

# Oxford Instruments NanoAnalysis MicroAnalysis System

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## MicroAnalysis System

### ABOUT THIS GUIDE

This guide provides basic information for using the Oxford Instruments NanoAnalysis (OINA) **MicroAnalysis** System for Scanning Electron Microscopes (SEM) and Transmission Electron Microscopes (TEM) in Energy Dispersive Spectroscopy (EDS) applications.

A **MicroAnalysis** System consists of:

- an OINA X-ray detector - **x-act**, **X-Max** or **X-Max TEM**.
- an x-stream module for X-ray acquisition and detector control.
- a mics module for sample image capture.
- a host personal computer (PC).
- OINA **AZtec** and/or **INCA** software running on the host PC.

### Support and training

For technical support or training, contact your system vendor. If the vendor is not known, details of your local Oxford Instruments NanoAnalysis office are available on the Oxford Instruments Customer Support web site.

Go to <http://www.oxford-instruments.com> then navigate to **Support > NanoAnalysis**.

### Further information

Full instructions for using your **MicroAnalysis** System are given in:

- the **MicroAnalysis** System User's Manual. A portable document format (PDF) edition of the manual is supplied on the system software DVD.
- the OINA User's Manual for your detector model - a portable document format (PDF) edition is supplied on the system software DVD.
- the OINA x-stream and mics Installation and Users Guide (51-1720-001).

Please read these documents and this Getting Started Guide before using your **MicroAnalysis** System.

## SAFETY INFORMATION

Read these safety instructions before installing and using Oxford Instruments NanoAnalysis Systems.

### General safety information

- The system must be earthed (grounded) in accordance with the Oxford Instruments installation guidelines.
- Do not use the equipment if any cable is damaged.
- Do not operate the equipment if failure or damage is suspected.
- Do not use a supply voltage that is outside the specified range.
- Do not operate the equipment with the covers removed.
- Do not operate the equipment in damp or wet conditions.
- Do not immerse the equipment in liquids.
- Do not use and store the equipment in corrosive or explosive atmospheres.
- Do not operate the equipment outside the specified temperature range of 10°C to 30°C.
- Provide proper ventilation to prevent overheating.
- Only use cables and parts provided by the manufacturer.
- Use a proper earth (ground) to connect to the equipment.
- Installation and service work must be performed by Oxford Instruments qualified personnel only.

### Electrical power and safety earth

Any interruption of the protective conductor inside or outside the host PC, the monitor and the electrical power supply for the x-stream unit, or disconnection of the protective earth terminal, could render the equipment dangerous.

Electrical power outlets are classified as a hazardous voltage. The power connection to OINA equipment must be made to an accessible power outlet within reach of the operator, so power can be easily disconnected if necessary.

Replace power cords if they are damaged. Contact your system vendor to ensure that the correct replacement part for your country is used.

Only Oxford Instruments qualified personnel should install OINA products. Contact your system vendor if hardware problems occur.

## MicroAnalysis System

## Hazard notices

**HIGH VOLTAGE**

The x-stream module contains a high voltage (HV) circuit that can generate voltages in excess of 1 kV. The covers of the x-stream module should not be removed.  
There are no user serviceable parts within the x-stream module.

**CRUSH INJURY**

Some detectors are fitted with a motorised slide. Keep your fingers clear of the slide when it is in motion, or injury may result. The slide of the detector may move without warning. There is a risk of crush injury if the motion of the detector is activated by remotely operated software.

**BERYLLIUM**

The windows of some detectors contain beryllium metal, which is extremely toxic by inhalation and ingestion and a probable human carcinogen. It is a serious respiratory irritant and may be harmful if in contact with the skin.

To avoid fatal or serious injury, always obey the precautions given below.

## Beryllium precautions

The windows of some detectors are made of Beryllium metal. The windows are safe unless broken but are hazardous if the window breaks and beryllium dust forms. Exercise extreme care to avoid damage to the window. If a window is damaged, always obey these precautions:

- Do not touch the window of an X-ray detector.
- Avoid breathing dust or powder when in the vicinity of the detector.
- If you suspect that a detector is damaged, wear safety glasses, protective gloves, a face mask and a respirator when in the vicinity of the detector. Ensure that the area is well ventilated.
- If you inhale, ingest or make skin contact with Beryllium particles, seek immediate medical assistance.

