

## ULTRA HIGH VACUUM TECHNOLOGY

## VSN40

# NexGeneration Ultra High Vacuum Suitcase

Instruction manual

Version 5.4

September 2021

Ferrovac AG, Thurgauerstrasse 72, CH-8050 Zurich Switzerland Phone +41 44 273 16 38 www.ferrovac.com / sales@ferrovac.com



## Warranty

Ferrovac AG warrants this product to be free of defects in material and workmanship for a period of 12 months from the date of shipment. In case of proof of any defective parts in the product, we will at our discretion, either repair or replace the product.

## Warranty limitations

Warranty for this product does not apply to defects resulting from:

- non-observance of operational- and safety instructions
- natural wear of components
- consumables
- modifications to our products without our written consent
- misuse of any product or part of the product

This warranty stands in place of all other warranties, implied or expressed, including any implied warranty of implied merchantability or fitness for a particular use. The remedies provided herein are buyer's sole and exclusive remedies.

Neither the company Ferrovac AG nor any of its employees shall be liable for any direct, indirect, incidental, consequential or special damages arising out of the use of its products, even if the company Ferrovac AG has been advised in advance of the possibility of such damages. Such excluded damages shall include but are not limited to: Costs of removal and installation, losses sustained as the result of injury to any person, or damage to property.

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A triangle with exclamation mark indicates a passage in the manual with information that is crucial for the operator. READ THESE PARAGRAPHS CARE FULLY or the product might be damaged by misuse.



The hot surface symbol refers to hot surfaces which can lead to injury and burns when touched.



The high voltage symbol, if found on a product or accessory of a product, indicates voltages that are potentially lethal. READ THESE PARAGRAPHS CAREFULLY in order to protect the operator from any injury.



A triangle with a snow flake indicates a passage in the manual with information that is crucial for the operator with respect to cryo cooling and handling of cryogenic liquids. READ THESE PARAGRAPHS CAREFULLY in order to protect the operator from any injury.

**WARNING** The WARNING heading in a manual explains dangers that may result in personal injury or death. Always read the associated information very carefully.

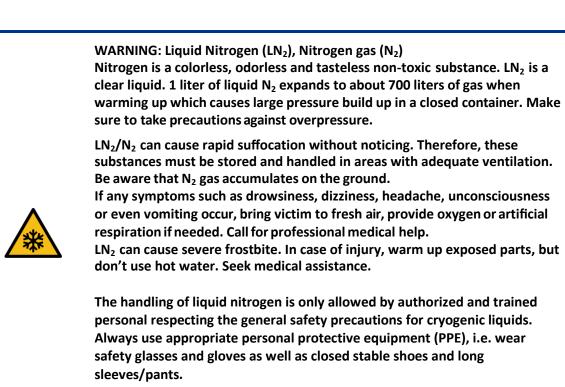
**CAUTION** The CAUTION heading in a manual explains hazardous situations that could damage the product. Such damage may invalidate warranty.



CAREFULLY READ THE SAFETY INFORMATION AND ALL RELEVANT MANUALS BEFORE USING THE PRODUCT AND ANY RELATED INSTRUMENTATION!



WARNING: High voltages!! Any adjustment, fault finding procedure, installation and maintenance of the products described in this manual must be carried out by authorized personnel, fully qualified to handle potentially lethal voltages.



Read all safety instruction given by your supplier of liquid nitrogen and storage vessels carefully.

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## Safety precautions

The following safety precautions must be observed at all times before using the product described in this manual and any associated instrumentation.

The product described in this manual is intended for use by qualified personnel who recognize hazards and are familiar with the precautions necessary to avoid possible injury.

**Responsible body** is the individual or group of persons that are responsible for the proper use and maintenance of the product, ensuring that the product is operated within its specifications and operating limits. The responsible body must ensure that users of the product are adequately trained.

**Operators** are using the product for its intended purpose. Operators must be trained in basic laboratory safety, basic electrical safety, handling of cryogenic liquids and adequate use of scientific instruments, in particular ultra high vacuum systems. They must be protected from electric shock, frostbite and contact with potentially dangerous situations.

**Maintenance Personnel** perform routine tasks on the product to keep it in proper operating conditions. Maintenance procedures are described in the manual and must be followed at all times.

**Service Personnel** are trained to work on live circuits, ultra high vacuum equipment, gas handling systems and handling cryogenic liquids, as well as perform fault finding measurements and repair work on the product. Only fully trained service personnel qualified to handle potentially lethal voltages may perform servicing and repair on electronic devices. Only fully trained service personnel set up the gas filling lines, gas bottles, etc. which are necessary for a proper operation of the product.

The American National Standards Institute states that a shock hazard exists when voltage levels are greater than 30 V RMS, 42.2 V peak or 60 VDC. A good safety practice is to assume that hazardous voltages are present in any unknown circuitry.



CAUTION: Check for correct mains voltage and connectors before connecting any equipment!



**CAUTION:** High Voltages (HV) up to 5 kV! Adjustments and fault finding measurements may only be carried out by authorized service personnel. High Voltages may be present at parts of the instrument during operation.

#### WARNING:

Always observe and strictly follow the safety notes and regulations given in this and related documentation.

Always use the configured cables delivered with the product for electrical connections. It is prohibited to use cables and connectors which are not rated for the respective voltage values or which are damaged.

Always disconnect the mains supplies and switch off all electrically connected units before venting, pumpdown, opening the vacuum chamber, touching any part of the in-vacuum components.



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Always observe and strictly follow the safety notes and regulations given in this and related documentation.

Never operate the ion pump in a pressure range above  $1 \times 10^{-5}$  mbar.

Read safety instructions first and be familiar with general safety precautions for cryogenic liquids and compressed gases.

Always strictly follow the safety notes and regulations for handling cryogenic liquids given by the vendor of cryogenic liquids.

Make sure that a proper ventilation is present in the laboratory while using cryogenic liquids.



CAUTION: Do not open electronic devices unless you fulfill the requirements of a fully trained service personnel and you are familiar with live circuits and potentially lethal voltages.

VSN40

### Normal use

The product described in this manual must always be used:

- With original sets supplied by Ferrovac AG which are explicitly specified for the use with the product described in this publication
- With all cabling connected and secured, if applicable
- With electronic equipment switched on only when cables are connected properly
- In an indoor research laboratory environment
- By personnel qualified for operation of delicate scientific equipment
- By personnel trained in using cryogenic liquids and pressurized gas handling, if applicable
- In accordance with all related manuals.

## Ambient conditions and environments:

This product is only to be used indoors, in laboratories meeting the following requirements:

- Room temperature between 5°C/41°F and 40°C/104°F.
- Relative humidity up to a maximum of 80% for temperatures up to 31°C decreasing linearly to 5% relative humidity at 40°C.
- Altitudes up to 2000 m.
- Pollution Degree 2 environments.
- Mains supply voltage fluctuations must not exceed ±10% of the nominal voltage
- The device must only be operated at the **indicated mains supply voltage.**

The responsible body needs to make sure that all general safety standards in a laboratory are fulfilled. Every collaborator in the laboratory needs access to personal protection equipment (PPE) according to the safety regulations. Appropriate safety training must be provided.

