



# Thermo Scientific ARL EQUINOX 100 X-Ray Diffractometer

## Instruction Notice

AA83838 - C • March 2019

# 1 Revision Table

Revision	Date	ECO number	Description of Changes
A	04-JUN-2018	SDR14163	Initial Release
B	21-JUN-2018	SDR15489	Minor update done.
C	11-MAR-2019	SDR17946	Appendix 5 - Gandolphi sample holder removed.
		SDR18055	Co X-Ray limit changed from 0.2 mA to 0.3 mA.

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**Note**      **This document is original manual.**

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The information in this document is subject to change without notice.

We assume no responsibility for any errors that may appear in this document.

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## 2 Introduction

ARL EQUINOX X-Ray diffractometers are designed for measuring of crystalline samples (powder, bulk, metals, thin layers, etc...), phase identification, nanoscale structural analysis, non-destructive quality control, as well as for many other applications.

This instrument is intended for professionals. The user is advised to read the following instructions and any other additional information supplied by Thermo Fisher Scientific before operating the instrument.

Thermo Fisher Scientific declines all responsibilities for improper use by unauthorized personnel or non-authorized operation of the instrument.

For any additional information, contact Thermo Fisher Scientific.

### Supplied Items

Before the shipment, ARL EQUINOX X-Ray diffractometers are tested and packed carefully. However damage can occur during the transport.

If the instrument or any additional supplied item has been damaged during transport, or if you notice any signs of a possible damage, please contact both the transport company representative and the Thermo Fisher Scientific representative as soon as possible.

Furthermore, if you notice that any part is missing or you have any questions about the delivered items, contact the Thermo Fisher Scientific representative.

### Storage

The storage where the instrument is stored has to meet following ambient specifications:

Temperature: 0 – 50°C, relative humidity: < 95% (without condensation).

### Assistance

The Thermo Fisher Scientific worldwide technical support network consists of highly trained field service engineers, technical support specialists and service coordinators who are ready to quickly assist the customers with answers and solutions to service needs and application questions. If you need any help, please contact the local Thermo Fisher Scientific representative.

### Manufacturer

Thermo Fisher Scientific Brno s.r.o.

Vlastimila Pecha 12, Brno 627 00

Czech Republic

## 3 Safety

### 3.1 General Safety Guidelines

- Before using your ARL EQUINOX X-Ray diffractometer, read this instruction manual completely and understand it. Please take notice of all warnings, cautions and notes to ensure the correct and safe functioning of the instrument.
- Never place the instrument near an open flame or in an excessively hot environment. The instrument must not be installed in an ATEX explosive hazard area.
- Never place the instrument in locations where excessive moisture or corrosive materials are present.
- The instrument construction provides protection against the risk of electrical shock by grounding the appropriate metal parts. The protection may not work properly unless the power cord is connected to a properly grounded socket. It is customer's responsibility to assure that a proper ground connection is provided.
- Never use flammable or corrosive fluids with this unit.
- Never use gases other than described in specifications.
- Never block the fan grid or the bottom of the instrument to allow a proper ventilation.
- If the instrument is used in a manner not specified by the Thermo Fisher Scientific, the protection provided by the instrument may be impaired.
- Only accessories that comply with Thermo Fisher Scientific specifications are allowed to be used with ARL EQUINOX X-Ray diffractometers.
- Transport of the instrument should be done with extreme care and should be done with an appropriate lifting system, ideally a forklift truck with forks height not exceeding 115 mm.
- Never operate a damaged or a leaking instrument.
- Always turn the instrument OFF and disconnect the power cord from the power source before performing any service or maintenance tasks, or before moving the instrument.
- Never operate the instrument with a damaged power cord.
- Never turn the instrument ON without a proper cooling fluid.
- Install the instrument on an appropriate table and make sure that the instrument can not move horizontally.

## 3.2 Hazard Statements and Symbols

The following messages are used throughout the manuals to highlight information.

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**Note** A Note message indicates the information in it requires special attention.

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**CAUTION!**



A Caution message indicates a potentially hazardous situation that, if not avoided, may result in moderate or minor injury. It may also be used to alert against unsafe practices.

---

**WARNING!**



A Warning message indicates a potentially hazardous situation that, if not avoided, may result in death or severe injury.

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**DANGER!**



A Danger message indicates an imminently hazardous situation that, if not avoided, will result in death or severe injury.

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**WARNING!**



Radiation danger – Indicates that X-Rays hazard may be present. Take necessary precautions.

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**SERVICE!**



A Service message indicates that the described procedure should be performed by qualified personnel only.

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## 3.3 Symbols on the Instrument

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**Note** The symbol of electrical danger is located on the rear panel.

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**DANGER!**



A danger of a potential risk of electrical shock in case of operation made on components placed behind the rear panel. Any task must be performed by qualified personnel only.

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### 3.4 Emergency Stop

In case of any problem (electrical, radiation or mechanical) there is an emergency stop button located on the front panel of the instrument. Pushing the emergency button immediately removes the power to the instrument, including the X-Ray generator. Be aware, that pushing the emergency stop button will result in communication loss between the control computer and the instrument.

**CAUTION!** The emergency stop should not be used for routine shut down of the generator since repeated rapid power shut downs may dramatically reduce the lifetime of your X-Ray tube and damage the X-Ray generator.

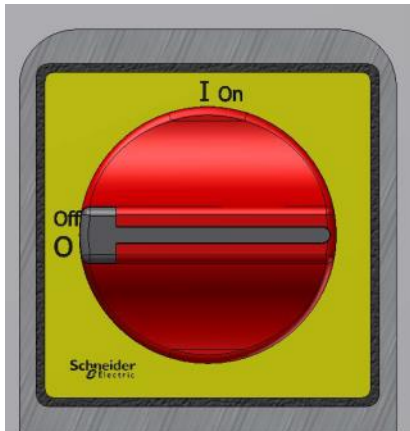


### 3.5 Electricity



The symbol of electrical danger is present on the left of the appliance inlet on the rear panel.

The switch disconnecter located on the rear of the ARL Equinox 100 allows disconnecting the power supply from the mains. The position “0” “Off” means that the system is disconnected from the mains. The position “I” “On” means that the system is connected to the mains.



### 3.6 High Voltage Generator

**DANGER!** DANGER OF DEATH BY ELECTROCUTION:



Any tasks on the electrical equipment (high voltage generator, electronics...) must be performed by certified and qualified personnel only.



All energized parts are accessible with specific tools only. Any service tasks must be performed only when the instrument is unplugged and by qualified personnel only. All conductive parts are usually insulated from the energized parts and are grounded to each other.

A safety beacon indicates that the X-Ray generator is in operation.

### 3.7 Protection Enclosure and X-Ray Tubeshield

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**WARNING! WARNING IONIZING RADIATION:**



**X-Ray is ionizing radiation which has effects on living tissue and on the organism. While the X-Ray generator is in operation and the shutter is open, no access inside the enclosure is permitted.**

---

Both the enclosure and the tubeshield of the ARL X-Ray diffractometer fulfill the function of a protection shielding against the ionizing radiation. The enclosure surrounds the instrument and helps to attenuate the diffused radiation emitted by the X-Ray tube. The X-Ray tube is mounted in a tubeshield which is the most important part of the X-Ray protection. The optics and collimation enable forming the X-Ray beam aperture to a directional beam. Never use the instrument with a damaged enclosure, tubeshield or optics line!

