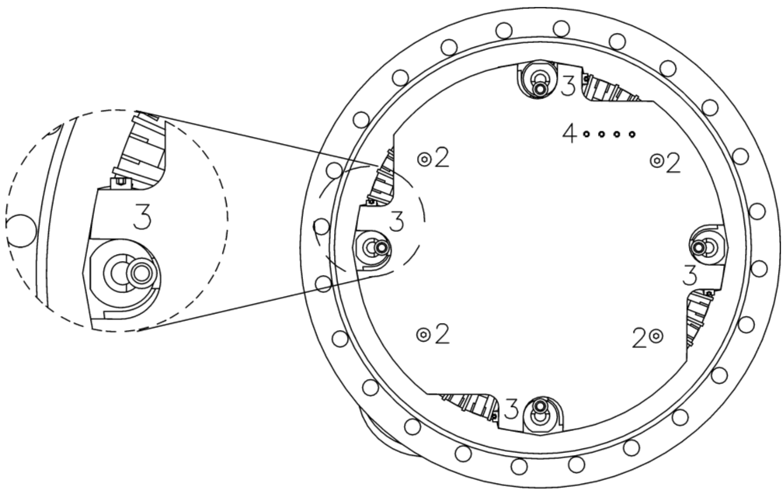


Figure 6. VT STM XA transport lock for the STM stage.

- For all versions: remove the four M4 screws and spring washers, see figure 6 (2).
- Now remove the four M3 screws and spring washers which hold the head plate to the spring suspension tubes, see figure 6 (3).
- Park these screws in the parking screw holes, see figure 6 (4), and carefully lift the head plate off.

- Unscrew the four spacing rods and keep them in a safe place, together with the loose screws and the head plate.

Notice
Please retain all parts of the transport lock, together with the packaging, in case the VT STM XA has to be shipped in the future, see page 123.

Reinstalling the Transport Locks

To reinstall the transport locks please wear suitable gloves and use clean, non-magnetic tools.

Attention
Take care not to drop any tool inside the VT STM XA assembly.

Notice
Reinstall transport locks in the following order: 1. Refit STM stage transport locks. 2. Refit X/Y table transport locks

Reinstalling the STM Stage Transport Locks

- Make sure the VT STM XA is upright and standing on its feet.
- Fit the four spacing rods and carefully place the head plate on top.
- Fit the four top screws (M4) and spring washers, see figure 6 (2), and tighten finger tight.
- Remove the screws and spring washers from the parking screw holes, see figure 6 (4), and use them to lock the head plate to the spring suspension tubes, see figure 6 (3).
- Fully tighten the four top screws, see figure 6 (2).
- Check that **all** screws have been fitted with spring washers and tighten them up.

Reinstalling the X/Y Table Transport Locks

Attention
When reinstalling the X/Y drive transport locks make sure not to tighten the lift screws to strongly in order to avoid damaging the coarse motion drive.

- Push the X/Y table until it touches a stopper in such a way that it does not slip when turning the instrument upside-down.

- Make sure the VT STM XA is sitting **upside down** on a clean work bench or table. (Do not push the instrument across the table in order to avoid abrasion on the UHV parts.)
- In order to have more room turn the handle of the push pull motion drive (PPM) clockwise about half a revolution. Pull the handle up. Lock the PPM in this position by turning counter clockwise to the limit.
- Fit the three transport locks (each consisting of screw, nut, washer and spacer) to the corresponding holes in the X/Y table.
- With the provided Allen screwdriver evenly turn the three lift screws clockwise. These pull the X/Y table plates away from each other. Attention, do not turn the lift screws too far or the fall-off stopper will be bent and damaged.
- When the X/Y table has come completely off evenly fix the position by tightening the counter nuts (restrain the screws while turning the counter nuts).
- Push the PPM to the locking position. It must lock the VT STM XA stage.
- Turn the VT STM XA over and carefully place it onto its feet.

Suspending the VT STM XA Stage

After removing the transport locks lower the VT STM XA stage.

1. Turn the handle of the push pull motion drive (PPM) clockwise about half a revolution.
2. Pull the handle down.
3. Lock the PPM in the down position by turning anticlockwise to the limit.

Locking the VT STM XA stage

- Push the PPM to the upper position. It must lock the VT STM XA stage so that sample exchange with the wobblestick can be carried out.

Inspecting the Spring Suspension

1. Place the VT STM XA onto a flat, strictly horizontal surface.
2. Make sure that the VT STM XA base flange is exactly horizontal, e.g. using a spirit-level.
3. With the PPM down check that the spring suspended VT STM XA stage can swing freely in all directions.
4. Check that all copper plates from the eddy current damping system are centred between the magnets to allow maximum free movement.
5. Check that the Kapton wires do not touch the locking mechanism or could be damaged by the motion of the PPM.

6. In cryo-versions check that the copper braid does not touch the stainless steel shielding.

Adjustment of the Vibration Isolation System

The lateral position and vertical alignment of the vibration isolation stage can be adjusted at the insert on top of the tubes housing the suspension springs.

Notice
The vibration isolation system has been preadjusted at Scienta Omicron. Normally no further adjustment should be necessary.

Touch the VT STM stage with a pair of tweezers to check free movement in vertical and lateral directions.

If necessary, adjust the position of the vibration isolation stage and of the VT STM base plate to fulfil the following requirements:

Attention
Make sure that the VT STM base flange is exactly horizontal before starting any adjustment procedure!

- The copper plates should be halfway down between the magnets.
- The copper plates should have equal distance to their adjacent magnets.
- The suspension springs must not touch the mounting tubes.

Height Adjustment of the VT STM

Height adjustment may be necessary after frequent or prolonged baking procedures.

- Slacken the countered nuts, see figure 7.
- Hold the spring insert in position and turn the nuts until you have reached the desired position.
- Counter the nuts properly to fix the vertical position.

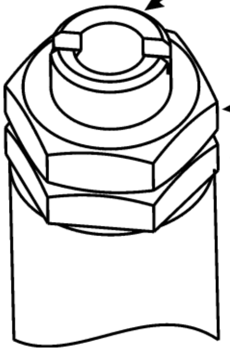


Figure 7. VT STM spring suspension adjustment, schematic diagram.

Notice

Do **not** unscrew the centre screws on these inserts because they hold the springs.

Lateral Adjustment

In addition you can turn the whole insert to **adjust the lateral position of the VT STM Base Plate**, see figure 7.

Notice

In cryo-versions the copper braid may push the base plate away from its laterally centred position. In this case please refer to page 95.

4. Electronics

Caution

Installation procedures and repair work may only be carried out by authorised personnel qualified to handle lethal voltages.

Switch off all units and wait for a few minutes (for discharge of the power supplies) before connecting or disconnecting any cables.

Make sure all high voltage plugs are secured before switching any one of the electronics units on.

All FT 12 connections have fixing screws which prevent accidental disconnection. The plugs must always be used with their fixing screw(s) tightened, i.e. plug secured. These screws, see figure 8, also prevent connecting a plug to the wrong FT 12 feedthrough and hence should not be removed when disconnecting the plug.

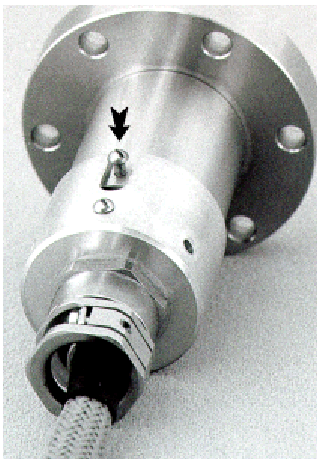


Figure 8. FT 12 connectors. always tighten the fixing screw.

Caution

All connectors which were originally supplied with fixing screws must always be used with their fixing screws attached and tightly secured.

The VT STM XA base flange has 12-pin connectors labelled VT TCC / VT HCC plus two 25-pin D-sub connectors labelled SPM PRE and PIC, see also figure 44 on page 107

Basic Wiring of MATRIX CU and Computer

- Connect the keyboard, mouse and monitor to the computer according to the manual supplied with the PC.
- Connect the Ethernet cable between the HUB and the Ethernet card of the computer (NOT the main-board Ethernet connection).

- Connect the Coarse Position remote Box to the MATRIX CU Coarse Positioning board.

Coarse Cable PIC

The coarse cable PIC connects the scan piezo and the coarse motion motors to the electronics system. It also connects the excite signals of the QPlus sensor.

⚠ Caution

The VT STM XA has been designed for UHV use. If connecting the VT STM XA outside a closed UHV chamber, lethal voltages are within reach presenting a potential danger. Note that using the VT STM XA other than according to the Normal Use definitions stated in your VT STM XA User's Guide renders the CE Declaration void. In this case the user is responsible for ensuring safe operating conditions and compliance with CE and other local directives.

- Connect the D-sub connector PIC to the respective socket on the VT STM XA base flange and fix the screws.
- Fix the grounding cables with the solder tags to the grounding screw on the VT STM XA base flange.
- Connect the sub-D connectors and the grounding connection at the other end of the PIC cable to the electronics according to table 1 below.
- Disconnect the two ground connections from each other (open the bridge) to avoid ground loops. Remember that the black system grounding cable is indispensable for system grounding when the grounding bridge is open.

for STM-only variants:

PIC cable	to MATRIX CU	socket	connector
sub-D PIEZO	piezo driver board	PIEZO OUT	5-pin sub-D
sub-D COARSE	coarse position board	COARSE	25-pin sub-D
black ground		⏚	grounding bolt

for QPlus variants:

PIC cable	to	socket	connector
sub-D PIEZO	PFU V2	PIEZO OUT	5-pin sub-D
sub-D COARSE	Coarse Position board	COARSE	25-pin sub-D
black ground		⏚	grounding bolt
POWER Mod	Preamp panel	Power	4-pin LEMO
EXCITE	Preamp panel	EXC OUT	BNC

Table 1. Connecting the coarse cable (PIC) to the electronics.

PDC6DQ Version (Optional, QPlus only)

Attention
Connecting to the wrong Piezo Driver board may destroy the scanner. Please see details below.

The QPlus option needs a special piezo driver board PDC6DQ. This board has a reduced bandwidth compared to the PDC6D in order to provide an enhanced signal-to-noise ratio. In principle you can also use the PDC6DQ for STM scanning (fast scanning not possible) but NOT the other way round.

For a VT STM XA with QPlus option ordered the MATRIX CU contains both PDC6 boards. These are connected to the Piezo Filter Unit (PFU) which provides some filtering and the means to switch between the two PDC6 boards – either manually or via software. The signal for PDC6 switching is generated on the CRTC and transferred to the PFU boards via the SBB. The signal for Z-gain switching is also generated on the CRTC and transferred to the PDC6 boards via the SBB and BUS.

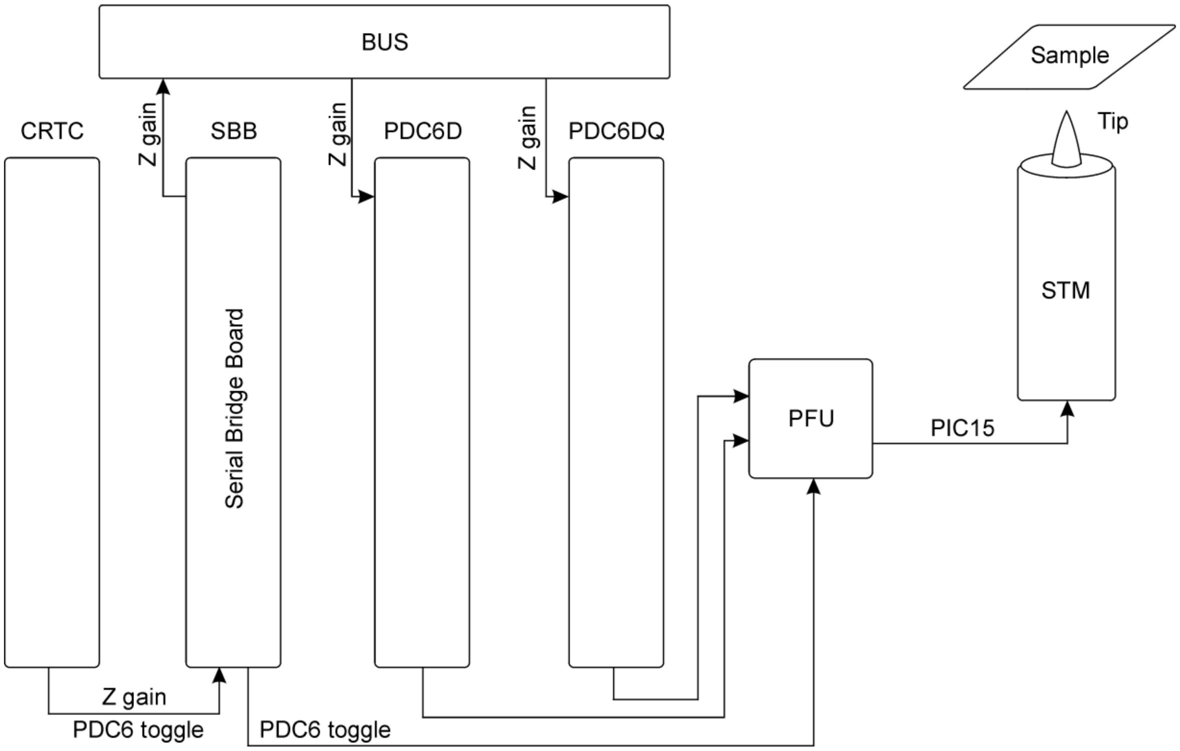


Figure 9. QPlus signal diagram for PDC6D and PDC6DQ.

In addition the Z-gain of the PDC6 boards can be set to 4 different values:

1 x 10.8	1/3 x 10.8	1/5 x 10.8	1/7 x 10.8

The Z-gain reduction leads to an increase in bit-resolution (Z-resolution) by reducing the minimum Z-step width of the scanner. Note that this also requires a reduction of the coarse step width during AUTO APPROACH. Example: Reducing the Z-gain to 1/5th reduces the entire scanner Z-range to 1/5th. In this case the coarse step width (SPEED dial on remote box) must also be reduced to 1/5th so that the scanner range is still larger than the coarse step width. Otherwise a tip crash during AUTO APPROACH might be the result.

Preamplifier SPM PRE4/(optional: SPM PRE4 QPlus)

The preamplifier is the first amplification stage for the tunnelling current signal. The amplification electronics has to be very close to the instrument to reduce electronic noise. The SPM PRE4 has the I/V converter (first stage) **inside the UHV chamber** and the preamp box mounted to the atmospheric side of the 25-way D-sub connector SPM PRE4.

The preamp box also translates the gap voltage between the MATRIX CU and the tip and serves as a power supply for the current-to-voltage converter (IVC) located inside the UHV chamber, see above.

The SPM PRE4 QPlus preamplifier comprises a second channel for QPlus AFM operation.

Attention

Make sure switch UOS is always in position "hybrid active", see figure 50 on page 117.

Attention

Never connect or disconnect the preamplifier supply voltage with the MATRIX CU switched on.

Connection Instructions

- Connect the SPM PRE 4 box to the respective socket on the VT STM XA base flange and fix the screw.
- Fix the green-and-yellow cable with the solder tag to the grounding socket on the VT STM XA base flange.
- Connect the other end of the preamp cable to the electronics according to table 2.
- Connect V GAP MON to the blue BNC connector on SCAR using the supplied Tee.
- The VTP-socket of the SPM PRE 4 box **must** be fitted with the **P**reamp **P**rogram **P**lug (VT PPP).

from preamp	to MATRIX board: plug		colour
red BNC	SCAR: IT	breakout cable PREAMP	red
blue BNC	SCAR: V GAP	breakout cable PREAMP*	blue
5-pin DIN	SCAR	breakout cable PREAMP	
3-pin XLR	DCOUT53 (top or centre)	MATRIX Power Supply	

Table 2. Connections between SPM PRE 4 and MATRIX CU. *) Use the supplied Tee.

Notice

All instruments fitted with SPM PRE 4 have the sample grounded while the tip is floating. Please do not connect anything other than the SPM PRE 4 to this feedthrough.

from SPM PRE4/QPlus	to MATRIX CU	Socket	Connector
POWER SUPPLY	DC18/5	DC OUT 2	5-pin mixed-D
ωout	AFM-SPU	FN IN	BNC, black

Table 3. Connections from SPM PRE4/QPlus to MATRIX CU.

Preamp Program Plug PPP

For the all versions the preamp box has an additional "preamp program plug" (PPP). This must always be connected.

- For STM version without QPlus: VT PPP
- For QPlus versions: VT PPP 3DQ

Heating and Temperature Measurement Cable TCC XA

VT STM XA with Heating only option

- Connect the FT12 connector to the TCC feedthrough on the base flange, see figure 10 on page 32.
- Connect the red and the black banana plug (Heater Sample) to the HC 600 heating power supply.
- Connect the PT100 plug to the Temperature display.
- The three cryostat plugs stay unconnected.

VT STM XA with LN2 cooling option

- Connect the FT12 connector to the TCC feedthrough on the base flange, see figure 10 on page 32.
- Connect the red and the black banana plug (Heater Sample) to the HC 600 heating power supply.
- Connect the PT100 plug to the Temperature display.
- The three cryostat plugs stay unconnected.

Notice
In the above variants the optional temperature controller can be used to replace the HC600 and the temperature display.

VT STM XA with LHe cooling option

- Connect the FT12 connector to the TCC feedthrough on the cryostat.
- Connect the red and the black banana plug (Heater Sample) to the HC 600 heating power supply or to the temperature controller (optional, see below).

Optional Temperature Controller

- We recommend to control the temperature directly on the sample reception. This means that the cryostat heater is only used for fast warm-up and not for counter heating.
- Connect the plug Pt100 to channel A of the temperature controller
- Connect the plug Cryostat to channel B of the temperature controller
- Connect the red and black banana plugs labelled "Heater Sample" to heater output 1 of the temperature controller
- Connect the two black bananas labelled "Heater Cryostat" to heater output 2 of the temperature controller, if available.

LakeShore Controller Model LS 335

The above wiring instructions refer to the LS 335 with the following settings.

- Out 1 controls channel A

  heater resistance setting 50 Ω

  maximum current 1 A

- Out 2 controls channel B

  current mode

  heater resistance setting 25 Ω

  maximum current 1 A

Note that Out 2 can also be used in voltage mode to yield an analogue signal proportional to temperature that MATRIX can be read and save with the measurement data.

Wiring Diagram

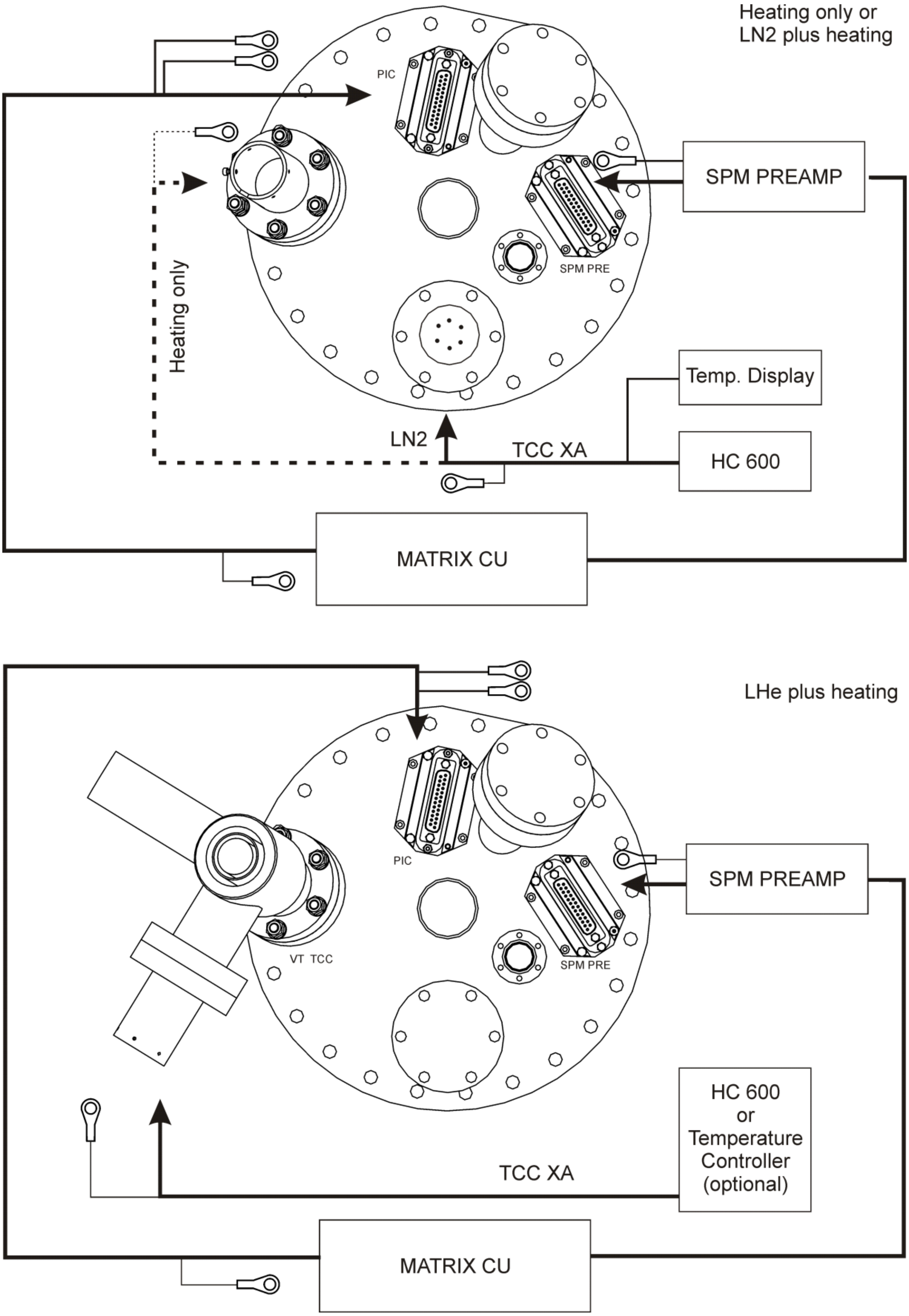


Figure 10. Cabling of the base flange.

Oscilloscope Wiring for Scienta Omicron SPMs

For monitoring purposes an oscilloscope is strongly recommended (interacting time scale typically around 1 ms/div). The oscilloscope is the best instrument to see most of the problems in AFM/STM work after you have gained some experience in interpreting its lines. Try switching the scan on and off with the Stop and Start buttons in the Scanner Window. Without scanning you should see a relatively stable DC-signal with only a little noise.

Connect the oscilloscope cables (including oscilloscope mains cable) to the MATRIX rack from the back using one of the provided openings on the inner side panel. Thread the signal cables through on the provided guide rails and connect to the connector terminal on the front. You can now easily access all signals of interest.

Notice

Initially only a few signals are connected to the terminal on the front. Please feel free to connect other signals of interest as described above.

Function generator, lock-in amplifier or similar devices can be connected in the same way as the oscilloscope.

Caution

Always use the Z MON socket on the BNC connector terminal for connecting the oscilloscope.

To connect an oscilloscope to your system please follow the table below.

mode	channel 1	channel 2
STM	BNC connector terminal IT MON (DC mode)	BNC connector terminal Z MON (AC mode)
QPlus	AFM-SPU FN IN (AC mode)	AFM-SPU DF OUT (DC mode)

Table 4. Oscilloscope cabling.

Power Supply

Notice

- Before connecting the electronics to the mains supply check for correct voltage setting.
- Connecting all mains cables to the same wall socket avoids ground loops.

The following units have to be connected to the power sockets inside the MATRIX rack:

- Computer and monitor
- MATRIX CU
- Heater control unit, if used

- Oscilloscope, if used.

Starting the Electronics

1. Check the cabling, see pages 26ff.
2. Switch on the MATRIX rack.
3. Switch on the computer.
4. Wait for about one minute for the control units to boot up before starting MATRIX software.

Notice
In case of problems or for further details please refer to the Matrix Application Manual.

5. Mechanical Details

STM Scanner

The Scienta Omicron VT STM XA uses a single tube scanner with a maximum scan range of about 12 µm × 12 µm with a Z-travel of about 1.5 µm. A Z-resolution of better than 0.01 nm can be achieved.

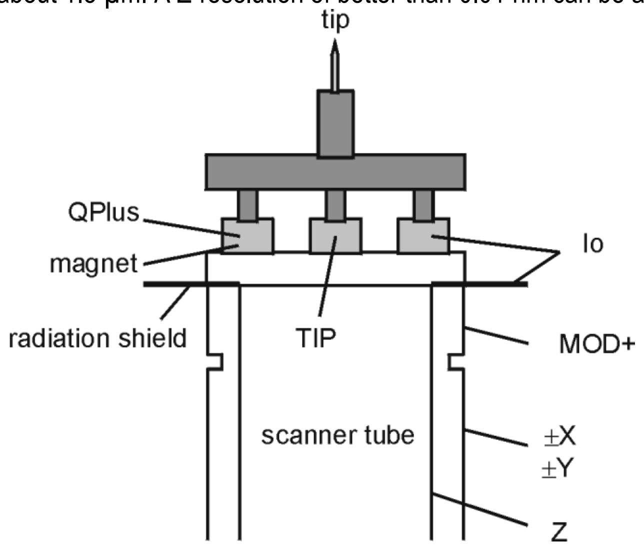


Figure 11. The VT STM XA scanner electrical contacts. Note that the scanner is shown with a tunnelling tip mounted.

Notice

The scanner has been calibrated for atomic resolution dimensions. When scanning large areas (i.e. in the µm range) the displayed distance values are **smaller** than the physical lengths. A new calibration may be necessary for measuring accurate long-distance values.

QPlus Option

If the instrument is equipped with a QPlus option, QPlus sensors can be mounted to the scanner instead of a tunnelling tip. It is based on a quartz tuning fork with one prong fixed and a tungsten tip or cantilever mounted on the other vibrating prong. The quartz is excited to swing in ±Z direction with a frequency of about 20-32 kHz (depending on tip/cantilever weight). A Z-resolution of better than 0.01 nm can be achieved with a QPlus sensor mounted.

The QPlus sensor design allows simultaneous tunnelling current measurements while scanning in AFM non-contact mode i.e. feedback loop regulation via df. The QPlus sensor can also be used for pure STM scanning.

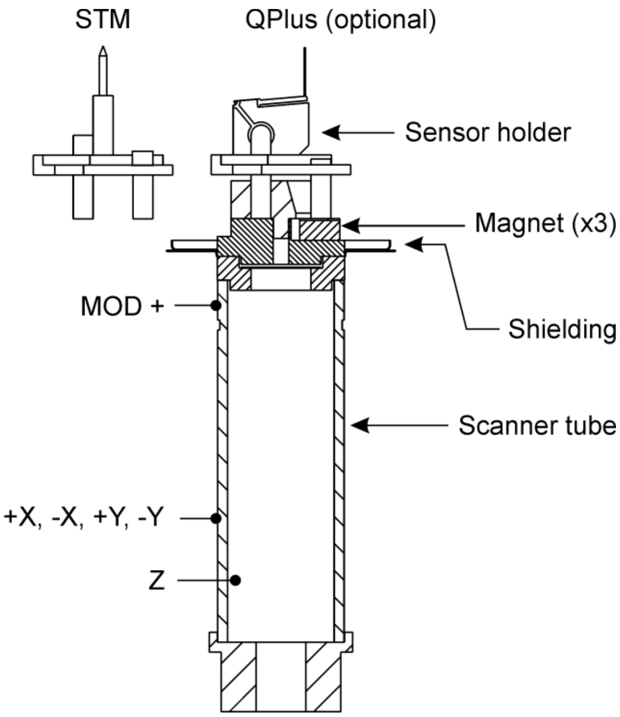


Figure 12. QPlus scanner mechanical details.

Sample Coarse Positioning Device

The scanner is positioned with a 3-axes linear piezo motor. It uses slip/stick effects related to inertia forces when a piezo is driven in a fast/slow sequence. The sliders are magnetically coupled to three shear piezos which are driven with a sawtooth voltage input. The sliders are transported during the slow movement of the piezo and slip during the fast piezo motion due to their inert mass. The following sketch shows the shape of the voltage ramp applied to the piezos during a single step.

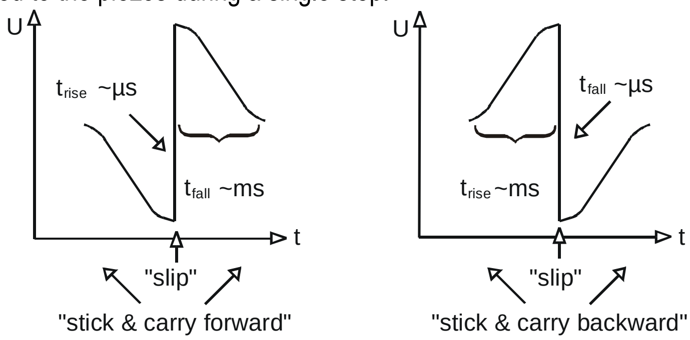


Figure 13. Typical voltage applied to piezos during slip/stick motion.