Besides wearing long sleeves and solid closed shoes for handling cryogenic liquids the PPE consists at least of

- Personal eye/face protection (see also EN 166)
- Protective gloves against cold (see also EN 511)
- Protective shoes (EN ISO 20345)



## **Technical Specifications**

For detailed specifications of e.g. the LSA (Battery Powered Ion Pump Controller), transfer arms and wobblesticks, please refer to the respective sub product manuals.

<b>Bakeout</b> : Maximum bakeout temperature	150°C
Chamber: He Leak rate Tolerances: • Machined parts	< 5 × 10 <sup>-10</sup> mbar x l x s <sup>-1</sup> ISO 2768 -m -K
<ul> <li>Welded structures</li> </ul>	$\pm 1 \text{ mm and } \pm 1^{\circ}$
<ul> <li>Pumping:</li> <li>SAES NEXTorr®D-100-5:</li> <li>Supply voltage ION section</li> <li>Activation current NEG section</li> <li>NEG section alloy</li> <li>NEG section getter surface</li> </ul>	+5 kV DC 5 A, max. 5 A (9 V) during activation St 172 (ZrVFe) 114 cm <sup>2</sup>
<ul> <li>SAES NEXTorr®D-200-5:</li> <li>Supply voltage ION section</li> <li>Activation current NEG section</li> <li>NEG section alloy</li> <li>NEG section getter surface</li> </ul>	+5 kV DC 4.8 A, max. 5 A (12.5 V) during activation St 172 (ZrVFe) 238 cm <sup>2</sup>
HV and NEG activation cables	bakeable to max. 150°C
Cryo options: Liquid nitrogen dewar Temperature sensor: • Type • Resistance • Tolerance • Temperature range • Supply current	filling volume: 0.6 liters Pt100 100 Ω at 0°C Class A -200°C to 170°C 1 mA

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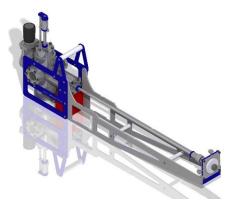


Figure 1: VSN40S configuration with integrated sample storage.

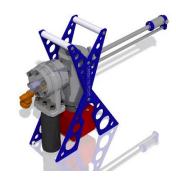


Figure 2: Ultra compact version VSN40M.

## 1 Introduction

The transport of samples under true UHV conditions from one instrument to another, sometimes from one institution to the other, has become an increasingly important task in experimental research.

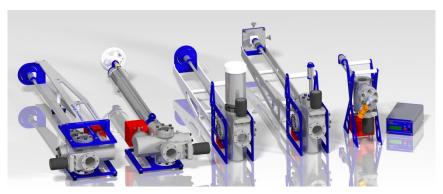
The VSN40 NexGeneration UHV Suitcase (examples in Fig. 1 and 2) is a fully featured, miniaturized UHV system for transport and storage of samples under ultraclean conditions. The vacuum suitcase is equipped with a combined Non Evaporable Getter (NEG) and ion pump driven by a battery-powered controller.

The usage of combined NEG/Ion pump technology allows us to build a truly portable, compact and lightweight (10 kg<sup>1</sup>) UHV system. The weight (2.2 kg) of the NEG/Ion pump used being less than one third of a 20 l/s ion pump, its nominal pumping speed is five to ten times higher. The NEG-element can be activated with any standard DC lab power supply. High voltage for the ion-pump element is delivered by our rechargeable battery-driven controller LSA, for up to 60 hours without mains power connection. Base pressures well below 10<sup>-10</sup> mbar are commonly reached.

The suitcase can be individually configured for vertical or horizontal usage, type and travel range of the transfer arm as well as the sample grabbing mechanism (examples in Fig. 3). It is fully compatible with our wide range of manipulators such as linear/rotary feedthroughs, wobblesticks and sample transporters.

<sup>&</sup>lt;sup>1</sup>Overall weight depends on the configuration of the suitcase.





**Figure 3:** Configuration examples of the VSN40.

The modular VSN40 is our state of the art model of NEG/Ion pumped UHV suitcases optimized in size, cost and performance.

## 2 Unpacking and inspection

Before unpacking, optically inspect the parcel. If damage is found, take pictures of the parcel and send them to Ferrovac AG immediately.

Prepare a very clean workspace. Carefully unpack the suitcase and perform a visual check for any damage of the package, its contents and accessories.

The UHV Suitcase is shipped under ultra high vacuum conditions and is ready for use.



CAUTION: Ferrovac suitcases are shipped under UHV! Do not open the gate valve before establishing controlled vacuum conditions. Sudden uncontrolled venting can cause damage to pumps and valve.



WARNING! Read the manual of the LSA pump controller before connecting and using it.



Package content depends on each specific suitcase configuration.

Compare content with the delivery note. Any damage or missing item must be reported to Ferrovac **within 48 hours after delivery**.



Figure 4: Typical package content of a complete system.

- 1. UHV suitcase
- 2. Battery powered ion pump controller
- 3. Battery charger including 4 country specific mains adapters
- 4. Bakeable cable for NEG activation
- 5. Bakeable HV cable for ion pump element
- 6. This manual and any manuals of sub-products

#### Depending on your configuration:

- 7. Temperature measurement cable (as an upgrade for the cryogenic version)
- 8. Additional accessories depending on selected options

#### CAUTION:

• Ensure enough work space on a clean table for unpacking and inspection.



- Never open the gate valve before the suitcase is mounted to a vacuum system at a working pressure below 1E-7mbar
- Read manuals carefully before using any device.
- Never expose the suitcase and its manipulators to physical shock or aggressive chemicals.
- Never hit the CF flange knife edge nor any bellows.



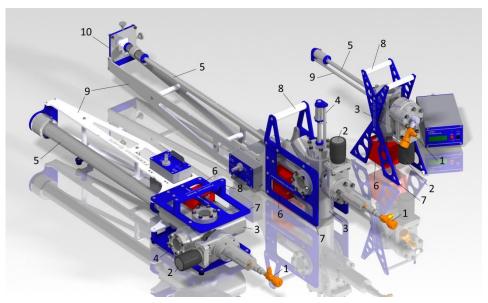


Figure 5: Overview of the NexGeneration UHV suitcase and its parts.