Control circuits are designed to monitor the state of the shutter (opened or closed) and adjust them accordingly while the generator is running.

Safety interlocks located on the enclosure's door enable to lock the door during the emission of X-Rays in the analysis area.

### 3.8 Security Beacons

The ARL Equinox 100 X-Ray diffractometer is equipped with two security beacons located on the right side of the front cover. The orange beacon "X-RAY" indicates that the X-Ray generator is in operation and emitting X-Ray radiation. The red beacon indicates that the shutter is opened and the X-Ray radiation is emitted inside of the enclosure.



Additional red LEDs to indicate the state of the shutter are located on the shutter as well (red LEDs ON represents an opened shutter).

In case of a malfunction of the safety beacons the voltage of the X-Ray generator will be limited and/or the shutter will remain closed.

## 3.9 Gas

The ARL EQUINOX 100 uses a mixture consisting of 85% of argon and 15% of ethane . A gas cylinder of 1 liter under 150 Bar can be installed in the system. During the operation of the instrument, a micro-leakage of about 30 to 40 mm<sup>3</sup> of the detector gas may be released every 20/30 seconds from the GAS OUT valve located at the rear of the instrument. This is a standard behavior.

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**WARNING!** Exposure to high concentrations of the detector gas may cause asphyxiation.



**Symptoms of the asphyxiation may include loss of mobility and/or loss of consciousness. The victim of the exposure of high concentrations of the detector gas may not be aware of the asphyxiation. In such a case, remove the victim from the contaminated area wearing self-contained breathing apparatus. Keep the victim warm and rested and seek medical assistance. Apply artificial respiration if the victim stopped breathing.**

**Ingestion is not considered as a potential route of exposure.**

**There are not any known toxicological effects related to this product.**

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## 4 General Information

### 4.1 ARL EQUINOX 100 Front View

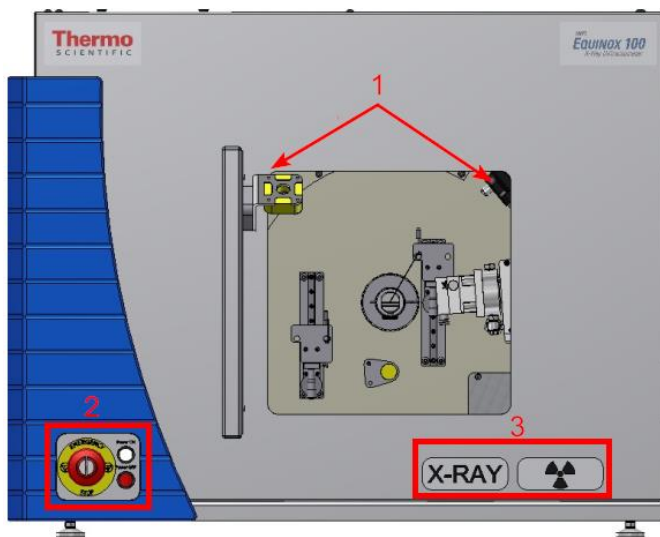


Height: 550 mm

Width: 750 mm

Depth: 560 mm

Weight ~80 kg



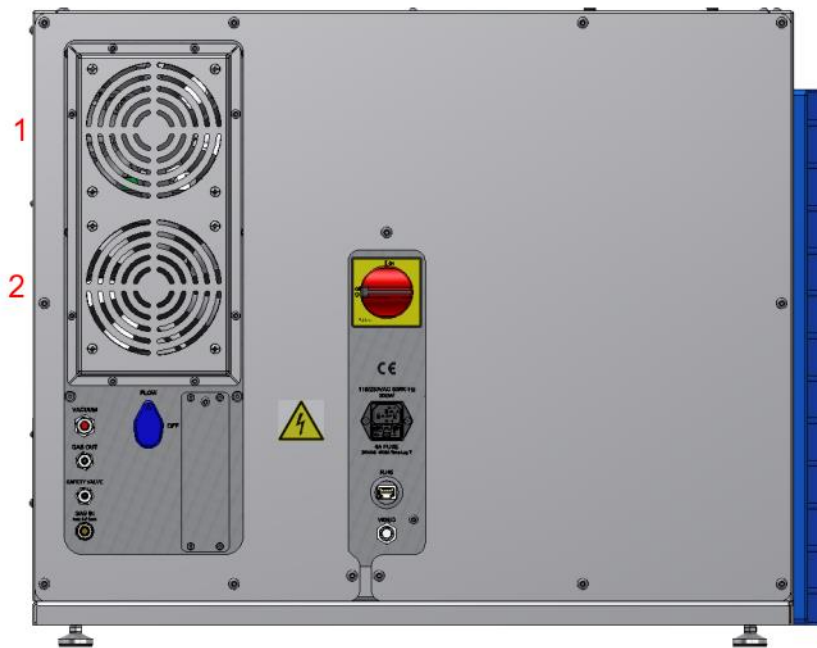
1 - Door locking system (safety interlock)

2 - Front panel

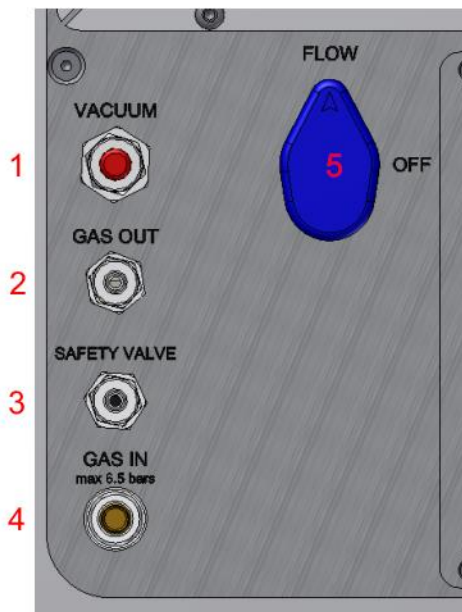
3 - Security beacons

The door can be opened by pulling. The door is blocked while the instrument is in operation, the door lock is released when the shutter is closed.

## 4.2 ARL EQUINOX 100 Rear View



- 1 - Heat exhaust fan
- 2 - Water cooling fan



- 1 - Vacuum connection for the mirror
- 2 - Gas outlet from the detector
- 3 - Gas outlet from the safety valve
- 4 - Gas inlet (for external gas cylinder) max 6.5 Bar
- 5 - Outlet gas valve
- 6 - Main breaker
- 7 - Power cord connector with a fuse
- 8 - RJ45 plug
- 9 - BNC video

**SERVICE!**

The connection of the instrument should be done by qualified personnel. The safety valve hose should never be extended using any tubing device consisting of components able to block the tubing.



