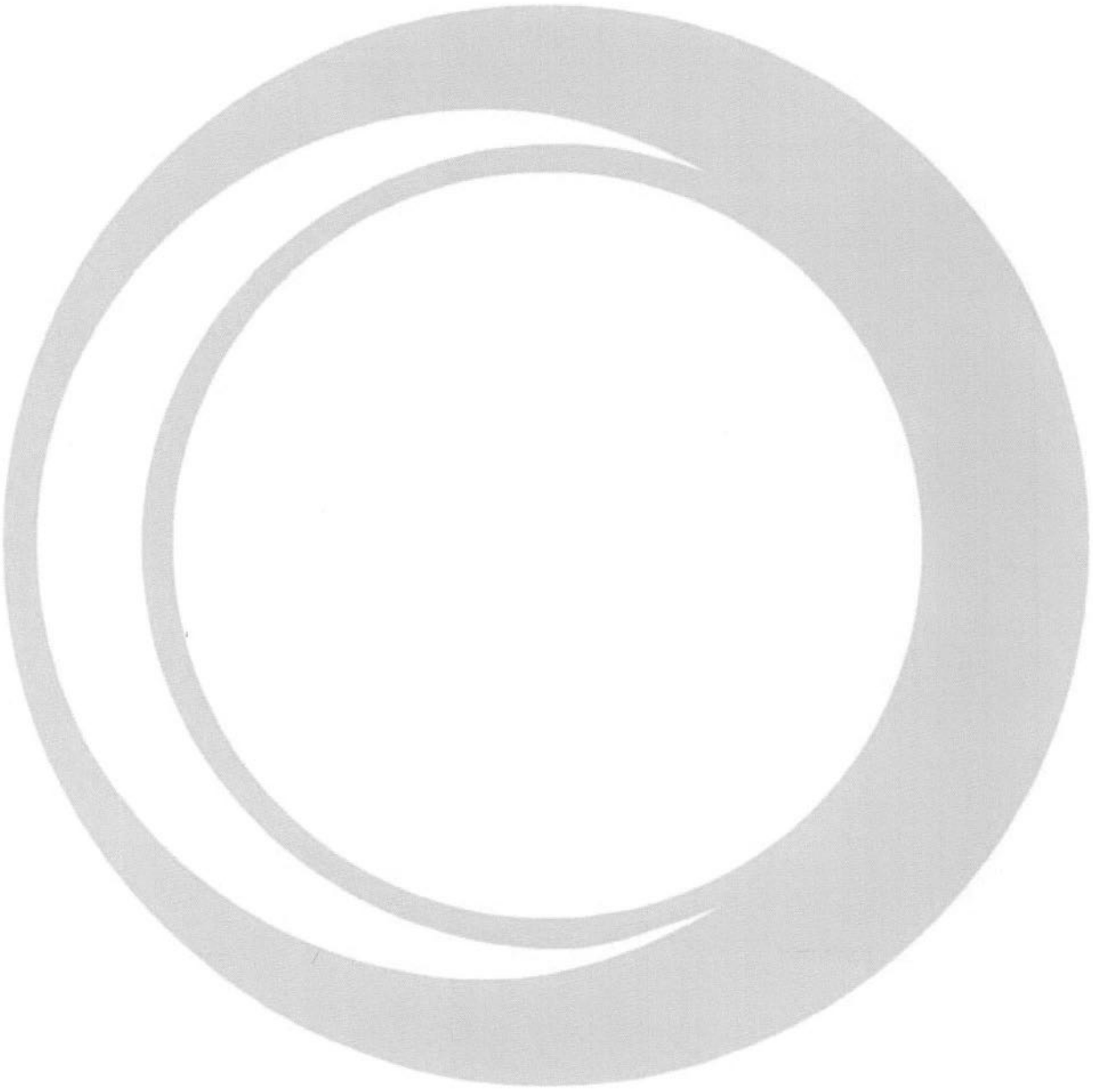
X-Supreme8000

User Guide



54-XSPOM1000E

February 2011 Revision 2

The Business of Science™

OXFORD INSTRUMENTS

CONTENTS

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Safety Information

IMPORTANT INFORMATION in this manual
Information easily overlooked—please read carefully.

Warning Symbols on labels

⚠ CAUTION
This instrument produces X-rays when energized. Do not insert any part of the body when energized– X-ray hazard.

⚠ CAUTION/ATTENTION
This equipment produces high intensity X-ray radiation when energized. To be operated only by qualified personnel. (Canadian

⚠ WARNING
This instrument contains hazardous voltages.
Voltage or current hazard sufficient to cause shock, burn or death.
Disconnect and lockout power before servicing.