**Electron Trap**

The electron trap must be fitted to the detector at all times when installed on an SEM microscope column. Failure to fit the electron trap will invalidate the warranty.

**Adjustment of Slide Movement Stop Nuts**

Do not adjust the position of the movement stop nuts on detectors fitted with a slide mechanism, otherwise damage to the detector damage may occur.

## GETTING STARTED

This section provides getting started information for the **INCA** and **AZtec** MicroAnalysis software applications.

### Switch ON the MicroAnalysis System

- 1 Switch the x-stream unit ON by using the **ON/OFF** switch on the rear panel of the unit. When switching x-stream on from cold, allow 30 minutes for the system to stabilize.
- 2 Switch ON the host PC and launch Microsoft® Windows®. The Windows Logon window opens.
- 3 Press **Ctrl + Alt + Delete** to log on to the PC. For the **INCA** application, the default Username is INCA and the Password is INCA. For the **AZtec** application, the default Username is Oxford and the Password is oxford. If you do not know your Username and Password, ask your system administrator.

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NOTE: Usernames are not case sensitive.

Passwords are case sensitive. The default password **INCA** is all upper-case and the default password **oxford** is all lower-case.

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When you are logged on, a green light should be visible on the front of the mics and x-stream units.

### Software licensing

The **AZtec** and **INCA** software must be licensed. To see the licence status, go to:

**All Programs > Oxford Instruments NanoAnalysis> Tools> License Manager**




A limited number of starts are allowed before a licence must be activated. For more information about software licensing, refer to the OINA Software Licensing User Guide (51-1720-061) and the Software Licensing Quick Guide (51-1624-129).

## MicroAnalysis System

## Getting started with INCA

- 1 Start the **MicroAnalysis** System application by going to **Start > Programs > Oxford Instruments > INCA** or double-click on the desktop icon.
- 2 Select **Getting Started Tutorial** from the Help option on the menu bar.  
Use **Analyzer** for analysis from the image on the SEM screen (Energy 250, 350 and 450).  
Use **Mapping** for elemental maps (Energy 250, 350 and 450).  
Use **Point & ID** for analysis from the digital image on the EDS screen (Energy 350 and 450).

## Getting help in INCA

- 1 At each step, you can open help bubbles by pressing the  button.  
The bubbles are numbered to indicate sequential operations at each step in the analysis.
- 2 Press  within the bubble to obtain more detailed help and advice.  
The full **MicroAnalysis** System Help encyclopedia can be accessed from the **MicroAnalysis** System menu bar.
- 3 Press  on the work area to see tutorials of the sequence of operations.  
The full **MicroAnalysis** System Help encyclopedia is also available when the tutorial is running.

## Detector control in INCA

- 1 View and control the operational state of the detector by choosing **Detector Control** from the Options menu.
- 2 The Detector Control dialogue box opens, showing the status of the detector.  
This dialogue box opens automatically whenever the operational state of the detector changes. The tabs in the dialogue box depend on the type of x-ray detector in use.
  - For **x-act** detectors, only the **Thermal** tab is available.
  - For **X-Max** detectors, the **Thermal** and **Slide** tabs are available.
  - For **X-Max TEM** detectors, the **Thermal**, **Slide** and **Flux Protection** tabs are available.
- 3 Use the **Thermal** tab to switch the operational state of the **x-act** or **X-Max** detector between **Standby** and **Operate**.

In **Standby** mode, the detector warms up.  
In **Operate** mode, the detector cools down





- 4 Use the **Slide** tab to change the position of the **X-Max** or **X-Max TEM** detector:

Click **Move In** to insert the detector.  
 Click **Move Out** to retract the detector.  
 Click **Stop** to halt the movement of the detector.



NOTE: **X-Max TEM** retracts automatically in the presence of high flux. To re-insert the detector, click **Move In** or use the manual push button.

- 5 Use the **Flux Protection** tab to protect the **X-Max TEM** detector from excessive x-ray flux: Select **Enabled** to automatically retract the detector when excessive x-ray flux is sensed. Enter a value in the **Threshold (MeV/s)** field to set the flux level at which the detector retracts. Enter a value in the **Delay (s)** field to set the time delay in seconds between the detection of excessive flux and retraction of the detector.