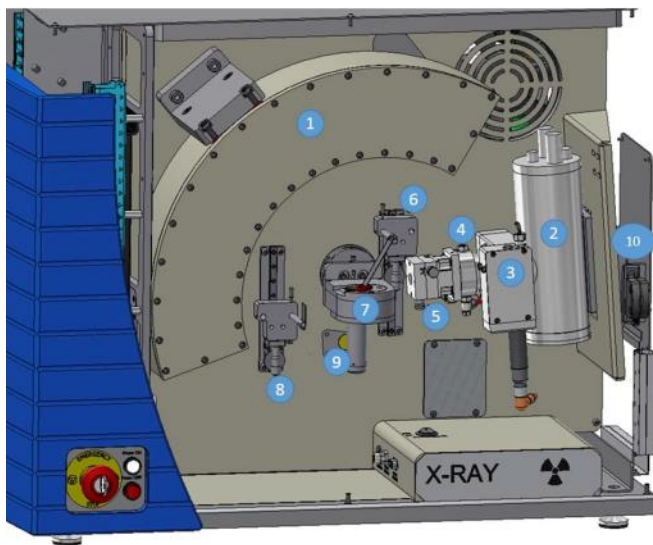
- Power: The electrical connection should be done by qualified personnel only.
- Vacuum: It is recommended to use a peristaltic pump in order to achieve vacuum in the optic chamber. This will minimize the background noise and preserve the quality of the layering of the mirror.
- Gas in: Gas in allows to connect the instrument to an external gas cylinder.
- Gas out: Gas out is the exhaust of the detector flush. By using the "flow valve" it is possible to close the flushing and lower the gas consumption.
- RJ45: The RJ45 connector serves to connect the instrument to a computer using the TCP/IP protocol.

**SERVICE!** The service engineer can connect the X-Ray video for beam alignment. The use of this connector is explained in the Service Guide.



### 4.3 ARL EQUINOX 100 Inside View

Sensitive components are protected by appropriate sensors to avoid radiation exposure: optical line assembly, alignment tooling, door and panels. The picture describes the main goniometric components of the instrument.



- 1 - CPS180, curved position sensitive detector
- 2 - Low power X-Ray source
- 3 - Shutter
- 4 - Elliptical mirror
- 5 - Crossed slits
- 6 - Anti-scattering knife
- 7 - Sample holder (SSRT type)
- 8 - Beam stop
- 9 - Safety sensor for service tasks
- 10 - Source alignment system

The operator is allowed to configure following parameters:

- beam size, by adjusting the crossed slits (5),
- anti-scattering knife, to optimize beam/signal ratio (6),
- beam-stop height, to optimize low angle detection range (8),
- sample holder inclination, to set the appropriate incidence angle,
- sample holder type in order to choose the appropriate analysis (transmission, reflection, thin film, ...). There are several sample holders available (see the Appendix 5: Sample Holders for ARL EQUINOX 100 on page 41 for further information).

### SERVICE!

**In service mode, appropriate tools are used by the qualified personnel. Sensor (9) allows to detect the service tooling.**



**Only the field service engineer is allowed to adjust the mirror housing (4) and perform the beam centering procedure, dismantle the CPS detector (1) and the X-Ray source and its components: slits (5), mirror housing (4), shutter (3) and the tubeshield (2).**

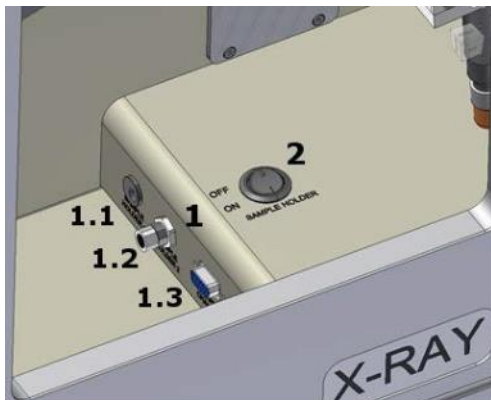
## 4.4 Front Panel Information

There is a panel with an emergency stop button, a Power ON button and a Power OFF button located on the front of the instrument. Please, see the section Operation on page 25 for further information.



## 4.5 Power Supply for Sample Holders

The power supply plug for the spinning sample holder is located below the sample area.



1. Set of plugs to connect the sample holder.
  - 1.1 A jack connector for the 12V power supply for spinning the sample holder.
  - 1.2 A 4 pin circular connector for the thin film sample holder
  - 1.3 A Sub D9 connector for the 6 positions sample changer
- 2 The switch to turn ON the sample holder power supply.

## 4.6 Selector Switch with Key

**SERVICE!** 3- Connector for safety sensors for service purpose only.



4- Video BNC connector for beam alignment.

5- Bypass key for service purposes.



The 3 positions selector switch with the key for maintenance and alignment mode must be used by Thermo Fisher Scientific FSE or other qualified personnel only. This key allows to activate the maintenance mode by adapting the safety loop for receiving the service tooling.

## 5 Connection Specifications

### 5.1 Electrical Connection

ARL EQUINOX X-Ray diffractometers have an Ingress Protection rating of IP20. This means that they are protected against the ingress of solid particles with a diameter over 12 mm (e.g.: a finger). ARL EQUINOX X-Ray diffractometers do not have any protection against the ingress of liquids.

All connections to the instrument have to comply with class SELV (Safety Extra Low Voltage) according to norm EN 61010-1:2010.

Equipment	Voltage (V)	Frequency (Hz)	Phase	Power (W)	Max consumption (A)
ARL EQUINOX	110/230 $\pm$ 10%	50/60	1	300	
Computer	100/240	50/60	1	250	
Screen	100/240	50/60	1		1

The ARL EQUINOX 100 complies with overvoltage category II (OVCII).

The ARL EQUINOX 100 complies with insulation class 1.

### 5.2 Cooling Fluid

The cooling circuit is integrated directly to the instrument. The composition of the cooling fluid is based on a mixture of distilled water and 6% of ethylene glycol.

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**Note**      **The cooling fluid level check should be performed regularly.**

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### 5.3 Gases

ARL EQUINOX curved position sensitive detector (PSD) belongs to the family of gaseous ionization detectors. A gas cylinder has to be constantly connected to the instrument in order to enable stable and optimal working conditions for a long term use.

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**DANGER!**

**FLAMMABLE GAS**



**The gas mixture contains ethane which is a flammable gas. Never place the instrument near an open flame or in an excessively hot environment. The instrument must not be installed in an ATEX explosive hazard area**

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ARL EQUINOX X-Ray diffractometer can be equipped with a gas cylinder integrated directly to the instrument. In this case, the cylinder should not exceed given dimensions based on the specific instrument. Our gas regulator is equipped with a safety valve adjusted to 6.5 Bar. In case of overpressure, a tubing allows to release the gas outside of the instrument.

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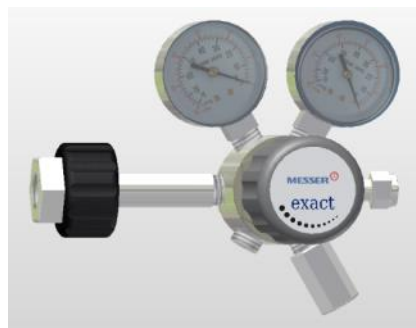
**Note**      **Always use a gas regulator with the instrument.**

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Maximum dimension of the gas cylinder:

Diffractometer model	Height – Valve included- (mm)	Diameter (mm)	Recommended Gas Regulator
ARL EQUINOX 100	380	85	Messer (type FM53 with indicators positioned next to each other)

If the gas cylinder cannot be installed as a part of the instrument, please follow the instructions in Section Installation of an External Gas Cylinder on page [23](#).