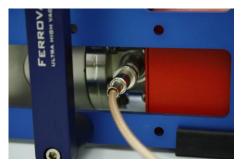
## **3** Overview and naming conventions

The main parts of the NexGeneration UHV suitcase are named as follows:

- 1. Protective tube with valve
- 2. Gate valve
- 3. Chamber with viewports
- 4. Storage manipulator (or viewport or dewar)
- 5. Transfer manipulator (or blank flange)
- 6. Pump
- 7. Side / baseplate(s)
- 8. Carrying handle
- 9. Protective rail (in combination with wobblestick transfer manipulator)
- 10. Axis alignment knob or XY precision axis aligner

## 4 Setup and installation

All our VSN40 vacuum suitcases are shipped under ultra high vacuum. During transport, the NEGelement maintains its base pressure in the  $10^{-10}$  mbar range. For operating the ion pump element and measuring the vacuum level, it needs to be connected to the battery powered ion pump controller (LSA). Please consult the manual of your specific LSA device before use.



LSA3 FERROVAC ON OFF press 5 sec.

Figure 6: Plug in the SHV cable

**Figure 7:** Front of the LSA controller.

Unpack the battery charger and plug its cable into the rear socket of the LSA ion pump controller. Then plug the battery charger into a mains supply socket.

Connect the LSA high voltage output to the NexTorr pump's HV input socket using the original high voltage cable delivered with the VSN suitcase.

Press the "ON" button of the LSA for five seconds.

Check the readings of the display. If vacuum conditions are sufficient, the ion pump will start and the display will indicate the vacuum level in mbar. An overrange ("1.") reading for the first few seconds after power on might occur and is normal. The pressure reading should drop to a value in the low  $10^{-10}$  mbar range.

Occasionally the vacuum reading drops down to "underrange". This means either that your suitcase is at a base pressure below  $1 \times 10^{-11}$  mbar or it means that the ion pump element did not yet start.

To figure out which is the case, move the transfer manipulator a few centimeters forth and back (which is possible with the gate valve closed). Moving the manipulator will cause some degassing which should change the display reading. It may also help to start the ion pump if the vacuum is extremely good.



Figure 8: Display reading on the LSA connected to the ion pump.

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## 5 Operation

This section describes the principle of operation. For using the suitcase as a transfer device and installation on a UHV system, please refer to section 6.

Ideally, the suitcase is operated in a pressure range below 10-9 mbar for clean sample surfaces and a long lifetime of the pump. Short exposures in the range of minutes to higher pressures up to about  $5 \times 10-6$  mbar during sample exchange are tolerable.

Depending on the readings of the LSA display, choose one of the following three options:

- 1. The pressure reading is higher than 1×10–7 mbar (i.e. the LSA shows "overrange"): Switch the ion pump controller off and proceed to section 8 to re-establish UHV conditions.
- 2. The pressure is between 10–9 mbar and 10–7 mbar: Proceed to section 8.3 "NEG activation".
- 3. The pressure is below 10–9 mbar: Proceed to chapter 6 "Installing the suitcase to a UHV System".

## 6 Installing the suitcase to a UHV system

This section contains step by step procedures for installing the suitcase to a host UHV system and transferring samples.

We recommend establishing a checklist depending on the steps necessary on your systems.

#### 6.2 Items to be provided by the customer

Tools and items the customer needs to take care of:

- Cu gaskets of the respective size (mainly for DN40CF and DN16CF flanges)
- Metric size 10 and size 7 wrenches or spanners
- Support structures for the suitcase if necessary

#### 6.3 Preparing host UHV system

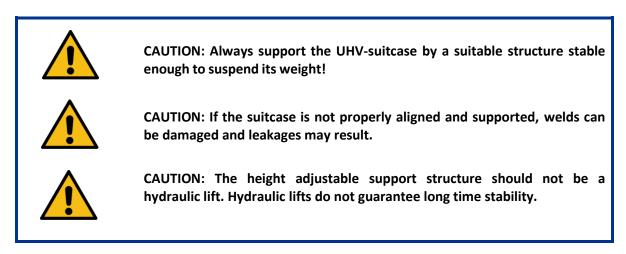
Your host UHV-System must be equipped with a DN40CF flange. Usually a gate valve and a short CF40 T-piece (e.g. VSCROSS40RL80, VSTEE40) or VSCTH40 docking station is installed as a buffer volume for fast pumpdown using a turbo pump. If the UHV System is equipped with a load lock system, the load lock can be used as buffer volume (i.e. a dock for installing the UHV suitcase).

Vent the protective vacuum tube of the UHV suitcase with dry Nitrogen by opening its vent valve.



CAUTION: Make sure that the gate valve of the UHV suitcase is and remains firmly closed before venting the protective vacuum tube!

The UHV suitcase must be properly aligned in height and position and its weight must be supported by a suitable structure depending on the selected configuration (type and length of manipulators). A lab jack or height adjustable spindle trolley for instance, provides a good solution.



#### 6.4 Pumpdown of buffer volume

After flanging the UHV suitcase (gate valve closed) to the buffer chamber (or T-piece), pump the buffer volume using a turbo pump until a sufficiently low vacuum level is reached. "Sufficiently low" in this context depends on the sensitivity (reactivity) of your sample surface. An initial bakeout of a new buffer volume might be necessary case by case. A Pressure at least in the low 10<sup>-7</sup> mbar range is highly recommended. Using dry Nitrogen gas for venting and keeping vented conditions short in time is always beneficial for achieving faster pumpdown.

A note on baking the buffer chamber: Collateral warmup of the UHV suitcase during bakeout of the intermediate chamber will degrade the vacuum level in the UHV suitcase to some extent. Baking the buffer chamber is therefore not an ideal solution.

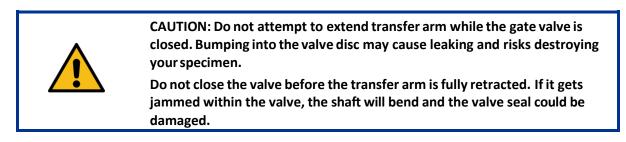
For this reason, we developed a liquid nitrogen cold trap CTDH40 (dewar version) and CTH40 (flow version) for fast pumpdown of the buffer chamber. Please visit our website for more information about these products. If the UHV Suitcase is still in ex-works condition, namely under true UHV, the system is now ready for transferring samples.



#### 6.5 Transfer of samples

Open the gate valve of the suitcase while keeping an eye on the LSA display. If the pressure reading (pump current reading on LSA2 units) is as expected you can now transfer samples between host UHV system and suitcase.

After transferring, make sure that your manipulator is fully retracted before you attempt to close the gate valve. If applicable secure it by the locking screw. Close the gate valve.



#### 6.6 Removing the suitcase from the UHV chamber

Firmly close the gate valve of the suitcase and vent the buffer chamber with dry Nitrogen. Open the CF40 flange connection between buffer volume and gate valve of the suitcase. For protection of the knife-edge of the gate valve, reinstall the protective vacuum tube VSN40SBT. See also section 7.2.

## 7 Transporting samples under UHV

#### 7.1 Preparing the suitcase for transport

Make sure that the sample won't move during transport. Secure the transfer manipulator by its lock screw or lock ring.