With the slip/stick technique the following movements can be achieved:

- X coarse movements parallel to the sample surface,
- Y coarse movements parallel to the sample surface
- Z coarse movements perpendicular to the sample surface.

For further details please refer to the MATRIX Electronics technical reference manual.

6. Mounting the VT STM XA

Work in accordance with UHV standards, i.e. avoid bringing dust or dirt into the chamber. Never touch any UHV surfaces with bare hands, always use clean polythene gloves and degreased tools.

Mounting the Chamber

1. Stand the VT STM XA on a workbench (do not fix the feet with bolts yet).

2. Insert a new copper gasket on the base flange.

3. Place 3 studs into the marked holes of the base flange before sitting the chamber onto it.

4. Now place the UHV-chamber onto the VT STM XA-unit. For this two people are needed, one to hold the base flange and one to set the chamber onto it. Take care not to damage the wobblestick (if already mounted) during this operation. Fix the bolts only provisionally.

5. The port for the wobblestick has to be perpendicular to the direction of inserting the sample, see figure 14. Seen from the wobblestick port, you look into the sample support stage with the main cooling stage to the right.

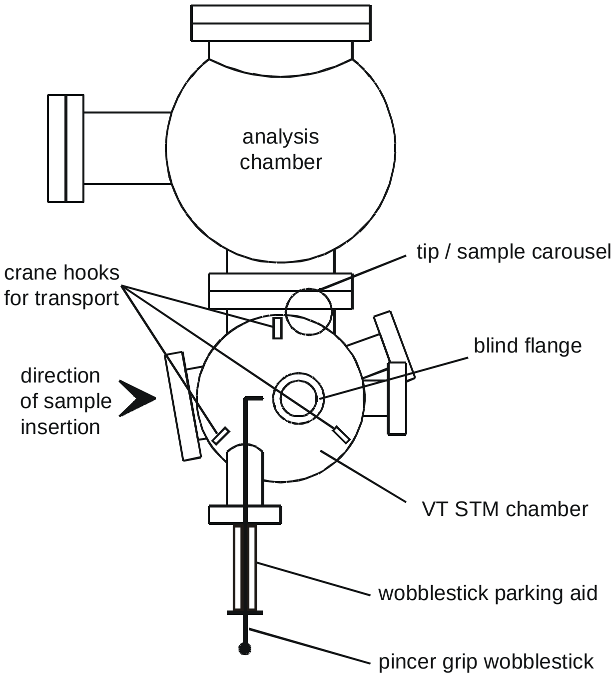


Figure 14. Mounting the VT STM XA chamber to the main system.

Mounting the Carousel

- Sit the carousel onto the nipple of the VT STM XA chamber, see figure 15, and fix it with two lateral screws using an Allen key.

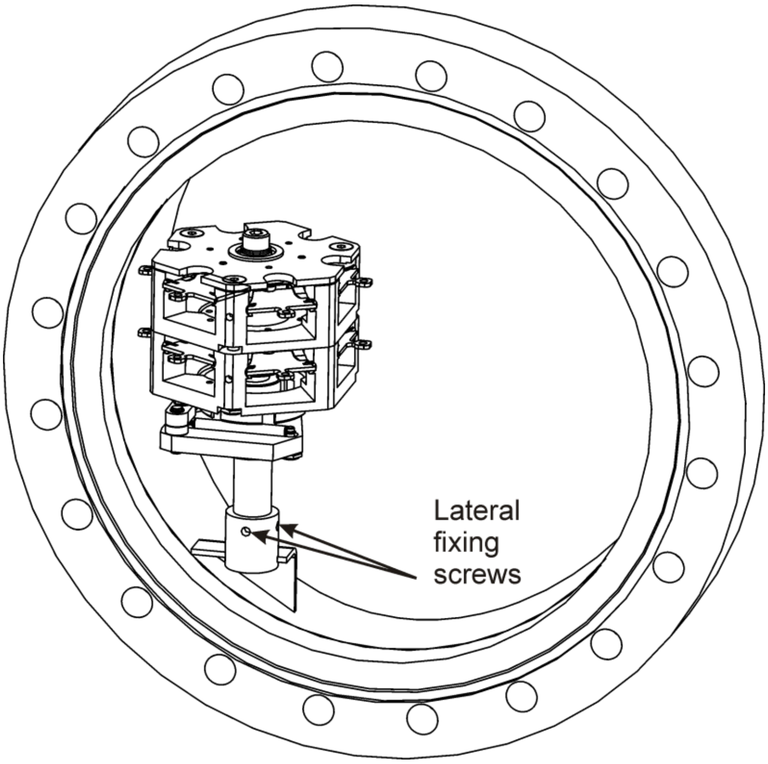


Figure 15. Mounting position of the carousel.

Mounting the Wobblestick

Before mounting the wobblestick familiarise yourself with its operation: press the knob against the handle to open the jaws.

- Slip a copper gasket over the wobblestick from the front. Mount the wobblestick to the VT STM XA chamber. It can be inserted through the flange without dismantling, see figure 16. Fix the bolts. (If you only want to practise wobblestick operation a used gasket will do.)

Notice
Do not bend the wobblestick bellows during the mounting procedure.

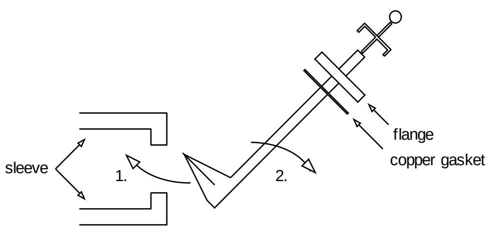


Figure 16. Mounting procedure for the wobblestick, top view.

Notice

The wobblestick can be moved to and fro, up and down and sideways. **It must not, however, be rotated!**

Notice

For initial wobblestick practising in air it is not necessary to fit the viewports. When fixing the viewports do not forget to attach the CCD camera holder, see below.

Mounting the CCD Camera

The CCD camera attachment is normally connected to the middle one of the three CF 35 flanges. To mount the CCD camera to the chamber you have to fix the viewports, see figure 17.

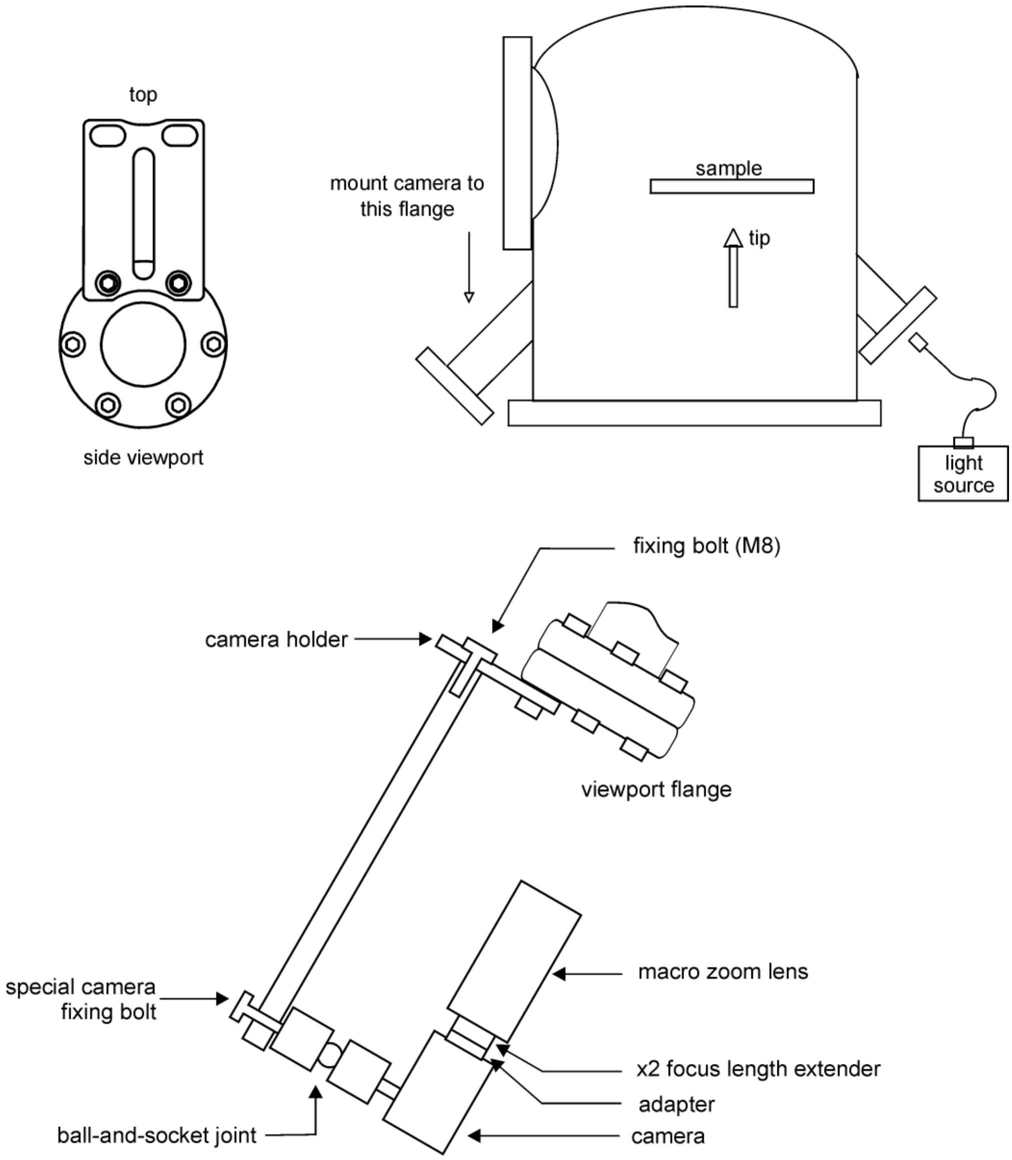


Figure 17. Mounting position for the CCD camera attachment.

The CCD camera can subsequently be mounted to the holder according to figure 17.

The following electrical connections have to be established:

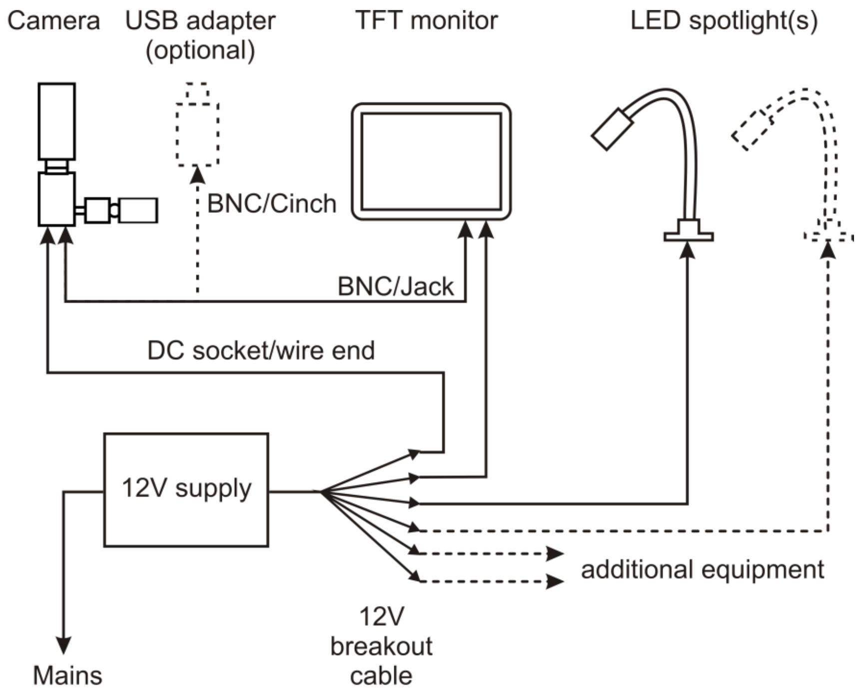


Figure 18. Wiring of CCD camera and LED spotlight. Depending on your system configuration there may be one or two LED spotlights.

Notice

Always connect the camera supply to the camera (check polarity) **before** connecting to mains in order to avoid short circuits.

Make sure the unused connectors of the breakout cable **do not touch ground/earth** (e.g. the UHV chamber) to avoid potential short circuits.

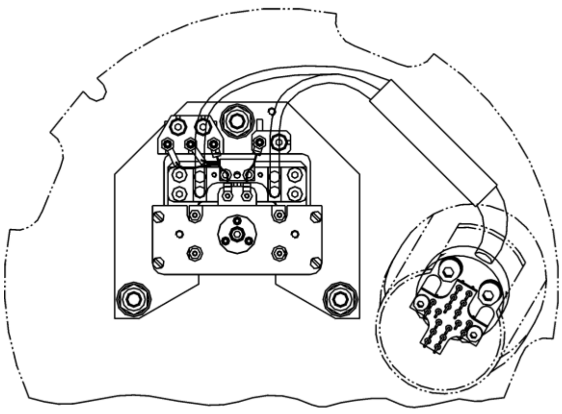
from	to	with
12 V power supply	mains	standard mains cable
12 V power supply	breakout cable	power supply output cable
breakout cable	LED spotlight(s)	low voltage cable
breakout cable	camera	low voltage cable (DC socket/wire end)
breakout cable	TFT display	low voltage cable
camera	TFT display	BNC (4 way Jack) cable
camera	USB adapter*	BNC/RCA (CINCH) cable

Table 5. Wiring of CCD camera and LED spotlight. *) The optional USB adapter can be used for connecting to your PC if you want to use your computer screen instead of or in addition to the TFT display.

7. Handling

Sample Stage

Try loading and unloading a sample plate to and from the VT SPM. Practise all steps with an empty sample plate first!



Sample stage with PT-100 resistor

Figure 19. Sample stage, detailed schematic diagram.

Loading a Sample to the VT SPM

- Retract the scanner as far as possible.
- Carefully slide the sample plate into the reception on the VT SPM sample stage horizontally.
- Push in until the plate is clamped rigidly. The springs will require some force to push the plate fully home.

Notice
Lack of rigidity can result in unstable or oscillating conditions during the experiment.

Tip Reception

With the initial delivery of the Scienta Omicron VT STM XA you get a set of tips already mounted to dedicated VT-tip holders. The same tip holder may be used several times if the tip is clamped by squeezing the tip holding tube. It is also possible to spot-weld a tip to the holder.

With the initial delivery of the Scienta Omicron QPlus sensor option you receive a set of QPlus sensors already mounted to dedicated VT QPlus sensor holders. Note that these QPlus sensors are extremely delicate and are therefore mounted to their holders in factory.

The tip holder is magnetically clamped on top of the scanner. To change the tip a specially designed tip transfer plate can be fitted into the sample stage. The shape of a transfer plate is similar to those of the sample plates.

Loading a Tip Transfer plate to the Sample Stage

Having practised sample exchange with the VT STM XA now try loading and unloading a tip to and from the VT STM XA. Practise all steps with an empty tip holder first!

1. Gently slide the carrier home sideways into the sample stage. The tag plate engages in the sample stage reception.

2. Open the pincer and withdraw the wobblestick.

3. Check that the tip transfer plate is fully home in the sample stage. If the tip transfer plate cannot be fully inserted with gentle pushing: remove it completely and start again.

4. To remove the tip transfer plate grip it by the tag and pull sideways.

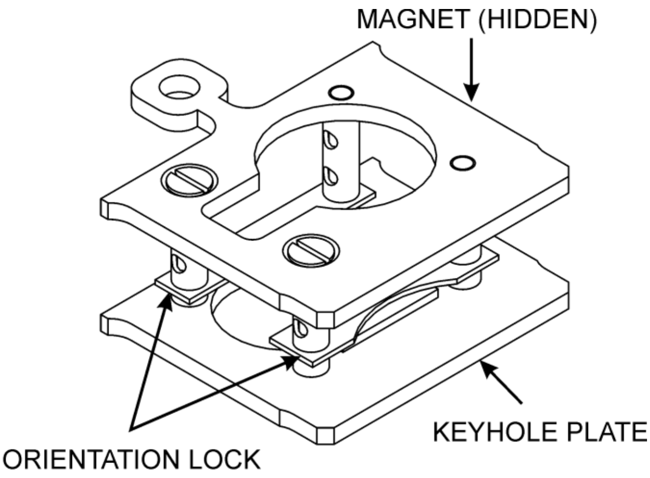


Figure 20. The VT STM XA tip transfer plate.

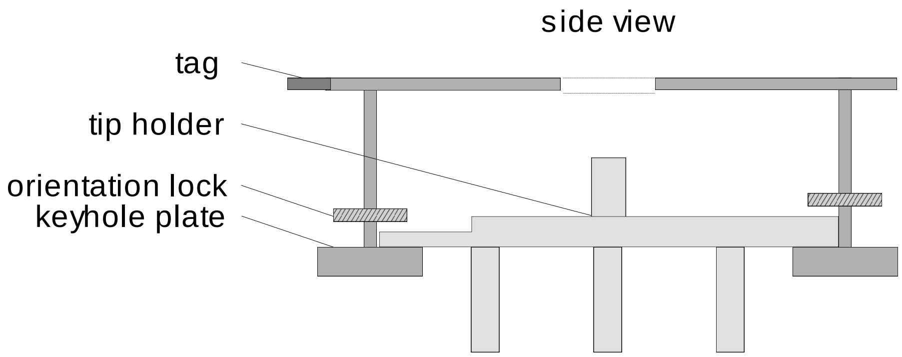


Figure 21. VT STM XA scanner with mounted tip holder, side view schematic diagram.

Mounting a Tip/Sensor Holder to the Scanner Reception

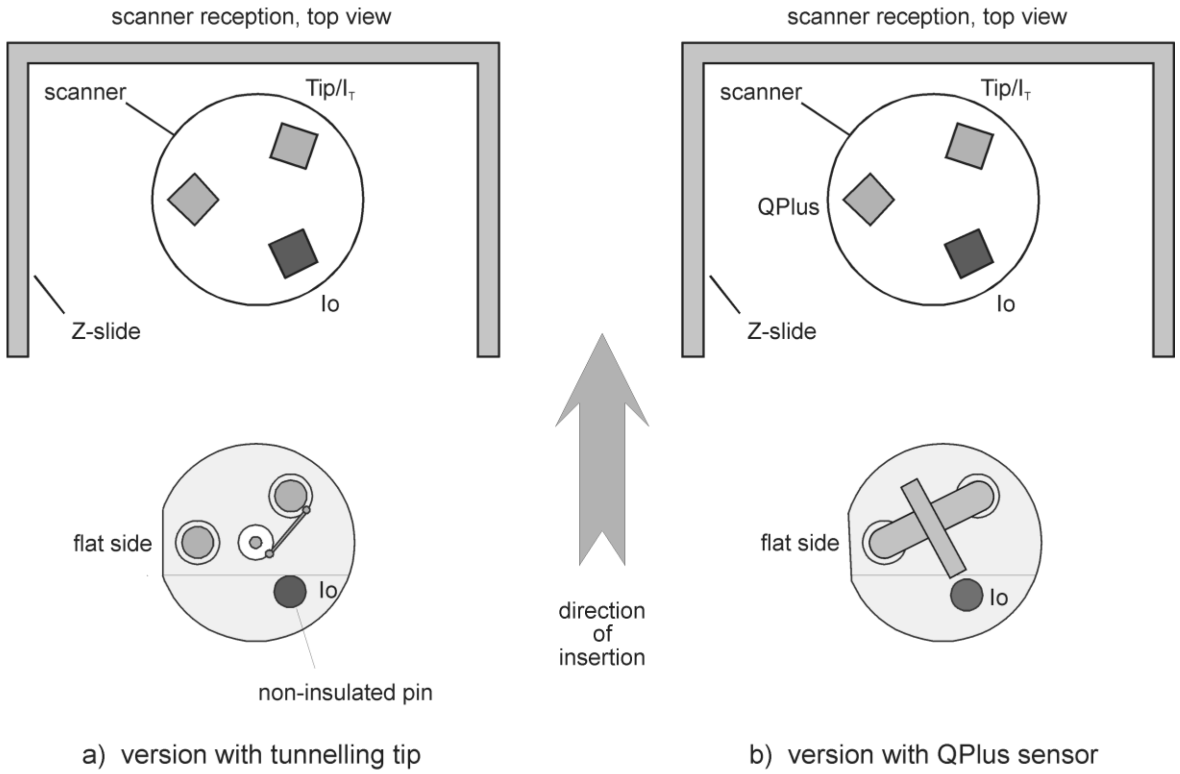


Figure 22. Orientation of the sensor holder, top view schematic diagram.

1. Mount a tip transfer plate with a tip holder to the sample stage, see above.

2. Approach the scanner to the tip holder by pressing the ±X, ±Y and APPR (-Z) buttons on the remote box until the tip holder is magnetically drawn onto the scanner. (Tip: fully extend the scanner and then withdraw a little bit.)

3. Disengage the tip holder from the keyhole opening of the tip transfer plate using the coarse motion drive (press ±X on the remote box). If it does not move in X

direction, press APPR / RETR (-Z/+Z) and/or ±Y once to make sure that the tip holder does not touch the tip transfer plate and try again with ±X.

4. Withdraw the scanner (press RETR (+Z) on the remote box) until the tip is completely retracted.

5. Remove the empty tip transfer plate from the sample stage and place it in the carousel.

Removing a Tip Holder from the Scanner

1. Mount an empty tip transfer plate to the sample reception (keyhole plate pointing downwards).

2. Engage the tip holder in the keyhole opening of the tip transfer plate using the coarse motion drive (press the ±X and ±Y buttons to centre the tip in the hole of the keyhole plate and -Z to move it in).

3. Move the scanner towards the narrow end of the keyhole (press ±X on the remote box) until the tip holder is attracted by the small magnet, see figure 20.

4. Withdraw the scanner (press +Z on the remote box) to leave the tip holder in the transfer plate, held in place by the small magnet. Since the scanner magnet is rather strong you may not be able to withdraw it at the first attempt. In this case move the tip back and forth (press the ±X and ±Y buttons) and try again.

5. Make sure the scanner is fully retracted and remove the tip transfer plate from the sample stage and place it in the carousel.

Wobble Stick

SCIENTA OMICRON tip and sample carriers are designed for handling within a UHV system using a specially designed pincer grip wobble stick.

- The wobble stick is a **double action pincer**, i.e. both jaws are movable.
- Press the **knob against the handle** to open the jaws.

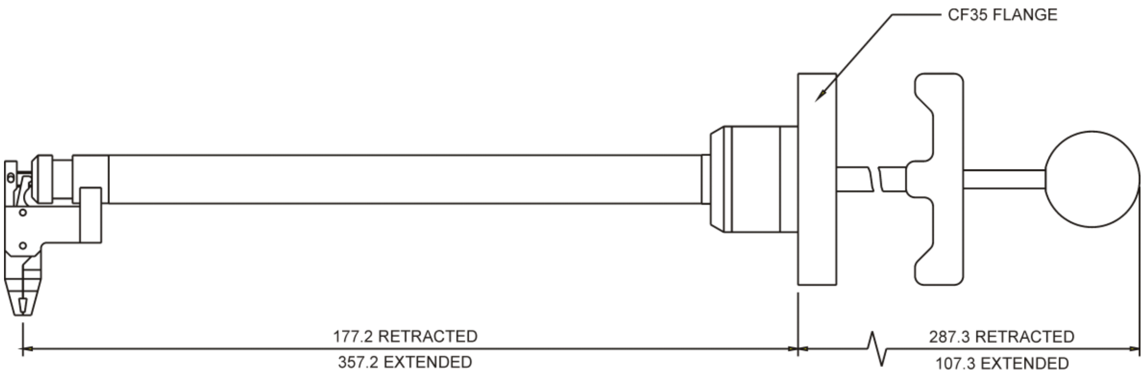


Figure 23. Wobble stick WS 180, top view. A longer wobble stick WS 270 and adapter chamber are also available optionally.

There are three possible motions with the wobble stick:

- a linear motion (Z),
- an angular motion (X,Y)
- and the pincer grip motion.

Attention
The wobble stick must never be rotated!

After mounting:

- Try each of the three motions separately from the others.
- Position the wobble stick close to the tip/sample carousel as well as in front of the sample stage.
- Open the pincer grip by **pushing the knob**. A lot of force is required to open the wobble stick jaws, so be careful not to move the position of the whole wobble stick during their operation.

Notice
Do not pull the handle against the knob as the handle controls the **position** of the whole wobble stick.

The aim of the exercise at this stage is to get familiar with the wobble stick motion, in particular to separate the pincer motion from the X, Y and Z motion of the wobble stick.

When not in use carefully pull out the wobblestick and secure it using the wobblestick parking aid, see figure 24 below.

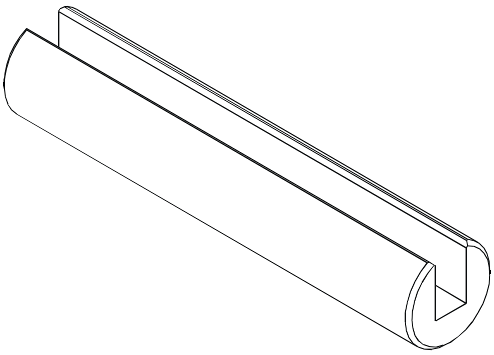


Figure 24. Wobblestick parking aid, isometric view.

Notice
Make sure not to damage fragile parts when moving the wobble stick. Do not bend the wobble stick too heavily to avoid deformations to the sleeve.

Notice

Do not scratch the wobble stick bellows at parts of the damping stage or chamber as this may cause vacuum leaks!

Make sure the heating current is zero (I_H = 0 mA) when using the wobble stick.

To get some experience with tip and sample handling using the wobble stick the following steps are recommended:

- Mount VT SPM-chamber to VT SPM and the wobble stick and carousel to the VT SPM-chamber, see page 37. Initially mount the base flange provisionally using an old copper gasket.
- Practice the tip/sample handling under ambient conditions.

Notice

The VT SPM is not very stable when mounted to its chamber and standing on its feet. Please take care not to tip it over when practising wobble stick operation.

Carousel

Try turning the carousel, then try loading and unloading a sample plate to and from the carousel.

- Use the wobblestick to turn the carousel by pushing or pulling at the sample reception of the carousel.

Loading/Unloading a Sample Plate

Notice

Practise with an empty sample plate, empty tip transfer plate or Ta-plate. Do all steps by hand first.

Initially load the carousel using a pair of tweezers.

Unloading the Carousel

1. Turn the carousel until a sample position becomes accessible.
2. Grip the tag of the sample plate with the wobble stick (make sure that the jaws are fully closed).
3. Pull the sample plate sideways to the left.

Loading the Carousel:

1. Turn the carousel until a free sample position is accessible.
2. Grasp the tag of a sample plate (e.g. from the manipulator) with the wobble stick and slip it sideways into the entry slot, see figure 18. For direct heating, radiative

heating and cooling sample plates only the molybdenum plate engages in the carousel reception slot.

3. Open the wobble stick jaws and let go of the tag.

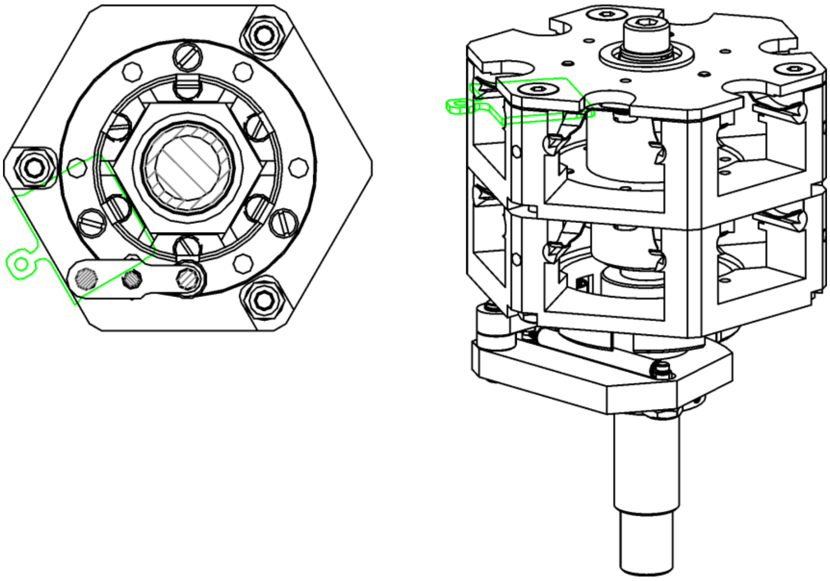


Figure 25. Tip/sample carousel, detailed schematic diagram.

Loading/Unloading a Tip Transfer plate

The tip transfer plate can be loaded and unloaded to and from the carousel in exactly the same way as a sample plate.

Unloading a Sample from the VT STM XA

Notice
Remember to lock the spring suspension before sample exchange.

- Retract the scanner as far as possible.
- Pull the tag of the sample plate. Move the wobble stick to and fro whilst pulling because the receptor gets sticky (more so under vacuum).