### 5.3.1 Specifications of the Detector Gas

Specification Parameter	Value, Comments
Gas	Argon / Ethane (C <sub>2</sub> H <sub>6</sub> ) Argon: Q.S. N56 Ethane : 15%+/-0,3% Ethane N35
Detector high voltage	9.8 kV
Gas Pressure	6 Bar
Gas Flow	450 mm <sup>3</sup> /min +/- 100 mm <sup>3</sup> /min (i.e. 2 bubbles/min)

### 5.3.2 Gas Impurities Specifications

The impurity levels are given in volumetric [ppm].

Argon > 99.9996 %	Ethane (C <sub>2</sub> H <sub>6</sub> ) > 99.9 %
O <sub>2</sub> < 0.5	O <sub>2</sub> < 10
H <sub>2</sub> O < 2	H <sub>2</sub> O < 5
N <sub>2</sub> < 0.5	N <sub>2</sub> < 40
H <sub>2</sub> < 0.01	H <sub>2</sub> < 5
C <sub>n</sub> H <sub>m</sub> < 0.2	C <sub>n</sub> H <sub>m</sub> < 20
CO+CO <sub>2</sub> < 0.1	CO <sub>2</sub> < 10
	C <sub>2</sub> H <sub>4</sub> < 400
	CH <sub>4</sub> < 10

### 5.3.3 Environment

The instrument is rated as a Pollution Degree 2 (PD2) equipment. For disposal, follow the legal requirements of your country.

## 6 Installation

### 6.1 Location

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**WARNING! LIFTING HAZARD!**

**When installing the EQUINOX 100, in the next step, either use lifting instruments or four person lift as the EQUINOX 100 weights 80 kg.**

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The ARL EQUINOX X-Ray diffractometer is designed for indoor use only at an altitude level below 2,000 m above sea level. The ARL EQUINOX X-Ray diffractometer must be placed in a clean environment with constant ambient temperature. We advise an air conditioned room where the room temperature is kept between 15 °C and 30 °C. The room must be ventilated sufficiently. The relative humidity has to be in range from 20% to 80% without condensation.

- Never place the instrument near an open flame or in an excessively hot environment. The instrument must not be installed in an ATEX explosive hazard area.
- Never place the instrument in locations where excessive moisture or corrosive materials are present.

The ARL EQUINOX X-Ray diffractometer should be positioned in such a way so there is a minimum clearance of 50 cm on each side of the instrument in order to keep access to the main breaker easily.

Excessively dusty areas should be avoided and a periodic cleaning schedule should be instituted.

The instrument should be placed on an appropriate surface. Accidental motion should be avoided.

### 6.2 Assembly Instructions of Diffractometer's Parts

Installation, adjustment procedures and calibration should be performed by Thermo Fisher Scientific service engineers only or by personnel certified by Thermo Fisher Scientific. Otherwise, Thermo Fisher Scientific does not take any responsibilities for any malfunction or damage caused by unauthorized installation of the instrument.

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**WARNING! During any assembly operation, the ARL EQUINOX X-Ray diffractometer must be shut down and disconnected from the power source!**

## 6.3 Gas Line Installation

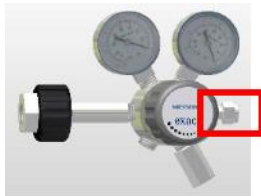
The following assembly operations should be performed only by Thermo Fisher Scientific service or one of its representative or properly trained users by Thermo Fisher Scientific.

If the gas cylinder cannot be installed that way inside the instrument, please follow the instructions in the Installation of an External Gas Cylinder on page 23 where you will find the procedure to install the cylinder outside of the instrument.

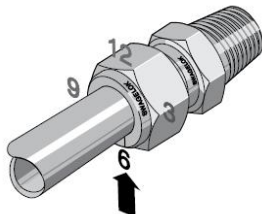
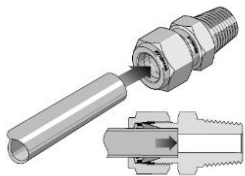
First, you will have to install the gas regulator on the gas cylinder. Simply screw the threaded fitting of the gas regulator on the gas cylinder (pay attention to the way of tightening it, the threads are reversed compare to a classical thread). Tighten it well in order to avoid any leaks but not too hard to avoid damage of the threads. Apply appropriate procedure for detecting gas leakage.



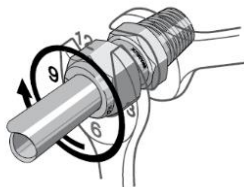
A short hose of  $\varnothing 6$  mm diameter with a male quick fitting already mounted is shipped with the instrument.



Connect the hose to the gas regulator. The connector is a standard Swagelok gas connector.



Insert the hose to the gas regulator outlet (without dismantling the nut) and tighten the nut by hand. Then, by using a wrench, tighten it by 1 full turn and  $\frac{1}{4}$  of turn.



Ensure the gas regulator is well closed (turn completely anti clockwise).

**SERVICE!**

Behind the bigger right side cover, you will find two rings with grip to maintain the gas cylinder.



1- Access for gas cylinder replacement and cooling circuit refilling.

2 - Access for X-Ray source alignment.

**WARNING!**

**PRESSURIZED GAS:**



If the gas regulator is not provided with the instrument, it is the responsibility of the customer to install his own gas regulator and, on the input gas circuitry, a safety valve preloaded at max 6.5 Bar to avoid any over pressure inside of the instrument.

## 6.4 Connection Specifications

### 6.4.1 Computer Communication

The ARL EQUINOX is shipped with a computer that already has all software required for operating the instrument. You just have to connect the RJ45 cable to the back panel of the ARL EQUINOX X-Ray diffractometer and to the Ethernet port of the computer. The RJ45 cable used is a patch cable (straight).

## 6.4.2 Electrical Power Supply Connection

**WARNING!** The ARL EQUINOX is equipped with a standard IEC 60320/C13 standard connector used also by computers.

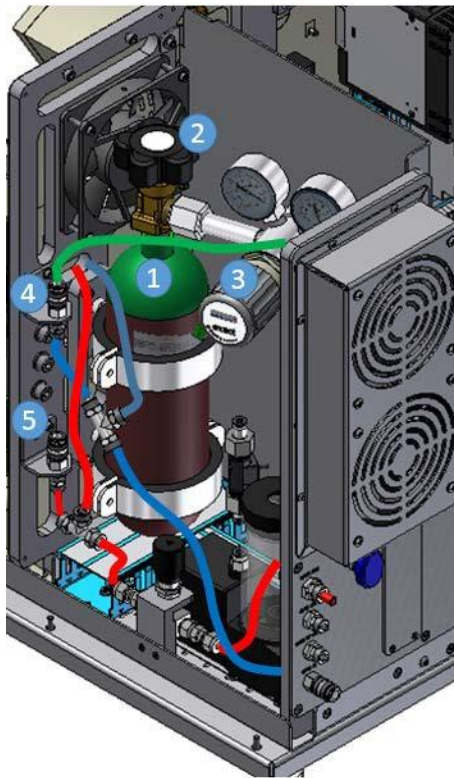


For french and US sockets, the power supply cord is shipped with the instrument. For other socket types, the power supply cord has to be obtained locally.

The socket providing the electric power supply for the instrument must have a proper ground connection.