For in-laboratory transport, disconnect all cabling except the HV cable of the ion pump element. After disconnecting the power adapter from the LSA, recheck that the high voltage is on and the ion pump is running properly. Your suitcase, now running on batteries, is ready for transport. The maximum power-up time on batteries is approximately 60 hours.

#### 7.2 Maintaining UHV with the ion pump disconnected

In a NexTorr D-100-5 combined NEG-Ion pump, 95% of the pumping capacity is achieved by the St172 getter alloy cartridge. Once a suitcase is baked and the getter activated as described in Section 8.3, the NEG cartridge maintains the vacuum level without any electrical power. Since the ion pump elements pumping capacity is only around 5% in comparison, in principle you can park, transport or store the suitcase under true UHV conditions even with the high voltage of the LSA pump controller switched off and disconnected. There are however two important arguments against switching the ion pump off:

- Every ion pump generates a pressure burst during power up.
- You have no control over the vacuum level when the ion pump is switched off, since the ion pump serves also as a vacuum gauge.

Details of NexTorr pump characteristics can be found on the <u>SAES website</u>.

Repeated testing over several weeks and months showed no significant increase of the base pressure in the suitcase. This applies however only if the suitcase is either connected to an evacuated buffer chamber or parked somewhere on a shelf with correctly sealed and evacuated, protective vacuum tube VSN40SBV. Please refer also to section 6.4.



IMPORTANT: The gate valve of your VSN40 is sealed with an FPM rubber gasket. There is always permeation through such gasket. Diffusion is a slow process but an increase of the vacuum level to the low 10<sup>-9</sup>mbar range will occur within around 24h in case the protective vacuum tube neither is mounted nor is the suitcase flanged to a pumped buffer chamber. For maintaining ultimate base pressure in the suitcase, keep it connected to any kind of oil-free vacuum available or install and evacuate the protective vacuum tube VSN40SBV, such that both sides of the gate valve remain under vacuum.

Connect the angle valve of the protective vacuum tube to any oil-free vacuum pump. Pump down the buffer volume and close the angle valve. A membrane pump with a base pressure around 5 mbar is sufficient.

#### 7.3 Transport of the UHV suitcase

Within the laboratory, you can carry the suitcase by hand with the LSA pump controller connected and switched on for monitoring the vacuum level. Make sure that the HV cable is well secured and does not become a tripping hazard.

For long distance transportation by car, truck or airplane, use of the professional flight case VSN40SFLIGHTCASE for protection against transport damage is strongly recommended.

Whether the high voltage of the battery driven pump controller needs to be switched off during transportation depends on local and eventually international safety regulations. Consider that the battery lifetime is limited to approximately 60 hours.



IMPORTANT: Always comply with the safety regulations for the transport of electric and electronic devices. In your country and any country where the system is shipped to. The LSA pump controller is powered with Nickel Metal Hydride rechargeable batteries. If in doubt, ask your carrier for explicit permission to transport the system with the pump controller switched on.

#### 7.4 Transport of the UHV suitcase with cryo option

When a dewar is filled with liquid nitrogen, it may only be transported over short distances within a laboratory or to a neighboring building. General safety precautions for cryogenic liquids apply. It is not permitted to transport the suitcase with filled dewar in a normal car. A UHV suitcase with  $LN_2$  filled dewar may not be packed inside any sealed enclosure.

#### 8 Maintenance

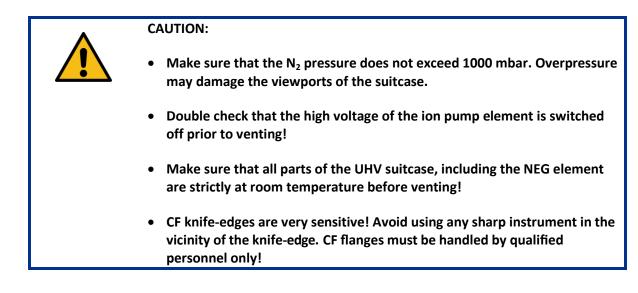
#### 8.1 Venting and pumpdown

#### 8.1.1 Venting using the protective vacuum tube valve

The suitcase is delivered with the gate valve closed and with evacuated protective vacuum tube VSN40SBV. In case the protective vacuum tube has been dismounted, re-install and pump it prior to venting.

Establish a tube connection with dry nitrogen gas flow to the angle valve of the protective vacuum tube.

Open the gate valve of the UHV suitcase, and then slowly open the angle valve of the protective vacuum tube.



#### 8.1.2 Venting using a turbo molecular pump

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For controlled venting of the UHV suitcase, establish a pumping connection between the gate valve of the suitcase and a turbo molecular pump. This is usually done by means of a CF40 flexible hose. The turbo pump can be either a mobile pumping station or the turbo pumping system installed on a load lock.

- Start the turbo pump and wait for normal operation at full speed.
- If possible, connect the vent valve of the turbo pump to a source of dry nitrogen.
- Nitrogen gas pressure must not exceed 1000 mbar.
- Make sure that the ion pump controller is switched off.
- Open the gate valve of the suitcase and subsequently turn the turbo pump off.
- Wait until the turbo pump vents automatically or vent it manually through its vent valve.

#### 8.1.3 Pumpdown

- Establish a pumping connection to a turbo molecular pump as described in section 8.1.2.
- Start the turbo pump and wait until it reaches its full speed.
- Start the pressure gauge of the turbo cart as described in the manual of the gauge.
- Wait until the ion gauge displays a vacuum level < 1 × 10<sup>-6</sup> mbar. If no pressure gauge is present, wait approximately 45 minutes.
- Press the "ON" button of the LSA ion pump controller for five seconds and wait until its power up sequence is completed.
- Confirm the vacuum reading (pump current on LSA2 units) and compare it with the displayed value of the vacuum gauge (if available).
- Proceed as described in section 8.2 when the vacuum level reaches at least 10<sup>-6</sup> mbar.



#### 8.2 Preparing Bakeout

#### 8.2.1 General procedure

Reaching vacuum levels below approximately  $5*10^{-9}$ mbar requires bakeout at a temperature above 100°C for a sufficiently long time. Your VSN40 system is factory baked for 24 hours at a temperature of 150°C and reached a confirmed base pressure in the  $10^{-10}$ mbar range according to the product test certificate. Ferrovac strongly recommends using the isolation jacket and heater VSN40SBTHE for baking the suitcase. Refer to section 8.2.2 for additional information about its usage.



CAUTION: Hot surfaces surface inside bakeout zone reach temperatures between 100°C - 150°C. Place the system on a flamenroof support and keep all flammable substance

Place the system on a flameproof support and keep all flammable substances and items well clear of the bakeout zone!