China RoHS (Restriction of Hazardous Substances) pollution control logo. Indicates a 50 year EFUP (Environmentally friendly use period of the product).

⚠ CAUTION
Beryllium present. Avoid contact. Can cause health problems.

Instruction to read the user manual.

Other warning symbols used in this manual

Toxic substance. Hazardous to life.

CAUTION

This instrument weighs approximately 43 kg (95 lbs). Move it with care. At least two (2) people should be available to move or handle it.

All servicing to this equipment must be done by an Oxford Instruments trained engineer. Use of controls or adjustments of performance or procedures other than those specified herein is not permitted.

If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment might be impaired.

Radiation safety

The X-Supreme contains one small X-ray tube housed in a fully shielded and interlocked radiation enclosure. A radiation warning light, on each side of the instrument, indicates when X-rays are being generated inside this enclosure. Failure of any of the interlocks to the enclosure immediately shuts off X-ray power.

A full radiation safety assessment has been carried out by an independent Radiation Protection Advisor. This assessment included a survey of the radiation dose rates at the exterior surface of the instrument carried out at maximum X-ray power (30 kV, 100 µA). The survey reported that during normal operation there are no accessible radiation dose rate levels above 0.5 µSv/hr (0.05 mrem/hr). The unit can be operated like any other piece of laboratory equipment, and users do not need to wear personal radiation dosimeters.

To maintain this level of safety, any servicing required, within the instrument, must be carried out by an Oxford Instruments-trained service engineer.

The following sections contain further details relating to the radiation safety of the instrument.

Notification

X-ray equipment is federally regulated in Canada under the Radiation Emitting Devices (RED) Act and companion regulations. The X-Supreme is subject to Part XIV of Schedule II of the RED Regulations.

Oxford Instruments will notify the Canadian Radiation Authority of the sale of each X -Supreme with details of the customer's name, address and telephone number. Canadian customers should contact the radiation protection authority that has jurisdiction of the facility in which the X-ray system (X-Supreme) will be in use in order to comply with the appropriate federal/provincial/territorial rules of operation, given that such rules differ from one jurisdiction to another.

Simple rules should be in place for the use of the equipment i.e.

- Users must be familiar with the operating instructions contained within this manual.
- **Users must not try to gain access to the radiation enclosure.**
- Users must contact Oxford Instruments if any damage to the enclosure occurs.
- The instrument must be serviced regularly by an Oxford Instruments appointed service engineer.

Users should contact their local Oxford Instruments representative for specific advice.

Principals of radiation protection

The principal of radiation protection is to protect workers and the general public from radiation exposure, to minimize radiation hazard, and to maintain radiation exposure to As Low As Reasonably Achievable (ALARA).

There are three standard ways to limit exposure:

Time: radiation exposure can be reduced by minimizing the time of exposure.

Distance: distance is a simple and very effective way of dose reduction. If a distance between a person and a source of radiation is doubled then the exposure rate is decreased by a factor of 4. This is called the "Inverse Square Law".

Shielding: shielding is to place a material which will interact with radiation between a source and a person (or location of interest) to reduce exposure. Shielding material selection is dependant on the type and energy of radiation. X-rays require a high density material such as steel or lead.

Safety features

The principles of radiation protection described in the previous section have been incorporated into the design on the X-Supreme. Engineering controls and design features have been put in place to ensure safe operation of the device.

X-rays are produced from an X-ray tube manufactured by the X-ray Technology Group of Oxford Instruments. The X-ray tube is operated with positive anode (target) and a grounded filament. The target material is either titanium, palladium,

The hot filament emits electrons which are accelerated into the anode where X-rays are then generated (see appendix B for a description of characteristic X-ray emission). The tube is encased with silicon rubber and as such is not radiation shielded. X-rays generated within the tube are emitted through the beryllium exit window. At higher anode voltages some X-rays are emitted through the glass/ rubber body of the tube. The X-ray tubes are contained within a fully shielded and interlocked radiation enclosure.

The top section of the radiation enclosure is formed by the metal lid that is opened by the operator for sample loading (Figure 1.1). The lid is fitted with two safety switches which prevent the X-ray tube from operating unless it is closed. The primary interlock is the safety switch at the front of the lid shown in Figure 1.3. An additional safety switch with a roller actuator is fitted at the rear of the lid and is shown in Figure 1.4.