## 6.4.3 Connection Gas

**SERVICE!** Open the valve (2) of the gas cylinder (1) and open slightly the gas regulator (3) in order to have a low gas flow. Then, connect the male quick fitting to the “Inside Gas Bottle” connector (4). This will avoid air pollution in the detector.



Adjust very slowly the outlet gas pressure on the regulator to the recommended value (6 bar). A gentle flush can be performed on connection (5). If the gas cylinder cannot be installed as a part of the instrument, please follow the instructions in Section Installation of an External Gas Cylinder on page 23.

### 6.4.4 Installation of an External Gas Cylinder

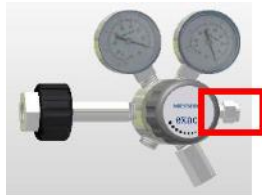
The gas regulator has to be mounted on the gas cylinder (be careful, the thread is anti-clockwise). If the gas regulator is not shipped with the instrument, it is the responsibility of the user to obtain and install the regulator locally as well as a safety valve (preloaded to max 6.5 Bar) on the input gas circuit to avoid any overpressure inside of the instrument.



A feedthrough with a female quick fitting is located on the back panel and labeled "GAS IN".



A short hose with a 6 mm diameter with a male quick fitting already mounted is shipped with the instrument.



Connect the hose to the gas regulator. The connector is a standard Swagelok gas connector.

1. Open the gas cylinder and slightly open the gas regulator in order to have a small gas flow.
2. Connect the long hose male fitting in the input receptacle "GAS IN" located on the back panel.
3. Adjust the outlet gas pressure on the regulator to the recommended value (6 Bar).

If the gas pressure is set to a too high value, a purging connector is located inside the instrument. Switch OFF the instrument. To access the purging connector "PURGE", remove the right side panel with the window. To decrease the pressure, close the gas regulator and insert a male quick fitting in the purging connector (consisting of a female quick fitting receptacle) for a few second. Increase the pressure again by opening slowly the gas regulator. Remount the side panel of the instrument.

## 6.5 Additional Documentation

Installation, adjustment procedures and calibration should be performed by Thermo Fisher Scientific service engineers only or by personnel certified by Thermo Fisher Scientific. Otherwise, Thermo Fisher Scientific does not take any responsibilities for any malfunction or damage caused by unauthorized installation of the instrument.

Additional documentation concerning the X-Ray diffraction theory, asymmetrical geometry, alignment, calibration and the adjustment of the ARL EQUINOX X-Ray diffractometer's accessories are supplied in PDF file format in the folder "C:\INEL\Documentation" or on demand to Thermo Fisher Scientific.



## 7 Operation

Before powering the instrument ON, double-check electrical, gas and water distributions. Also check if the cooling fluid tank is full and all protection panels and covers are mounted.

### 7.1 Start Up

1. Turn the main breaker located on the rear panel to the "I-ON" position.
2. If the instrument is equipped with secondary breakers, make sure they are in ON position as well.
3. Check that the emergency stop button is released.
4. Push the white button marked "Power ON" located on the front panel.
5. After powering the instrument ON, the red light of the "Power OFF" button turns ON.
6. If the instrument is connected to an external cooling circuit, make sure the source of the cooling water is opened.
7. After several tens of seconds the fan will turn ON to cool the instrument and the system is ready for operation.

---

**Note**      **The ARL EQUINOX X-Ray diffractometer needs to stabilize for about thirty minutes after starting the instrument. This time is necessary for the best performance of the instrument.**

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**Note**      **The ARL EQUINOX X-Ray diffractometer driving operations are made by Symphonix software. Please, read the Symphonix user guide to for further information on how to use the instrument. The Symphonix user's guide is available in PDF file format in the folder c:/inel/Documentation/SymphoniX user guide.pdf**

---

## 7.2 Shut Down

The instrument has been designed to run continuously. So it's not necessary to turn it OFF unless needed.

The maximum working values are 45 kV, 0.9 mA for a Cu and Mo tube and 45 kV, 0.3 mA for a Co tube.

When the X-Ray diffractometer is not used for more than an hour, we advise to run the generator at a power saving mode, 20 kV, 0.2 mA.

When the instrument is not used for more than two weeks, it can be shut down completely.

1. Reduce the generator power to 0 kV and 0 mA.
2. After the generator has reached 0 kV and 0 mA, quit all the software and terminate the communication between the instrument and the computer (refer to the SymphoniX user guide for more information).
3. Push the red button labeled "POWER OFF" located on the front panel. After you pushed it, this red button will turn OFF and the white button "POWER ON" will illuminate.
4. Turn the main breaker located on the rear of the unit to the "0-OFF" position.

## 7.3 Emergency Stop

In case of any problem (electrical, radiation or mechanical) there is an emergency stop button located on the front panel of the instrument. Pushing the emergency button immediately removes the power to the instrument, including the X-Ray generator. Be aware, that pushing the emergency stop button will result in communication loss between the control computer and the instrument.

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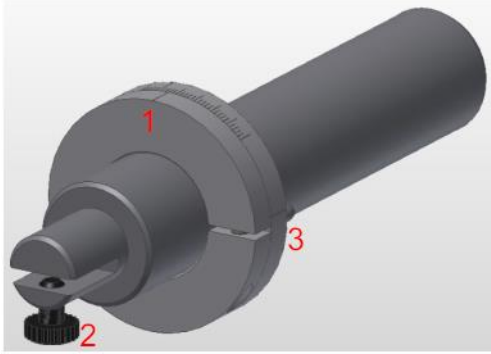
**CAUTION!**

**The emergency stop should not be used for routine shut down of the generator since repeated rapid power shut downs may dramatically reduce the lifetime of your X-Ray tube and damage the X-Ray generator.**

---

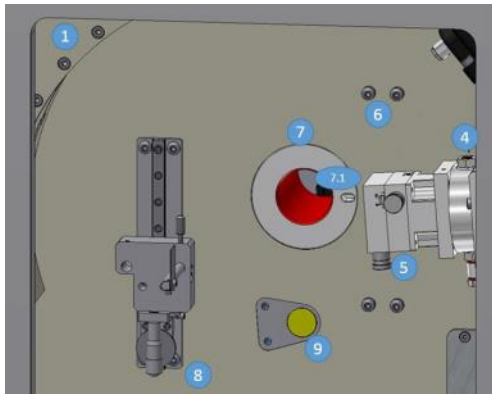
## 7.4 Sample Holder

All ARL EQUINOX instruments are shipped with a basic non-spinning sample holder suited for powder measurement in reflection mode. This sample holder is necessary for adjustment and alignment and can be used for measurement as well.



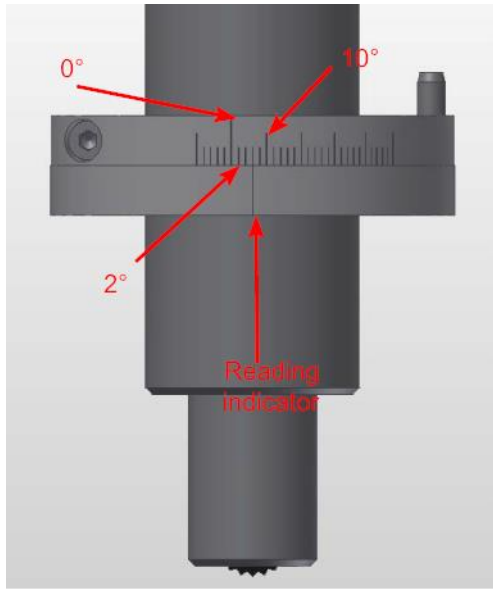
- 1 - Indicator of the incidence angle  $\Omega$
- 2 - Sample clamp
- 3 - Positioning pin

The non-spinning sample holder is designed to be easily used. The sample holder is equipped with a positioning pin to avoid any position mistake, an incidence angle indicator to read the incidence angle and a sample clamp to maintain the powder sample mount.