- Perform pumpdown as described in section 8.1.3 "Pumpdown". Use a pumping system that does not automatically vent in case of power failure. The latter could lead to damage of the NexTorr pump due to rapid oxidation.
- Confirm a vacuum level below  $5 \times 10^{-6}$  mbar
- Remove all non-bakeable items from the bakeout zone (i.e. plastic lids, packing material, tools, non-bakeable cables, pressure gauge electronics, etc.).
- Factory installed manipulators are bakeable up to 150°C. Do not attempt to remove magnetic couplings of manipulators prior to bakeout.
- Plug the bakeable cable for NEG activation into the matching socket of the NexTorr pump.
- Confirm that the HV cable of the ion pump element is **connected** to the LSA HV-supply. **Be sure to keep the high voltage switched off!**
- Make sure that the bakeout temperature never exceeds 150°C. Original bakeout accessories for VSN suitcases supplied by Ferrovac AG are self-limiting to safe temperatures. When using any other heating equipment, limit the temperature using a suitable temperature controller. Place its temperature sensor in a secure way on the chamber wall of the suitcase or one of its CF flanges.
- Cover all viewports and unused electrical feedthroughs with aluminum foil.
- In case no Ferrovac original isolation jacket and heater is used, install heating and isolation equipment according to you laboratory's standard procedures for baking UHV systems. Ferrovac declines all warranty for damages that may occur due to overheating if third party equipment is used for bakeout.
- Recheck that the turbopump is running normally and that the vacuum level is in the low 10<sup>-6</sup> mbar range.





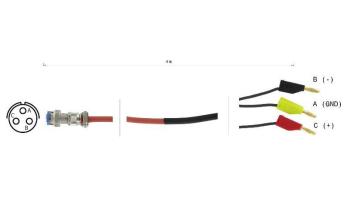


Figure 9: Bakeable NEG activation cable (Colored 4mm laboratory plugs are NOT bakeable).

- Double check that the gate valve of the VSN40 is open.
- Double check that the high voltage of the ion pump element is switched off and activate the NEG cartridge according to section 8.3.



CAUTION: Never remove magnetic couplings of manipulators for bakeout. Make sure bakeout temperature never exceeds 150°C!

#### 8.2.2 Preparing system for bake using isolation jacket VSN40SBTHE and heater VSN40HE

A tailored isolation jacket and a resistive heating element are provided for easy and reliable bakeout. Before installing the isolation jacket VSN40SBT, prepare the vacuum suitcase as described above. Make sure to use the originally supplied heater model with the correct operating voltage.



WARNING: Only use an original Ferrovac heater with the correct operating voltage. Never remove the operating voltage marking.





Figure 10: Isolation jacket placement.

- Place the suitcase onto the (long rectangular) bottom part of the jacket
- Place the heater onto the support rails of the wobblestick with the heater facing downwards as shown in image 10. Pass bakeable cables through one of the corners of the isolation jacket.
- Original bakeout equipment for VSN UHV suitcases supplied by Ferrovac is self-limiting to safe temperatures. It is however advisable to place a thermocouple on the suitcase for monitoring the temperature in the bakeout zone.
- Carefully put together all Velcro strips of the isolation jacket as shown in the figure. The little hat on top might be redundant depending on your exact suitcase configuration e.g. a sample storage with vertical manipulator or LN<sub>2</sub> dewar installed.

#### 8.3 NEG activation

After exposing a NEG pump to air, subsequent activation of the getter material is required. The same applies if the pumping speed falls below acceptable limits.

During NEG activation, the getter element is heated up to a maximum temperature of 500°C. The chamber of the suitcase warms up. Although temperature increase of the suitcase chamber is well below 100°C during NEG activation, keep the system clear of any flammable material.

During NEG activation, the UHV suitcase must be turbo pumped at all times. A lot of H2 gas desorbs from the getter alloy during the activation process, causing a pressure rise in the system well up to the  $10^{-4}$  mbar range.

Usually a full NEG activation is combined with a bakeout.

Ferrovac recommends to perform a NEG activation cycle prior to bakeout and once again at the end of the bake, during cooldown of the system.



CAUTION: Always keep the high voltage of the ion pump element off during NEG activation!

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- Plug the 4mm banana plugs of the NEG activation cable into a regulated DC lab power supply with a minimum output range of 10VDC / 5A (D-100-5 pump) or 12.5V /5A (D-200-5 pump). Operate the power supply in current limit mode. Do not exceed the maximum voltage and current limits specified by the pump manufacturer listed in table 1.
- A pressure burst up to 1 x 10<sup>-3</sup> mbar is not unusual.
- The suggested current/time values for a 100% activation of a St 172 getter alloy cartridge is given in table 1.
- If the reactivation follows normal operation in vacuum without venting, it can be shorter and carried out at lower temperature (approximately 25% lower).
- For more information, please consult the authoritative operating instructions of the pump manufacturer.

Pump model	Voltage	Current	Power	Time	Max. Current
NEXTorr®D-100-5	9 V	5 A	45 W	60 minutes	5 A
NEXTorr®D-200-5	12.5 V	4.8 A	60 W	60 minutes	5 A

**Table 1:** Table of NEG activation data for different pumps.



IMPORTANT: Never exceed the maximum applicable current for NEG activation specified in the manual of the pump manufacturer SAES getters.

#### 8.4 Performing Bakeout

#### 8.4.1 Heat up and baking cycle

After preparing the system for bakeout according to Section 8.2 and subsequent NEG activation, perform a bakeout cycle as described below.

- Start heating the UHV suitcase. The temperature inside the bakeout zone should reach 120°C 150°C within approximately 4 hours.
- At this stage, note vacuum conditions and the starting time.
- Keep the suitcase at a stable temperature between 120 and 150°C for at least 24 hours.



CAUTION: The surface of the bakeout zone reaches a temperature of 100°C - 150°C. Keep all flammable substances and items well clear of the bakeout zone.

#### 8.4.2 Cooldown

- Turn off heater.
- During cooldown of the UHV system a second NEG activation cycle (section 8.3) must be performed, ideally while the system is still warm at a temperature of around 80°C.
- Subsequently let the system cool down to about 40°C until you can touch the handle of the gate valve.
- Checking the vacuum gauge of the Turbo cart, confirm that the vacuum level is in the  $10^{-8}$ mbar range.



CAUTION: Do not touch the handle of the gate valve without checking if its temperature is below 40°C. Use a laboratory thermometer.

- Start the ion pump element by pressing the LSA "ON" switch for 5 sec. The display of the LSA controller might temporarily show "overrange". After a few seconds, a vacuum reading comparable to the value measured with the ion gauge of the turbo cart is displayed.
- Confirm that the vacuum level displayed by the LSA is in the same order of magnitude as the ion gauge reading.
- Degas the manipulator(s) by moving them forth and back while the system is still warm.
- Close the gate valve as soon as the vacuum in the suitcase becomes better than the vacuum in your turbo cart.
- Wait for approximately 24 hours and let it settle until ultimate base pressure is reached.
- During "settle in" keep the turbo pump connected and running, even though the gate valve is closed.
- After bakeout and NEG activation the system is ready for operation. Please return to chapter 5 "Operation" or 6 "Installing the suitcase to a UHV system".
- In case the suitcase is not immediately used, reinstall the protective vacuum tube and evacuate it using a membrane pump.