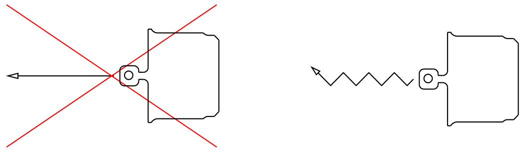


Figure 26. Unloading the sample from the VT STM XA reception, top view.

Notice
During sample exchange be careful not to bend the wobble stick too heavily. Keep it horizontal at all times. The sample plate must stay in the horizontal insertion plane.

8. Setup and Test Procedure

When setting up the VT STM XA for the first time, reinstalling the instrument to the main UHV system after transport and after extensive on-site repair work it is highly recommended to perform some scans in air in order to check all major functions of the instrument before pumping down.

Notice

If your VT STM XA has been installed and set up ready for use by an Scienta Omicron service representative you may skip this chapter and continue on page 53.

Caution

Warning: Lethal Voltages!!

Installation procedures and repair work, including SPM experiments in environments other than UHV, may only be carried out by authorised personnel qualified to handle lethal voltages.

Take care not to touch any wires or piezo elements.

Attention

When connecting the preamp box to the VT STM XA in air, one support leg must be fixed to the base flange using the provided extension piece in order to allow enough space between the legs.

Attention

The VT STM XA, particularly the QPlus variant, is very sensitive to **electrostatic discharge (ESD).**

- Always apply appropriate ESD protection when inserting sensors. Use only tools certified for CMOS handling.

For first images in air we recommend loading an old tip (Pt/Ir recommended) and an evaporated gold sample. These can be used to check the coarse motion drive and scanning function in air.

Due to the annealing capabilities of the VT STM XA sample plates the samples are limited in size. Maximum lateral dimensions are 3 mm × 9 mm for a rectangular sample, maximum sample height is 2 mm.

Notice

It is strongly advisable for monitoring purposes to have an **oscilloscope connected** to the VT STM XA during operations (a simple two channel scope is sufficient). Normally monitor the current signal (IT MON, time scale about 1 ms/div) and the Z- voltage (Z MON from the SCAR board). On the oscilloscope look for a Z-voltage in the range ± 140 V during approach and AC-coupled with about 200 mV/division whilst measuring corrugations below 1 nm. For connection instructions see also page 33.

Getting Started

Before starting a check-up experiment the following steps have to be performed:

1. Check that the VT STM XA is installed correctly, see page 37 and connect the electronics, see chapter 4. For this check-up experiment the VT STM XA chamber needs not to be mounted to the main UHV system. However, in this case be very careful not to tip it over when operating the wobblestick.

2. Make sure all green-and-yellow grounding cables (earth) are connected to the grounding screws at the base flange.

3. Mount the provisional tip and sample, see chapter 7.

4. Lower the VT STM XA stage, see page 23.

5. Check that the vibration isolation works properly, see page 24.

6. Start MATRIX now.

7. Make sure the appropriate parameter set is loaded.

Scanning in air can be carried out in the same way as scanning in UHV. For a detailed description of the coarse approach and scanning procedure please refer to page 55ff. Once you are sure the VT STM XA is working as it should, proceed with chapter 9.

9. The UHV System

For wobblestick and tip/sample handling purposes and during the air test the VT STM XA chamber was initially mounted provisionally with used copper gaskets. Do this procedure now carefully with new gaskets and tighten all bolts.

To mount the VT STM XA chamber to the main UHV system please follow the steps below.

1. Check if the base plate is horizontal. If not align the VT STM XA base plate by adjusting the springs, see page 24.

2. Mount the VT STM XA-chamber to the VT STM XA and fix the bolts provisionally see page 37.

3. Mount the wobblestick and carousel to the VT STM XA-chamber, see page 37.

4. Load carousel with tips and samples, see page 46. Choose your tips and samples carefully since the set-up time for UHV experiments is quite long! Load at least one evaporated gold sample and possibly an old tip for initial experiments.

5. Mount the so prepared VT STM XA-chamber to your UHV-system (main flange), see page 37. The main flange rotates. Lower the spring suspension and rotate the flange to centre the VT STM XA stage in the ring of magnets. Centre the other direction using the UHV-system height-adjustable feet.

6. Remove the feet and fix **all** bolts.

7. Mount VT STM XA viewports together with the CCD camera holder, see page 39.

Pumping

Attention
Never have the VT STM XA connected to the electronics in the corona pressure region, i.e. between 10^{+1} to 10^{-3} mbar as to avoid damage due to corona discharge.

1. Carefully pull out the wobblestick and secure it using the wobblestick parking aid, see figure 24 on page 45. Otherwise it may cause serious damage when being sucked in due to pressure reduction.

2. Do this also before venting the system (for the same reason).

3. Pump down your vacuum system as stated in the relevant system manual.

Bakeout

Attention

Bring the PPM to its upper position before bakeout or the suspension springs may extend and cause major problems to the eddy current damping system.

Never leave a sample plate in the STM during bakeout!

In order to **avoid charge build up** during bakeout due to the **pyroelectric effect** fit all electrical feedthroughs with their **short circuit plugs.**

Maximum bakeout temperature: 150°C.

True UHV conditions in the range 10^{-9} to 10^{-11} mbar can only be achieved when the system is baked for a sufficient amount of time.

1. Switch off all units, and remove all cables that are not necessary during bakeout.
2. Fit all FT12 and sub-D feedthroughs with their respective short circuit plugs. Note that
3. **Bring the PPM to its upper position.**
4. Remove the sample from the VT STM XA stage and park it in the carousel.
5. Remove all non-bakeable parts from the system **(including the LHe cryostat fast coupling port)**.
6. Shield all viewports and the continuous flow cryostat (particularly the helium input and output lines) with aluminium foil.
7. Enclose your vacuum system with a bakeout tent or other heat isolation housing. Be careful not to touch the wobblestick by this operation.
8. Before baking, check that all systems are under vacuum and that all pumps are running during bakeout.
9. For the Scienta Omicron VT STM XA chamber the approximate bakeout time will be 12 hours at a temperature of 150°C. If the VT STM XA is connected to an Scienta Omicron UHV-system see the UHV SPM Lab User’s Guide.
10. If you install the VT STM XA for the first time or if it was exposed to air for a longer period of time the bakeout time should be increased. Set your thermostat and timer accordingly.
11. A vacuum interlock system which automatically stops the bakeout procedure and shuts the gate valve in case of turbo or other failure is recommended.

12. After bakeout leave to cool down until the chamber has adapted to room temperature before disconnecting the short circuit plugs and re-installing removed parts.

13. Only re-connect and operate the VT STM XA after the system has completely cooled down. Remember that the VT STM XA inside needs longer (at least 24 hours) to cool than the UHV chamber.

14. Do not forget to reinstall all removed parts and cables after bakeout.

Attention

The sample stage must stay **locked** during cool-down. Before connecting the SPM control unit make sure that the temperature of the scanner has come down **well below 50°C**. Operating the scanner at temperatures above 50°C may lead to a depolarisation.

Notice

We recommend degassing the heater element of the temperature controller after bakeout (T < 170°C, I < 1 A).

Venting

The VT STM XA chamber has no venting valve of its own and must always be vented through the main UHV chamber.

⚠ Caution

The pressure within the system must never exceed 1.2 bar! Fit venting line with pressure relief valve if using pressurised gas containers for venting.

1. Close the gate valves to all chambers which are not be vented.

2. Switch off all control units and wait for a few minutes for any stored energy to discharge. Otherwise the piezo scanner may be severely damaged due to corona discharge.

3. Check that all parts of the VT STM XA have gained **room temperature**.

4. Connect a reservoir of dry nitrogen (< 4 ppm impurities) to the venting inlet tube.

5. Hold the wobblestick to avoid damage due to sudden movements.

6. Open the venting valve and wait until atmospheric pressure is reached.

7. Close all venting valves.

10. STM Mode

For first images in UHV we recommend loading at least one old tip and at least one gold coated silicon sample, see below. This can be used for STM experiments and to practise sample exchange and/or tip exchange under UHV conditions: mechanical handling is more difficult in UHV due to increased friction.

Notice
Be particularly careful when operating the wobblestick in UHV for the first time.

Samples and Tips

For successful AFM/STM experiments clean and UHV compatible samples and sample plates are essential.

- For a first test run evaporated gold is a suitable sample for scanning. Evaporated gold samples need no further treatment prior to mounting to the UHV system.
- If you want to achieve **atomic resolution** with STM **clean samples are important.** For this reason many samples need preparation and scanning under UHV conditions.
- If the sample **corrugation is too high** (microscopic steps) you will not achieve atomic resolution because even a good tip is only an atomic tip on a very fine scale.
- A good starting point for atomic resolution experiments with STM are HOPG samples (highly oriented pyrolytic graphite) imaged in air. Preparing an atomically flat surface with HOPG is easily done in air by cleaving with adhesive tape.
- Silicon samples need extensive treatment before they can be used for STM experiments. For Si(111)7x7 sample preparation procedures see page 86.
- Not all tips are capable of atomic resolution scans. You will have to **try several for a good image** at atomic resolution. For some samples (e.g. silicon) the tips need to be heated in UHV, e.g. on the manipulator (max. 200°C) or baked with the system, in order to remove the water film always present in air.

Notice
Check fitting of all sample plates in air before pumping down.

Notice
It is strongly advisable for monitoring purposes to have an **oscilloscope connected** to the VT STM XA during operations (a simple two channel scope is sufficient). Normally monitor the current signal (IT MON, time scale about 1 ms/div) and the Z- voltage (Z MON from the SCAR board). On the oscilloscope look for a Z-voltage in the range ± 140 V during approach and AC-coupled with about 200 mV/division whilst measuring corrugations below 1 nm. For connection instructions see also page 33.

Setting up for UHV Experiments

Before starting a VT STM XA experiment the following steps have to be performed:

- Check that the VT STM XA is installed correctly with new copper gaskets, see page 37/50.
- Pump the chamber down, see page 50.
- Connect the electronics, see chapter 4.
- Make sure all green-and-yellow grounding cables (earth) are connected to the grounding screws at the base flange.

Starting the Experiment

- Mount a tip and a sample, see chapter 7. If you try this procedure for the first time, we recommend that you use an old tip and an old sample.
- Lower the VT STM XA stage, see page 23.
- Switch on the MATRIX rack and computer and log in.
- Start the MATRIX software now.
- Load the STM experiment.
- In MATRIX check the parameter set, see MATRIX software documentation.

  Before using the VT STM XA scanner for the first time activate the relevant parameter set via the Experiment Options window, see MATRIX Application Manual, and save the configured hardware setup.

Coarse Positioning

Attention

The software must have already been started and the correct Experiment loaded before starting any adjustment or tip approach! **Otherwise a tip crash may be the result.**

Make sure the PPM is at its lower limit, i.e. the stage is unlocked.

Notice

Always watch the scanner/sensor when operating the coarse approach to avoid crashing the tip.

As a first step try operating the coarse motion drive (remote box) without or with an old sample and tip mounted. Try to observe the shadow which the tip casts on the sample / sample plate. For further details on the remote box see the appendix.

1. Upon switching on the MATRIX CU the remote box display will come on and display the Scienta Omicron logo together with the head that has been configured. Press DOWN to proceed to the BACK menu, i.e. scan piezo fully retracted and coarse positioning functions active.

2. Operate the ±X, ±Y, APPR (approach) and RETR (retract) coarse motion buttons (±F1 to ±F3) on the remote box.

3. Turn regulator "SPEED" to the maximum ("10"). As the coarse steps are so small (40 nm to 400 nm at room temperature) you have to look closely to see the motion. Different directions normally have different speeds (up to a factor of 3).

4. When you are close to the sample reduce the step width and approach very slowly.

Notice

If the step size ("SPEED") is too small, i.e. below "2", the coarse drive may not move. This has to do with overcoming the inertia of the coarse drive and is no reason for concern.

Attention

Do not leave the remote box tip positioning switch in FORWARD position if tunnelling cannot be achieved. Note that this condition in combination with extreme scan range/frame positioning parameters and long duration or scanner temperatures above 50°C **may lead to scanner depolarisation**.

Attention

For STM operation a special coarse approach procedure is necessary. Manual approach can cause a tip crash, so please **use the auto approach with STM tips!**

STM Coarse Approach

Now you can start bringing the tip into tunnelling distance with the sample. For details on operating the coarse motion drive please refer to the instructions on page 55f.

Attention

The apex of a good tip cannot be resolved in an optical microscope at "× 30" magnification, i.e. **is not visible with a CCD camera**. In other words: the tip is normally longer than it appears.

1. Bring tip close to the sample holder, looking through one of the viewports.
2. Use +Y/-Y buttons and adjust the tip position until the tip can be seen in front of the sample on the CCD camera screen.
3. Carefully adjust the CCD camera and light source such that the sample appears bright on the screen and the tip is dark. **Note**: this can be quite tricky!
4. Turn the CCD zoom to maximum and repeat steps A and B, see figure 27, until you see the tip and its mirror image on the sample:
5. A) Operate the push buttons on the remote box until the tip appears close to the sample edge. When you are close use the SPEED regulator on the remote box to reduce the speed to about "5".
6. B) If the mirror image of the tip cannot be seen on the sample: press the +Y / -Y buttons on the remote box to enlarge the virtual distance between the tip and the sample.

Attention

The tip reflection can only be seen on reflecting samples. For non-reflecting sample materials keep a safe distance and start the AUTO APPROACH early.

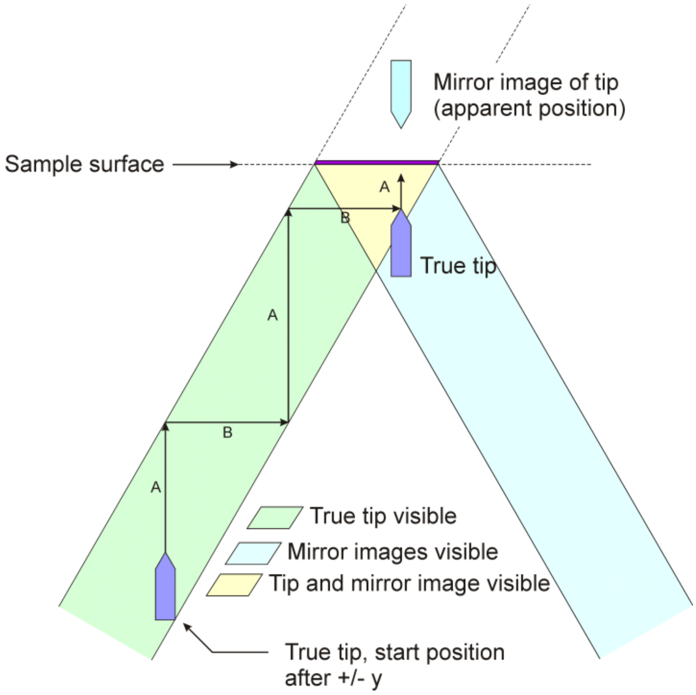


Figure 27. Tip-sample approach, schematic diagram.

7. Use the +X/-X and +Y/-Y buttons to select the scanning area, e.g. a macroscopically clean spot on the sample.

8. Bring tip and its mirror image as close as possible **without** mechanical contact (reduced speed!). Since you cannot see the end of a good tip there will be a gap between the visible end of the tip and its mirror image.

9. In MATRIX select a current range and bandwidth, see table 7 on page 59. Note the minimum currents required for AUTO APPROACH, see table 17 on page 103.

10. Adjust the gap voltage. Increase the loop gain to ≥1%.

11. On the remote box increase SPEED to "10" and press the AUTO button.

12. When the tip is close enough a tunnelling current will be detected and the AUTO APPROACH stops.

Adjusting Fine Piezo Play

After a coarse approach the surface is only just in the reach of the tip since the coarse step width (≈0.2 µm) is smaller than the Z-range of the scanner (>1 µm). The green tip shape of the Z-meter in the MATRIX is hence close to the yellow region.

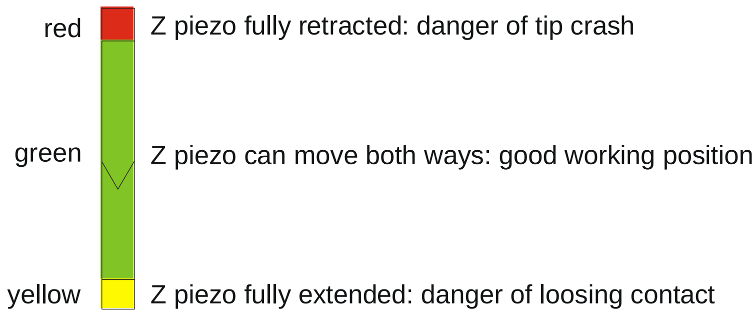


Figure 28. The software Z-meter display.

In order to have piezo play in both directions during scanning the green tip shape in the software Z-meter display should be in the centre between the red and yellow regions.

1. On the remote box set SPEED to maximum.
2. With the remote box in FORWARD mode watch the software Z-meter.
3. If the green tip shape is closer to yellow: switch to BACKWARD and press APPR once.
4. If the green tip shape is closer to red: switch to BACKWARD and press RETR once.
5. After each step switch to FORWARD and check the position of the green tip shape.
6. Stop this process when the green tip shape has reached a nearly central position between yellow and red, see figure 28.

Attention

The yellow warning areas of the Z-meter and of the frame position display may indicate **unsafe conditions**. Parameter settings in these warning areas shall always be limited to **durations far less than one hour**, particularly if the SPM is not actually scanning.

Notice

When scanning with temperature gradients (particularly towards higher temperatures) adjust the working position closer to the yellow region of the Z-meter.

Data Acquisition

A continuous scan can be started after the tip is in tunnelling distance of the sample. Before you try to get atomic resolution (e.g. on graphite) you should start with large frames (> 300 nm × 300 nm) on relatively flat samples such as evaporated gold (on silicon) or an optical grid.

- In the Scanner window choose the raster size (i.e. number of points and lines) to be measured.
- Select frame size, frame angle, frame position and raster time.
- In the Regulator window select a current setpoint, see table 7.

- Do not switch the range button with the tip in tunnelling condition (FORW on remote box) as this causes preamplifier relays to switch. During switching the feedback loop is undefined which may lead to a tip crash.
- Set a loop gain, see table 7 on page 59.
- Check the bandwidth (switch FSEL on the SPM PRE 4, see figure 50 on page 117). Attention, do not change the FSEL switch setting with the tip in forward direction.
- In the Gap Voltage window set a gap voltage, see table 7 on page 59.
- Start a measurement.
- Configure the online display to suit your needs.
- Fine-adjust the loop gain and possibly the current setpoint and gap voltage.
- To start saving measurement data check the "Store" boxes of the respective channels in the channel list window and the "Store" box in the scanner window.

Channel	Comment	Loop Gain
Z	topography (adjust Z-gain according to sample roughness, see Software Manual)	as high as possible without visible vibrations in the on-line display in line mode (0.1% to 1%)
I	tunnelling current	as low as possible

Table 6. Recommended acquisition channels and loop gain settings for STM measurements.

	gold	HOPG	Si (in UHV)
Channels	Z	I	Z
No of points/lines	400	100	400
V GAP	+0.5 V to +2 V	+0.1 V to +0.3 V	+2 V or -2 V
Range / bandwidth	330 nA / 80 kHz	330 nA / 80 kHz	330 nA / 80 kHz
I_T	1 nA	0.5 nA	0.2 nA
Loop gain	1%	0.5%	1%
Raster time	1.25 ms	1 ms	0.5 ms … 1.25 ms
(Scan speed	1000 nm/s	50 nm/s	100 nm/s …1000 nm/s)
scan range	500 nm	5 nm	20 nm … 500 nm

Table 7. STM settings, please adjust to optimise the image.

For further information on loop gain settings please refer to the Software Manual.

Notice

You can also do image processing during a measurement. This may slow down the on-line display but does not affect the data acquisition process.

If the obtained image does not match the expected surface structure try adjusting the tunnelling current by changing the feedback setpoint or polarity. Play with the parameters given in table 7. You may also want to try changing the scan area by adjusting the scanner's X and Y offset or even retracting and using the coarse motion drive.

The final solution may be changing the tip/tip material or the sample, or improving sample and tip preparation. Often additional methods along with UHV-STM operation are necessary for defining the surface condition.

STM imaging really needs patience. Sometimes, especially on relatively dirty samples, quality results are only obtained after a long period of scanning and searching for a clean area of the sample, i.e. adjusting the X and Y offset. Occasionally quality results are achieved at the first attempt. If this is not the case leaving the instrument scanning a clean surface area unattended for a while may lead to a cleaning effect on the tip.

Notice

For Delay time and Acquisition time values as well as the extra delay times T1 to T4 please refer to the table below.

Delay Time	Acquisition Time	T1	T2	T3	T4
60 µs	160…640 µs	200 µs	300 µs	300 µs	50 µs

Table 8. Spectroscopy times for the VT STM XA, I(V) channel. Note. These times are valid for 80 kHz bandwidth and typical voltage changes of about ±0.2 V. For smaller bandwidths or more substantial voltage changes these times may need to be increased.

dI/dV Spectroscopy with Lock-In Technique

When measuring dI/dV curves using a lock-in amplifier, crosstalk of the modulated gap voltage onto the tunnelling current may become a serious problem. The preamplifier box offers a trimmer potentiometer (CC = crosstalk compensation) that allows compensating this crosstalk before it is amplified by the IVC.

Notice

The procedure below assumes that the lock-in amplifier includes a function generator. If using an external function generator this has to be connected to V EXT instead of Sine OUT.

Attention

Always use a non-conducting (e.g. ceramic) trimming tool for adjusting the trimmer potentiometers.

Additional Wiring for dI/dV Spectroscopy

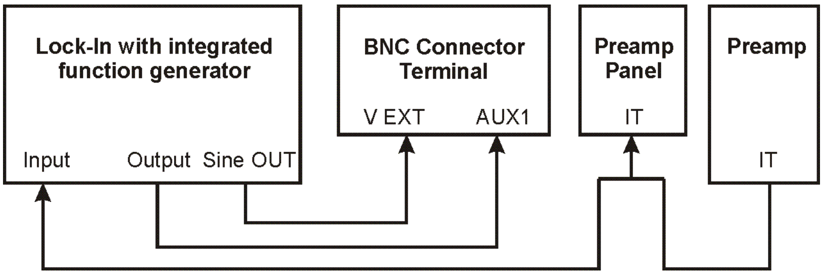


Figure 29. Wiring diagram for experiments with modulated gap voltage.

The amount of capacitive crosstalk, and with it the amount of required compensation, depends on the distance between the scanner and the sample, i.e. the tip length. Fine tuning is therefore necessary for every new tip or experimental setup.

Fine-Tuning Procedure

- Perform the AUTO APPROACH as usual.
- On the SPM Remote Box then switch to Backwards and retract the tip two coarse steps.
- Connect Sine OUT of the lock-in amplifier to V EXT on the MATRIX CU and tick the selection box next to V Ext in the External Inputs window (may be hidden under the Scanner window).
- On the lock-in amplifier select a modulation frequency (e.g. 1 kHz to 7 kHz in 333 nA range, maximum bandwidth, amplitude about 1-2 V). Make sure that the FSEL switch on the SPM PRE 4 is in the correct position.
- Connect IT of the preamplifier (red BNC plug) to the input of the lock-in amplifier and to IT of the SCAR cable connector using a BNC-Tee on the preamp panel.

Notice

Remove the additional cabling and the BNC Tee after finishing your spectroscopy measurement as it may otherwise pick up noise.

- Connect IT MON (BNC connector terminal) to the signal input of an oscilloscope (you may want to use a 10 kHz low pass filter).
- Connect Sine Out to the trigger input of the oscilloscope.
- Switch the lock-in amplifier on.
- The oscilloscope should now display the crosstalk. Adjust the lock-in setting such that you have no overmodulation/clipping. Set Phase such that the signal on the output of the lock-in is maximised.

- Adjust potentiometer CC until the displayed crosstalk is minimised (typically <5 mVpp at 5 kHz/2V excitation).
- Note that crosstalk and wanted signal are phase shifted by 90° (capacitive crosstalk). You therefore need to rotate the phase for measurements.

Setting up for dI/dV Spectroscopy

- Connect the lock-in output to one of the external input connectors (BNC) Aux1 / Aux2 on the MATRIX BNC connector terminal.
- Select a time constant τ on the lock-in amplifier (the value depends on the filter type, modulation frequency and function mode)
- On the lock-in amplifier select the input filter (switch mains filter on if available).
- On the SPM Remote Box move two coarse steps forward and switch to Forward.
- On the lock-in amplifier adjust the sensitivity.
- In Matrix select the delay and acquisition times (Spectroscopy window) to fulfil:

  Delay = Acquisition Time and both between τ and about 10 x τ

11. AFM Noncontact Mode QPlus With AFM-SPU

In AFM noncontact mode the feedback signal is derived from the force induced shift in resonance frequency of the vibrating QPlus sensor (tuning fork).

The QPlus sensor is a free, self-starting oscillator, the amplitude of which is setpoint regulated. The regulation works in a regenerative loop (positive feedback) in order to compensate energy loss, e.g. due to tip-sample interaction. If the reference oscillator is set to the correct frequency to begin with, the FM detector monitors the changes in QPlus sensor resonance frequency.

The interaction of the vibrating sensor with the sample results in a shift Δf of the tuning fork resonance frequency. Since the interaction force strongly depends on the tip-surface distance, this frequency shift can be used as feedback signal for a distance regulation (Z-loop). Depending on the sample the QPlus sensor frequency may decrease or increase due to van-der-Waals, magnetic or electro-static forces.

Notice

The **Δf sign** buttons **in the Z-Regulation window** allow selecting the correct polarity for attractive or repulsive interaction.

Δf sign "-" : attractive interaction: frequency decreases with decreasing gap width (normal case).

Δf sign "+" : repulsive interaction: frequency increases with decreasing gap width.

Attention

Tip cleaning with field emission carries the risk of causing damage (ESD) to the input stage.

Until further notice we strongly advise not to supply any external voltages to the tip-sample-gap exceeding 25 V – not even with Scienta Omicron supplied field emission adapters. This warning relates to all SPMs equipped with the QPlus option (i.e. LT STM QPlus, VT STM QPlus, MultiScan STM VT QPlus) and is true with simple STM tips as well as QPlus sensors mounted.

Notice

Since the STM mode is much easier to use, you may want to mount a QPlus sensor with a conducting tip and a conducting sample and scan it in STM mode initially in order to check for drift and vibration stability.

AFM experiments in noncontact mode might not work properly in air.

For optimum performance we recommend setting the I-Range to the 333 nA regime for QPlus measurements on conducting samples.

Notice

The AFM-SPU comprises a digitally phase-locked loop (PLL) that can be run in constant amplitude or constant excitation mode.

PLL supports extremely small amplitudes at a detection bandwidth between 4 KHz to 3 MHz. At input signal-to-noise ratios of 1:10 or worse atomic resolution is still possible.

The AFM-SPU supports a flexible demodulation bandwidth resulting in "adjustable" output signal frequency noise. For example, at a signal-to-noise ratio of 25 and a demodulation bandwidth of 400 Hz the output signal noise is 2 Hz_{pp}. When reducing the demodulation bandwidth down to 1 Hz, the achievable frequency noise at a S/N ratio of 25 will be even better (0.03 Hz_{pp}).

The AFM-SPU provides excellent frequency stability with respect to temperature changes over time and an average drift of just 10 µHz/K.

The QPlus Project is opened from the main window via the  icon, see MATRIX Application Manual. They provide a number of Experiments, each with a complete set of GUI controls for AFM noncontact mode measurements.

PLL Control Modes

The PLL control window offers three modes of operation: Self Excitation, Constant Amplitude and Constant Excitation.

- In **Self Excitation** mode the PLL is kept inactive and the frequency shift is detected via a mixed demodulator. This mode is mainly used for the frequency finder. The excitation is achieved by coupling the phase-shifted FN IN input to the excitation piezo. The vibration amplitude is kept constant by gain-controlling the excitation.

  In Self Excitation mode select the **f-Res. Sensor** range where you suspect the resonance frequency of the sensor. The manufacturer's documentation should give you a sufficiently accurate hint even for unfamiliar sensors. Scienta Omicron QPlus sensors have a resonance frequency in the range of 25 kHz.

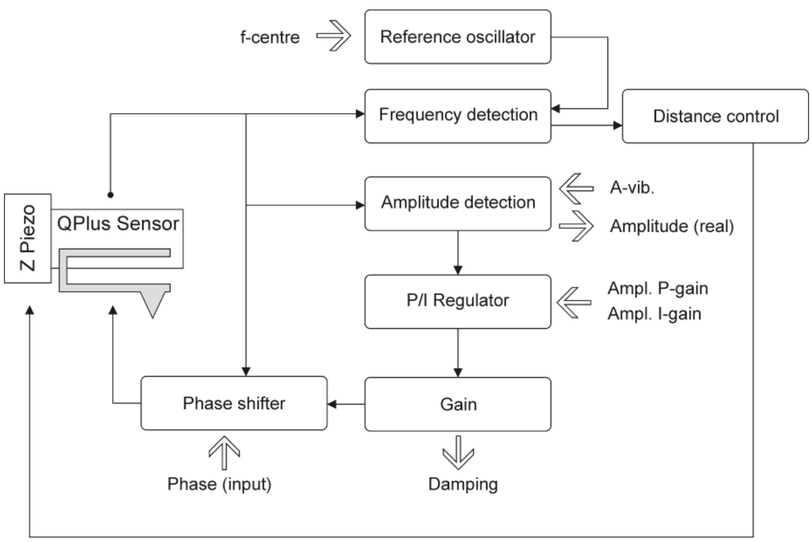


Figure 30. Self excitation mode, schematic diagram.