Insert the sample holder in the sample holder mounting hole of the instrument (7), do not use any unnecessary force. The sample holder and the mounting hole ( $\varnothing$  30 mm) are designed to avoid any mechanical clearance resulting in irreproducible results. The pin of the sample holder has to be inserted in the reference hole (7.1).

The incidence angle indicator consists of two rings. One ring is fixed while the second one is free. This allows the sample holder to rotate in order to change the incidence angle. This second ring can be tightened to avoid changes of the incidence angle. The way of reading the incidence angle indicator is described in the picture below. The longest line represents the incidence angle equal to  $0^\circ$ . Each short line represents  $2^\circ$  of incidence angle. The medium long lines represent  $10^\circ$  of incidence angle. The indicator then serves to read the total incidence angle.

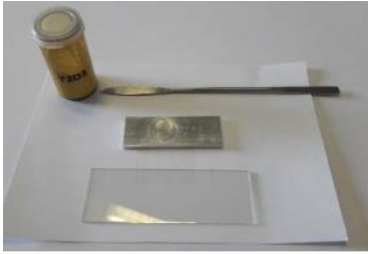


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**Note** In the above configuration, the incidence angle is equal to 6°.

---

## 7.5 Sample Preparation Procedure



In order to prepare your sample, you will need a powder mount, a spatula and a glass slide (as used for microscopes).

If the powder to be measured is too coarse, it's necessary; to grind it a bit using a small mortar.



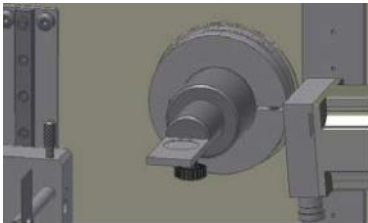
Fill the cavity with the powder.



Use a glass slide to make the surface flat and homogeneous.



The surface of the powder must not exceed the level of the surface of the powder mount, in order to avoid line displacement error due to height error.



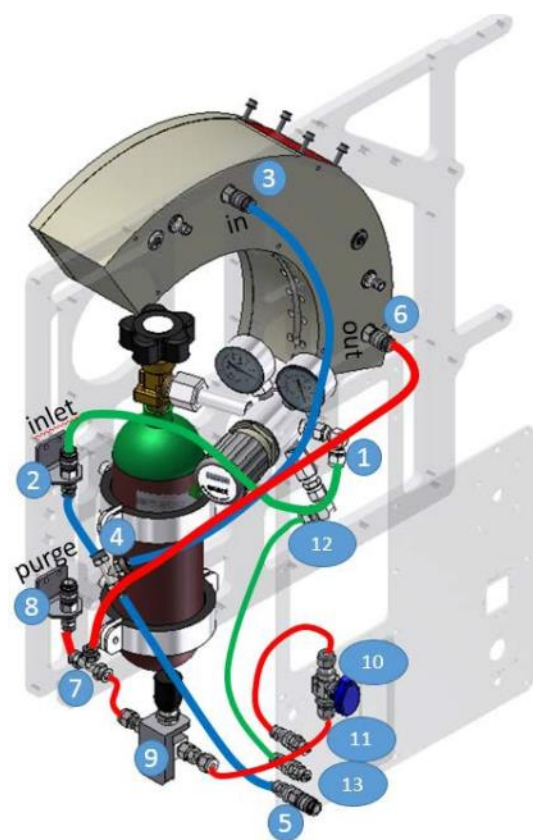
The powder mount is clamped in the sample holder by tightening lightly the screw of the non-spinning sample holder.

## 8 Maintenance and Cleaning

The ARL EQUINOX X-Ray diffractometer is designed to require only minimal maintenance operations. However, several preventive maintenance tasks should be performed regularly to keep an optimal working state of your instrument. Please refer to Appendix 4: Periodic Preventive Maintenance on page 39 to find the reminder of the periodic preventive maintenance operations. Furthermore, the cooling fluid and the detector gas cylinder must be replaced periodically.

### 8.1 Replacing the Gas Cylinder

**Note** This task should be performed by qualified personnel only



We advise to replace the gas cylinder as soon as it reaches 20 Bar. The cylinder pressure can be checked using either the side window or by opening the side cover and reading the indicator (the left indicator in case of using the FM53 gas regulator).

#### Gas circuit

The gas cylinder (1) is connected to the instrument gas inlet (2) fast connector. The T (4) distributes the gas in on the detector (3) and the external gas inlet (5). The detector out (6) is connected to the T (7) distributing the fast connector of purging (8) and the micro-leak valve (9). This one is connected to the isolating valve (10) and go to external exhaust (11). The last circuit is the overpressure line from the safety valve (12) to external exhaust (13).

#### **CAUTION!**



The gas leak valve (9) is adjusted in the factory and should not be modified. It allows to control the right consumption of gas, allowing an optimum regeneration of the gas inside detector.

1. Power down the instrument and unplug the power supply cord from the power supply. See Section Operation on page 25 for more information.
2. Close the isolating gas valve (10).

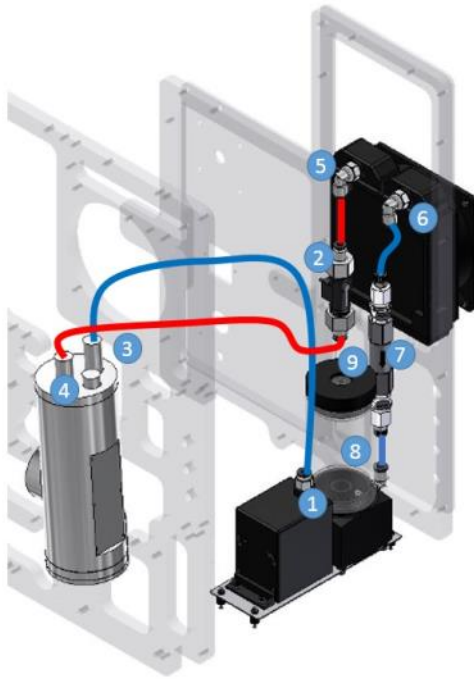
3. Remove the side/back cover of the instrument to have access to the cylinder (based on the specific model of the instrument).
4. Close the gas cylinder and the gas regulator.
5. Unplug the inlet fitting from the receptacle “Inside Gas Bottle” (2).
6. Open the gas regulator in order to purge it to avoid pressure inside before dismounting it. It must be closed again after this purge.
7. Remove the cylinder from the grips.
8. Dismount the regulator from the old cylinder and remount it on the new cylinder.
9. Install the new cylinder in place of the old one.
10. Open the gas cylinder and open slightly the gas regulator in order to have a small gas flow. Connect the inlet fitting to the input receptacle “Inside Gas Bottle” and adjust the outlet gas pressure on the regulator to the recommended value (6 Bar).
11. If the gas pressure is set too high, use the purging connector named “Purge” (8) which is located next to the cylinder. To decrease the pressure, close the gas regulator and insert a male quick fitting in the purging connector (consisting in a female quick fitting receptacle) for a few seconds. Increase again the pressure by opening slowly the gas regulator.
12. Remount the respective side/back cover of the ARL EQUINOX.
13. Start up the ARL EQUINOX (see Section Operation).
14. If necessary, proceed to the calibration with the software.