The bottom cover of the radiation enclosure (underneath the instrument) is locked and keys are only provided to suitably qualified and trained service personnel. The radiation enclosure therefore prevents direct access to the tube, increasing the distance between the operator and the source of X-rays, and the shielding material attenuates the stray radiation.

X-ray tubes are widely used in XRF analysers but these usually operate at higher potentials and power than on the X-Supreme and often remain energised between measurements to maintain instrument stability. A consequence of using a low power tube in the X-Supreme is it can be switched off when not needed, a condition which obviously gives zero external dose rate. This minimises the time of any exposure.

There is a key activated control at the front of the instrument (Figure 1.5) which prevents X-ray production when the key is removed or in the disable position.

The operator has a visual identification of the generation of X-rays within the instrument by two LED panel warning lamps on the top cover (Figure 1.1).

Figure 1.1 X-Supreme shielded lid and the two X-ray warning lamps.

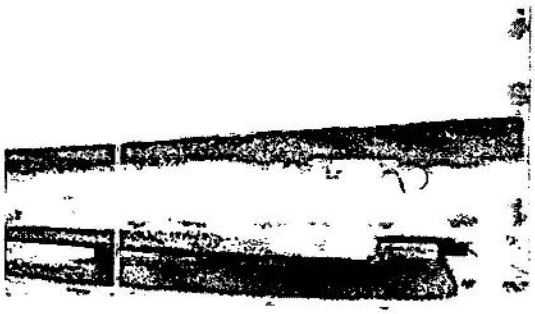

Figure 1.2 'Power On' light

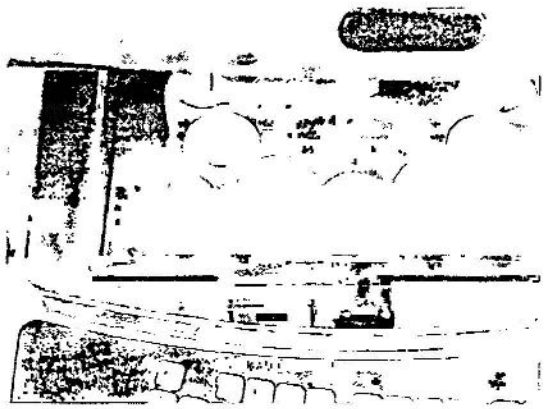

Figure 1.3 Front safety switch (lid)

Figure 1.4 Rear safety switch (lid)

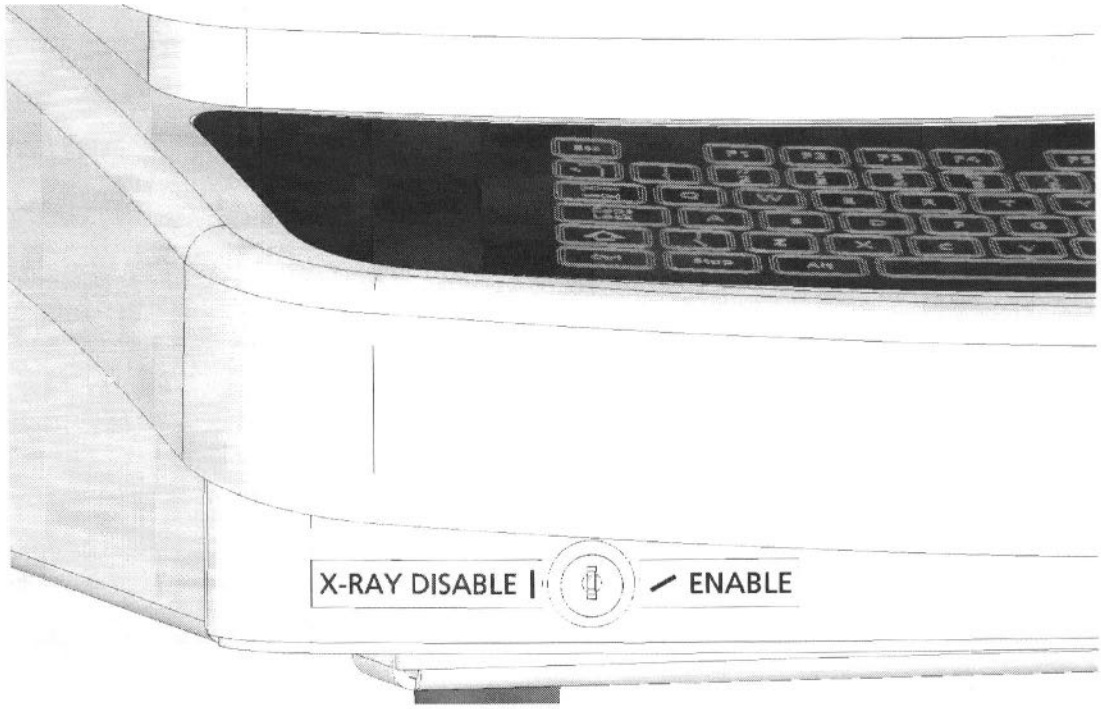

Figure 1.5 Key switch to enable or disable the generation of X-rays.