- In **Constant Amplitude** mode the PLL is active. The frequency shift is detected via the PLL. The vibration amplitude is kept at a user defined setpoint by the amplitude regulator. The excitation source is the internal reference oscillator.

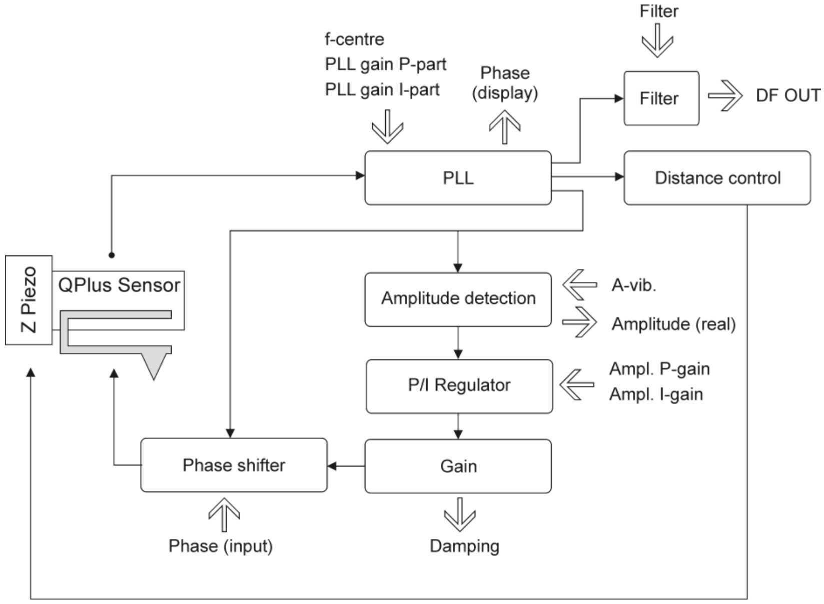


Figure 31. Constant amplitude mode, schematic diagram.

- In **Constant Excitation** mode the PLL is active. The frequency shift is detected via the PLL. The excitation signal amplitude is predefined by the user and fed to the gain control as a fix parameter. In this case no damping signal is available. The excitation source is the internal reference oscillator.

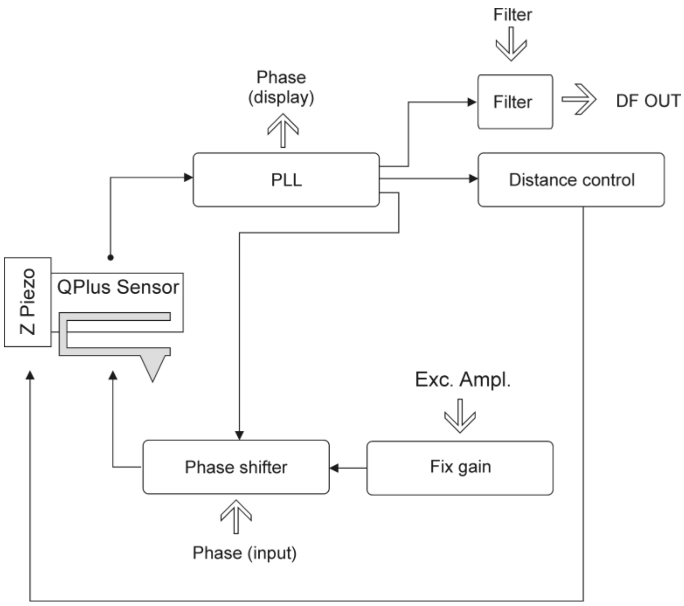


Figure 32. Constant excitation mode, schematic diagram.

Measurements in QPlus Non-Contact Mode

Frequency Finder

Use the frequency finder to determine the resonance frequency of the free sensor.

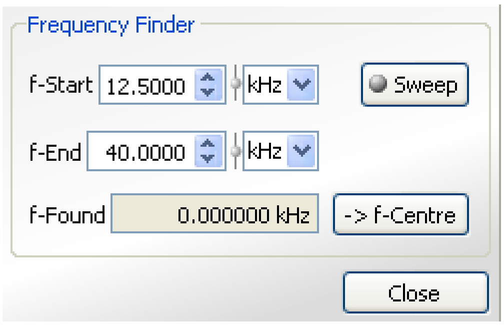


Figure 33. PLL Frequency Finder.

- In the Mode Select panel choose Self Excitation and pick a suitable f-Res. Sensor range.
- Select a vibration amplitude: 0.02 V is a good initial value for QPlus sensors.
- Set Amplitude P Gain and Amplitude I Gain. The I Gain should be smaller or equal the P Gain; 5% is a good initial value for both.
- In the Amplitude control panel click the Excitation button to switch the excitation on.
- Adjust Phase to get stable oscillation and minimize Damping (choose appropriate Exc.-Attenuation for Damping between 0.1 and 1 V).
- In the Frequency Finder panel select start and end values inside the selected range.
- Now click Sweep in the frequency finder panel. After a successful sweep use the "-> f-Centre" button to set the PLL centre frequency to the found value with a click.

PLL Adjustment

- Make sure the tip is in "backward" position
- Set f-Centre to the resonance frequency of the free sensor, see above.
- Set the PLL mode to Constant Amplitude and the Amplitude Mode to Frequency Selective.
- Select a vibration amplitude:

  0.02 V is a good initial value for QPlus sensors.
- Set Amplitude P Gain and Amplitude I Gain:

  5% is a good initial value for P.

  1% is a good initial value for I.

- In the Amplitude control panel click the Excitation button to switch the excitation on.
- Select Exc. Attenuation to have the smallest possible Damping (0.05 V to 0.1 V).
- Check that the true sensor amplitude is about the same as the amplitude setpoint A-Vib, see MATRIX Application Manual. This indicates that the amplitude control works properly.
- Adjust f-Centre until Damping is at its minimum value.
- Adjust Phase until the measured phase is as close to zero as possible.
- Choose the proper frequency detection bandwidth by setting the PLL Gain. We recommend setting P-Part = I-Part using the "wedding rings" icon. Note: Larger gain values result in a wider bandwidth and a faster maximum scan speed. Unfortunately, they also result in increased frequency noise. The optimum PLL bandwidth depends on surface corrugation, scan speed, loop gain of the z-regulator and many more.
- Set the tip protection conditions. This mode is activated by default but can be switched off, if required.
- Switch PLL on: the Locked indicator should now be green and Δf and phase should be about zero.
- In the Regulation window enter an appropriate Δf-Setpoint for Auto Approach, e.g.

  -0.5 Hz to -1 Hz for QPlus sensors.
- Set an appropriate loop gain for Auto Approach, e. g. 100% for QPlus sensors.
- Now start your Auto Approach.

Data Acquisition

- Select a slow scan speed (much slower than in STM mode), see table 9 on page 68.
- You may want to monitor the ΔFN OUT and Z MON signals on an oscilloscope.
- Start a measurement.
- Configure the on-line display to suit your needs.
- Fine-adjust the loop gain.

If the contrast is very bad in the on-line image try one of the following.

- Increase the feedback setpoint.
- Increase the loop gain for a Z-image.
- Decrease the loop gain for a Df-image.
- Decrease the amplitude setpoint.
- Change the gap voltage (compensation of contact potential and surface charges).

If the gap regulation is disturbed during scanning (on-line image or green tip shape in the Z-meter jumps during scanning) try one of the following.

- Reduce the scan speed; recommended value: ≈20 nm/s for QPlus sensors
- Reduce the feedback setpoint.
- Change the amplitude setpoint.
- Apply a gap voltage or change the gap voltage setting.
- Change the loop gain.

Notice

A spectroscopy curve Δf versus V GAP or Δf versus Z (channels Df(V) and Df(Z), respectively) can be used to find a suitable setting.

Channels	Z	Df
No of points/lines	400	
Δf Sign	negative	
Damping	0.05 V to 2 V	
Feedback Set	-0.5 Hz to -25 Hz	
Loop gain	40%	3%
Raster time	5 ms	2 ms
(Related scan speed)	(25 nm/s)	(50 nm/s)
Scan range	100 nm	

Table 9. AFM noncontact settings for HOPG samples, may be changed to optimise the image. Note that the optimum feedback set depends on the tip geometry. For sharp tips you should be closer to –0.5 Hz, for blunt tips closer to -25 Hz.

Notice

For monitoring purposes an oscilloscope is strongly recommended.

Channel	**Comment**	**Loop Gain**
Z	sample topography (adjust Z-input gain according to sample roughness)	as high as possible without visible oscillation (oscilloscope or on-line display in line mode)
Df	frequency shift	as low as possible
Damping	local damping	as low as possible

Table 10. Acquisition channels and loop gain settings for AFM noncontact measurements.

12. Scanning at Elevated Temperatures

At temperatures above room temperature scanning and surface imaging become more difficult, the problems increasing with increasing temperature. We therefore recommend that you try imaging at elevated temperatures only after you have become familiar with STM at room temperature.

Notice

The interpretation of surface images taken at elevated temperatures requires a sound knowledge of temperature induced surface effects.

Temperature changes generally result in thermal drift effects due to thermal expansion or contraction of the sample. This may lead to a change in gap width (vertical drift, ±Z) as well as to a shift of the scanning area (lateral drift, ±X/Y).

At elevated temperatures the influence of surface contamination increases due to the activation of chemical reactions with the sample material. Adsorbates, surface coatings and even the sample material may become mobile, possibly leading to difficulties in surface imaging.

Heating Facilities

Radiative sample heating is possible for all sample types and achieves peak temperatures of 500 K (650 K for heating-only version).

Attention

Attention:

- **Never exceed 8 W or 0.5 A (whichever occurs first)** when heating samples in the VT STM XA **cooling version**.
- **Never exceed 5 W or 0.5 A (whichever occurs first)** when heating samples in the VT STM XA **heating only version**.

Radiative Heating

For radiative heating a solid-state heating element is integrated into the sample stage. This facility opens a wide field of applications e.g. the investigation of diffusion, adsorption or desorption processes of atoms and molecules at metallic surfaces.

For optimum performance a low noise power supply (HC600) is supplied and operated in current control mode.

Radiative Heating Procedure

Heating Procedure for Small Temperature Changes ($\Delta T < 20$ K)

- Check the cabling, see page 32.
- On the HC600 set the current and voltage potentiometers to zero.

- Switch on the HC600.
- Start a continuous frame STM scan at room temperature or with the desired starting temperature in thermal equilibrium until you have a stable image.
- Keep on scanning and slowly increase the heating current to the desired value while constantly observing the software Z-meter.

Attention
When changing temperatures with the tip in tunnelling distance there is an increased danger of tip crash. Constantly observe the software Z-meter and be prepared to take the tip back with the coarse motion drive (+Z button on the remote box).

- Observe the sample drift. On stepped surfaces you will notice that the "step velocity" depends on the temperature gradient.
- Do not increase the temperature any further if Z variations become uncontrollable or the STM image resolution disappears.

Heating Procedure for Extensive Temperature Changes ($\Delta T > 20$ K)

Attention
For extensive temperature changes always take the tip a few coarse steps away from the surface!

1. Check the cabling, see page 32. Recommendation: perform the heating-up procedure without a sample mounted.
2. On the HC600 set the current and voltage potentiometers to zero.
3. Switch on the HC600 and set it to current control mode.
4. Set the HC600 to the desired heating power.
5. Regularly check the temperature and wait for about two hours for the thermal equilibrium to be reached.
6. Now insert the sample and perform the AUTO APPROACH procedure.

To keep high flexibility in sample exchange no direct temperature measurements are possible at the sample itself. For the radiative heating method the temperature depends only weakly on the employed sample material and geometry. Therefore a standard calibration can be used, see the appendix.

Notice
Temperature regulation can also be done using a temperature controller (e.g. LS 335) instead of the HC600. Always use the PT100 sensor on the sample reception for reference; for wiring instructions see page 30.

Data Acquisition

Normally the scanning procedure at elevated temperatures is the same as at room temperature. However, a number of facts have to be taken into account both, before and during the experiment to achieve quality results.

The employed tips must be mechanically and electronically stable at elevated temperatures. The tip material (e.g. tungsten) should have a sufficiently high melting point. Generally speaking slender, regularly V-shaped tips tend to be more stable at high temperatures than broad tips with small clusters or whiskers.

To achieve atomic resolution at high temperatures:

- Tips must be extremely clean and surface contamination removed (e.g. by annealing in UHV). The mean tip radius must be sufficiently small, i.e. < 10 nm.
- Tips which cannot be vacuum annealed must be thoroughly washed in distilled water (several changes of water). Make sure no water drop remains on the tunnelling end of the tip.
- Samples also must be very clean: surface contaminants may become very mobile at elevated temperatures.

Notice

Deposited surface coatings, particularly islands, may also become mobile on hot substrates!

Due to increased diffusion/drift effects we recommend scanning with relatively high scan speeds, particularly during temperature changes.

Apart from the drift problems mentioned above, the data acquisition method at elevated temperatures is identical to data acquisition at room temperature.

Notice

The resonance frequency of QPlus sensor shifts slightly with increasing temperature. It is therefore recommended to allow the cantilever to reach thermal equilibrium close to the surface before starting a measurement.

13. Scanning at Reduced Temperatures

Scanning at low temperatures has a number of advantages:

- Tunnelling stability increases because thermal effects like surface diffusion are mostly suppressed.
- Atoms or molecules which would not adsorb at room temperature can be studied at low temperatures.
- Mobile adatoms can be frozen to defined sites and may be atomically resolved at low temperatures.

Attention

Never cool the cryostat down with the VT SPM in air! The vacuum in the UHV chamber must be better than 10^{-9} mbar **before cooling down** to avoid residual gas particles adsorbing upon the cold sample surface and the copper braid. The surface sticking probability is close to 1 for temperatures of 50 K and below.

Notice

Before venting the VT STM XA make sure that the sample stage is at room temperature!

Experiments at very low temperatures require cooling with liquid helium. Temperature measurements are taken by means of a PT-100 sensor at the sample stage. The temperature difference between the sample stage and the sample is less than 10 K.

The Liquid Nitrogen Bath Cryostat (VT STM XA 100)

⚠ Caution

Please read the safety information on liquid gases (see manufacturer's documentation and page 15 of this manual) before handling LN_2 or LHe.

For applications which require temperatures not lower than 130 K, cooling can be carried out with liquid nitrogen. A bath cryostat is mounted on the base flange of the VT AFM XA. The cooling power is transferred to the sample via a copper cold finger connecting the liquid nitrogen to a highly flexible copper braid.

Freezing the liquid nitrogen solid (by pumping) further reduces the LN2 temperature by about 14 K (from 77 K to 63 K) reducing the sample stage temperature to below 100 K.

Higher temperatures may be achieved with counter heating using the sample stage heater. Under equilibrium conditions the temperature stability will be some 0.1 K.

Attention

When changing temperatures with the tip in tunnelling distance there is an increased danger of tip crash. Constantly observe the software Z-meter and be prepared to take the tip back with the coarse motion drive (+Z button on the remote box).

LN_2 Cooling Procedure

- Check all cabling and start the electronics, see page 32.
- Start a continuous frame STM scan at room temperature (large area, scan speed 1000 nm/s) until you have a stable image.
- Although you may keep on scanning during cool-down (while constantly observing the software Z-meter) we recommend that you take the tip a few coarse steps away to protect both, the tip and the surface.
- Fill the LN_2 bath cryostat with liquid nitrogen using the supplied KF-to-tubing adapter, see figure 34.

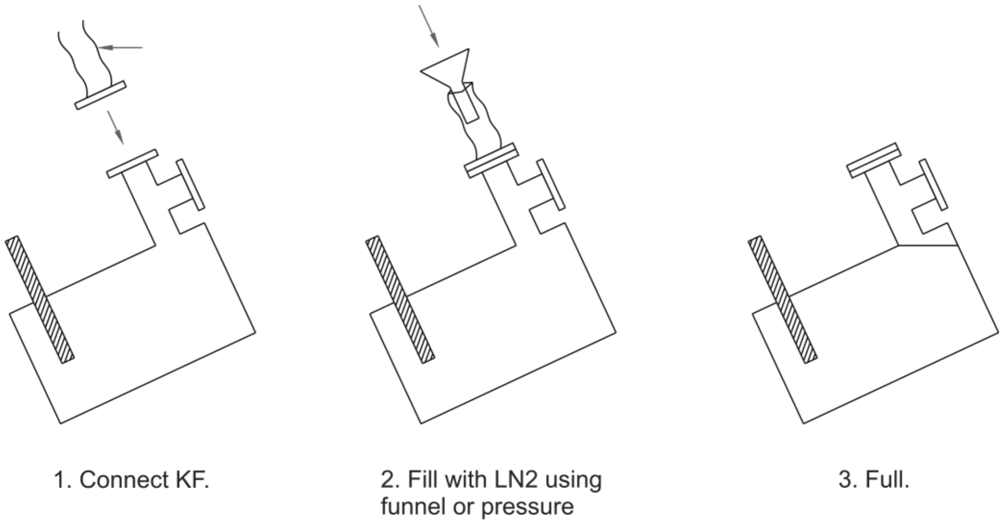


Figure 34. Filling the LN_2 bath cryostat, schematic diagram. Please make sure not to spill any liquid nitrogen onto skin or clothing through the airing flange.

- In order to avoid H_2O sublimation inside the cryostat connect the open KF 16 connector (LN_2 exhaust line) to the supplied 3 m of tubing using the KF 25/16 adapter, see figure 35.

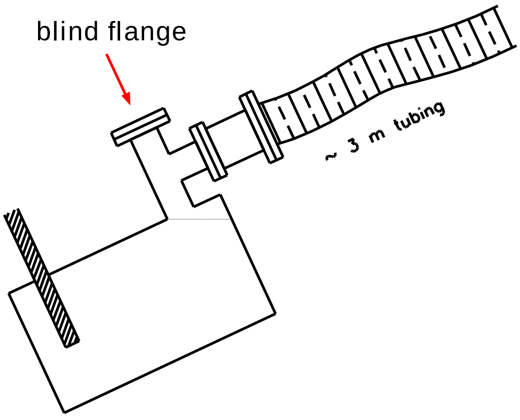


Figure 35. Connect the cryostat to the LN_2 exhaust line.

- Check the temperature at the sample stage using the temperature display VT TD (optional).
- Nearly constant temperature is reached after about 45 minutes but drift may continue for a while. Fairly stable conditions should be reached about 2 hours after filling the cryostat.
- The cryostat will hold the sample temperature for more than 6 hours without re-filling, depending on the thermal input.
- To achieve even lower temperatures the liquid nitrogen may be frozen solid by pumping the reservoir, see figure 36.

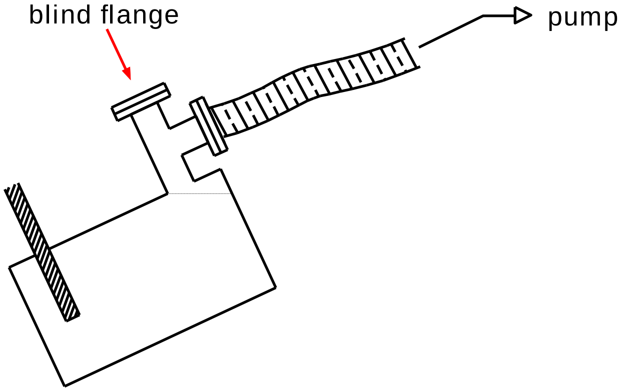


Figure 36. Freezing the LN_2 by pumping the reservoir.

Notice

Always use the LN_2 roughing pump with an oil mist filter. Check oil contents of the pump and oil trap regularly.

The LHe Flow Cryostat (VT STM XA 50)

Caution

Please read the safety information on liquid gases (see manufacturer's documentation and page 15 of this manual) before handling LN_2 or LHe.

Experiments at very low temperatures require cooling with liquid helium. For these applications a UHV compatible continuous flow cryostat is fitted to the stage. The heat exchanger of this cryostat ends at the level of the base plate. Thermal coupling between the cryostat and the sample is realised by a copper braid.

Compared to a bath cryostat a continuous flow cryostat leads to a reduced consumption of the cryogenic liquid and allows cooling the sample to intermediate temperatures. This can be done very accurately by varying the flow through the cryostat.

After reaching thermal balance a temperature stability at the heat exchanger of about 0.1 K is achieved in the 10 K to 270 K regime.

Notice

The flow cryostat can also be used with liquid nitrogen. In this case make sure that the LN_2 stays liquid at all times (p > 200 mbar abs).

The cryostat comprises a counter heating element which can for example be use to speed up the warming up process. The heater element is fitted on the cryostat top together with a silicon diode as a temperature sensor. Using a temperature controller, e.g. VT TC, any temperature below room temperature can be chosen by flow regulation and counter heating the heat exchanger.

Attention

The maximum temperature on the cryostat head is limited to 190°C by the durability of the Si-diode.

Notice

We recommend degassing the heater element of the cryostat after bakeout (T < 170°C, I < 1 A).

It makes sense to stabilise the temperature directly at the sample reception using the Pt100 sensor for temperature measurements and the PBN heater of the sample reception for counter heating. With a temperature controller (e.g. VT TC) the sample temperature can be set with an accuracy of <0.1 K (relative).

The temperature controller VT TC has a microprocessor based PID controller adjusting the output power of the heater with respect to the setpoint and the measured temperature. Recommended start values for the coefficients of the PID control loop are P=50, I=100 and D=100. These values should be fine-adjusted manually. The temperature at both, the cryostat and the sample stage, are displayed simultaneously on the digital display of the controller. For further information please refer to the temperature controller manual.

For further information on the cryostat please refer to the cryostat manual. Read the cryostat and transfer tube instructions carefully before mounting or using the parts.

Notice

Always use the LHe roughing pump with an oil trap. Check oil contents of the pump and oil trap regularly.

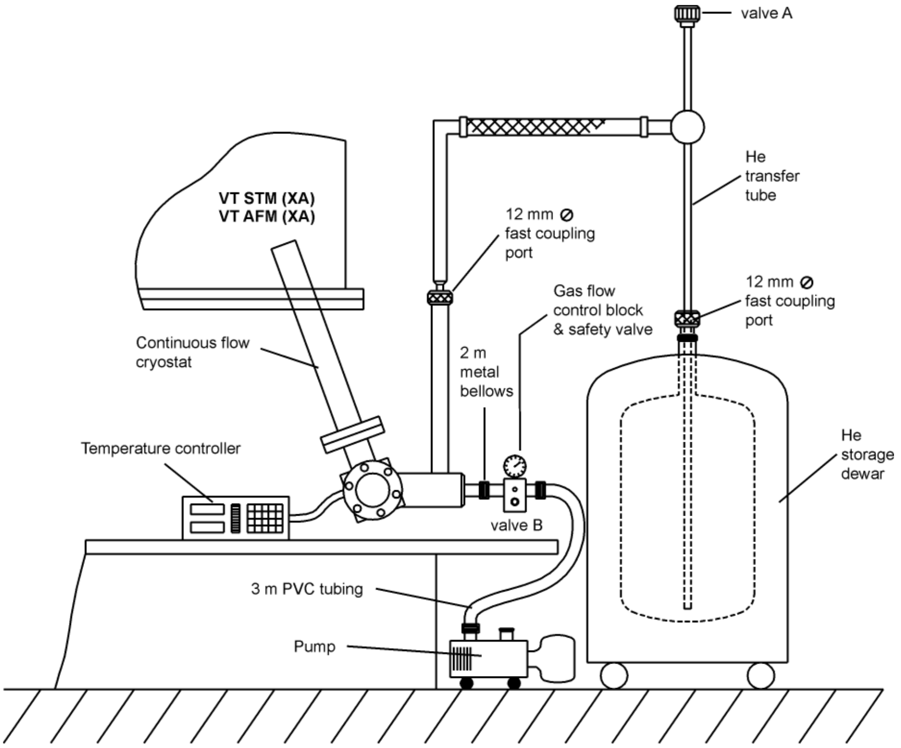


Figure 37. Schematic setup diagram for LHe cooling.

LHe Cooling Procedure

For simplicity we recommend to use the transfer tube without the cold-gas-supplement, initially. For further information you may also want to consult the cryostat manual.

Preparation

- Check the cabling and connections, see page 32 and figure 37.
- Connect the flow cryostat to the liquid helium dewar using the transfer tube, see cryostat manual.
- Start a continuous frame STM scan at room temperature (large area, scan speed 1000 nm/s) until you have a stable image.
- Although you may keep on scanning during cool-down (while constantly observing the software Z-meter) we recommend that you take the tip a few coarse steps away to protect both, the tip and the surface.
- Flush the helium system several times, see below, to prevent icing inside the heat exchanger.

Notice

Temperature controller VT TC:

- Channel A = PT 100 sensor at sample reception;
- Channel B = Si diode at cryostat.

Attention

When changing temperatures with the tip in tunnelling distance there is an increased danger of tip crash. Constantly observe the software Z-meter and be prepared to take the tip back with the coarse motion drive (+Z button on the remote box).

Attention

The gas flow control block should stay close to room temperature at all times in order to allow safe working conditions for the overpressure and adjustment valves. Make sure the safety valves are not blocked by ice (you may want to use a hot air blower).

Flushing

1. Close all valves, i.e. valve A on the transfer tube and valve B on the gas flow control block, see figure 37.
2. Switch on the pump.
3. Open valve B.
4. Watch the pressure display at the gas flow control block. When the pressure comes down below -0.8 bar close valve B.
5. Check that the pressure stays about constant for one minute (leak test of He line).
6. Open valve A until the pressure reaches 0 bar.
7. Close valve A.
8. Open valve B until the pressure at the control block reaches minimum and close again.
9. Repeat steps 6 to 8 at least three times.
10. Now start the cool-down procedures outlined below.

Cool-Down

- Set valve B to 6.0 on the scale (about 6 turns) for a sample temperature of 50 K (for other sample temperatures adjust accordingly).
- Partly open valve A.

Hints and Tips

- Take care to avoid pressure changes in the sub-Hz region (visible from the manometer). If necessary, further reduce the He flow at valve A (this might be quite tricky). Recommended pressure: -0.2 bar.

- Temperature stability is reached after about 45 minutes but drift may continue for a while. Fairly stable conditions should be reached about 60 minutes after starting the cool-down.

Temperature Stabilisation

For temperature stabilisation by counter heating we recommend using the Pt100 at the sample reception (VT TC, channel A) as feedback signal because of the close thermal coupling between temperature sensor, heating element and sample.

For further information on the counter heating procedure please refer to the temperature controller manual.

Data Acquisition at Low Temperatures

Good UHV conditions ($p \leq 10^{-10}$ mbar) are particularly important to achieve atomic resolution at low temperatures as the relative sticking probability for all gaseous substances rises towards 1 with sinking temperature.

- In case of desorption from the STM tip or cantilever keep the tip in tunnelling distance and change its lateral position frequently until the desorption process stops (i.e. the tip becomes clean). This process may, however, take some hours.

At low temperatures the sample resistance of semiconductors increases. This may cause problems for STM on weakly doped semiconductors as the resistance may become too high to allow stable tunnelling. We generally recommend scanning weakly doped semiconductor samples with relatively high bias voltages (either polarity).

At low temperatures diving board cantilevers or QPlus sensors show a slight variation in resonance frequency. For contact mode cantilevers no changes were observed.

Apart from the problems mentioned above, the data acquisition method at reduced temperatures is identical to data acquisition at room temperature.

Using the LHe Cold Gas Supplement

The helium transfer tube comes with a cold gas supplement to reduce consumption and bubbling, particularly when used with LN_2. The cold gas supplement may simply be screwed to the end of the transfer tube before insertion into the He dewar. The cooling and data acquisition procedures are unchanged.