NOTE: The threshold and delay values are set by the engineer during installation. It is strongly recommended that you do not change the default delay or threshold values. Doing so may reduce the life of your detector.

## INCA Monitor system check

If a problem occurs, use this software feature with its comprehensive help guide. The purpose of the system check utility is to confirm that the mics and x-stream units are operating correctly. When run, the process cycles through a series of checks, measurements and questions to validate the system's performance. This procedure identifies faults and, if possible, diagnoses the probable cause.

Launch Monitor by double-clicking this icon on the task bar




If a hardware problem is suspected, double-click on the icon and click the **System Check** step. Detector thermal cycling and calibration are accessible via INCA Monitor.

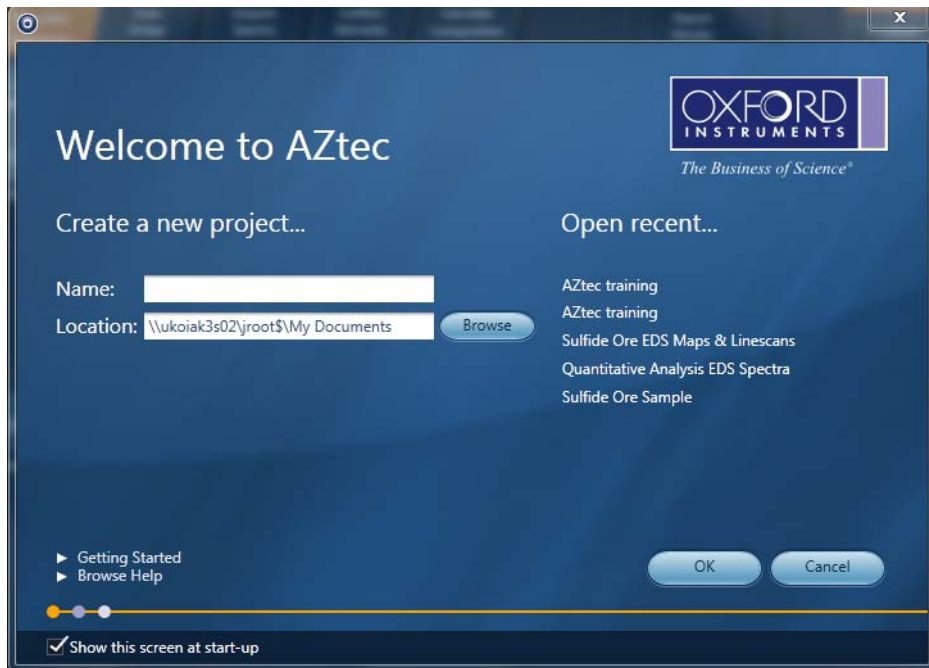
Detector slide, shutter and LN<sub>2</sub> alarm controls are available in **MicroAnalysis System** Microanalysis under **Options > Detector Control**.

## MicroAnalysis System

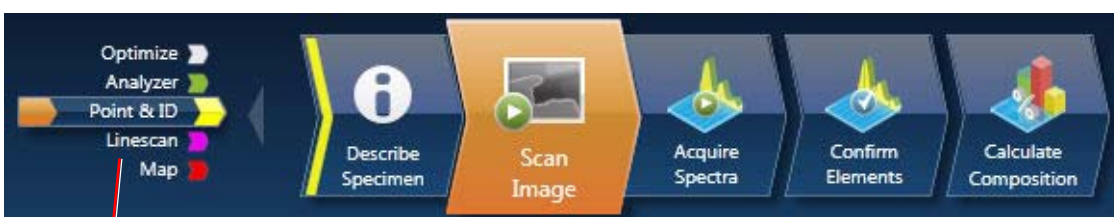
## Getting started with AZtec

## Using AZtec

- 1 Click the  shortcut.
- 2 The **AZtec** welcome screen opens.

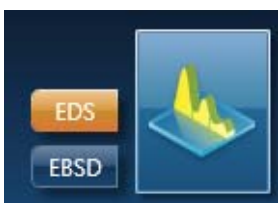


- 3 Create a new project and enter the project **Name** and **Location**, or click **Open recent** to open a recent project. Then click **OK**.