Biological effects of ionising radiation

Ionising radiation consists of subatomic particles or electromagnetic waves that are energetic enough to detach electrons from atoms or molecules, ionising them. The negatively-charged electrons and positively charged ions created by ionising radiation may cause damage in living tissue.
The biological effects of radiation are thought of in terms of their effects on living cells. For low levels of radiation, the biological effects are so small they may not be detected.

The body repairs many types of radiation and chemical damage. Biological effects of radiation on living cells may result in a variety of outcomes, including:

1. Injured or damaged cells repair themselves, resulting in no residual damage.
2. Cells die, much like millions of body cells do every day, being replaced through normal biological processes.
3. Cells incorrectly repair themselves resulting in a mutation. This mutation may contribute to the formation of a disease or diseases such as cancer.

Natural and artificial radiation sources are similar in their effects on matter. The worldwide average background dose for a human being is about 2.4 mSv per year (Report of the United Nations Scientific Committee on the Effects of Atomic Radiation to the General Assembly, 2000) which corresponds to an average dose rate of approximately 0.3 µSv/hr. This exposure is mostly from cosmic radiation and natural radionuclide in the environment.

Applicable dose rate limit

The 1990 Recommendations of the International Commission on Radiological Protection (ICRP) have been widely adopted by countries around the world. These specify an annual effective dose limit of 1 mSv for members of the public.

The Sievert and the rem (roentgen equivalent for man) are units of dose equivalent.

1 Sv = 100 rem

The dose equivalent is the product of the amount of energy deposited (J/Kg) and a radiation weighting factor which depends on the type of radiation. For example, for X-rays the weighting factor is equal to 1, for alpha particles the weighting factor is

Radiation survey of the X-Supreme

An independent radiation safety assessment has been conducted by a Radiation Protection Advisor from the Health Protection Agency (HPA) in the UK. A total of three radiation surveys were carried out on three instruments, each fitted with either a titanium, palladium or tungsten target X-ray tube operating at full power (30 kV, 100 µA). Dose rate measurements were carried out with the measurement probe in contact with the surface of the instruments, i.e. 0 cm distance from the instrument to the radiation monitoring probe.
Measurements of radiation dose rate were made around the X-Supreme using a Mini Instruments 900 D (H*10) (S/N 35567). The 900 D is an end window energy compensated Geiger Muller dose rate meter which is scaled over the range 0.5 to 1000 µSv/hr (0.05 to 100 mR/hr). Its response is maintained down to X-ray energies of at least 17 keV. A Mini Instruments 900 fitted with a 44B scintillation probe (S/N E0000596) capable of responding to X-rays at or above 5 keV was used as a search instrument to detect any elevated dose rate around the unit. All radiation monitoring instruments used by the HPA are tested by a United Kingdom Accreditation Service (UKAS) accredited calibration facility and the measurements are directly traceable to national standards. The UKAS is the sole national accreditation body recognised by government to assess, against internationally agreed standards, organisations that provide certification, testing, inspection and calibration services.

The dose rates measured at all accessible exterior and interior areas of the instruments were less than 0.5 µSv/hr.

The low level of stray radiation emission from the X-Supreme means any dose received by the instrument operator will be below the ICRP annual dose rate limit of 1 mSv under all foreseeable circumstances.

CE marking

CE	The **X-Supreme** system conforms with the appropriate EEC device directive for this type of instrument and has been subject to the conformity assurance procedures laid down in the Council Directive.

CE

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OXFORD INSTRUMENTS

DECLARATION OF CONFORMITY

This Declaration of Conformity is suitable to the European Standard EN 45014, "General criteria for supplier's declaration of conformity." The basis for the criteria has been found in international documentation, particularly in: European Commission Draft Certif 2005 – 2.

Oxford Instruments Analytical's liability under this declaration is limited to that set forth in the current Oxford Instruments Analytical's Terms and Conditions of Sale.