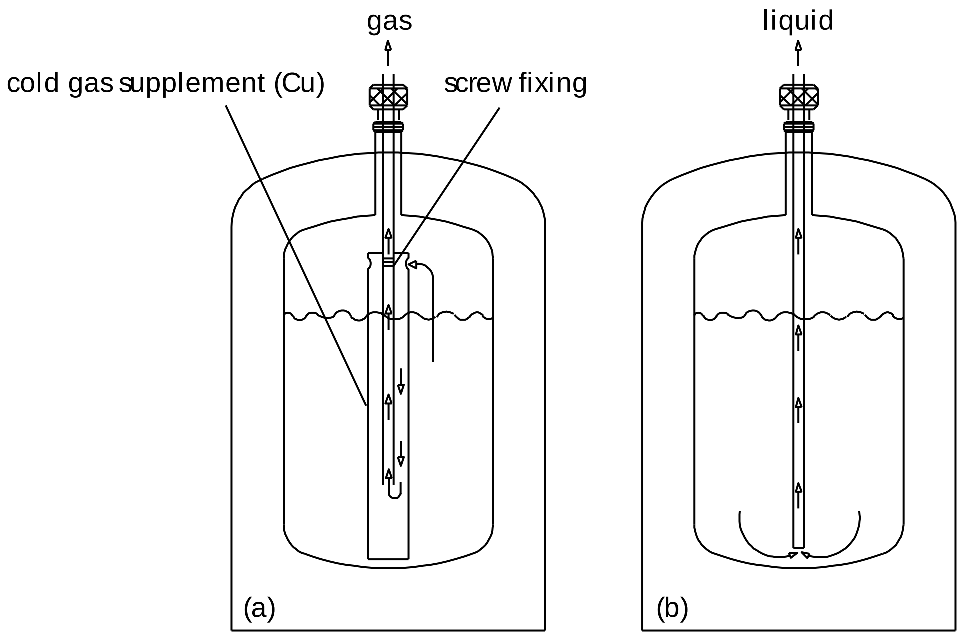


Figure 38. The transfer tube (a) with and (b) without the cold gas supplement.

> **Caution**
>
> When pumping liquid helium or cold gas make sure to avoid negative pressure in the helium dewar, see also page 94.
>
> Always store the LHe transfer tube with valve A open in order to prevent damage caused by thermal expansion.

Warming up

When you are finished with your low-temperature experiment it is important to follow the indicated warm-up procedure in order to avoid pressure build-up in the cryostat or its connection tubes. For reference of parts refer to figure 37 on page 76.

> **Caution**
>
> - Cold He vapour can cause severe burns to skin and eyes. Always wear splash goggles or a face shield when inserting or removing a transfer tube.
> - Cold surfaces require handling with cryogenic gloves! If in doubt please refer to the safety instructions on page 13.

- Switch off the pump.
- Open valve B on the gas flow control block.

- Increase the pressure at the gas flow control block to about 0 bar.
- Now close valve A on the He transfer tube.

In case of shutting down for a longer period of time

- Carefully lift the transfer tube out of the cryostat.
- Close the cryostat opening using the provided plug in order to prevent ice formation on the capillaries.
- Release any overpressure from the LHe dewar.
- Carefully lift the He transfer tube out of the LHe dewar, see also dewar manual.
- Open valve A on the He transfer tube and keep it open during warm-up to avoid damage to the needle valve due to thermal expansion.

14. Hints and Tips

Adjustments for New Preamplifiers

DC-Offset Compensation

Warning

Lethal Voltages!!

Adjustments and fault finding measurements as well as **installation procedures and repair work** may only be carried out by authorised personnel qualified to handle lethal voltages.

No preamplifier tuning with the HC1100 connected!! Always connect the VT PPP plug before tuning the preamplifier.

Attention

The preamplifier has been fine-tuned with respect to its dedicated SPM head. This offset compensation procedure only needs to be repeated after exchanging the in-vacuum IVC or the SPM PRE 4 preamplifier.

All compensation steps have to be performed in the presented consecutive sequence!

Attention

Always use the supplied or a similar non-conducting (e.g. ceramic) trimming tool for adjusting the trimmer potentiometers.

Preparations

- Make sure the UHV system is pumped down and wired up according to the manual (all electronics connected).
- Make sure the electronics has been running continuously for at least 1 hour (including the new preamp).
- Connect a non-grounded multimeter (measuring accuracy 0.01 mV or better) to IT (red BNC on the preamp panel of the MATRIX rack) via BNC-Tee.

Attention

Do not use a grounded oscilloscope because ground loops may produce an additional offset of a few millivolts.

- In the MATRIX software set V GAP to 0 V
- Make sure that the recessed switch UOS on the SPM PRE 4 is in the upper position (default position) , see figure 50 on page 117.

- Make sure the VT PPP plug is connected.
- Please use an insulated tuning probe for the adjustments.

Total Zero Offset Adjustment

- In the regulator window switch to the 333 nA range.
- Make sure the FSEL switch on the SPM PRE 4 is also in the upper position (max. bandwidth).
- Adjust trimmer OS2 until the multimeter reads <±0.02 mV DC.

Compensating IVC Residual Input Current

- In the regulator window switch to the 3.3 nA range.
- On the preamp box set switch FSEL to the lower position
- Adjust trimmer BCC until the multimeter reads <±0.02 mV DC.

Other Filters

The compensation procedure detailed above compensates the offsets by using the passive low pass filters in the SPM PRE 4. If using the active low pass filters (3 kHz and 1 kHz) their output offsets of about 0.5 mV have to be added to remaining offset of 0.02 mV.

While it is possible to compensate this offset (3 kHz region: trimmer OS2; 1 kHz region: trimmer BCC) this procedure automatically leads to a compensation maladjustment in the passive low pass filters.

Crosstalk Compensation - Coarse Adjustment

Attention
This crosstalk coarse adjustment procedure only needs to be performed after exchanging the in-vacuum IVC or the SPM PRE 4 preamplifier. All compensation steps have to be performed in the presented consecutive sequence!

Attention
Always use the supplied or a similar non-conducting (e.g. ceramic) trimming tool for adjusting the trimmer potentiometers.

The procedure is the same as for fine tuning but can be done in air and with an old tip and an old sample. Instead of a careful AUTO APPROACH the tip can be approached manually to speed up the procedure.

- Make sure the lock-in amplifier is connected according to figure 29 on page 61.
- Follow the procedure detailed for fine adjustment on page 61.

Notice

In case you have no lock-in amplifier available use a sine generator for the modulation (V EXT) and an oscilloscope for detection (IT MON).

Notice

This procedure provides a coarse crosstalk compensation adjustment for your instrument. For best results, a fine-adjustment of the crosstalk compensation should be performed for every tip-surface pair individually as described on page 61.

HC 600 Operation Modes

The HC 600 can be operated in constant current mode (CC mode) and in constant voltage mode (CV mode). Normally the constant voltage mode is applied for loads where the resistivity increases with increasing temperature (light bulb with metal filament). The constant current mode is applied for loads where the resistivity decreases with increasing temperature (graphite layer in PBN heaters). The idea is to make sure that the power input decreases with increasing temperature if the system is left alone.

Ripple and noise generation are different for the two operation modes:

CC mode: ripple & noise < 1 mA rms, i.e. < 2.8 mA pp.
At 65 Ohms (VT XA version) this means ripple & noise < 184 mV pp
At 8 Ohms (VT RH sample plate) this means ripple & noise < 22 mV pp

CV mode: ripple & noise < 0.5 mV rms, i.e. < 1.4 mV pp

Notice

If you encounter a 50/60 Hz interference on your VT images in CC mode you may want to try switching to CV mode for less noise input.

In-Situ Evaporation Using the EFM 3

The Scienta Omicron UHV Evaporator EFM 3 is the ideal tool for in-situ deposition of thin films while at the same time recording SPM images at varying temperatures.

Setup

Please read the EFM3/4 Instruction Manual for installation assistance and adjustment procedures.

Attention

Make sure to mount the VT STM chamber on the VT STM before attempting to install the EFM evaporator.

Notice

It is desirable to aim for the smallest possible evaporation spot in order to avoid coating parts of the VT STM sample stage.

- Make sure the smallest aperture is mounted on the EFM 3 in order to minimise the evaporation spot.
- Connect the EFM 3 with 46 mm port aligner and 50 mm straight connector to the CF 35 flange as indicated in figure 39 on page 85.
- Make sure the shutter moves correctly before tightening the screws.
- Now mount the VT STM XA chamber with EFM 3 in place on the main UHV system. Adjust the rotating flange in order to allow free movement of the spring suspension, see on page 23.
- On the EFM remove the HV feedthrough (CF 16) and inspect the position of the port aligner by looking through.
- Adjust the port aligner such that you can see the sample with the VT STM XA suspended, i.e. hanging freely in the damping stage.
- Mark the setting of the port aligner so that it can be reproduced and refit the HV feedthrough.

Attention

Take special care to correctly adjust the EFM and port aligner in order to avoid coating parts of the VT STM sample stage (particularly electrical contacts)!

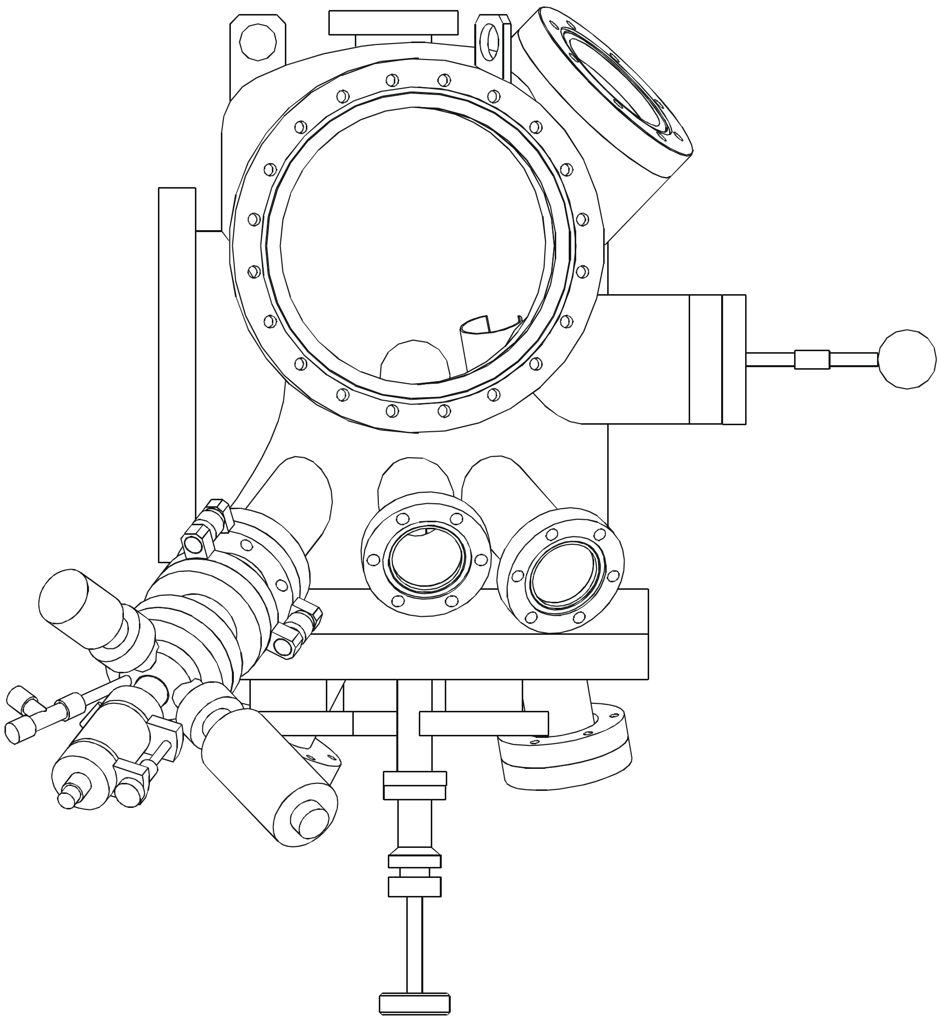


Figure 39. VT STM XA chamber with EFM 3 mounted.

Specifications	
Distance EFM-nozzle to sample	91 mm
Spot diameter with 4 mm aperture	≈ 9 mm
Angle between EFM and sample	31 degrees

Table 11. EFM on VT STM XA, specific lengths and angles.

Notice

In vacuum the target area can be determined observing the luminous spot of the EFM filament, see EFM3/4 instruction manual.

Tip. Use longer STM tips in order to minimise the risk of vapour-depositing the tip holder and allow free access to the sample. Note however, that longer tips are mechanically less stable. The maximum allowed tip length is stated in the appendix, see page 104.

Preparing Si(111)7x7

Attention

Do not use tools made from Ni-alloys when handling silicon samples! **Please note, that stainless steel contains sufficient amounts of nickel to contaminate your sample**. We recommend using molybdenum or ceramic tools.

Notice

Silicon sample temperature measurement will be required at several stages throughout the procedure. This may for example be achieved using a disappearing filament optical pyrometer.

A standard test sample for UHV STM operation is the Si(111)7x7 reconstruction. This structure is relatively easy to image because of its large unit cell and high corrugation. However, sample preparation is very critical for Si samples since the (7×7)-reconstruction is very sensitive to even small amounts of contaminants, e.g. nickel.

- The crystal plane orientation of the wafer should be better than 0.5°, the thickness 0.3 to 0.5 mm. Wafers must be clean and free of any visible contamination (optical microscope).
- Make sure that you use nickel-free instruments to cut your Si-wafer and also for mounting the Si-sample.
- Use Mo sample plates which have been annealed in UHV prior to sample mounting.
- For samples preferably use SiO_2 passivated standard wafers. These should not be treated chemically unless you have extremely clean chemical facilities.
- Blow away (pure, dry nitrogen) any small particles on the sample surface before inserting it into the vacuum system: carbon contained in these particles might otherwise contaminate your sample.

In most studies of silicon surface one or a combination of several surface preparation methods is used. These can broadly be categorised as follows.

1. Chemical etching with subsequent low-temperature annealing (900°C) in UHV.
2. Sputter etching and annealing to a variety of temperatures up to 1250°C in UHV.
3. Annealing-only treatments in UHV on samples that have not previously been chemically or sputter etched.

We recommend an annealing-only procedure according to [10] using a direct current heating facility in UHV. This procedure is best for Si(111) and Si(001) surfaces.

Degassing

After pumping down and baking the vacuum system, degassing of all filaments (ion gauge, TSP, etc.) should be carried out. Special care has to be taken with the manipulator(s) and the sample heater.

Degassing the Integral Heater Element of the Manipulator(s) (Scienta Omicron UHV Systems, only)

Attention

Never use a stainless steel sample plate at such high temperatures in order to **avoid nickel contamination** in the UHV system.

- Degas the integral heater element of the manipulator(s) at a power of about 60 W for 1-2 hours, preferably with an OMICRON tantalum plate in place.

- Shortly after applying the heating power the pressure will rise and then slowly decrease again. Apply the heating power until the pressure stabilises in the mid 10^{-9} mbar regime.

Degassing New STM Sample Plates

New sample plates or those stored for a long time must be degassed prior to sample annealing.

- Insert your empty sample plate (or a sample plate with a dummy silicon sample fitted) into the manipulator and apply 60 W of heating power to the resistive heating.

- Shortly after applying the heating power the pressure will rise and then slowly decrease again. Apply the heating power until the pressure stabilises in the mid 10^{-9} mbar regime. (The dummy silicon sample will adsorb contamination.)
  Leave to cool down and remove the sample plate from the vacuum system through the fast entry lock and make sure that all subsequent handling is carried out under absolute clean conditions using clean tools, clean storage boxes, no skin contact, no grease, no oil, no stainless steel etc. being in contact with the sample plate.

Initial Preparation of a Si Sample for STM Investigations

We do not recommend any wet etching or sputter cleaning procedures for Si. The following is only sensible for wafers with native oxide. The crystal plane orientation of the wafer should be better than 0.5°, the thickness 0.3 to 0.5 mm. For direct heating applications a doping level of the order of 10^{16} cm^{-3} to 10^{18} cm^{-3} has been found to be most applicable and the wafers must be clean and free of any visible contamination (optical microscope).

Never handle silicon with steel or stainless steel tools, use only molybdenum or ceramic tools.

Use only the originally supplied molybdenum wrench for tightening the molybdenum nuts.

1. Cut the wafer to size: lay the wafer down on its face onto a dust free tissue (lens tissue). Scratch the back of the wafer with a diamond tip guided by a ruler, which is held down to the backside of the wafer being separated by a dust free tissue. Break the wafer along the scratches by bending.

2. Any dust should be blown off the Si pieces with clean pressurised nitrogen. In case there is any indication of remaining contamination apply ultrasonic clean in acetone

p.a. and a final rinse in methanol VLSI. It is important to remove the sample on-end out of the solvent in order not to carry any liquid on the face.

3. Mount the sample into the sample holder which should have been degassed prior to mounting in accordance with the procedure given above. Mount the sample as described in the manual.

4. Introduce the sample plate to the system through the fast entry lock, or put it directly on the carrousel or the sample manipulator.

5. Without prior *ex situ* treatment outgas the sample and sample plate on the system manipulator by resistive heating just below the glowing temperature for several hours until the pressure reaches the mid 10^{-10} mbar region.

6. Outgas the sample over night by direct current heating at a temperature, low enough not to remove the native oxide (i.e. very dark red glowing sample, < 600°C). The pressure should stay in the low 10^{-10} mbar region.

   With very long annealing times and/or high doping levels a segregation of dopants may occur at the surface. These can destroy surface reconstructions (as can Ni and other surface contaminants). In case of surface segregation limit the degassing procedure to the necessary minimum.

7. Flash the sample up to 1250°C by direct current heating watching the pressure not to exceed the low 10^{-9} mbar range. If the pressure goes up turn the heating power down, wait for the pressure to recover and try again. Alternatively turn the heating current down and apply a sequence of short heating cycles until you can stay at 1250°C for about 3 s.

8. Decrease sample temperature quickly to 950°C and then more slowly cool down to room temperature at a rate of about 2°C per second.

It is **important to go through the 950°C to 1250°C range quickly** the first time the oxide is removed. Otherwise the sample may take on a hazy appearance which can be ascribed to µ-scale roughness. Once a sample has been cleaned in this way it can be cleaned repeatedly. Overnight the sample will adsorb contamination which coalesces into 3D islands if heated to 950°C. These can in turn be removed by flash-annealing the sample to 1250°C.

Notice
Si(001) is more sensitive to the correct procedure than is Si(111).

After successful sample preparation the surface condition is normally stable for several hours.

A very helpful method for testing surface preparation is LEED. The silicon sample has to show a really good LEED pattern without too much background (similar to those found e.g. in the Scienta Omicron SPECTALEED brochure) in order to give a good chance of seeing atomic resolution with a scanning probe microscope.

Preparing STM Tips

STM tips are normally made from cut-to-size pieces of high purity metal wire. There are many different ways to make tips for atomic resolution, the very first STM tips simply were mechanically ground. A standard method for STM tip fabrication is electro-chemical etching. This method has the advantage of being straight forward and is most easily accomplished using the Scienta Omicron Tip Etching Kit.

Different tip materials and diameters require different etching methods while the tip shape can be influenced by varying the etching parameters. Although dc-etching results in sharper tips, very long and slender tips also tend to be less stable than short tips with a large cone angle. The metal wire for tip fabrication may be vacuum-annealed prior to etching, but polycrystalline tungsten wire as received from the manufacturer also yields atomic resolution tips. Tungsten tips may be etched in KOH or NaOH. This is most easily done using the Scienta Omicron Tip Etching Kit. Table 12 gives a number of etching recipes.

Notice

Cut wire pieces non-perpendicular to the wire axis prior to etching. **Do not cut the wire after etching!**

After etching rinse the tips in distilled water (semiconductor grade) and check for irregularities using an optical microscope. Additionally in-vacuum tip treatments, e.g. field emission, may be used if required.

Platinum-iridium wire also yields very good tips. Cut Pt/Ir wires at a small angle with a pair of scissors and use without etching. STM tips may also be bought from a variety of vendors. For further reading see the literature listing from page 122.

The prepared STM tips are inserted into the tip carrier tube. Use a pair of pliers to fix the tip in the tube.

Safety Warnings

⚠ **Caution**

Safety Goggles

Tungsten wire is very hard and has a high spring constant, i.e. cut-off pieces may fly rather far. Always wear protection goggles when cutting wire and clamping tips to the holder tube (you may cut the tip accidentally when pinching too hard).

Wear safety goggles and protective clothing while handling etching liquids.

⚠ **Caution**

Etched tunnelling tips are extremely sharp! Take care of your fingers...

AC Tip Etching Procedure

- Use annealed W-wire (e.g. ∅ 0.38 mm, remove clamped ends).
- Break/cut off pieces for tunnel tips: add about 1.5 mm to the desired final tip length. The maximum allowed tip length is stated in the appendix, see page 104.
- Put 0.6 N KOH-solution into a bowl beneath the etching device. The solution must be renewed daily.
- Fix a piece of tungsten into the clip of the etching device.
- Move down the piece of tungsten into the centre of the ring electrode until the current reaches 150 mA (check with ammeter).
- Leave the tip in the solution until the current ceases to flow.

- Remove the tip and dip it into high purity distilled water for a short time.
- Check the shape of the tip with a microscope: for optimum quality: the tip should be a symmetric V-shape without deposits and the apex should not be resolved in an optical microscope at "× 500" magnification.
- Clean the tip by running high purity distilled water vertically from back to point in order to remove any residual KOH deposits. Use about 30 ml per tip.
- Remove remaining water by touching the side of the tip with a lint-free tissue (lens tissue).

U	15 V AC
I_{ini}	150 mA
I_{fin}	0 mA
Solution	0.6 N KOH (3.36 g KOH, 100 ml H_2O distilled.)
Ring electrode	W, diameter app. 100 mm
alternatively	
U	3 V…8 V DC, connect "+" to tip
I_{ini}	30-200 mA
I_{fin}	app. 15 mA depending on insertion depth
Solution	5 N NaOH (8.5 g NaOH, 50ml H_2O distilled)
Ring electrode	stainless steel, diameter app. 45 mm, thickness 0.5 mm

Table 12. Tip etching parameters for polycrystalline tungsten tips.

DC Tip Etching

Remarks concerning DC-etching of tunnel tips

- Length of the wire pieces is desired final tip length plus insertion depth (about 2 mm). The maximum allowed tip length is stated in the appendix, see page 104.
- The cathode must touch the surface of the solution.
- Vibration of the etching device should be avoided during etching.
- At the end of the etching process the end of the tungsten piece will drop. The power supply must be cut in this instant to ensure a sharp point.
- Thoroughly clean the tips with distilled water before use, otherwise KOH/NaOH crystallites may remain on the point.

Fixing the Tip to the Holder

There are different ways to fix a tip to the tip holder such that you can re-use the holder after the actual tip has gone blunt. Whatever method you use, make sure that the tip has perfect electrical contact and sits absolutely tight.

- Put the tip into the tube of the tip holder and squeeze the tube with a pair of pliers or a dented side cutter.

- Spot weld the tip to the outside of the tube.

AFM QPlus Mode

In AFM noncontact mode the feedback signal is derived from the force induced shift in resonance frequency of the vibrating QPlus sensor.

$$\frac{\Delta f}{f} = \sqrt{1 + \frac{1}{C} \cdot \frac{\partial F}{\partial z}} - 1 \approx \frac{1}{2C} \cdot \frac{\partial F}{\partial z}.$$

As the frequency resolution depends on the Q-value of the resonator this method is particularly interesting in vacuum (baking not required). The minimum detectable force gradient is

$$F'_{\min} = \left.\frac{\partial F}{\partial z}\right|_{\min} = \sqrt{\frac{4 \cdot C \cdot kT \cdot B}{2\pi \cdot f_0 \cdot Q \cdot A^2}},$$

where	C	=	cantilever spring constant (≈1800 N/m)
	kT	=	thermal energy
	B	=	PLL demodulation bandwidth (≈2 kHz)
	f_0	=	QPlus sensor resonance frequency (20 - 30 kHz)
	Q	=	QPlus sensor Q-value (500 - 4000 at RT)
	A	=	QPlus sensor vibration amplitude (0.3 - 2 nm).

The pressure which can be obtained by pumping with a turbo pump is sufficient to achieve high enough Q-values for a good frequency resolution. Baking is not normally necessary.

15. Trouble Shooting

Caution

Warning: Lethal Voltages!!

Adjustments and fault finding measurements as well as SPM experiments in environments other than UHV may only be carried out by authorised personnel qualified to handle lethal voltages.

Leg Support for 100 K Variants

Attention

When setting up a VT STM XA with bath cryostat in air, one support leg must be fixed to the base flange using the provided extension piece in order to ensure tilt resistance. Otherwise the weight of the bath cryostat can pull the entire setup over.

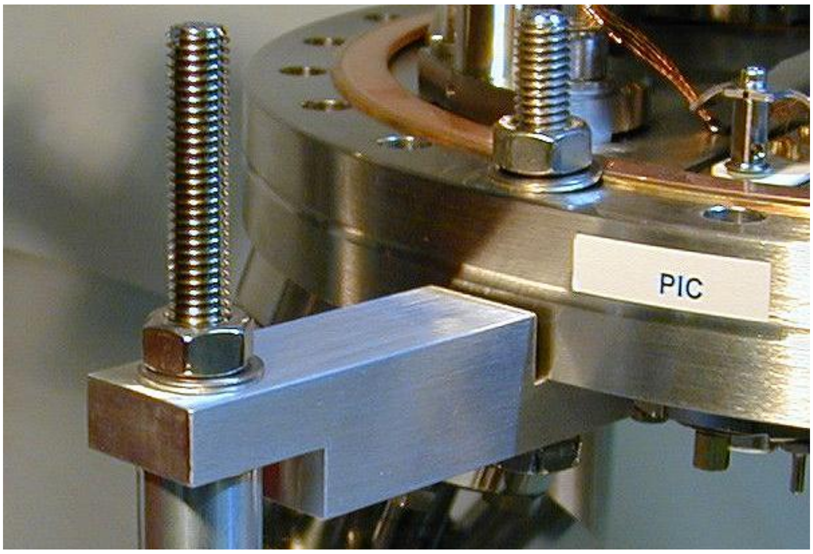


Figure 40. Extension piece for support leg.

Loop Oscillations

The speed of the feedback loop can be altered by changing the loop gain. If the value set is too high then the loop begins to oscillate. This can be seen in the IT MON signal (STM mode) or in the Delta FN OUT signal (AFM mode). The frequency is typically a resonance frequency of the scanner assembly (typically a few kHz). Be sure to set the regulation speed below the onset of oscillation.

If the value is set too low Z-image structures may become blurred out, take the form of asymmetric ramps or the tip may crash into the surface when scanning fast.

50/60 Hz Noise

The noise level depends on the condition of the tunnelling gap, i.e. on the quality of tip and sample. With an open feedback loop (i.e. tip fully retracted or fully driven forward) the electronics will exhibit distinct ripple and noise resulting from the overload condition at the Z outputs of the Regulator or of the Piezo Driver. This is normal and no need for concern. While in STM feedback, Z-noise should not be above mV-level with a clean sample and good tip.

Notice
Connect all mains cables to the same system rack socket to avoid ground loops (including oscilloscope, if used). You may also want to employ a mains filter.

If you detect a 50/60 Hz noise signal in your images or on the oscilloscope (IT MON, Z) this may have a number of reasons.

- The remote box switch is set to BACKWARD. In this case switch to FORWARD.
- No STM tunnelling contact with FORWARD position. Note that this condition in combination with extreme scan range/frame positioning parameters and long duration or temperatures above 50°C may lead to scanner depolarisation.
- Earth wires are not or not properly connected.
- The "grounding bridge" on the back of the MATRIX CU is not open.
- The UHV system is not grounded properly. In this case connect the system to protective earth (PE) of the employed mains supply line or of the mains rack..
- Interference from other devices connected to the UHV system, e.g. vacuum pumps. In this case try to find out which device causes the interference by switching them off one by one. Replace or remove the device in question or try grounding it and its power supply with a thick cable to earth.
- Interference from strong sources of radio noise in adjacent rooms. This may be airborne or transmitted via the mains connection.
- If you encounter a 50/60 Hz interference on your images while heating in constant current mode you may want to try switching to constant voltage mode for less noise input.

Mechanical Instabilities

Instabilities in the tunnelling current may also result from mechanical instabilities.

- Check if the damping stage is adjusted correctly.
- Check if the sample plate is clamped correctly.
- Check if the sample and tip have been correctly fixed.

Pressure Instabilities / Negative Pressure

In case of pressure instabilities / negative pressure in the LHe dewar due to helium consumption you may impose a constant dewar pressure using an additional He gas bottle.

- Connect a He gas bottle and reducing regulator to the spare KF flange on the LHe dewar outlet.
- Open the He gas bottle carefully until you read a pressure of just above atmospheric pressure on the gas bottle manometer.

Coarse Motion Failure

If the coarse motion does not work

- Check if the coarse drive is on its mechanical limit.
- Check the cabling.
- Check if the electronics is on.
- Check if the remote box is set to BACKWARD.
- Set SPEED potentiometer to "10" and check again, possibly with different frequencies. If the step size is too small, e.g. below "5", the coarse drive may not move.

In case of a coarse motor failure this problem normally requires repair at Scienta Omicron. However, there are cases which can be fixed on-site with standard Scienta Omicron spare parts. Please contact the Scienta Omicron service department (see on page 123) to decide the correct maintenance procedure.