## 8.2 Replacing the Cooling Fluid

---

**Note** We advise to replace the cooling fluid every two years.

---



This operation has to be performed with the instrument powered ON and by removing the cover on the right side of the instrument. Only qualified personnel are allowed to perform this task.

The cooling fluid level should be checked and adjusted if necessary. The low level of cooling fluid is detected by the control software.

### Cooling fluid circuit:

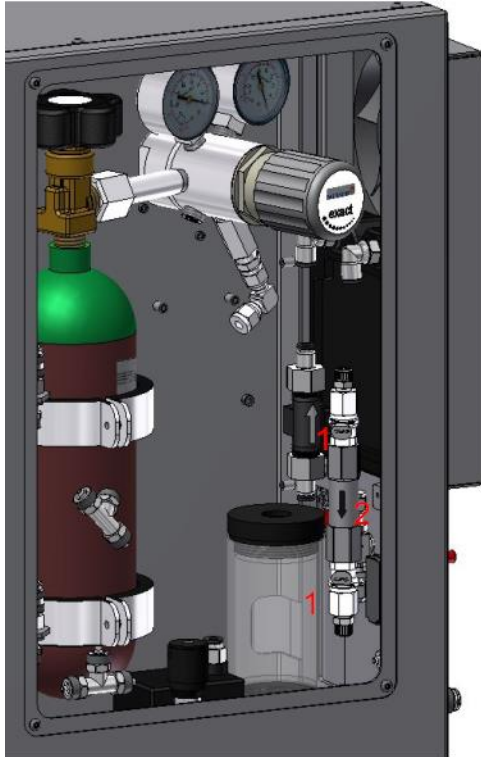
The pump (1) pumps the fluid to the X-Ray tube cooling inlet (3). The flowmeter (2) enables to control the right functioning of the device. The hot fluid (4) flows to the heat exchanger (5), and returns back (6) to the cooling fluid tank (8) by passing through the water filter (7).

The reservoir can be refilled by removing the top cap (9).



## 8.3 Cleaning and Replacing the Water Filter

When the cooling fluid needs to be replaced, it is advised to clean the water filter as well, or if in a case of a damaged filter, to replace it. The filter is accessible through the panel with the window on the right side of the system.



- 1 - Quick connect/disconnect fittings with valve
- 2 - The water filter. Mind the direction of the flow

The filter housing can be removed by disconnecting the two white quick fittings on both sides (press on the metallic part labeled CPC). The quick fittings are both equipped with a valve, thus no leakage of cooling fluid is possible. To clean or change the filter follow the instructions described in Appendix 2: Cleaning and Replacing the Water Filter on page [37](#).

---

**Note**      **Mind the direction of the flow, the direction of the flow has to be downwards.**

---

## 8.4 Checking the Emergency Stop Button

The proper function of the emergency stop button has to be checked on a yearly basis. The procedure is follows:

- The instrument has to be switched ON.
- Set the X-Ray generator to 0 kV and 0 mA (to avoid any damage of the tube).
- End the control software and quit the server by right clicking on the radiation icon that is blinking in the bottom right of the task bar in Windows.
- Push the emergency stop button.
- The instrument must switch OFF: no noise from the instrument can be heard, the red POWER OFF button switches OFF and the white POWER ON button switches ON.

- Pressing the POWER ON button should not allow powering ON the Equinox.
- The emergency stop button has to be unlocked with the key.

## 8.5 Replacing the Main Power Supply Cord

If the shipped power supply cord has to be replaced, the new power supply cord must comply with following specifications:

- 250VAC 10A or 125VAC 10A
- Cross section of wires: 3x0.75 mm<sup>2</sup>
- IEC 60320/C13 plug type

## 8.6 Replacing the Fuses

The fuseholder is located below the appliance inlet. To access the fuses, use a screw driver to pull the tray with the fuses.



Two fuses are used and with following specifications:

Size 5x20mm

4A fuse

Ratings 250VAC 1500A

Time-Lag T

This type of fuse is manufactured for example by Schurter under the reference 0001.2510.

## 8.7 Cleaning

External parts and the enclosure can be cleaned with a soft and dry duster. Domestic alcohol is forbidden as it can damage paint and markings.

## 9 Troubleshooting

### **The instrument does not start:**

- Check the state of the power supply cord, be sure it is properly plugged in.
- Check if the emergency stop button is released.
- Check the position of the main power breaker on the back of the instrument, it must be in "I-ON" position.
- If the problem persists, contact Thermo Fisher Scientific.

### **An error message appears when the control software is launched:**

- Close the error message by clicking on OK.
- Terminate the communication between the instrument and the computer by right clicking on the "radiation" icon in the task bar of Windows. Select "Quit".
- Push the OFF button of the instrument, wait for few minutes and turn the instrument ON and launch software again.
- If the problem persists, contact Thermo Fisher Scientific.

## 10 Appendix 1: Conformity Certificate

Thermo Fisher Scientific has certified by an independent control authority that the ARL EQUINOX X-Ray diffractometer series and related accessories comply with:

- French regulation NF C 74-100 related with manufacturing instruments that emit X-Ray radiations.
- 

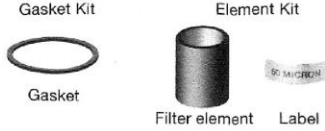
Thermo Fisher Scientific declares under its own responsibility that the ARL EQUINOX X-Ray diffractometers series and respective accessories comply with the CE mark according to the following standards and directives:

- EN 61010-1 (2010): Safety requirements for electrical equipment for measurement, control, and laboratory use - Part 1: General requirements,
- EN 61326-1 (2013): Electrical equipment for measurement, control and laboratory use - EMC requirements - Part 1: General requirements,
- 2011/65/EC: restriction of the use of certain hazardous substances in electrical and electronic equipment, RoHS2.

# 11 Appendix 2: Cleaning and Replacing the Water Filter

## F Series Inline Filter Service Instructions

### Kit Contents



### WARNING

Before removing a filter from the system for service, you must

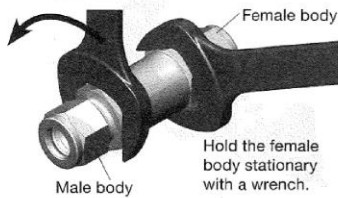
- depressurize system
- purge the filter to remove any residual system media.

### WARNING

Residual system media may be left in the filter.

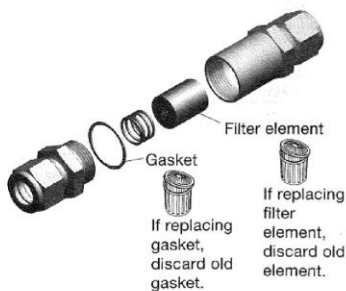
### Disassembly

1. Remove the filter from the system.
2. Loosen the **male body** from the **female body**.



3. Remove the components.

Note: Use a blunt tool to loosen the filter element if necessary.