## Navigator selector

- 4 **AZtec** is controlled by using horizontal navigators at the top of the screen. Click a navigator selector to choose the respective function.  
You can switch between navigators for EDS and EBSD by using the **EDS** and **EBSD** buttons to the left of the navigator.

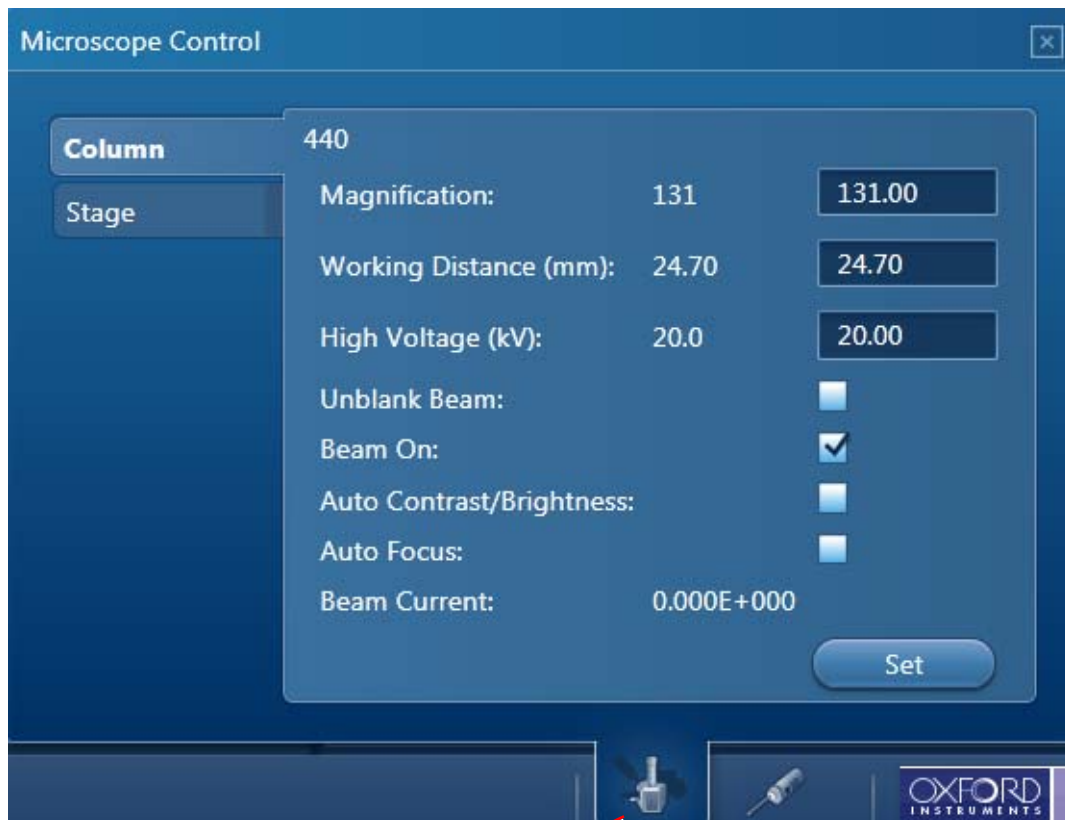


## Microscope control in AZtec

Microscope operating conditions are monitored on the status bar.

Mag: 131x | HV: 20.0 kV | WD: 24.70 mm | Input Rate: 83890 cps

Click the microscope tab to open a dialogue box where you can control the microscope.



Microscope tab

On most microscopes, these parameters are read automatically.