#### 8.5 **NEG flashing**

After some time of normal use, saturation effects of the NEG element may be the reason for an increase of base pressure in the suitcase. If and when this happens, depends on what the getter element "experiences" during sample transfer, namely on the vacuum level it was exposed to while attached to the intermediate chamber or T-piece with the gate valves open.

Provided the UHV suitcase was not exposed to air but a decrease in pumping performance is observed, a shorter reactivation cycle (so called "NEG flash") of the getter element is advisable. September 2021



A NEG flash is the same procedure as described in section 8.3 "NEG activation" but for a shorter time and carried out at lower temperature. We recommend flashing the getter material with a heating power of 32W for 30 minutes for a D-100-5 and 42W/30min for a D-200-5 pump.

The NEG flash procedure does reactivate the getter surface, while not releasing all the absorbed Hydrogen from its bulk material.

#### 8.6 Periodic functional inspection and preventive maintenance

#### 8.6.1 Cabling

Check the cabling regularly for damaged insulation or sharp kinks.

Shield resistance between the outer metallic parts measured between the two plugs must be below 100 m  $\!\Omega\!.$ 



WARNING: Any damaged cabling is not fit for purpose and has to be replaced immediately.

#### 8.6.2 Transfer manipulator

For maintenance of the transfer and storage manipulators, please refer to their respective manuals. It is recommend inspecting the haptics of the manipulators regularly. Their motion should feel smooth, without excessive force and without metallic scratching. If a scratching noise is observed a factory overhaul of the manipulator is required.

A preventive exchange of the slide bearings every five years is recommended.

## 9 LN<sub>2</sub> cryo cooling option

This section only applies to suitcases with cryo cooling. For cold and ultraclean storage and transport of samples, the UHV suitcase is equipped with a liquid nitrogen  $(LN_2)$ <sup>c</sup> dewar. Cold thermal shields surround the sample holders and a cold block thermally links to the specimens parked.

The cryo version is meant to transport sensitive, mainly biological and usually vitrified specimens at temperatures close to  $LN_2$  temperature and at the same time under true UHV conditions. The cryo shield protects the sample from the influence of the outer warm surfaces. Additionally the sample is actively cooled by either the cold sample grabber or a cooling block inside the shields. A Pt100 sensor is mounted on the shields for temperature monitoring.

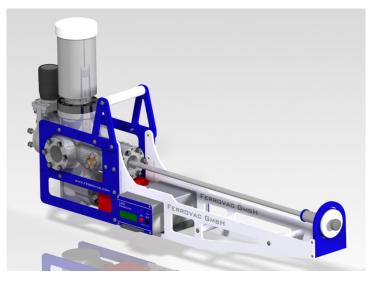


Figure 11: Cryo-UHV suitcase with LN<sub>2</sub> dewar.



WARNING: When using liquid nitrogen it is important to respect the safety measures for cryogenic liquids and nitrogen gas. Proper training for handling liquid nitrogen is needed.

Specimen transfer in the cryo cooled UHV suitcase must be done with great care. Thermal shields, sample grabber and sample cooling are precisely linked to optimize thermal contact while internal space is limited.

For retracting the transfer arm to its parking position, it is important to align the marking/screw of the magnet upright in order to match the guidance pin of the end piece.



CAUTION: Ensure correct orientation of the transfer manipulator when retracting to the parking position.

The screw/marking of the outer magnet needs to be kept strictly upright to move it into its guided end position.



#### 9.1 Cryo-cooling

Filling the dewar with liquid nitrogen will cool down the shields and sample parking station inside the chamber to temperatures below -150°C.

CAUTION: Always use the personal protective equipment (PPE) i.e. safety goggles and gloves when handling cryogenic liquids. Wear long sleeves/pants and stable closed shoes. Make sure a proper ventilation of the room is given.

Prepare a small portable dewar filled with approximately 1 liter  $LN_2$ . Pour  $LN_2$  slowly into the dewar of the Cryo-UHV suitcase. It is not recommended to fill the suitcase dewar directly from a pressurized  $LN_2$  cylinder since a lot of  $LN_2$  might spill. Be careful not to spill excessive  $LN_2$  over the UHV suitcase as this may crack the viewports by thermic shock.

Allow a cooldown time of 30-40 min until the shields reach their base temperature. The shields remain at a stable base temperature as long as there is  $LN_2$  in the dewar.

Place the lid onto the dewar. For transporting the suitcase while cold, it is strongly recommended to use a trolley. Move with great care, in order not to spill any liquid nitrogen.

#### 9.2 Temperature sensor

A Pt100 resistor inside the chamber measures the temperature on the sample-cooling block or shield (tolerance class A, DIN EN 60751 F 0.15:  $0.15 + 0.002 \times |T|$  with T in °C).

Pt 100 resistors have a resistance of  $100 \Omega$  at 0 °C. The calibration curve is well known (in °C):

$R(T) = R_0(1 + A \times T + B \times T),$	$O^{\circ}C - 850^{\circ}C$ (1)	
$R(T) = R_0 (1 + A \times T + B \times T_2 + C \times [T - 100] \times T_3),$	$-200^{\circ}C - 0^{\circ}C$ (2)	

With  $R_0 = 100\Omega$ ,  $A = 3.9083 \times 10^{-3} \circ C^{-1}$ ,  $B = -5.775 \times 10^{-7} \circ C^{-1}$  and  $C = -4.183 \times 10^{-12} \circ C^{-1}$ .

Four-wire temperature measurement with a current of approximately 1 mA is recommended. Fig. 13 shows the pin layout for the feedthrough.

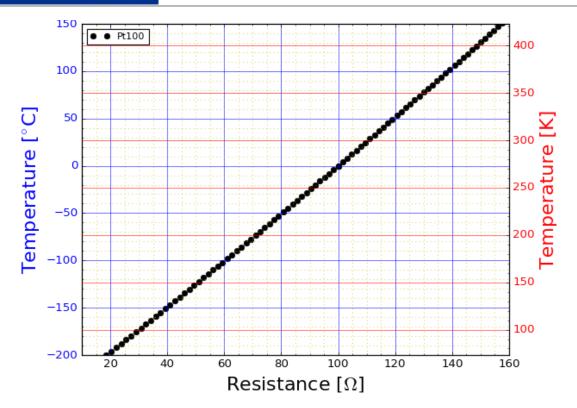
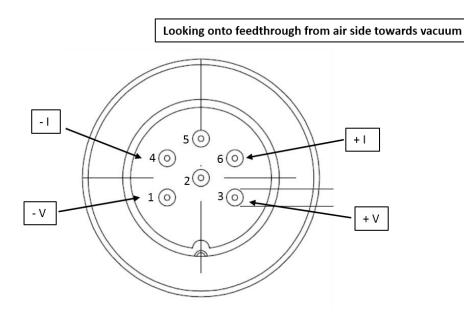


Figure 13: Temperature versus resistance curve for a Pt100 resistor.