Piezo Testing

In order to check the operativeness of the piezo tube(s) measure the capacities between the Z-electrode and any other electrode of the scanner piezo and for the coarse motion piezo between the electrode and its respective ground connection. For reference values please refer to page 104.

> **Notice**
>
> Always measure capacitance values at the end of the PIC cable to avoid damaging any fragile wiring with the probes. The cable capacity is about 0.4 nF.

- **Check the capacities if you have a capacitance meter available.**
- **Check the plug for short circuits with an ohm-meter.**

The capacitance values are only valid with the standard scanner version. In any other case please ask your service representative or refer to supplementary sheets delivered with your instrument.

Scanner Depolarisation

If a scanner depolarisation is suspected,

- take the tip a few coarse steps away from the surface (RETR),
- check that the correct hardware setup file has been loaded.
- On the remote box switch from tip BACKWARD to tip FORWARD while observing the effect on the monitor of the CCD camera. With a normally functioning scanner the forward movement of the tip should be just about visible on the screen and have no significant components in X- or Y directions.

In case of a scanner depolarisation please contact the Scienta Omicron service department (see page 123) to discuss the problem.

Overextended Suspension Springs

When the VT STM XA is baked with the PPM down, i.e. the base plate is not fixed, the suspension springs become overextended. If you cannot bring the base plate to a properly suspended position, see page 24, the springs need to be exchanged. In this case please contact the Scienta Omicron service department (see page 123) to discuss the problem.

Copper Braid Interferes with Base Plate Alignment

In cryo versions the copper braid may push the base plate away from its laterally centred position. This problem may disappear after bakeout. If it does not you may **gently** bend the copper braid. Be very careful in doing so as it is be rather brittle.

The copper braid should not touch any other parts, particularly those that stay at room temperature.

Adjusting the Sample Clamping Springs

- Remove the sample and tip and park them in the carousel.
- Switch off all electronics equipment and remove the cabling.
- Vent the chamber.
- Unbolt the VT STM XA chamber from the UHV system.
- Unbolt the VT STM XA chamber from the VT STM XA base flange and lift off carefully.
- Using a clean pair of angular tweezers push the springs upwards vertically.
- Insert a single sample plate and check that it is clamped correctly at both sides.

- Re-mount the chamber to the VT STM XA and bolt it to UHV system, see mounting procedure from page 37.

Instabilities After Vapour Deposition (QPlus sensor Version)

- Check the tip holder for accidental deposits. This may lead to an electrical short cut.
- Correct the EFM adjustment, see on page 84.
- Mount a new tip.

16. Appendix

Chamber Layout

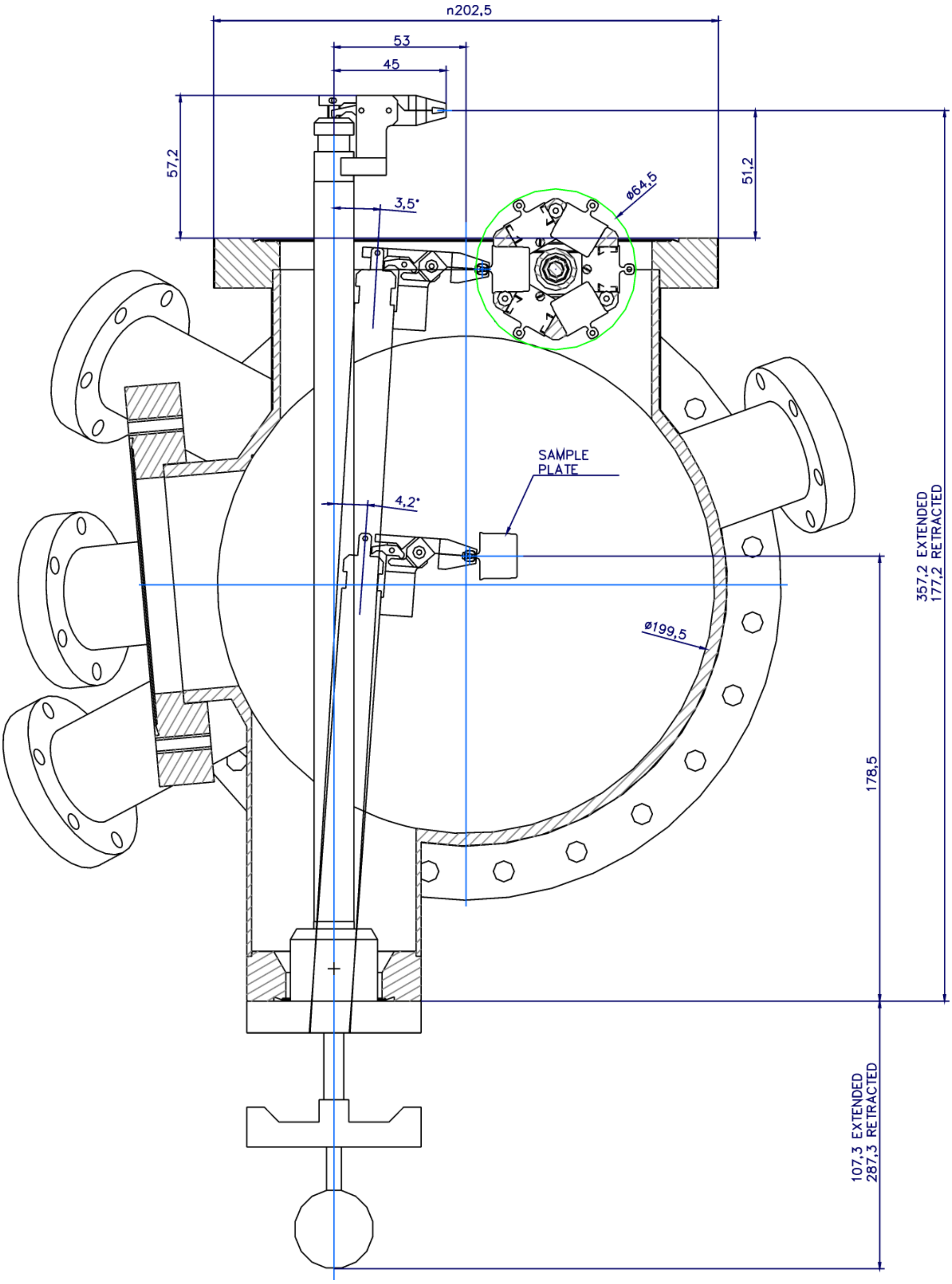


Figure 41. Bolt-on chamber for wobblestick WS 180, standard configuration, schematic diagram.

Si-Diode Technical Data

Sensor Type	Silicon Diode DT-670C-SD from Lake Shore
Sensor Temperature Coefficient	Negative
Sensor Units	Volts (V)
Maximum Reverse Voltage	60 V
Sensor Excitation	10 µA ±0.1% constant current
Sensor Temp. Range	1.4 - 500 K
Tolerance Band C	±1 K at 2 K to 305 K ±0.50% of temp at 305 K to 500 K
Sensor Curve	see page 2
Typical Sensor Sensitivity	-12.5 mV/K at 1.4 K -31.6 mV/K at 4.2 K -26.8 mV/K at 10 K -1.73 mV/K at 77 K -2.30 mV/K at 300 K

Table 13. Electronic information on the Si diode for various temperature ranges.

T (K)	Voltage (V)	dV/dT (mV/K)	T (K)	Voltage (V)	dV/dT (mV/K)	T (K)	Voltage (V)	dV/dT (mV/K)	T (K)	Voltage (V)	dV/dT (mV/K)
1.4	1.644290	-12.5	6.0	1.51541	-36.7	28.0	1.110421	-2.25	160.0	0.868518	-2.07
1.5	1.642990	-13.6	6.5	1.49698	-36.9	29.0	1.108261	-2.08	170.0	0.847659	-2.10
1.6	1.641570	-14.8	7.0	1.47868	-36.2	30.0	1.106244	-1.96	180.0	0.826560	-2.12
1.7	1.640030	-16.0	7.5	1.46086	-35.0	31.0	1.104324	-1.88	190.0	0.805242	-2.14
1.8	1.638370	-17.1	8.0	1.44374	-33.4	32.0	1.102476	-1.82	200.0	0.783720	-2.16
1.9	1.636600	-18.3	8.5	1.42747	-31.7	33.0	1.100681	-1.77	210.0	0.762007	-2.18
2.0	1.634720	-19.3	9.0	1.41207	-29.9	34.0	1.098930	-1.73	220.0	0.740115	-2.20
2.1	1.632740	-20.3	9.5	1.39751	-28.3	35.0	1.097216	-1.70	230.0	0.718054	-2.21
2.2	1.630670	-21.1	10.0	1.38373	-26.8	36.0	1.095534	-1.69	240.0	0.695834	-2.23
2.3	1.628520	-21.9	10.5	1.37065	-25.5	37.0	1.093878	-1.64	250.0	0.673462	-2.24
2.4	1.626290	-22.6	11.0	1.35820	-24.3	38.0	1.092244	-1.62	260.0	0.650949	-2.26
2.5	1.624000	-23.2	11.5	1.34632	-23.2	39.0	1.090627	-1.61	270.0	0.628302	-2.27
2.6	1.621660	-23.6	12.0	1.33499	-22.1	40.0	1.089024	-1.60	273.0	0.621141	-2.28
2.7	1.619280	-24.0	12.5	1.32416	-21.2	42.0	1.085842	-1.59	280.0	0.605528	-2.28
2.8	1.616870	-24.2	13.0	1.31381	-20.3	44.0	1.082669	-1.59	290.0	0.582637	-2.29
2.9	1.614450	-24.4	13.5	1.30390	-19.4	46.0	1.079492	-1.59	300.0	0.559639	-2.30
3.0	1.612000	-24.7	14.0	1.29439	-18.6	48.0	1.076303	-1.60	310.0	0.536542	-2.31
3.1	1.609510	-25.1	14.5	1.28526	-17.9	50.0	1.073099	-1.61	320.0	0.513361	-2.32
3.2	1.606970	-25.6	15.0	1.27645	-17.3	52.0	1.069881	-1.61	330.0	0.490106	-2.33
3.3	1.604380	-26.2	15.5	1.26794	-16.8	54.0	1.066650	-1.62	340.0	0.466760	-2.34
3.4	1.601730	-26.8	16.0	1.25967	-16.3	56.0	1.063403	-1.63	350.0	0.443371	-2.34
3.5	1.599020	-27.4	16.5	1.25161	-15.9	58.0	1.060141	-1.64	360.0	0.419960	-2.34
3.6	1.596260	-27.9	17.0	1.24372	-15.6	60.0	1.056862	-1.64	370.0	0.396503	-2.35
3.7	1.59344	-28.4	17.5	1.23596	-15.4	65.0	1.048584	-1.67	380.0	0.373002	-2.35
3.8	1.59057	-29.0	18.0	1.22830	-15.3	70.0	1.040183	-1.69	390.0	0.349453	-2.36
3.9	1.58764	-29.6	18.5	1.22070	-15.2	75.0	1.031651	-1.72	400.0	0.325839	-2.36
4.0	1.58465	-30.2	19.0	1.21311	-15.2	77.35	1.027594	-1.73	410.0	0.302161	-2.37
4.2	1.57848	-31.6	19.5	1.20548	-15.3	80.0	1.022984	-1.75	420.0	0.278416	-2.38
4.4	1.57202	-32.9	20.0	1.197748	-15.6	85.0	1.014181	-1.77	430.0	0.254592	-2.39
4.6	1.56533	-34.0	21.0	1.181548	-17.0	90.0	1.005244	-1.80	440.0	0.230697	-2.39
4.8	1.55845	-34.7	22.0	1.162797	-21.1	100.0	0.986974	-1.85	450.0	0.206758	-2.39
5.0	1.55145	-35.2	23.0	1.140817	-20.8	110.0	0.968209	-1.90	460.0	0.182832	-2.39
5.2	1.54436	-35.6	24.0	1.125923	-9.42	120.0	0.949000	-1.94	470.0	0159010	-2.37
5.4	1.53721	-35.9	25.0	1.119448	-4.60	130.0	0.929390	-1.98	480.0	0.135480	-2.33
5.6	1.53000	-36.2	26.0	1.115658	-3.19	140.0	0.909416	-2.01	490.0	0.112553	-2.25
5.8	1.52273	-36.5	27.0	1.112810	-2.58	150.0	0.889114	-2.05	500.0	0.90681	-2.12

Table 14. DT-670 Series Expanded Temperature Response Data Table. Measurement current 10 µA ± 0.1%.

Pt-100 Resistor Temperature Curve

T in °C	-0	-1	-2	-3	-4	-5	-6	-7	-8	-9
-250°	2.51									
-240°	4.26	4.03	3.81	3.60	3.40	3.21	3.04	2.88	2.74	2.61
-230°	6.99	6.68	6.38	6.08	5.80	5.52	5.25	4.99	4.74	4.49
-220°	10.49	10.11	9.74	9.37	9.01	8.65	8.30	7.96	7.63	7.31
-210°	14.45	14.05	13.65	13.25	12.85	12.45	12.05	11.66	11.27	10.88
-200°	18.49	18.07	17.65	17.24	16.84	16.44	16.04	16.64	15.24	14.84
-190°	22.83	22.40	21.97	21.54	21.11	20.68	20.25	19.82	19.38	18.95
-180°	27.10	26.67	26.24	25.82	25.39	24.97	24.54	24.11	23.68	23.25
-170°	31.34	30.91	30.49	30.07	29.64	29.22	28.80	28.37	27.95	27.52
-160°	35.54	35.12	34.70	34.28	33.86	33.44	33.02	32.60	32.18	31.76
-150°	39.72	39.31	38.89	38.47	38.05	37.64	37.22	36.80	36.38	35.96
-140°	43.88	43.46	43.05	42.63	42.22	41.80	41.39	40.97	40.56	40.14
-130°	48.00	47.59	47.18	46.77	46.36	45.94	45.53	45.12	44.70	44.29
-120°	52.11	51.70	51.29	50.88	50.47	50.06	49.65	49.24	48.83	48.42
-110°	56.19	55.79	55.38	54.97	54.56	54.15	53.75	53.34	52.93	52.52
-100°	60.26	59.85	59.44	59.04	58.63	58.23	57.82	57.41	57.01	56.60
-90°	64.30	63.90	63.49	63.09	62.68	62.28	61.88	61.47	61.07	60.66
-80°	68.33	67.92	67.52	67.12	66.72	66.31	65.91	65.51	65.11	64.70
-70°	72.33	71.93	71.53	71.13	70.73	70.33	69.93	69.53	69.13	68.73
-60°	76.33	75.93	75.53	75.13	74.73	74.33	73.93	73.53	73.13	72.73
-50°	80.31	79.91	79.51	79.11	78.72	78.32	77.92	77.52	77.12	76.73
-40°	84.27	83.87	83.48	83.08	82.69	82.29	81.89	81.50	81.10	80.70
-30°	88.22	87.83	87.43	87.04	86.64	86.25	85.85	85.46	85.06	84.67
-20°	92.16	91.77	91.37	90.98	90.59	90.19	89.80	89.40	89.01	88.62
-10°	96.09	95.69	95.30	94.91	94.52	94.12	93.73	93.34	92.95	92.55
-0°	100.00	99.61	99.22	98.83	98.44	98.04	97.65	97.26	96.87	96.48
0°	100.00	100.39	100.78	101.17	101.56	101.95	102.34	102.73	103.12	103.51
10°	103.90	104.29	104.68	105.07	105.46	105.85	106.24	106.63	107.02	107.40
20°	107.79	108.18	108.57	108.96	109.35	109.73	110.12	110.51	110.90	111.29
30°	111.67	112.06	112.45	112.83	113.22	113.61	114.00	114.38	114.77	115.15
40°	115.54	115.93	116.31	116.70	117.08	117.47	117.86	118.24	118.63	119.01
50°	119.40	119.78	120.17	120.55	120.94	121.32	121.71	122.09	122.47	122.86
60°	123.24	123.63	124.01	124.39	124.78	125.16	125.54	125.93	126.31	126.69
70°	127.08	127.46	127.84	128.22	128.61	128.99	129.37	129.75	130.13	130.52
80°	130.90	131.28	131.66	132.04	132.42	132.80	133.18	133.57	133.95	134.33
90°	134.71	135.09	135.47	135.85	136.23	136.61	136.99	137.37	137.75	138.13
100°	138.51	138.88	139.26	139.64	140.02	140.40	140.78	141.16	141.54	141.91
110°	142.29	142.67	143.05	143.43	143.80	144.18	144.56	144.94	145.31	145.69
120°	146.07	146.44	146.82	147.20	147.57	147.95	148.33	148.70	149.08	149.46
130°	149.83	150.21	150.58	150.96	151.33	151.71	152.08	152.46	152.83	153.21
140°	153.58	153.96	154.33	154.71	155.08	155.46	155.83	156.20	156.58	156.95
150°	157.33	157.70	158.07	158.45	158.82	159.19	159.56	159.94	160.31	160.68
160°	161.05	161.43	161.80	162.17	162.54	162.91	163.29	163.66	164.03	164.40
170°	164.77	165.14	165.51	165.89	166.26	166.63	167.00	167.37	167.74	168.11
180°	168.48	168.85	169.22	169.59	169.96	170.33	170.70	171.07	171.43	171.80
190°	172.17	172.54	172.91	173.28	173.65	174.02	174.38	174.75	175.12	175.49
200°	175.86	176.22	176.59	176.96	177.33	177.69	178.06	178.43	178.79	179.16

Table 15. Continued.

T in °C	-0	-1	-2	-3	-4	-5	-6	-7	-8	-9
210°	179.53	179.89	180.26	180.63	180.99	181.36	181.72	182.09	182.46	182.82
220°	183.19	183.55	183.92	184.28	184.65	185.01	185.38	185.74	186.11	186.47
230°	186.84	187.20	187.56	187.93	188.29	188.66	189.02	189.38	189.75	190.11
240°	190.47	190.84	191.20	191.56	191.92	192.29	192.65	193.01	193.37	193.74
250°	194.10	194.46	194.82	195.18	195.55	195.91	196.27	196.63	196.99	197.35
260°	197.71	198.07	198.43	198.79	199.15	199.51	199.87	200.23	200.59	200.95
270°	201.31	201.67	202.03	202.39	202.75	203.11	203.47	203.83	204.19	204.55
280°	204.90	205.26	205.62	205.98	206.34	206.70	207.05	207.41	207.77	208.13
290°	208.48	208.84	209.20	209.56	209.91	210.27	210.63	210.98	211.34	211.70
300°	212.05	212.41	212.76	213.12	213.48	213.83	214.19	214.54	214.90	215.25
310°	215.61	215.96	216.32	216.67	217.03	217.38	217.74	218.09	218.44	218.80
320°	219.15	219.51	219.86	220.21	220.57	220.92	221.27	221.63	221.98	222.33
330°	222.68	223.04	223.39	223.74	224.09	224.45	224.80	225.15	225.50	225.85
340°	226.21	226.56	226.91	227.26	227.61	227.96	228.31	228.66	229.02	229.37
350°	229.72	230.07	230.42	230.77	231.12	231.47	231.82	232.17	232.52	232.87
360°	233.21	233.56	233.91	234.26	234.61	234.96	235.31	235.66	236.00	236.35
370°	236.70	237.05	237.40	237.74	238.09	238.44	238.79	239.13	239.48	239.83
380°	240.18	240.52	240.87	241.22	241.56	241.91	242.26	242.60	242.95	243.29
390°	243.64	243.99	244.33	244.68	245.02	245.37	245.71	246.06	246.40	246.75
400°	247.09	247.44	247.78	248.13	248.47	248.81	249.16	249.50	249.85	250.19

Table 15. Pt-100 resistor data table, resistances in Ohms.

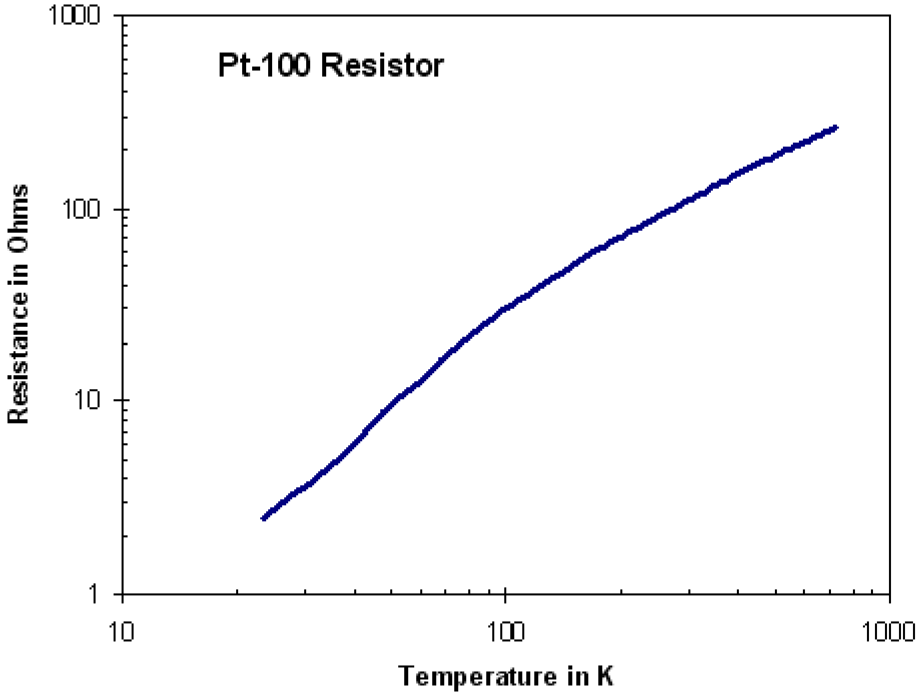


Figure 42. Pt-100 resistance values. Diagram according to Table 15.

Units	Temperature [K]
2.04000	16.200
2.32000	19.900
2.71000	24.300
3.48000	29.900
4.70000	35.300
6.17000	40.900
10.0000	52.500
12.1800	58.000
15.0150	65.000
19.2230	75.000

Table 16. Modification data for the PT-100 standard.340 curve of the LS 335 temperature controller for Scienta Omicron PT-100 sensors. The required program curvehandler.exe can be downloaded from the Lake Shore homepage.

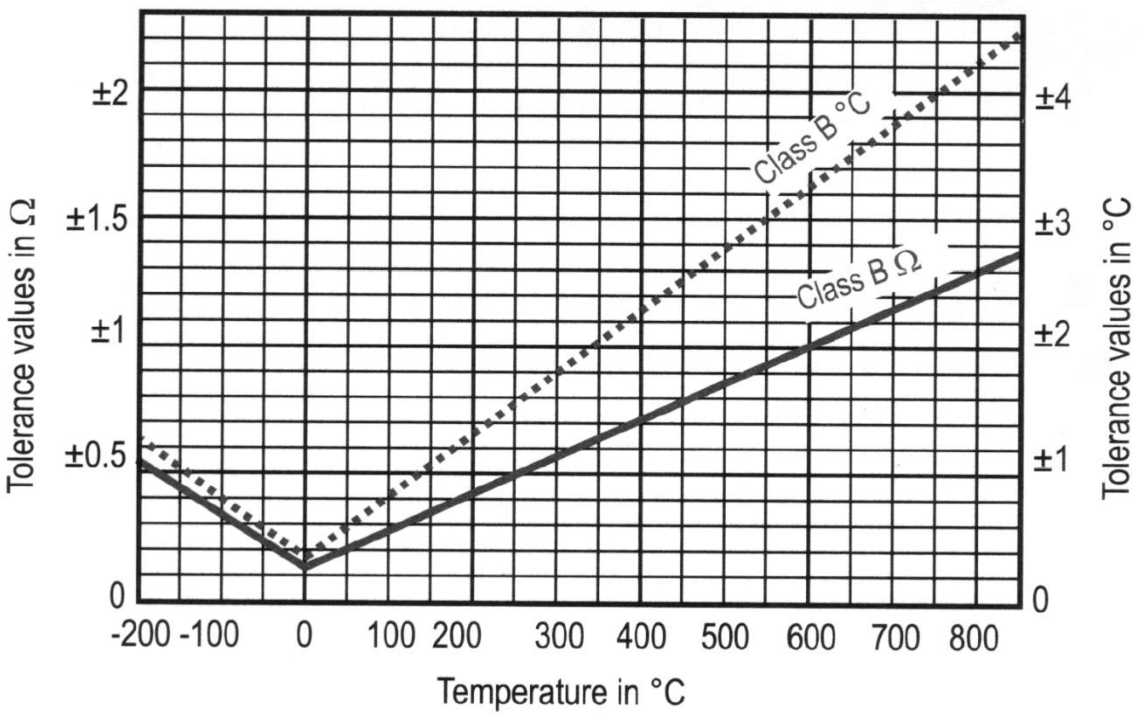
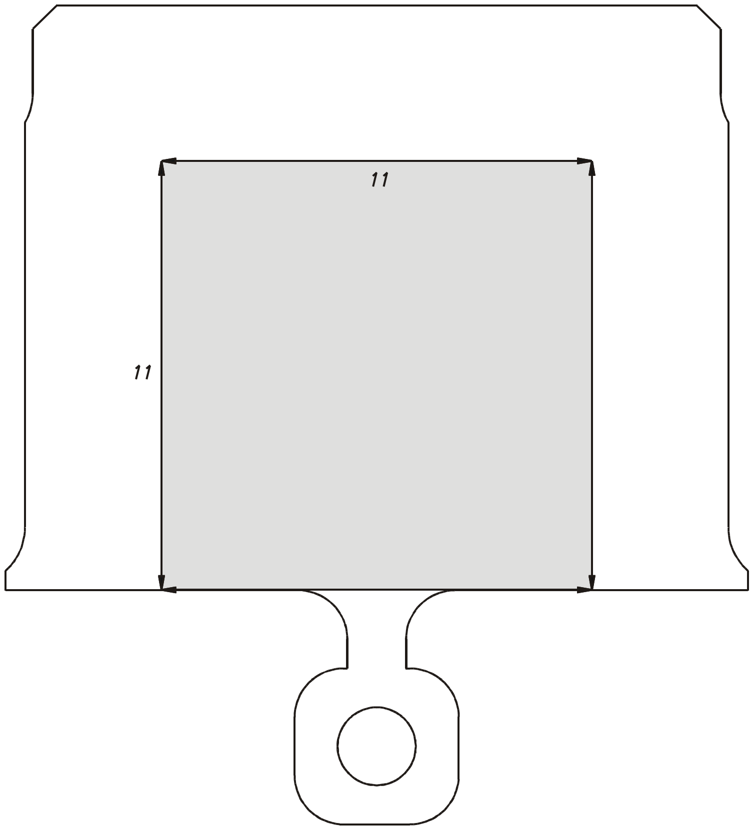


Figure 43. PT-100 permissible deviations according to IEC 751 and EN 60751, class B. (©2000 Lake Shore Cryotronics, Inc.)

VT STM XA Sample Plate Dimensions

sample not to exceed grey area, sample thickness < 3mm



maximum sample size standard sample plates, drawing not to scale

AUTO APPROACH Required Currents

Range /Bandwidth	$I_{AUTOAPPROACH}$
330 nA / 80 kHz	≥0.5 nA
330 nA / 3 kHz	≥0.5 nA
3 nA / 800 Hz	≥5 pA
3 nA / 200 Hz	≥5 pA

Table 17. Current values required during AUTO APPROACH for different range settings.

Coarse Motion Drive

Specifications	
X travel	±5 mm
Y travel	±5 mm
Z travel	10 mm
Capacity between electrodes and respective ground	≈ 0.9 nF

Standard Scanner

Specifications	
Maximum voltages for X, Y and Z	±140 V at T ≤ 40°C
Maximum scan range (X,Y)	nominal 19.8 µm ×19.8 µm
Maximum scan range (Z)	1.9 µm
Capacity between Z and X,Y	≈ 4 nF including the cable in UHV
MATRIX parameter set	VT STM

Notice

Scan ranges are with respect to small signal calibration (scan area ≤ 100 nm × 100 nm). If you need to re-calibrate the scanner for your typical measurement parameters please refer to the instructions in the "MATRIX Application manual for SPM".