## MicroAnalysis System

**Detector control in AZtec**

Click the EDS detector tab to open a dialogue box where you can control the detector. The tabs and details on the Detector Control dialogue box depend on the type of detector installed:

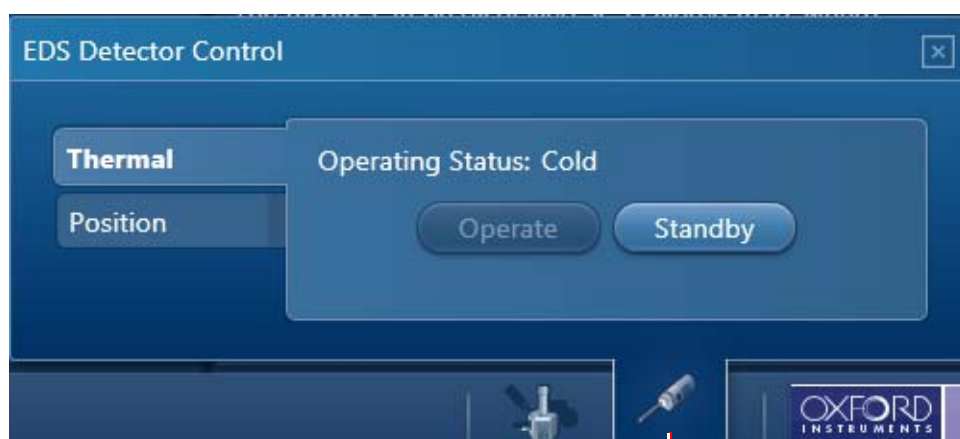
If an EDS detector is installed, the EDS Detector tab is displayed.

If an EBSD detector is installed, the EBSD Detector tab is also displayed. For EBSD detectors, refer to the EBSD Getting Started Guide.

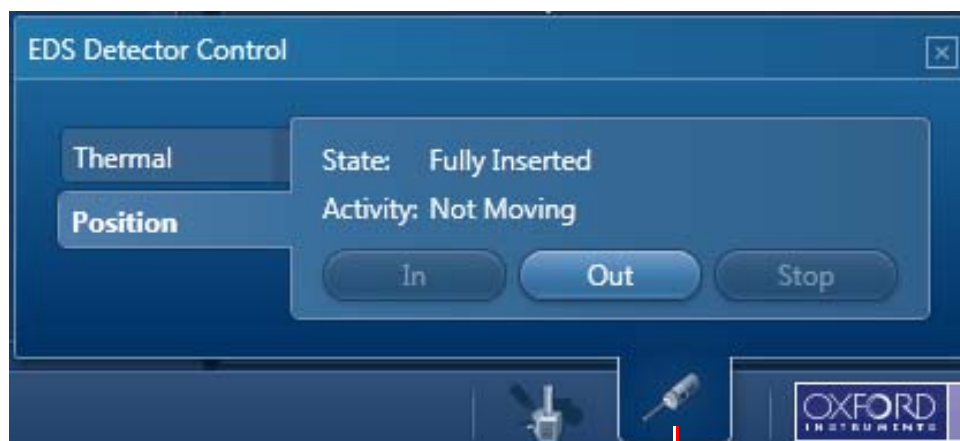
Detector control in **AZtec** is similar to **INCA**. See [Detector control in INCA](#).

For **x-act** detectors, only the **Thermal** tab is available and for **X-Max** detectors, the **Thermal** and **Position** tabs are available.

**X-Max TEM** is not currently supported so there is no detector control **Flux Protection** tab.



EDS  
detector  
tab



## Getting Help in AZtec

Help can be obtained from the Help menu.

Use the search function to find the topic you want.

Type your  
question here



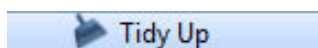
Click here to  
start the  
search

Alternatively, press **F1** in any screen for context sensitive Help. The User Manual in PDF form can be launched from the main Help menu.


## Troubleshooting in AZtec

In the event of a problem:

- 1 Go to **All Programs > Oxford Instruments NanoAnalysis > Tools**



The Tidy Up function clears and restarts the hardware modules.

- 2 Restart the host PC.
- 3 Launch Monitor  from the Task Bar and run the System Check utility.

## Software updates

To re-install the software, ensure that your data is archived, then refer to the instructions on the **MicroAnalysis** System compact disk (CD).

## Data backup

The installation engineer will make a backup of the entire system after installation and calibration are finished. The backup must be kept in a safe place.

It is your responsibility to back up your data as appropriate.

## MicroAnalysis System

## X-RAY DETECTORS

This section provides getting started information for the **x-act**, **X-Max** and **X-Max TEM** detectors of the **MicroAnalysis** System.



### Electron Trap

The electron trap must be fitted to the detector at all times when installed on an SEM microscope column. Failure to fit the electron trap will invalidate the warranty.