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# Cable layout Allectra CM 6-Pin feedthrough for temperature measurement with Pt-Sensor



Name	Туре	Spec	PIN on Allectra	Notes
-	Resistor current	About 1mA	#4	red
+	Resistor current		#6	yellow
- V	Resistor voltage	About 0.1V to 5.0V	#1	green
+ V	Resistor voltage		#3	blue

+ I/V cables on same resistor leg, - I/V cable on same resistor leg.

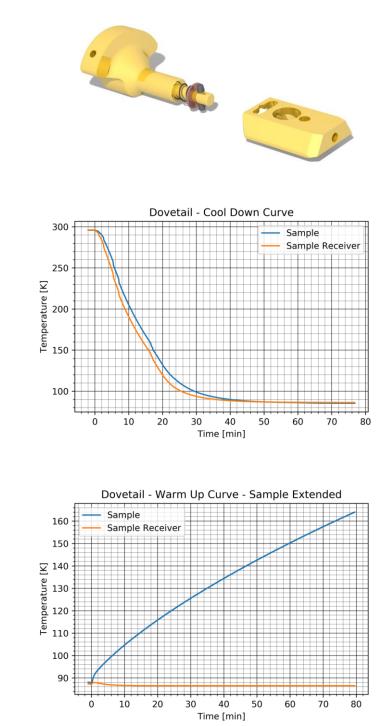
Figure 14: Pinout of the feedthrough for the Pt100 resistor connection with four-wire measurement.

#### 9.3 Cryogenic tests of selected sample holders and transfer types

Whenever samples are highly sensitive to heat and surface contamination, keeping the temperature as low as possible may be crucial. Exposure to non-cooled areas and surfaces must be avoided or kept as short as possible (Be aware that the inner setup and the thermal coupling of the sample holder to the shields varies with the type of sample holders.) Within the shields, the sample is protected from warm surfaces and heat irradiation. Once the sample is moved away from the cold storage and outside the shields, it will warm up due to the heat impact. In addition, atoms/molecules in the warm space might condense on the sample surface. Therefore, a fast transfer within a few minutes or even seconds is advisable. In this sub-chapter, cool down and warm up curves for specific transfer types provided by Ferrovac AG are presented.



#### 9.3.1 Dovetail Sample Holder (CuBe)



#### c)

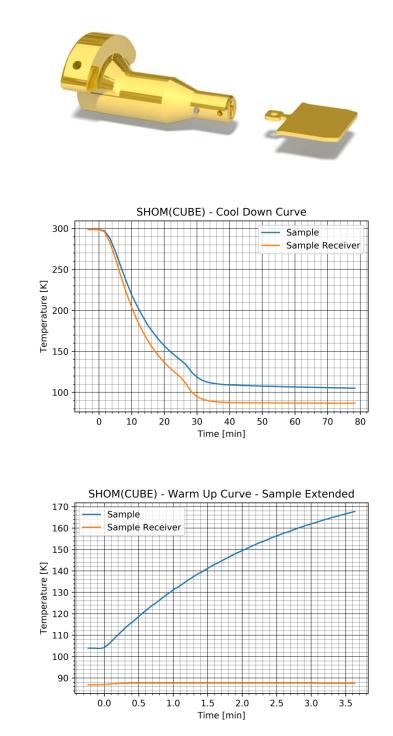
a)

b)

**Figure 15:** Cool down – and warm up curves for the dovetail sample holder: a) representation of sample holder (CuBe) and end-effector (Au plated CuBe) used in the tests. b) Cool down curve for both sample and sample receiver. Both reach their base temperatures (sample: 86K, sample receiver: 86K) within 50min. c) Warm up curve of the sample when transferred. The sample reaches the often critical temperature of 110K after 11.5min.



#### 9.3.2 Omicron Sample Holder (Au plated CuBe)



a)

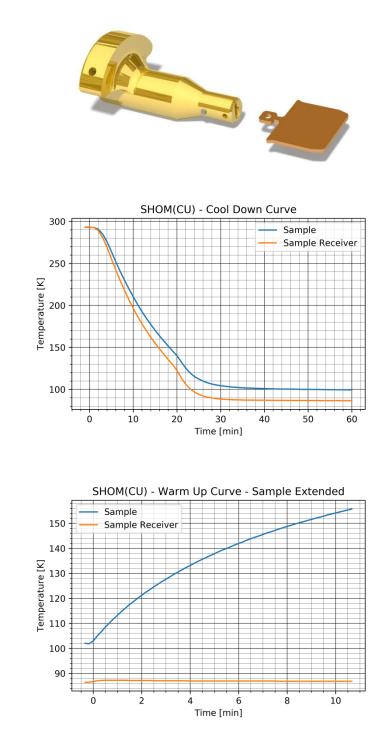


c)

**Figure 16:** Cool down – and warm up curves for the Omicron (CuBe) sample holder: a) representation of sample holder (Au plated CuBe) and end-effector (Au plated CuBe) used in the tests. b) Cool down curve for both sample and sample receiver. Both reach their base temperatures (sample: 104K, sample receiver: 87K) within 55min. c) Warm up curve of the sample when transferred. The sample reaches the often critical temperature of 110K after 15sec.



#### 9.3.3 Omicron Sample Holder (Cu)



a)



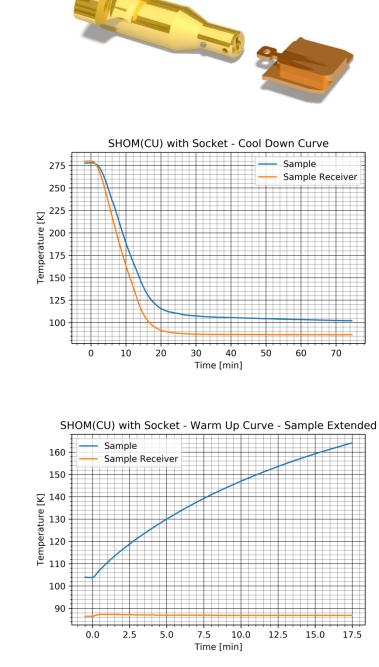
c)

**Figure 17:** Cool down – and warm up curves for the Omicron (Cu) sample holder: a) representation of sample holder (Cu) and end-effector (Au plated CuBe) used in the tests. b) Cool down curve for both sample and sample receiver. Both reach their base temperatures (sample: 101K, sample receiver: 87K) within 40min. c) Warm up curve of the sample when transferred. The sample reaches the often critical temperature of 110K after 40sec.