Applied Council Directive(s):
2004/108/EC Electromagnetic Compatibility.
2006/95/EC Low Voltage Directive

We, The Manufacturer:

Oxford Instruments Analytical
Halifax Road
High Wycombe
HP12 3SE
UK

declare under our sole responsibility that the following equipment

X-Supreme8000

to which this declaration relates is in conformity with the relevant provisions of the following standard(s) or other normative document(s) when installed in conformance with the installation instructions contained in the product documentation:

Emissions: EN 61326 : Classes A and B

Immunity: EN61326 : For light industrial and industrial equipment

Pertinent LVD sections of:
EN 61010-1:2002-02. Safety requirements for electrical equipment for measurement, control and laboratory use Part 1: General requirements.

Technical Information is maintained at:
Oxford Instruments Analytical
Halifax Road
High Wycombe
HP12 3SE
UK

08

We, the undersigned, hereby declare that the product(s) specified above conforms to the listed directive(s) and standard(s).

Signature:

Dan Varnam

Full Name: Daniel Arthur Varnam
Position: Marketing Director
Date: 11th February 2009

Oxford Instruments Analytical Limited
Registered office Tubney Woods
Abingdon, Oxon OX13 5QX
Registered in England, number 1044063
A subsidiary of Oxford Instruments plc
WEEE Producer Registration No. WEE/KJ0116XU

Safety systems

Electrical safety

The X-Supreme unit incorporates high and low voltage power supplies. It is not necessary to remove the covers during routine use, and the instrument should never be operated with its outer case removed.

Never assume that this equipment is isolated from the mains. Always check that the mains cord is disconnected from the mains supply.

Service work should only be carried out by trained engineers.

Safety and electrical compliance

The X-Supreme has been designed in accordance with IEC1010 ('Safety requirements for electrical equipment for measurement, control and laboratory use, Part 1'), which encompasses the European Low Voltage Directive. The instrument must be correctly installed and used, by trained personnel, only for the purposes described in this manual.

If you have any doubts about the installation or use of this instrument, contact either Oxford Instruments Analytical or your local Oxford Instruments representative.

Toxic materials

The X-ray tube windows and detector windows are made from pure beryllium. Under normal circumstances the windows are not considered to be harmful. However, if they become damaged in any way there is potentially an increased risk of contact with the beryllium. Inhalation of beryllium dust is harmful. In the event of a beryllium window becoming damaged, IMMEDIATELY CONTACT Oxford Instruments Analytical or your local Oxford Instruments representative for advice.

Always avoid contact with the X-ray tube and detector windows. These are very fragile and breakages are expensive as well as potentially harmful. Whenever possible use secondary safety windows in the tray for added protection.

Do not attempt to clean the beryllium windows following sample breakage or spillage. Contact Oxford Instruments Analytical or your local Oxford Instruments representative for advice.

Safety with samples

Always handle samples in accordance with the appropriate published Oxford Instruments method sheets and normal safety guidelines. If in doubt about the suitability of a sample for analysis, refer to the material safety data sheet.
Exercise caution when handling and analysing samples that give off flammable vapours
This equipment is not designed to be used in explosive atmospheres.

Spillage

Spillage on the outer surface of the X-Supreme can be wiped off using a dry or damp cloth. Do not use organic solvents.

If liquid is spilled from a sample cell, first check whether the liquid has been retained within the secondary (safety) window cell. If so, remove the liquid and replace the secondary window film.

If there is any possibility that liquid has seeped into the interior of the unit, isolate the instrument from the mains supply immediately and contact the customer support department at Oxford Instruments Analytical or your local Oxford Instruments service representative.

Fuse replacement

WARNING

The only replaceable fuse is in the plug (UK) and this could go open circuit in the event of an electrical fault. The fuse is easy to remove and replace using another of the same rating.

Unpacking the instrument

The X-Supreme is shipped in a single large box, and the contents of the shipment are listed in the shipping documentation. KEEP ALL PACKAGING. This will be required if it is necessary to return the instrument.

***Warning**: the instrument weighs approximately 43Kg /95 lbs and should be lifted by at least two people.*

- Remove the upper section of the packing.
- Carefully extract the instrument.
- Position the instrument on a suitable bench away from direct sunlight.
- Position the instrument so that the power cord is readily available for disconnection.
- Inspect for any visible damage. Inform your Oxford Instruments representative if damage is seen.
- Check all content against packing list. The accessories provided, will be packed separately and will vary with customer specification.