### Tools Required

Tool	Size	Component
Open-ended wrenches	2F: 9/16 in. 4F: 3/4 in. 6F, 8F: 1 in.	Body hex
Crow's foot	2F: 9/16 in. 4F: 3/4 in. 6F, 8F: 1 in.	Body hex
Torque wrench	Capable of 500 in.-lb (56.5 N-m)	Body hex

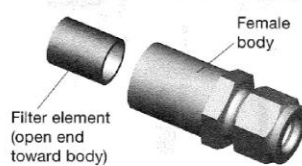


### Definition of Symbols

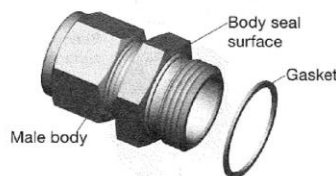


### Reassembly

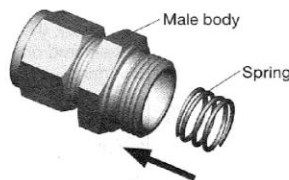
4. Clean all components.
5. Press the open end of the **filter element** into the **female body**.



6. Place the **gasket** on the **body seal surface** of the **male body**.



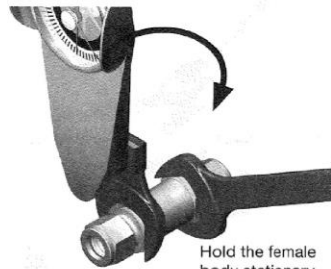
7. Insert the **spring** into the **male body**.



8. Thread the bodies together.

Note: There will be no space between the gasket and the male body hex when the bodies are fully threaded.

9. Tighten the male body according to table below.

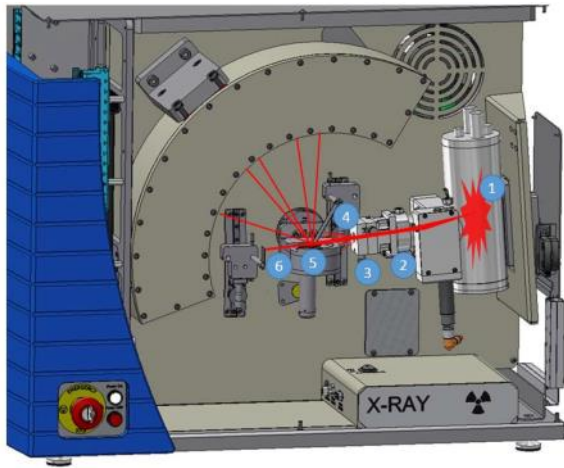


Series	Torque, in.-lb (N-m)	
	Stainless Steel, Alloy 400, Alloy C-276, Alloy 600	Brass
1F, 2F, 3F-MM	135 (15.2)	125 (14.1)
4F, 6F-MM	350 (39.6)	325 (36.7)
6F, 8F, 10F-MM, 12F-MM	500 (56.5)	450 (50.8)

10. Place the new label on the female body.

11. Test the product for proper operation prior to reinstallation in system.

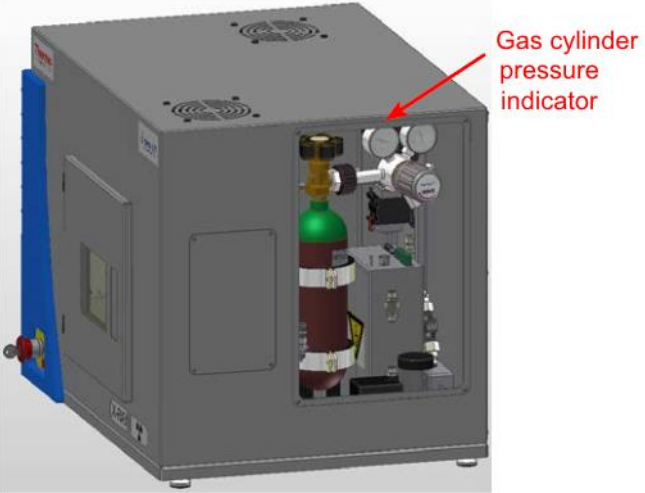

## 12 Appendix 3: Optical paths of X-Rays in ARL EQUINOX 100



The figure describes the X-Ray beam propagation inside of the enclosure when the shutter is opened. During this operation, the enclosure is closed and door is blocked.

1. X-Ray source emission. Scattering is emitted in all directions. The tube shield is limiting the X-Ray.
2. X-Ray mirror allows to capture a given solid angle from the direct beam, and emits a narrow focused and monochromatic beam.
3. Primary crossed slits can shape the incident beam from 0.5 to 0.02 mm.
4. The anti-scattering knife decreases the diffusion from the slits
5. The sample is able to diffract the beam according to its atomic structure
6. The beam stop blocks the direct beam. A proper adjustment of this plate allows to improve the low angle detection.

## 13 Appendix 4: Periodic Preventive Maintenance

Period	Action	Indication	Schedule
Monthly	Check the detector calibration	Refer to the SymphoniX user guide	
Monthly	Check the remaining gas pressure in the gas cylinder		
When necessary	Replace the gas cylinder	Contact Thermo Fisher Scientific or an authorized person.	
Monthly	Check the level of the cooling fluid		
When necessary	Change the cooling fluid and clean filter	Contact Thermo Fisher Scientific or an authorized person.	

<b>Period</b>	<b>Action</b>	<b>Indication</b>	<b>Schedule</b>
Annually	Check the emergency stop button	Pushing the emergency stop button must switch off the instrument. This procedure can be done by the customer, refer to Section Checking the Emergency Stop Button on page <a href="#">33</a> .	
Annually	Check the beam alignment	Contact Thermo Fisher Scientific or an authorized person.	
Annually	Check the gas circuit tightness	Contact Thermo Fisher Scientific or an authorized person.	
Annually	Check the safety interlocks	Contact Thermo Fisher Scientific or an authorized person.	



## 14 Appendix 5: Sample Holders for ARL EQUINOX 100

ARL EQUINOX 100 is the most compact Thermo Fisher Scientific X-Ray diffractometer which allows several types of configurations. Thanks to the number of proposed sample holders, user can perform conventional XRD powder analysis for quality control or quantitative analysis (reflection or transmission). The user can also identify phase transition by using Anton-Paar BTS furnaces, characterize thin film structures or acquire automatically up to 6 samples with our sample changer.



Fixed non-spinning sample holder  
Reflection & transmission mode



Spinning sample holder for powder sample



Spinning sample holder for powder & bulk sample  
Reflection mode



Spinning sample holder for cement studies  
Reflection mode



Spinning sample holder for powder in capillary  
Transmission mode



Spinning sample holder for filter studies  
Reflection mode



Motorized thin layer sample holder  
Bulk sample also Reflection mode



6 positions sample changer with spinner  
Reflection mode



High temperature camera  
BTS500  
From ambient to 500 °C  
Reflection mode



High temperature camera  
BTS150  
From -10°C to 150 °C  
Reflection mode

---

**CAUTION!** Hot surface on BTS500 and BTS 150



**Do not touch this surface without adequate protective measures. Refer to Anton Paar instruction manual of BTS500 and BTS150 for more information.**

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Thermo Fisher Scientific (Ecublens) SARL,  
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