Bakeout

Specifications	
Max. bakeout temperature without QPlus	170°C (voltage supplies disconnected)
Max. bakeout temperature with QPlus	150°C (voltage supplies disconnected)

Tip Length

Specifications	
Maximum free tip length	2.5 mm
Maximum overall tip length	6.5 mm

Eddy Current Damping Stage

Specifications	
Suspension resonance	≈ 2 Hz
Magnetic stray field at the tip	< 2.5×10^{-3} T (25 Gauss)
Magnetic stray field in 70 mm distance from the permanent magnets	< 10^{-4} T (1 Gauss)

Hybrid Preamplifier (for SPM PRE 4)

Specifications		
Supply voltage	±8.0 V	
Range	**333 nA**	**3.3 nA**
Bandwidth (-3 dB)	≥ 80 kHz	≥ 800 Hz
Feedback resistance	30 MΩ	3 GΩ
IVC conversion	3×10^{7} V/A	3×10^{9} V/A

Bias Voltage

Specifications	
Sample	grounded
Tip	V GAP

Radiative Heating (cooling version)

Specifications	
max heating current in stage	1.0 A, never exceeding 8 W
max heating power in stage	8 W, never exceeding 1.0 A
max temperature in stage	500 K

Cryostat Counter Heater

Specifications	
Resistance at room temperature	25 Ω
Maximum heater current	1 A
Maximum heating power	25 W
Max. temperature on cryostat head	190°C (limited by Si-diode durability)

Radiative Heating (heating-only version)

Specifications	
max heating current in stage	1.0 A, never exceeding 5 W
max heating power in stage	5 W, never exceeding 1.0 A
max temperature in stage	650 K

Cooling Stage

Specifications	
min. sample temperature (flow cryostat with LHe)	50 K
min. sample temperature (bath cryostat with frozen LN2)	110 K
min. sample temperature (bath cryostat with liquid LN2)	130 K

LHe Cryostat Counter Heating

Specifications	
max heating current in stage	1 A
max heating voltage in stage	25 V
max heating power in stage	25 W

Flange Layout

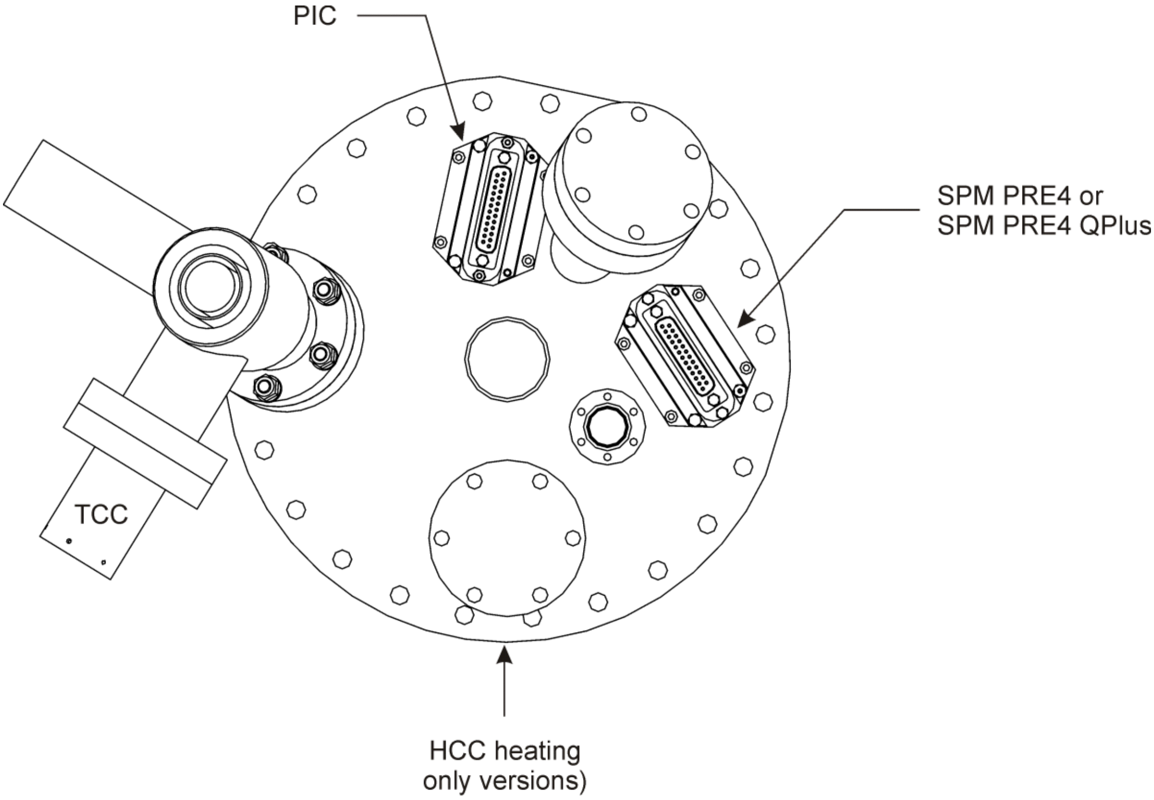


Figure 44. Base flange layout.

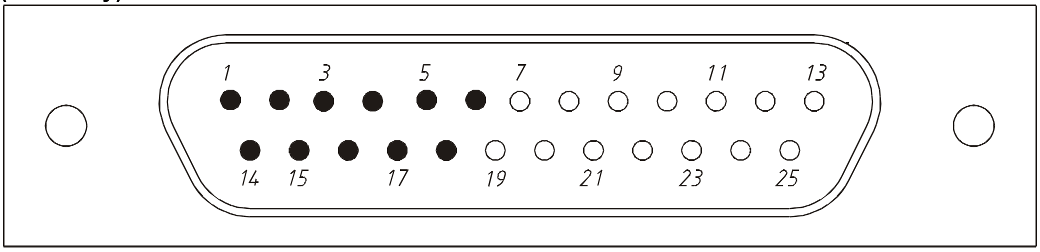
Notice

The plugs and sockets for SPM PRE and PIC are hardware coded to prevent connection mistakes.

SPM PRE 4 (type 3/4) PIC (type 1/3)

Connector Pinouts

PIC 9 (STM-only)

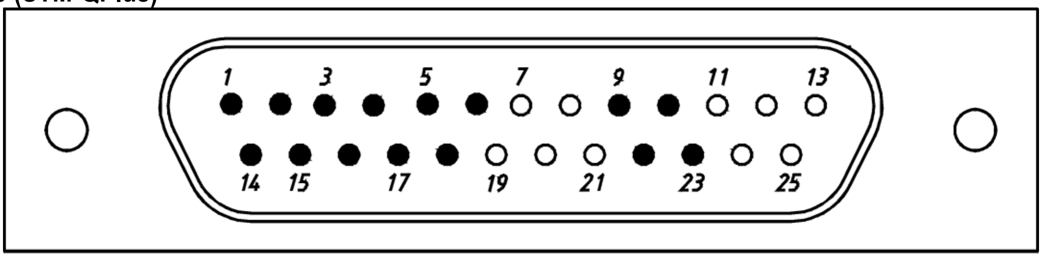


PIC 9

Figure 45. Feedthrough pinout PIC for PIC 9, atmospheric side.

Pin	1	2	3	4	5	6	7	8	9	10	11	12	13
Signal	-X	+Y	Zo	0cxy	Xc	Yc							
Pin	14	15	16	17	18	19	20	21	22	23	24	25	
Signal	+X	-Y	Z	0cz	Zc								

PIC 15 (STM-QPlus)

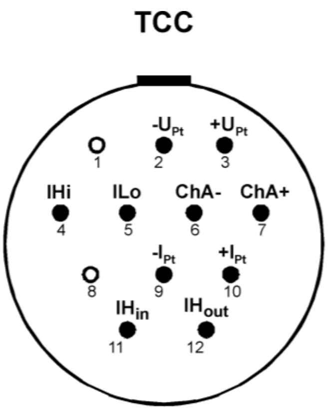


PIC15

Figure 46. Feedthrough pinout PIC for PIC 15, atmospheric side.

Pin	1	2	3	4	5	6	7	8	9	10	11	12	13
Signal	-X	+Y	Zo	0Cxy	Xc	Yc			MODo	MOD-			
Pin	14	15	16	17	18	19	20	21	22	23	24	25	
Signal	+X	-Y	Z	0cz	Zc				MODo	MOD+			

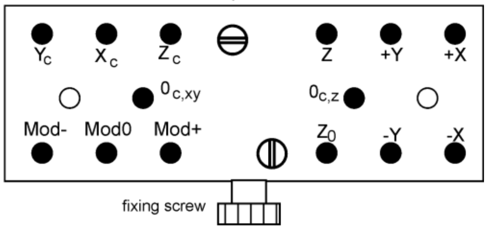
TCC Connector Pinout

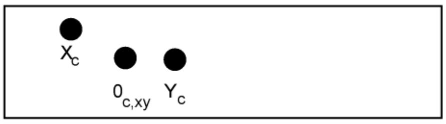


TEMPERATURE CONTROL CABLE
50/500 K VARIANT
ATMOSPHERIC SIDE OF FEEDTHROUGH
-UPt, +UPt, -IPt, +IPt: PT100 AT SAMPLE STAGE
ChA- AND ChA+: Si DIODE ON CRYOSTAT

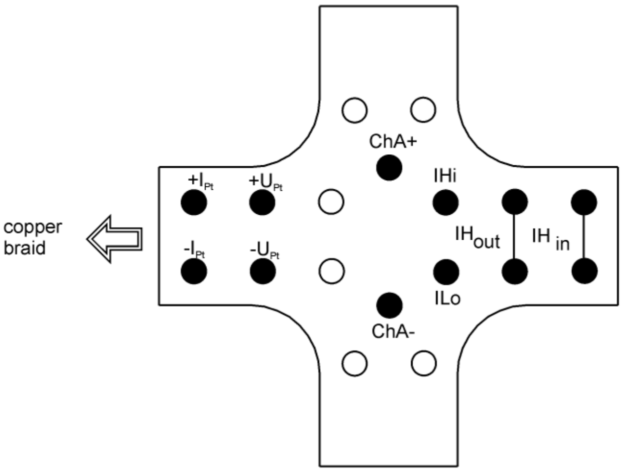
Connector Plates

connector plate VT STM





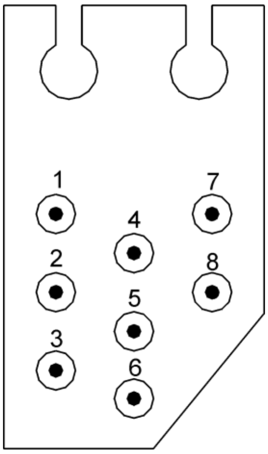
connector plate X-Y-table



connector plate cryostat, top view

Notice

Please note that for versions without QPlus preamplifier MOD+ is connected to Z_0 .



Pin	Signal
1	
2	
3	
4	Out
5	Q0
6	+8 V
7	
8	Q0

Special Plugs

PREAMP PROGRAM Plug VT PPP (9-pin D-sub)

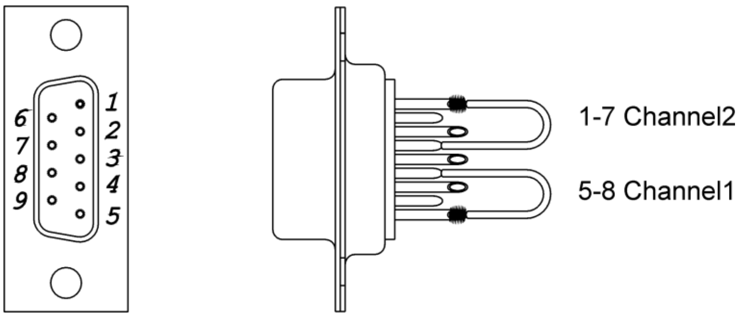


Figure 47. VT PPP pinout.

1	GND2	4		7	GND2
2		5	GND1	8	GND1
3		6		9	

PREAMP PROGRAM Plug VT PPP 3DQ(9-pin D-sub)

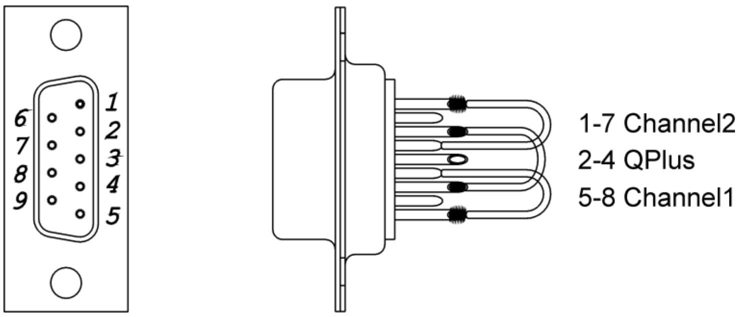
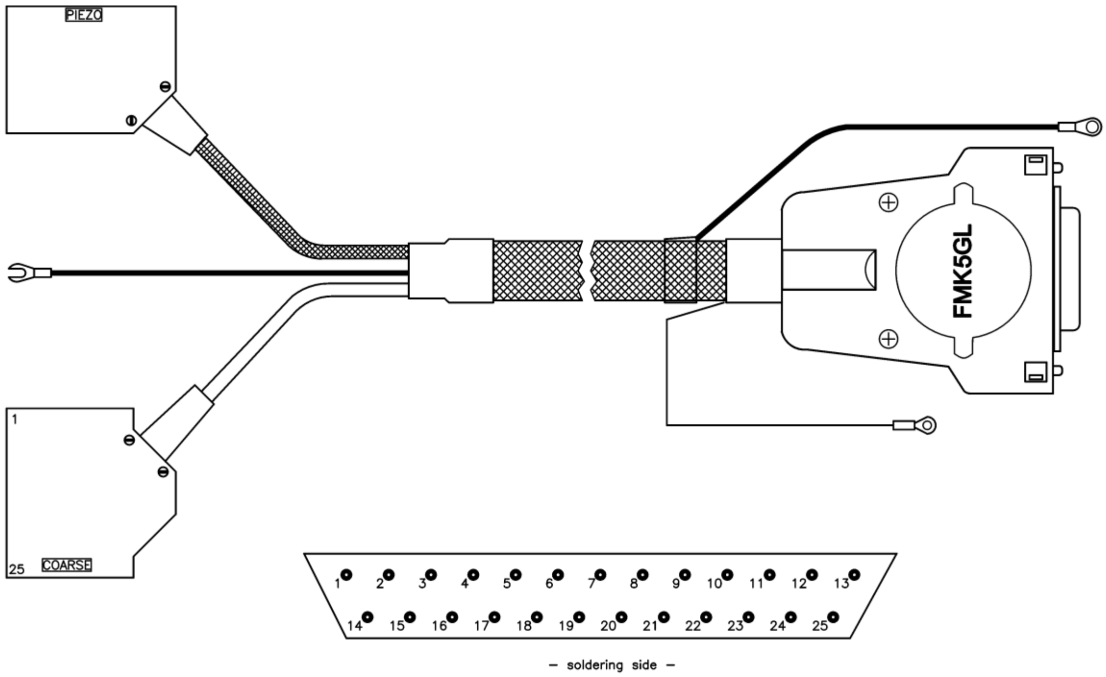


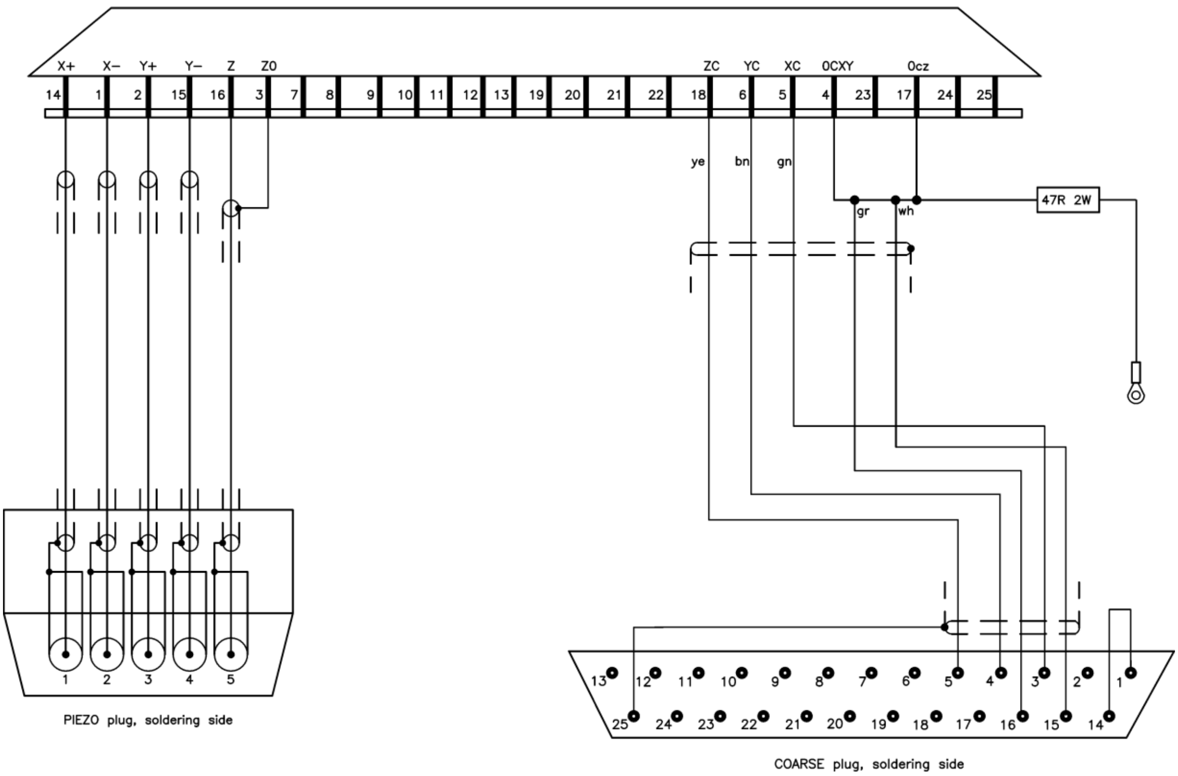
Figure 48. VT PPP 3DQ pinout.

1	GND2	4	Io	7	GND2
2	Io	5	GND1	8	GND1
3		6		9	

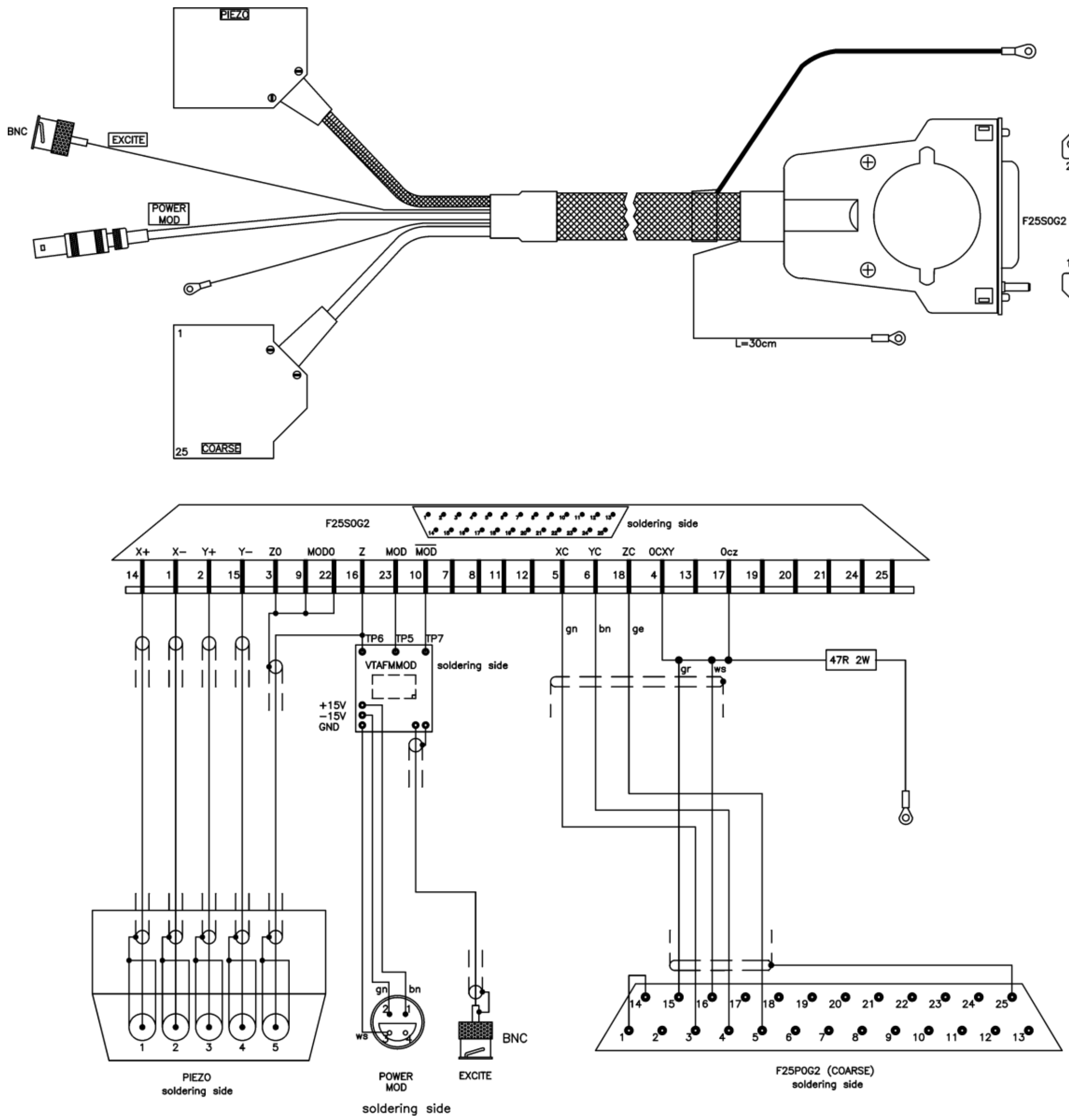
Cable Drawings

PIC 9 Cable



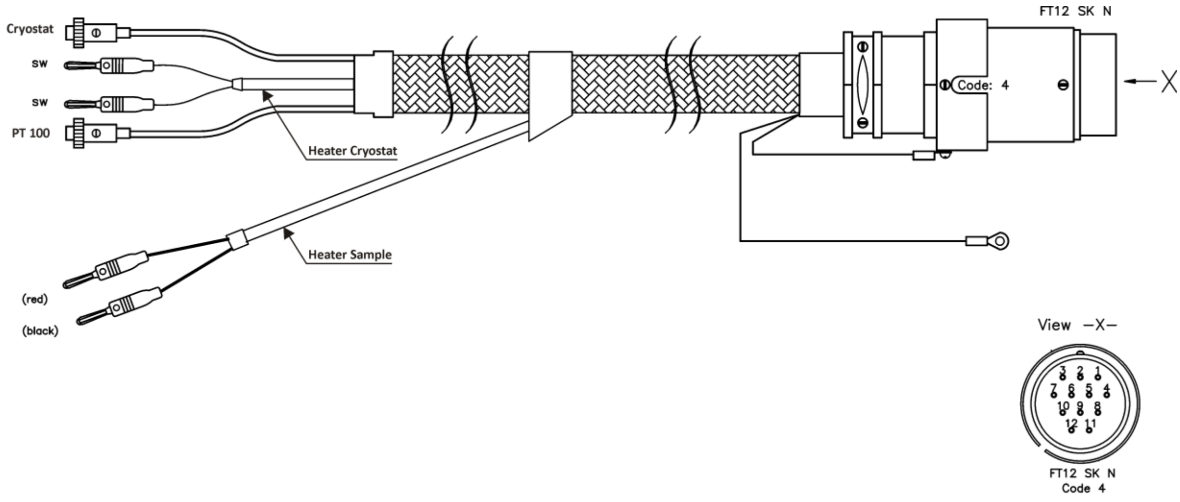


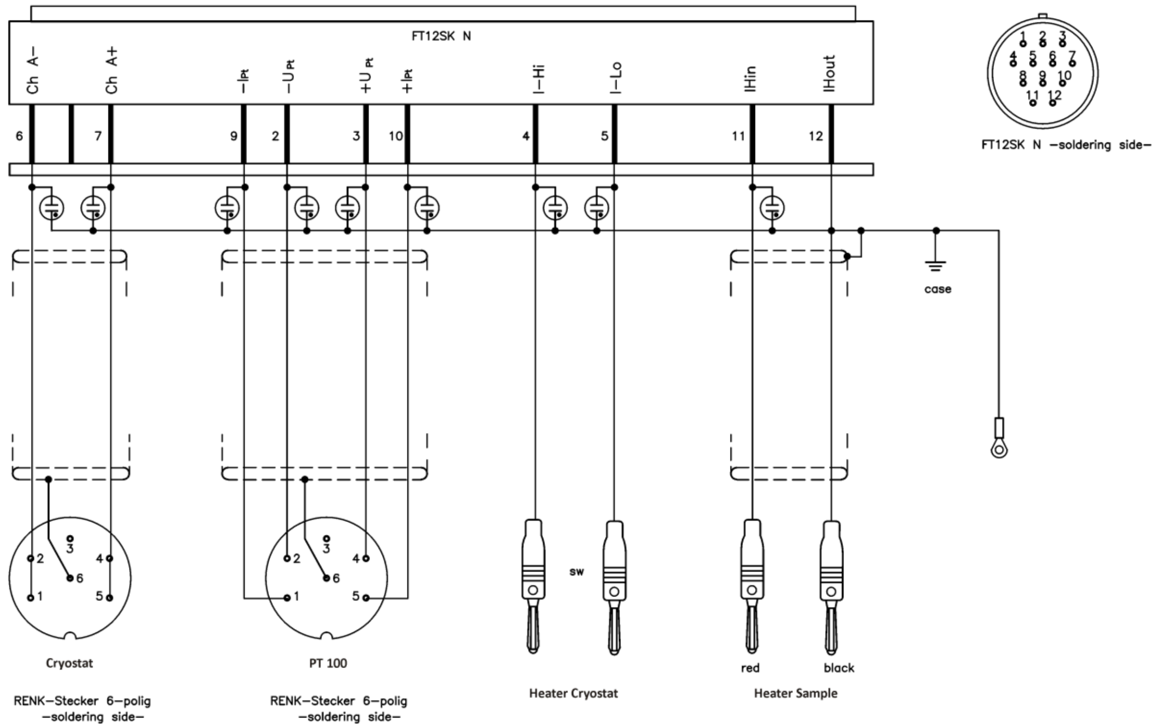
PIC 15 Cable (for QPlus variant)



Note: in the above drawing MOD = MOD+ and $\overline{\text{MOD}}$ = MOD-

Cable TCC XA (PN01977)