## General information about the detectors

This information applies to all three detector models.

### Switching the detector ON

There is no ON/OFF switch on the **x-act**, **X-Max** or **X-Max TEM** detector.

- 1 Switch ON electrical power at the wall socket.
- 2 Switch ON the x-stream unit using the rocker switch at the rear of the unit.
- 3 Switch ON the host PC and launch the software.  
When x-stream power is ON, the detector warms and cools under software control as required.

### Switching the detector OFF

- 1 Retract the **x-act**, **X-Max** or **X-Max TEM** detector until it reaches the end of travel.
- 2 Close the software application and the Windows® operating system and shut down the host PC.
- 3 Switch OFF the x-stream unit using the rocker switch at the rear of the unit.
- 4 Switch OFF electrical power at the wall socket.  
When power is removed from x-stream, the detector warms up.

## Getting started with x-act detectors

### Cooling the x-act detector

The **x-act** detector is a *cool on demand* device. Unless in STANDBY mode, the detector cools when electrical power is ON and the microscope column is at vacuum. The detector is fully operational within 5 minutes of column evacuation. For optimum **Quant** results, it is recommended that the **x-act** detector and x-stream are ON for at least 30 minutes before use. See [Detector control in INCA](#) and [Detector control in AZtec](#).

### Warming the x-act detector

The **x-act** detector warms when the microscope column is evacuated or electrical power is switched OFF.

### LEDs on the x-act detector

There are two LEDs on the cover of the **x-act** detector. They indicate the status of the device as shown below.

A message is displayed by the software if an error occurs.



LED 1	Description
Off	No +24 V supply
Green	+24 V supply OK
Amber	+24 V supply OK, unit on STANDBY
Red flashing	Error detected
LED 2	Description
Off	No electrical supply
Blue flashing	Detector cooling
Blue	Detector ready for use

### Inserting and retracting the x-act detector

Turn the handle at the rear of the **x-act** detector to insert and retract the device. A **Stop** position on the manual slide is set by the engineer at installation.



Caution

#### Adjustment of Slide Movement Stop Nuts

Do not adjust the position of the slide movement stop nuts of the x-act detector, otherwise damage to the detector may occur.

## MicroAnalysis System

## Getting started with X-Max detectors

## Cooling the X-Max detector

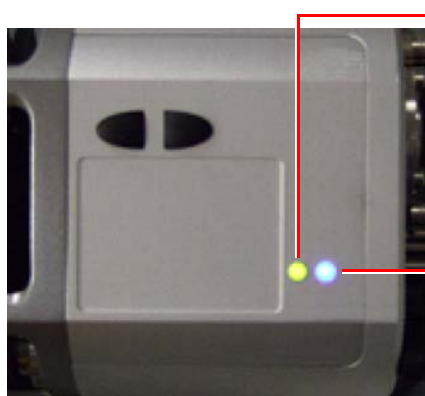
The **X-Max** detector begins to cool when electrical power is switched ON or the **Operate** button in the detector control panel is selected. When the detector has finished cooling, the blue light emitting diode (LED) on the detector cover illuminates steadily. The detector cools and is fully operational within 5 minutes. For optimum **Quant** results, it is recommended that the detector and x-stream are ON for at least 30 minutes before use.

## Warming the X-Max detector

- 1 Select **Standby** at the Detector Control dialogue box, or remove the electrical power supply to the detector. See [Detector control in INCA](#) and [Detector control in AZtec](#).

## LEDs on X-Max detectors

There are two LEDs on the cover of **X-Max**. They indicate the status of the device as shown below. A message is displayed by the software if an error occurs.



LED 1	Description
Off	No +24 V supply
Green	+24 V supply OK
Amber	+24 V supply OK, unit on STANDBY
Red flashing	Error detected
LED 2	Description
Off	No electrical supply
Blue flashing	Detector cooling
Blue	Detector ready for use

## Inserting and retracting the X-Max detector manually

**RISK OF CRUSH INJURY**

**Take care when operating the slide of the X-Max detector. There is a risk of crush injury when the motion of the detector is activated by remotely operated software.**

The slide of the **X-Max** detector is motor driven. Two buttons on the body of the detector control the slide movement:

- 1 To nudge the **X-Max** detector in, tap the **IN** button.
- 2 To insert the **X-Max** detector slowly, press and hold the **IN** button.
- 3 To retract the **X-Max** detector, press the **OUT** button.
- 4 Press the **IN** or **OUT** button to stop retraction or software controlled insertion.