Prior to Installation

Operating conditions

Temperature	The temperature range which produces optimum performance of the X-Supreme is 10°C to 35°C.
Humidity	15% to 80% non condensing.
Maximum Altitude	2000 metres
Storage Temperature	-30^0 C to 50^0 C
Power requirements	85 to 264 V AC 47—63 Hz 400 VA max

Power conditioners

Power conditioners are recommended to provide maximum stability of the input voltage. Power conditioners should be specified in conjunction with your Oxford Instruments representative and according to local power conditions.

Bench requirements

The X-Supreme has the following external dimensions:

- Height : 575 mm (23") at maximum height of lid and monitor.
- Width : 784 mm (31")
- Depth : 604 mm (24")

Note: *A minimum bench width of 1140mm (45") and a minimum height allowance of 1020mm (45") is recommended.*

Installation

An Oxford Instruments representative will normally install and connect the instrument and carry out the basic functional checks necessary to ensure that the system is performing satisfactorily. The remainder of this manual assumes that this has already been done.

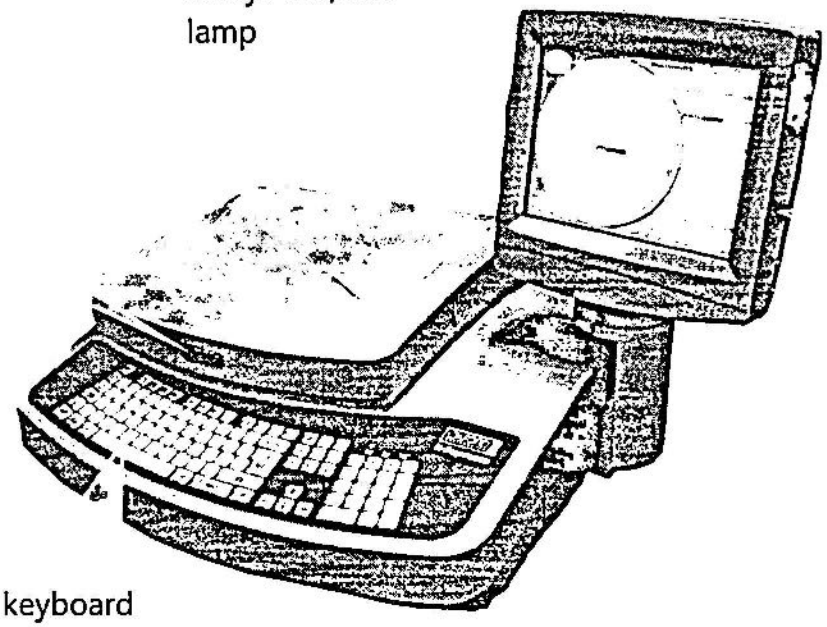
Note: *It is required that the instrument has been powered up for at least 2 hours before the first measurement is taken.*

Introduction to the X-Supreme

X-rays ON/OFF lamp

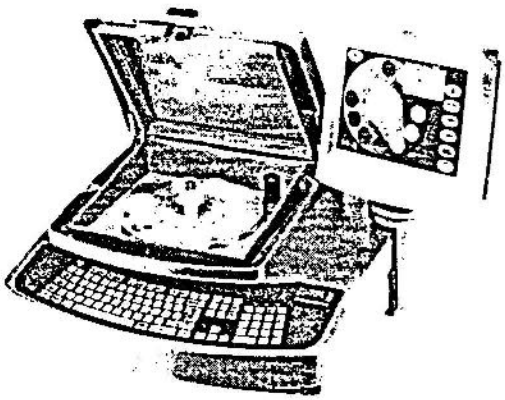
Press in to open lid

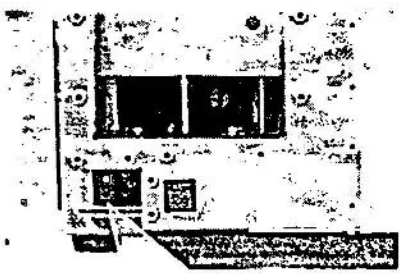
PC and monitor



Integrated keyboard with dedicated STOP/ START buttons.

Instrument tray—up to ten (10) samples may be loaded





External ON/OFF switch and mains supply connector.

Mains Power ON light

The instrument is identified by model and serial numbers located on plates at the rear of the instrument. Any enquiries and correspondence relating to the instrument must quote these numbers.

External connections

USB (Universal Serial bus)	3 off
Ethernet connector (RJ45)	1 off

The X-Supreme is shipped with a USB mouse. USB ports are located in the underside and side of the PC/Monitor (Figure 2.1).