Scanner Orientation

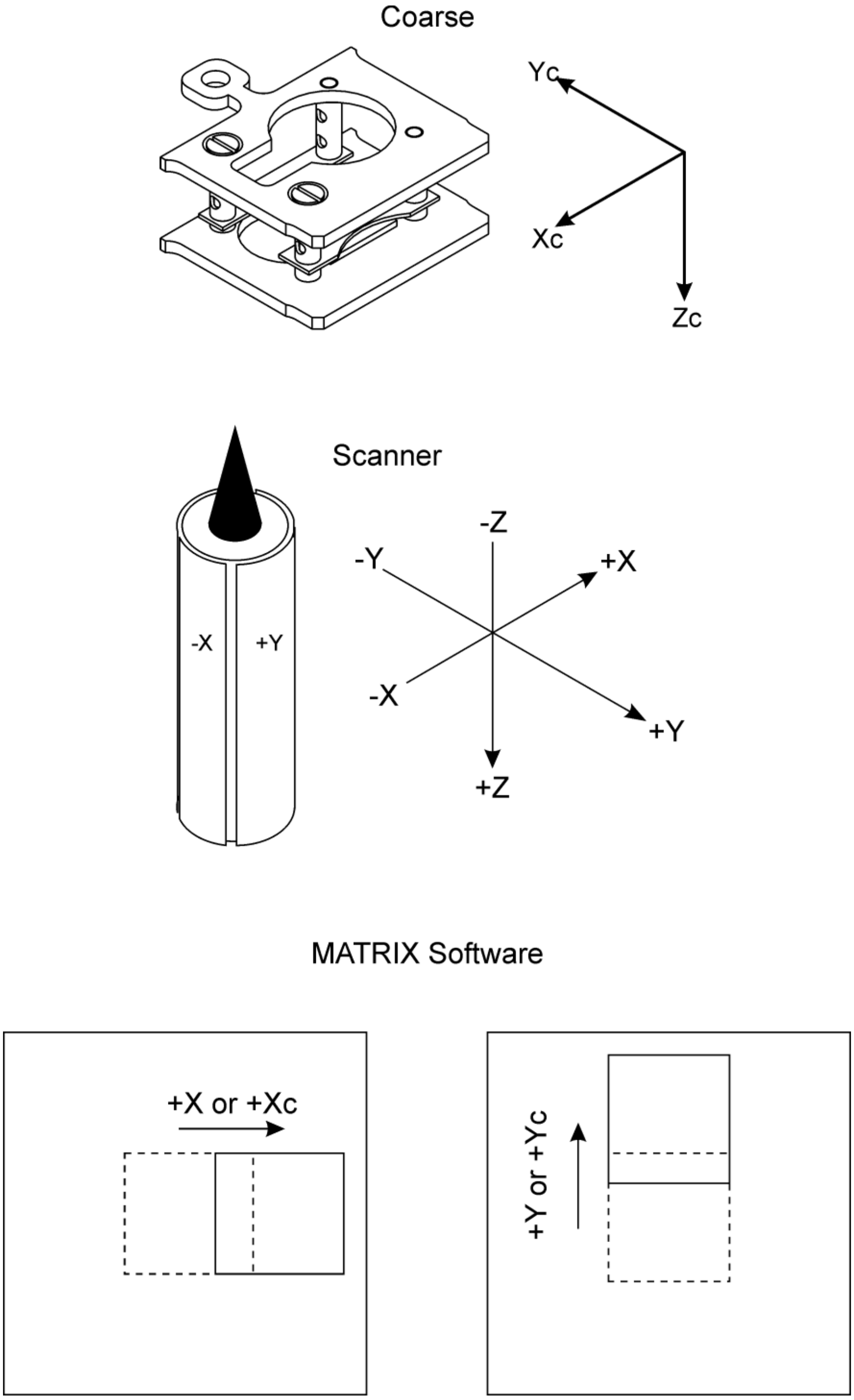
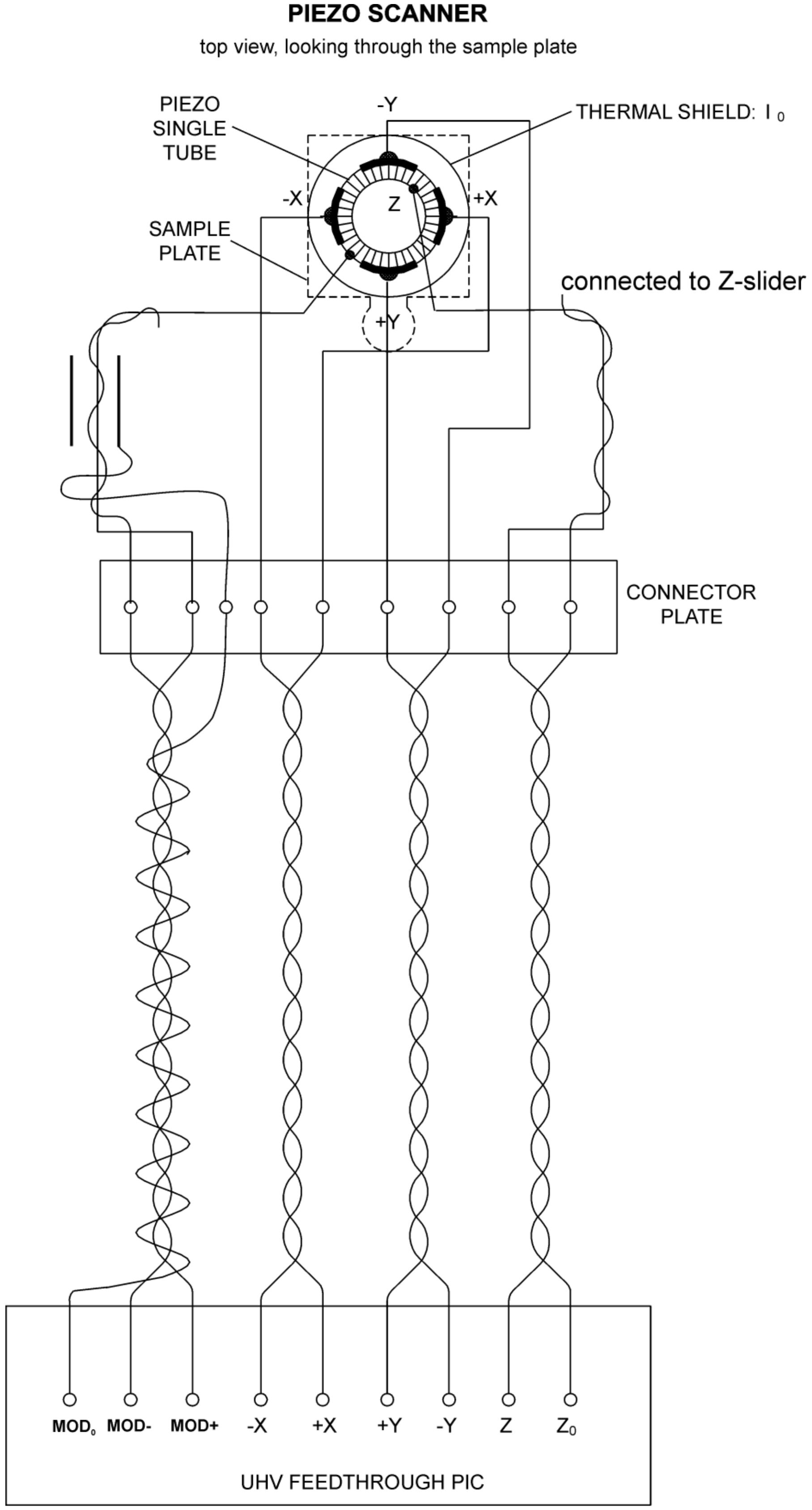


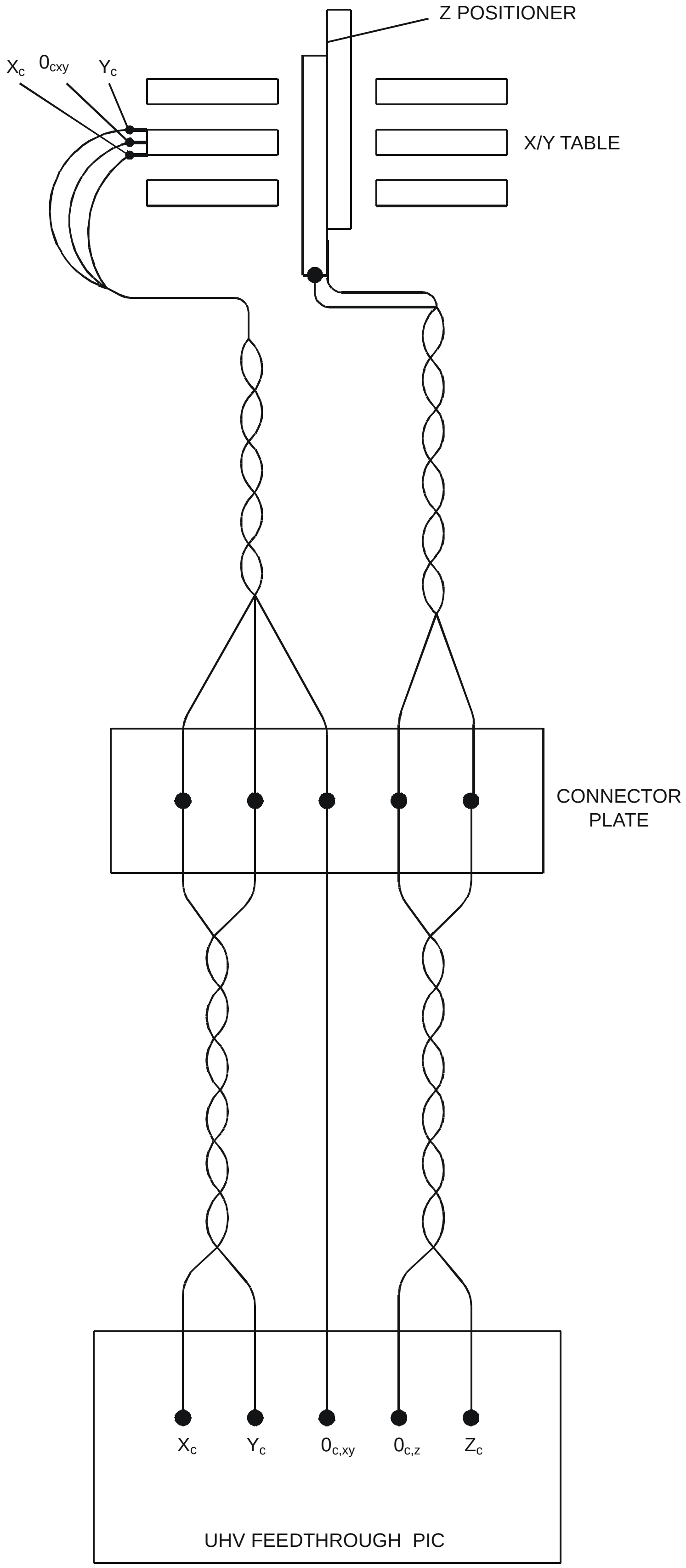
Figure 49. System of coordinates for coarse and scanner movements for scan angle = 0.

Internal Wiring



Note: Z_0 is connected to the coarse Z slider.

COARSE MOTION DRIVE



Preamplifier SPM PRE4 QPlus

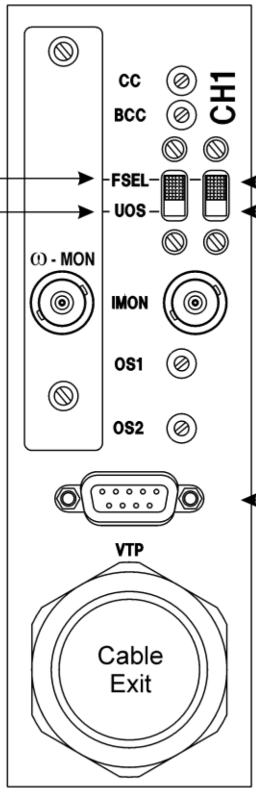


Figure 50. SPM PRE4/QPlus case.

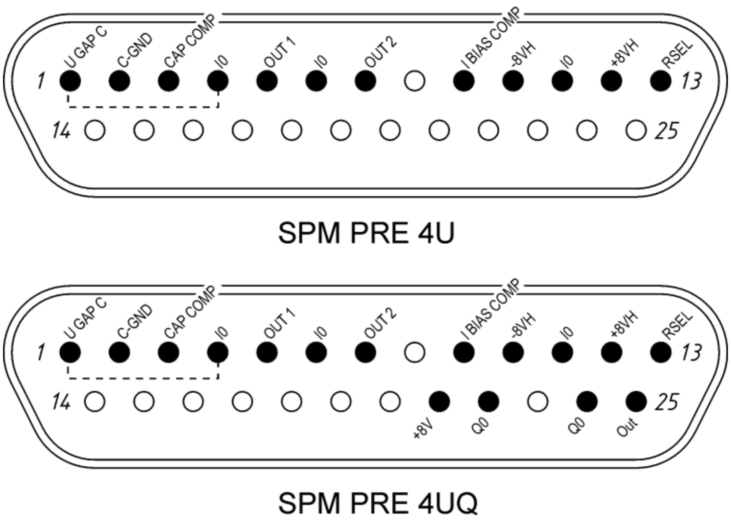


Figure 51. SPM PRE4/QPlus pinout, feedthrough atmospheric side. Note: pins 1 and 4 are connected inside the vacuum chamber.

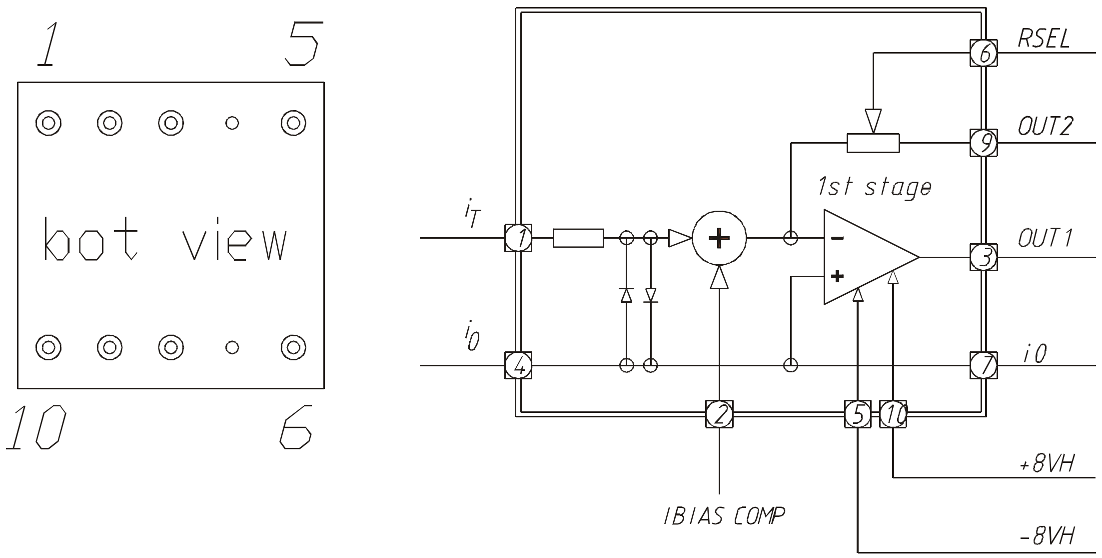


Figure 52. Hybrid I/V converter, schematic diagram. Case connected to pin 4, 7.

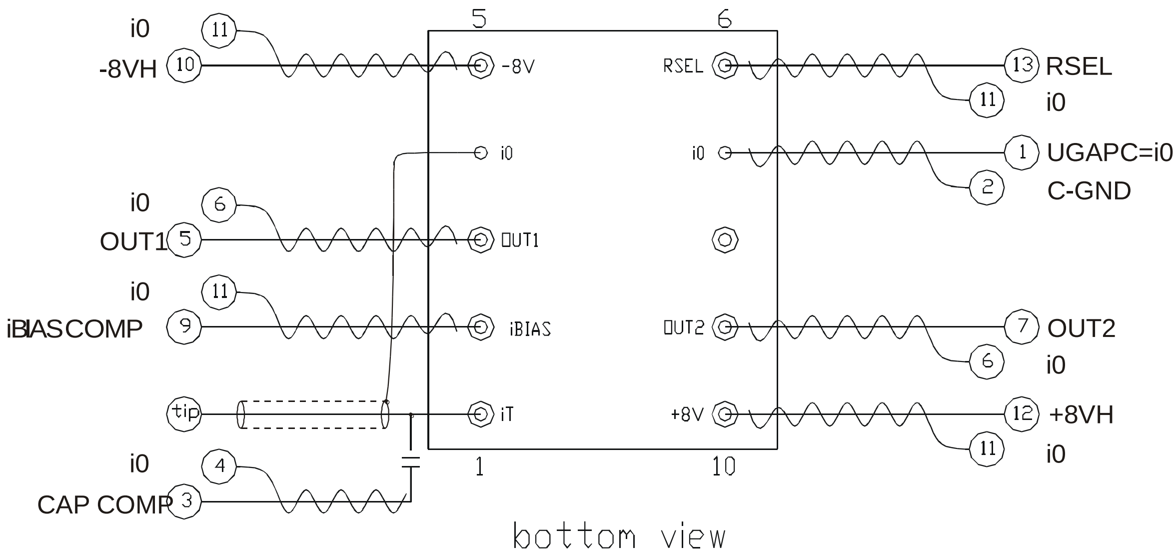


Figure 53. Hybrid I/V converter internal wiring. The circled numbers denote the pins of the vacuum feedthrough, see also figure 51 on page 117.

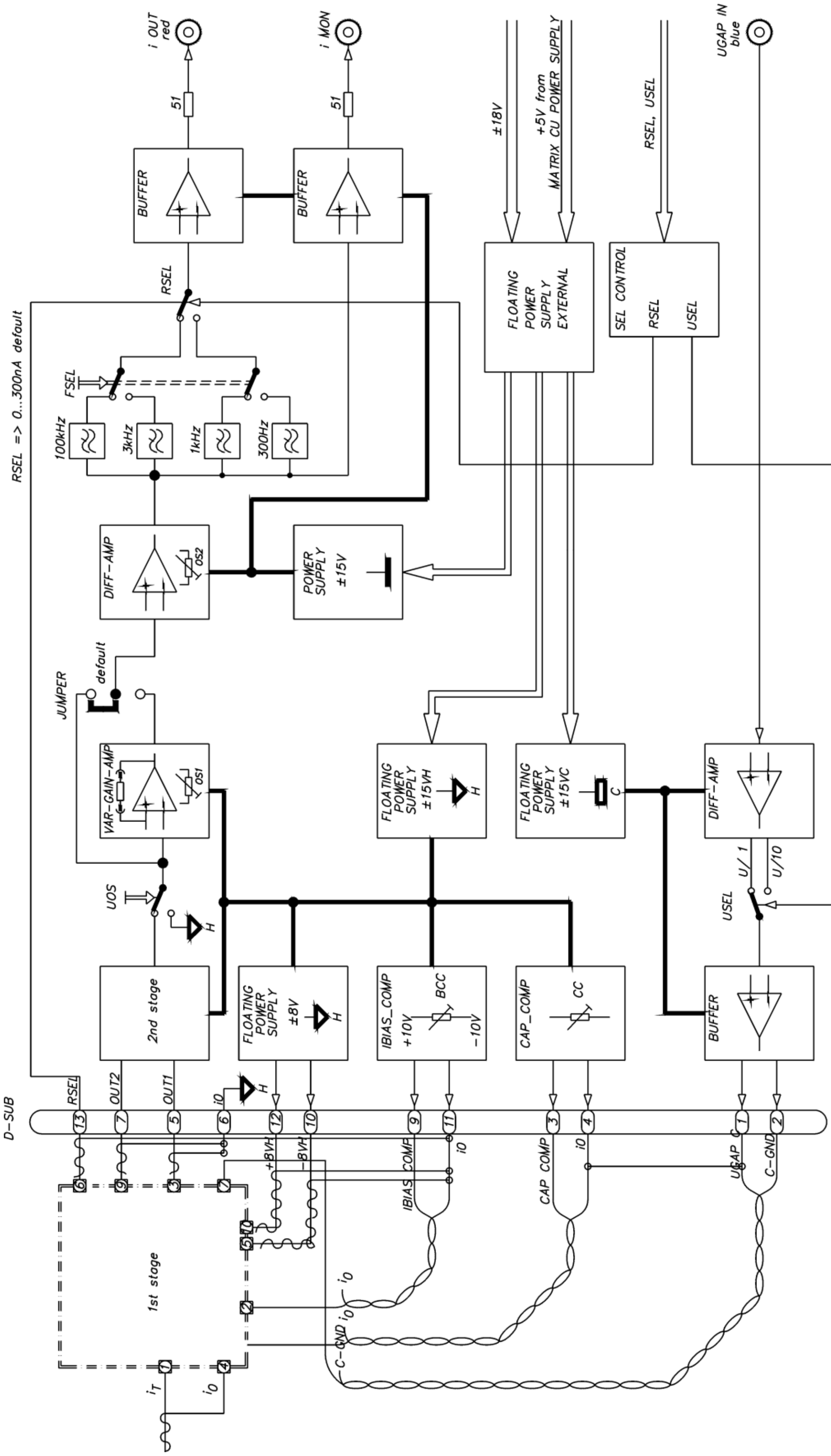


Figure 54. SPM PRE 4 schematic circuit diagram.

Coarse Position Remote Box

The Coarse Position Remote Box has a 4-line liquid crystal display (LCD) and a 12-button membrane keypad which gives access to several configuration- and operating menus. A "SPEED" potentiometer allows manual regulation of the coarse motor speed.

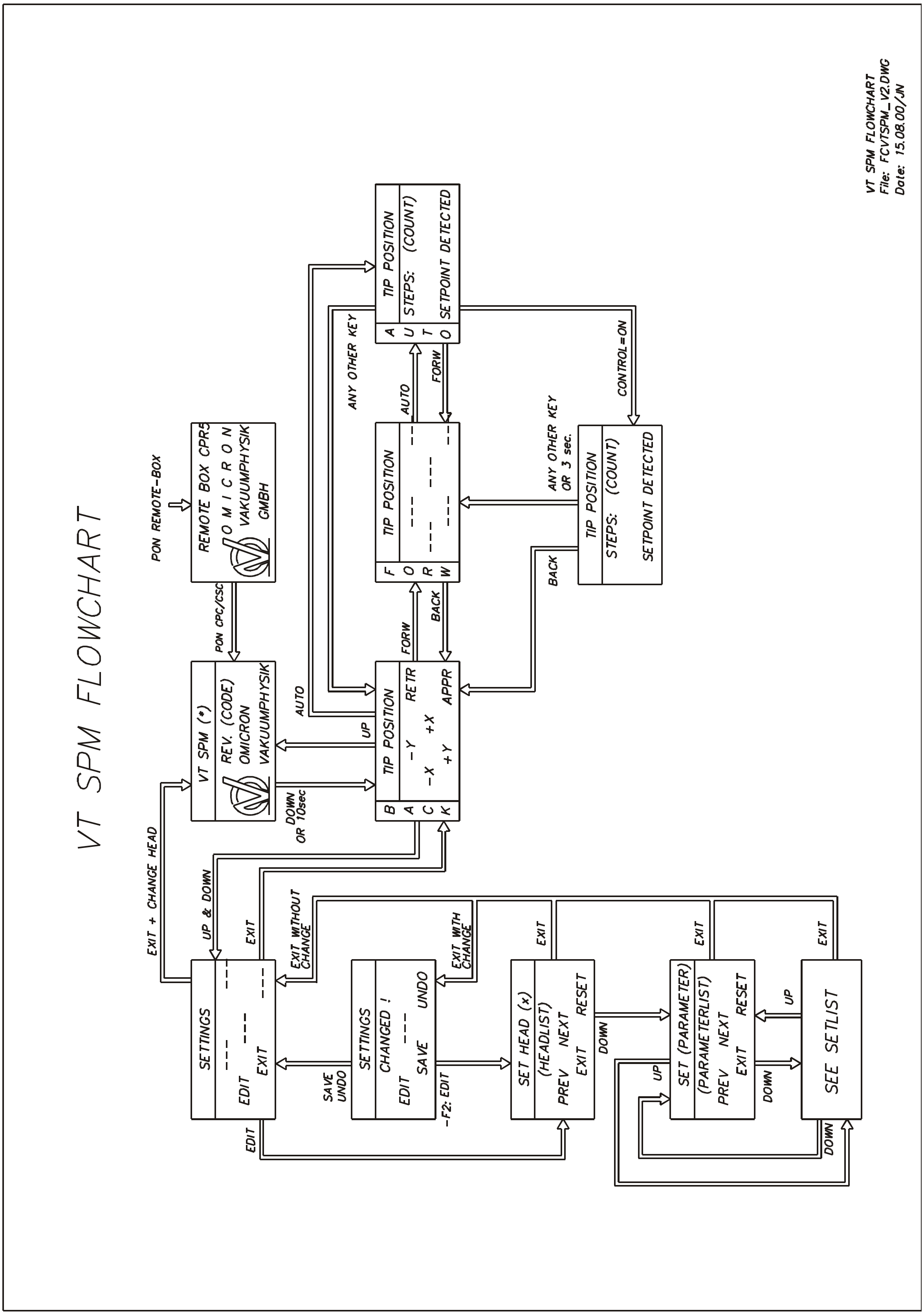
Before you start we recommend that you study the flowchart on page 121. This gives a short pictorial overview of the functions and scope of the Coarse Position Remote Box.

Notice
When you receive your Remote Box together with your SPM head there is no need to enter the settings menu, since all relevant parameters have already been configured at Scienta Omicron.

Upon switching on the MATRIX CU the remote box display shows an initialisation array for 30 seconds (can be skipped by pressing the DOWN button). This consists of the Scienta Omicron Logo together with the name of the SPM head that has been configured. The remote box then switches to the BACKWARD menu.

Setting	**Values**	**Comment**
HEAD	VT STM	
FREQUENCY	DIAL, 0.5 kHz, <u>1 kHz,</u> 2 kHz, 3 kHz, 4 kHz.	"DIAL-Range" 500 Hz - 4 kHz
VOLTAGE	<u>DIAL</u>, 20%, 40%, 60%, 80%, 100%	"DIAL-Range" 20% - 100%
STEPS	<u>1</u> to 10 in steps of 1	
Z-DIRECTION	<u>-</u>, +	
DELAYTIME	<u>0.6 sec</u> to 2 sec in steps of 0.2 sec	

Table 18. Remote box settings VT STM XA, default values underlined.


VT SPM FLOWCHART
PON REMOTE-BOX
REMOTE BOX CPR5
O M I C R O N
VAKUUMPHYSIK
GMBH
PON CPC/CSC
VT SPM (*)
REV. (CODE)
OMICRON
VAKUUMPHYSIK
AUTO
UP
DOWN
OR 10sec
TIP POSITION
-Y +X RETR
-X +Y APPR
B A C K
FORW
BACK
TIP POSITION
F O R W
AUTO
FORW
ANY OTHER KEY
TIP POSITION
STEPS: (COUNT)
SETPOINT DETECTED
A U T O
CONTROL=ON
ANY OTHER KEY
OR 3 sec.
BACK
TIP POSITION
STEPS: (COUNT)
SETPOINT DETECTED
EXIT + CHANGE HEAD
UP & DOWN
EXIT
EXIT WITHOUT CHANGE
SETTINGS
EDIT EXIT
SAVE
UNDO
SETTINGS
CHANGED !
EDIT
SAVE UNDO
EXIT WITH CHANGE
-F2: EDIT
EDIT
SET HEAD (x)
(HEADLIST)
PREV NEXT
EXIT RESET
EXIT
DOWN
UP
SET (PARAMETER)
(PARAMETERLIST)
PREV NEXT
EXIT RESET
EXIT
UP
DOWN
SEE SETLIST
EXIT
DOWN
VT SPM FLOWCHART
File: FCVTSPM_V2.DWG
Date: 15.08.00/JN

Literature

Books on Scanning Probe Microscopy

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QPlus References

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[13] Melmed A J (1991). Art and science and other aspects of making sharp tips. J. Vac. Sci. Technol. B 9, 601.

Service at Scienta Omicron

Should your equipment **require service**

- Please **contact Scienta Omicron** headquarters or your local Scienta Omicron representative to discuss the problem. An up-to-date address list is available on our website

  http://www.scientaomicron.com/

- Make sure all necessary information is supplied. Always **note the serial number(s)** of your instrument and related equipment (e.g. head, electronics, preamp…) or have it at hand when calling.

If you have to **send any equipment back to Scienta Omicron**

- Please contact **Scienta Omicron headquarters** before shipping any equipment.
- Place the instrument in a polythene bag.
- **Reuse the original packaging and transport locks.**
- Take out a **transport insurance policy.**

For ALL vacuum equipment:

- Include a filled-in and signed copy of the "Declaration of Decontamination" form which can be found at the back of the equipment manual.

STOP **No repair of vacuum equipment without a legally binding signed decontamination declaration !**

- Wear suitable cotton or polythene gloves when handling the equipment.
- **Re-insert all transport locks (**if applicable**).**
- Cover the instrument with aluminium foil and/or place it in a polythene bag. Make sure no dust or packaging materials can contaminate the instrument
- Make sure the **plastic transport cylinder (**if applicable**) is clean**.
- Fix the instrument to its plastic cylinder (if applicable).

Index

Decontamination Declaration

If performing repair or maintenance work on instruments which have come into contact with substances detrimental to health, please observe the relevant regulations.

If returning instruments to us for repair or maintenance work, please follow the instructions below:

- ***Contaminated units*** (radioactively, chemically etc.) must be decontaminated in accordance with the radiation protection regulations before they are returned.
- ***Units returned*** for repair or maintenance must bear a clearly visible note "free from dangerous substances". This note must also be provided on the delivery note and accompanying letter.
- Please use the attached attestation declaration at the end of this manual.
- **"*Dangerous substances*"** are defined in European Community Countries as materials and preparations according to Article 2 of the Dangerous Preparations Directive **1999/45/EC**.

No repair will be carried out without a legally binding signed declaration !

Declaration of Decontamination of Vacuum Equipment and Components

The repair and/or service of vacuum equipment/components can only be carried out if a correctly completed declaration has been submitted. **Non-completion will result in delay.** The manufacturer reserves the right to refuse acceptance of consignments submitted for repair or maintenance work where the declaration has been omitted.

This declaration may only be completed and signed by authorised and qualified staff.

1. Description of components

Type: ____ Serial No: ____

2. Reason for return/service ____

3. Equipment condition

Has the equipment ever come into contact with the following (e.g. gases, liquids, evaporation products, sputtering products…)

- toxic substances? Yes ☐ No ☐
- corrosive substances ? Yes ☐ No ☐
- flammable substances? Yes ☐ No ☐
- explosive substances? Yes ☐ No ☐
- microbiological substances (incl. sample material)? Yes ☐ No ☐
- radioactive substances (incl. sample material)? Yes ☐ No ☐
- ionising particles/radiation (α,β,γ, neutrons, …)? Yes ☐ No ☐

For all dangerous substances, gases and dangerous by-products which have come into contact with the vacuum equipment/components please list the following information on (a) separate sheet(s): trade name, product name, manufacturer, chemical name and symbol, danger class, precautions associated with substance, first aid measures in the event of an accident.

Is the equipment free from potentially dangerous substances? **Yes** ☐ **No** ☐

The manufacturer reserves the right to refuse any contaminated equipment / component without written evidence that such equipment/component has been decontaminated in the prescribed manner.

4. Decontamination Procedure

Please list **all dangerous substances, gases and by-products** which have come into contact with the vacuum equipment/components together with the decontamination method used.

SUBSTANCE	DECONTAMINATION METHOD

(continue on a separate sheet if necessary)

5. Legally Binding Declaration

Organisation: ____

Address: ____

____

Tel.: ____________________________________ **Fax:** ____

Name: ___________________________________ **Job title:** _______________________________________

I hereby declare that the information supplied on this form is complete and accurate.

Date: ________________ **Signature:** ______________________ **Company stamp:**