The maximum insertion distance and the **Stop** position are set by stop nuts that are adjusted by the engineer during installation.



Caution

#### Adjustment of Slide Movement Stop Nuts

Do not adjust the position of the slide movement stop nuts of the X-Max detector, otherwise damage to the detector may occur.

### Automatic retraction of the X-Max detectors



WARNING

#### CRUSH INJURY

The slide of the X-Max detector may move without warning. There is a risk of crush injury when the motion of the detector is activated by remotely operated software.

The **X-Max** detector retracts automatically under software control when selecting the **Move Out** command on the **Slide** tab in Detector Control. See [Detector control in INCA](#) and [Detector control in AZtec](#).

## Getting started with X-Max TEM detectors

### Cooling and warming X-Max TEM detectors

The cooling and warming of **X-Max TEM** detectors operates in the same way as in **X-Max** detectors. See [Cooling the X-Max detector](#) and [Warming the X-Max detector](#).

### Best practice

Some operating conditions on TEMs scatter high energy electrons which can potentially harm the X-ray detector over its lifetime. To optimise detector life, the following precautions are recommended:

- **Retract** the detector behind the protective flap when it is not being used for EDS work.
- On some microscopes, the detector retracts automatically when the microscope operates in low magnification mode. **As a precaution, always retract the detector before entering this mode.**
- **Do not leave the beam on a grid bar for longer than necessary.** If flux remains high for an extended period (typically 5 seconds) the detector retracts automatically, but it is better to avoid auto-retractions if possible.
- **Avoid very high beam currents and consequent high electron flux** when routinely scanning a sample in normal TEM imaging mode. In some positions, this may damage the detector over time. When possible, retract the detector.
- **Do not operate the detector with the objective aperture in place** (unless it is the high contrast objective aperture in JEOL Ltd. TEMs) as this will cause major artefacts in the X-ray spectrum.

## MicroAnalysis System

### X-Max TEM retraction

The **X-Max TEM** detector retracts automatically under software control in these conditions:

- When selecting the **Move Out** command on the **Slide** tab in Detector Control.
- If the flux exceeds the preset threshold shown on the **Flux Protection** tab in Detector Control.
- On some microscopes, when the low magnification setting is selected during setup.

Flux protection and Low Mag protection protect the **X-Max TEM** detector from excessive flux which could potentially harm the detector over its lifetime.

When the **X-Max TEM** retracts automatically, click **Move In** on the **Slide** tab of Detector Control, or use the manual push button to re-insert the detector.

### LEDs on X-Max TEM detectors

The LEDs on **X-Max TEM** detectors are identical to those on **X-Max** detectors. See [LEDs on X-Max detectors](#).

### Detector handling

This section provides important instructions for handling Oxford Instruments NanoAnalysis X-ray detectors. It applies to **x-act**, **X-Max** and **X-Max TEM** detectors.

**THIS DOCUMENT IS FOR GUIDANCE ONLY. IT DOES NOT CONSTITUTE A SPECIFICATION OR ADMIT ANY LIABILITY FOR PRODUCT PERFORMANCE.**

Obeying the guidelines suggested here will help to extend the life of the detector.

### Fragile components

The components of OINA detectors are very fragile - exercise great care when handling any part. Never touch the window of a detector. The detector window is usually highly stressed and even the light brush of a human hair can cause failure. You must not attempt to clean the window. The detector must be returned to the manufacturer to be cleaned.

**The windows of some detectors contain beryllium metal, which is a hazardous substance. To avoid fatal or serious injury, always obey the precautions given in [SAFETY INFORMATION](#).**

### Keeping the detector clean

The detector and interface components have been thoroughly cleaned by a multi-bath cleaning process in the factory, and are sealed in protective covers to prevent contamination during transportation.

**KEEP ALL PARTS CLEAN AND FREE FROM GREASE.**

Always wear powder-free nitrile gloves when handling detector parts.