Additional items may include a printer or a separate keyboard. These all have USB connectors. If customers wish to connect more than three USB devices to the instrument at the same time, they will need to add an externally powered USB hub.

Note 1: Where customers use their own peripherals – printer, keyboard, CD read/ writer, label printer – in conjunction with the X-Supreme unit, they need to specify USB connections and follow the relevant manufacturers' instructions.
Oxford Instruments is not responsible for problems arising from or with such equipment fitted by users.

General operation / Switching ON / OFF

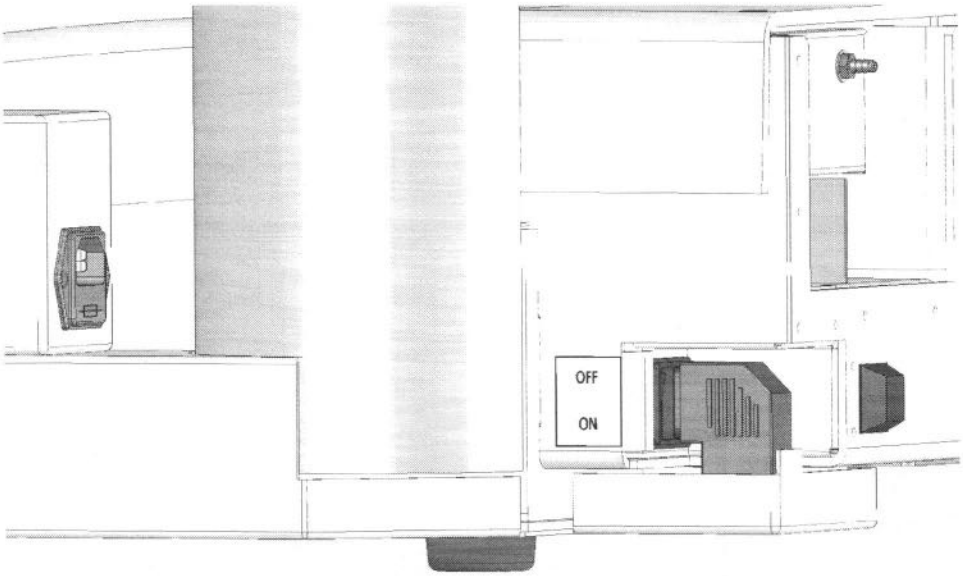


Figure 2.1 The mains input socket is at the rear of the Canadian Control Module (shown towards the left of the image)

The mains power cable connects to the input at the rear of the Canadian Control Module (Figure 2.1 and Figure 1.6). A cable connects the control module to the input of the X-Supreme at the rear of the unit, shown on the right of Figure 2.2. A panel fixed to the base of the X-Supreme by tamper proof screws prevents this

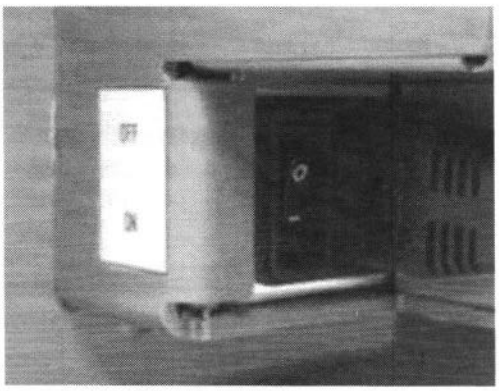


Figure 2.2 On / Off switch

Two ON/OFF switches are used to power the spectrometer ON and OFF. The first switch is located at the back of the spectrometer on the right-hand side (Figure 2.2). The '0' position is the OFF position and the 'I' position is the ON position. A second power ON/OFF button is located on the control panel (Figure 1.6). Both switches must be ON for power to be applied to the X-Supreme.

Once the X-Supreme has been correctly connected to the mains supply, set the switch at the rear of the instrument (Figure 2.2) to the ON position (I) and then press ON/OFF button on the control panel (Figure 1.6) once to provide power to the spectrometer and its computer. When the spectrometer and computer are switched on, the green light ('Power' LED – see Figure 1.2) at the front of the instrument is lit. At this point, Windows and the X-Supreme software will automatically be loaded, and a Password Login dialog box will appear on the screen (Figure 2.3).

Note: If the 'Power' LED does not light, i.e. power is not supplied to the instrument, check the Emergency Stop button is not in the down position.