#### 9.3.4 Omicron Sample Holder (Cu with extra socket)





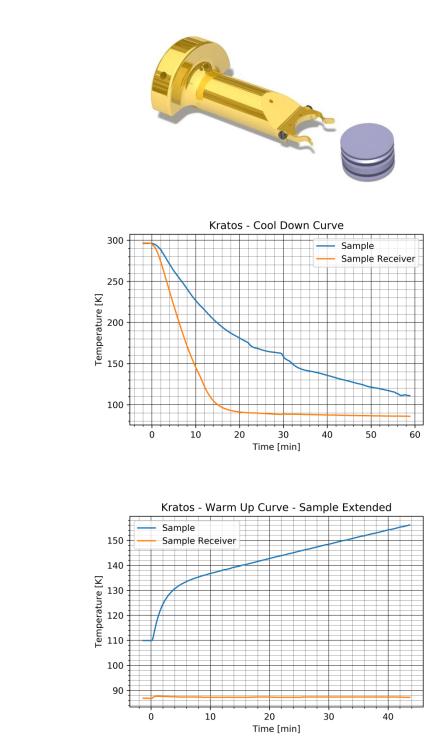
c)

b)

**Figure 18:** Cool down – and warm up curves for the Omicron (Cu) sample holder with an extra socket (Cu): a) representation of sample holder (Cu) with socket and end-effector (Au plated CuBe) used in the tests. b) Cool down curve for both sample and sample receiver. Both reach base temperatures (sample: 106K, sample receiver: 87K) within 40min. c) Warm up curve of the sample when transferred. The sample reaches the often critical temperature of 110K after 55sec.



#### 9.3.5 Kratos Sample Stub (Al)



a)

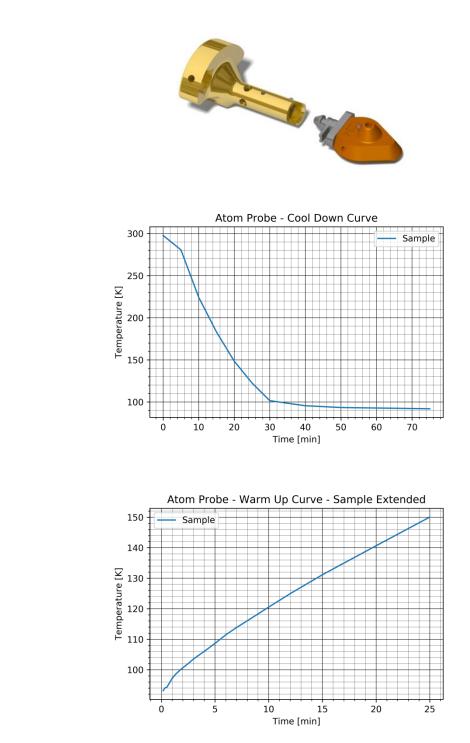




**Figure 19:** Cool down – and warm up curves for the Kratos (Al) sample stub: a) representation of sample holder (Al) and end-effector (Au plated CuBe) used in the tests. b) Cool down curve for both sample and sample receiver. Both reach base temperatures (sample: 111K, sample receiver: 88K) after 58min and 40min, respectively. c) Warm up curve of the sample when transferred.



#### 9.3.6 Atom Probe Puck (CuBe)



a)

b)

## c)

**Figure 20:** Cool down – and warm up curves for the Atom Probe (CuBe) puck: a) representation of sample holder (CuBe) and end-effector (Au plated CuBe) used in the tests. b) Cool down curve of the sample. The sample reaches its base temperature (95K) after 40min. c) Warm up curve of the sample when transferred.

## **10 Options and Upgrades**

Options and upgrades for the UHV suitcase are listed below. For details, visit our website www.ferrovac.com.

#### 10.1 Upgrade to 200 l/s nominal pumping speed

Product code: UPGRADEVSN40S(+D200)

NexTorr D-200-5 combined NEG-Ion pump with 200 I/s pumping speed. The 5 I/s ion pump element remains identical.

#### **10.2** Additional CF-ports

Add additional CF flanges to vacuum chamber at the time of order:

#### 10.2.1 Service port DN40CFT

Product code: UPGRADEVSN40S(+SP40) Additional DN40CF flange, tapped M6 for a vacuum gauge, electrical feedthrough, etc. Not compatible with rear view port option UPGRADEVSN40S(+RV).

#### **10.2.2** 2x Service port DN16CFT

Product code: UPGRADEVSN40S(+SP2X16) Two additional DN16CFT service ports for installing electrical feedthroughs. Mandatory for cryo configurations.

#### 10.2.3 Rear view port

Product code: UPGRADEVSN40S(+RV) The RV option provides a better view during sample exchange. Strongly recommended if the configuration includes a multiple sample storage. Not compatible with SP40 or cryo options.

#### **10.3** Bakeout isolation jacket and heater module

Product code: VSN40SBTHE-XXXX

Multi-piece isolation tent with Velcro<sup>™</sup> closures. Material: Two layers of glass fiber fabric with aluminum coating and an insulating interlayer. A resistive heater module featuring a K-type thermocouple for temperature measurement is included.

#### 10.4 Flight case

Product codes: VSN40SFLIGHTCASE-XXXX-YYYY and VSN40MFLIGHTCASE-XXXX-YYYY Professional Flight case for travelling around the world with your NexGeneration UHV Suitcase.

#### **10.5** Docking accessories

#### **10.5.1** Fast pump down dock with LN2 cold trap

Product code: VSCTH40, VSCTDH40

Our docking chambers are optimized for fast pumpdown to an acceptable vacuum level for clean sample transfer ( $^{2}10^{-8}$  mbar) without baking. In addition to a directly mounted turbo pump, an LN<sub>2</sub> cold trap condenses water vapor, effectively "boosting" pumpdown.

#### 10.5.2 Compact DN40CF T-piece

Product code: VSTEE40 Compact DN40CF T-piece used as buffer volume for UHV suitcases. Insertion length: 90mm

#### 10.5.3 Ultra-Compact DN40CF Cross

Product code: VSCROSS40RL80

Ultra compact DN40CF cross used as buffer volume between a UHV-system and a vacuum suitcase. Insert length 80mm, with a port for pumpdown using a turbo pump and an additional CF40 for a vacuum gauge.

#### **11** Additional information

#### **11.1** Return of defective items

Ferrovac AG requires a completed declaration of contamination form and will issue an RMA (Return of Materials Authorization) form, before any items are factory returned. Please contact us beforehand. You will be given an RMA number and information on how to proceed with the return of defective items.

#### 11.2 Downloads

The latest version of this manual can be downloaded from our website Ferrovac.com. For any suggestions or questions concerning this manual, please don't hesitate to contact us.

Ferrovac AG Thurgauerstrasse 72 8050 Zürich Phone +41 (0) 44 273 16 38 E-Mail: sales@ferrovac.com