Never place the components on the floor or a dirty surface.

Do not attempt to clean the detector window. The detector must be returned to the manufacturer to be cleaned.

## Unpacking/packing the detector

The detector has been carefully transported in dedicated packaging. A protective cover and a transport cover shield the detector window - the most fragile part of the device.

### **KEEP ALL PACKAGING SUPPLIED WITH THE DETECTOR.**

It is important to keep the protective cover and cover shield clean and dry - preferably stored in a new, sealed polythene bag. If the detector is returned to the manufacturer, use all the packaging supplied. Badly-packed detectors often incur further damage which will void the product warranty.

## Venting the column/chamber of the electron microscope

The column/chamber of the microscope must be vented in a slow, controlled manner. If venting is too rapid or the pressure is too high, the detector window may break. Do not force the chamber door open after venting, and shut the door gently before pump-down. Remove any loose material/particles from the chamber which, on venting, may impact and damage the detector window. Retract the detector before venting (NanoAnalysis products) to minimise the risk of breakage.

Suitable venting should take at least 30 seconds to reach atmospheric pressure. Take special care on chambers fitted with Nitrogen back-fill, and make sure that the back-fill does not exceed the atmospheric pressure. To minimise potential damage, ensure that the venting line is not in direct line of sight with the detector window.

If your vacuum system uses oil-filled diffusion pumps, take care to ensure that oil vapour does not enter the microscope chamber. Oil vapour condensing on the detector window will reduce system performance and may damage the window.

## Detector operation

All OINA detectors are designed for thermal recycling using appropriate cool-down and warm-up routines within the software. Do not attempt to cool the detector in any way except as explained in [Cooling the x-act detector](#), [Cooling the X-Max detector](#) and [Cooling and warming X-Max TEM detectors](#).

## Detector use with a hot stage fitted

Only specially designed detectors can be used for analysis at high temperatures. Please consult your Oxford Instruments representative for more detailed information and advice. Until verified by OINA, detectors cannot be used in combination with hot stages, even if the hot stage is shielded or the detector is retracted.

## Field servicing by qualified engineer/technician

The system contains no user serviceable parts. Service must be done by an Oxford Instruments qualified engineer only.

## MicroAnalysis System

### Protecting the detector when it is not in use

The detector must be removed from the microscope column by trained personnel only, using correct handling procedures to avoid contamination or damage.

When the detector is not attached to the microscope column, it must be stored in the protective cover provided, and handled using powder-free, nitrile gloves. See [Unpacking/packing the detector](#) above.

### Electron trap

The detector may be equipped with an electron trap. If so, the electron trap must be fitted by an Oxford Instruments qualified engineer only.

**CARELESS MANIPULATION OF THE ELECTRON TRAP CAN BREAK THE DETECTOR WINDOW.**

### Collimators

The detector may be equipped with a collimator. If so, the collimator must be fitted by an Oxford Instruments qualified engineer only.

**CARELESS MANIPULATION OF A COLLIMATOR CAN BREAK THE DETECTOR WINDOW.**

### Returning items to Oxford Instruments

In the unlikely event that a detector must be returned to Oxford Instruments for repair. Please pack it correctly in the original transportation box. To protect the detector, use the protective cover and transport cover provided, even if you think the part is already damaged. Further damage may occur if the detector is not packed correctly.

Ask for the correct packaging if it has been mislaid. Do not force the detector into the box - it will fit neatly if packed correctly.

When shipping a NanoAnalysis WDX spectrometer, the detector set **MUST** be removed prior to shipping. The spectrometer and the detector set must be placed in the dedicated shipping boxes.

Items can only be returned to Oxford Instruments using the prevailing Returns/RMA procedure. Contact your local OINA office for further details.

Complete the RMA form in full to ensure that faults in the detector can be assessed quickly and precisely, and to speed up the repair time.

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# Getting Started Guide

## MicroAnalysis System

### Technical support

#### United Kingdom

Halifax Road  
High Wycombe  
Bucks  
HP12 3SE  
United Kingdom  
Support email: [na.techsupport@oxinst.com](mailto:na.techsupport@oxinst.com)  
Training email: [na.training@oxinst.com](mailto:na.training@oxinst.com)  
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