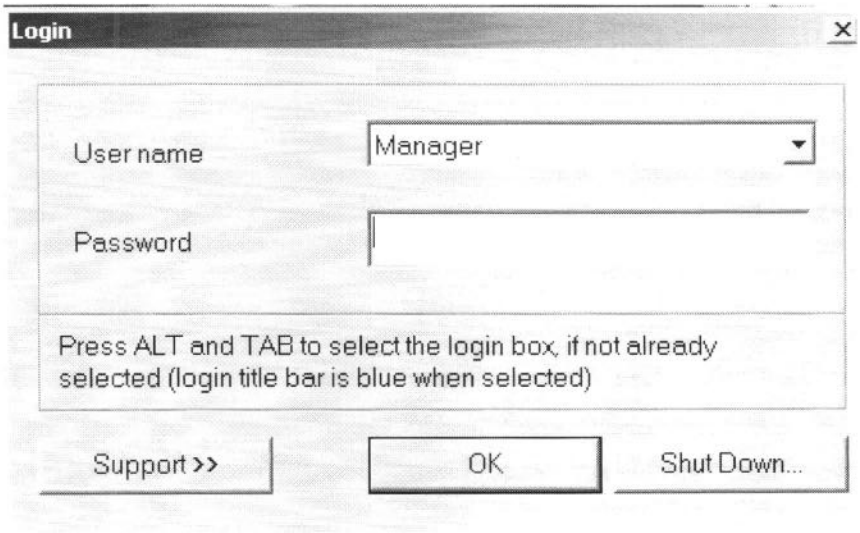


Figure 2.3 Login screen

No password will have been set when the X-Supreme is switched on for the first time.

Under User name, use the drop-down menu and select 'Manager'.

Click on the Password box and type 'manager' as the default password.

Click on 'OK' or press 'Enter' to continue.

The X-Supreme Main Analysis menu will now appear on-screen.

Note: New user passwords and levels of access (security) to new users may be set up (manager level access required to do this). From the Main Analysis menu select 'Other Functions F12', Preference Tab, User Setup (F8).

On subsequent occasions users may log in to the system using their given password.

- To select the appropriate user name, press the ↑ and ↓ keys OR type in the first letter of the name OR click on the arrow to display the drop-down menu.
- To move to the password window, click on the window or press Tab.
- Type in password.
- Click on OK or press 'Enter' to bring up the X-Supreme main analysis menu.

To change a password see 'Other Functions'/ Preferences/ Change Password.

Oxford Instruments recommends that the X-Supreme remains switched on at all times. When the spectrometer is not analysing, it remains in standby mode (the X-rays are off, and analysis positions for both heads are covered). If it becomes necessary to turn the X-Supreme off, exit the X-Supreme software (Main Analysis Menu) and choose 'Shut Down' in the Login screen (Figure 2.3). Windows will auto shut down. Press the Power ON/OFF button on the control panel once. This will power off the spectrometer and the PC.

General operation / X-Rays Enable/Disable key switch

There is a key activated control at the front of the instrument (Figure 1.5) which prevents X-ray production when the key is removed. The key must be inserted and in the X-ray 'enable' position for X-rays to be generated. The key can not be removed when it is in the 'enable' position.

General operation / Standby mode

When the spectrometer is not analysing, it remains in automatic Standby mode: X-rays off, and the sample tray covers the analysis positions.

General operation / X-Rays ON/OFF

When the key is inserted and in the 'enable' position then X-rays are automatically switched on when the spectrometer begins to analyse and are automatically switched off at the end of the analysis.

General operation / X-ray warning lamps

The X-ray warning lamps will be lit when the X-rays are switched on. These lamps are located on the sample chamber lid (Figure 1.1). The lamps are LED units and are expected to operate for the lifetime of the X-Supreme unit. In the event of one lamp failing, the unit will continue to operate. In the very unlikely event of both lamps failing, any X-rays are automatically switched off. In this case, you should call in your Oxford Instruments engineer.

Cleaning the instrument

Outer cover: Use a damp, not wet, cloth. Do not allow water to get

Computer screen: Use a dedicated screen wipe.

Annual service

An annual service should be performed by an Oxford Instruments qualified Service Engineer.

> ***Note**: Routine radiation leakage surveys may be required by the state or country. See the governing body's regulations or the agency.*

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Please read the X-Supreme Safety sections before using this section of the User's guide.

How to use the X-Supreme software

Introduction

Basic user training is provided at installation. The safety guide provides an overview of the basic operation of the X-Supreme. Application help, in the form of 'Method Sheets' is provided as part of an X-Supreme package. More advanced training can be arranged through your Oxford Instruments representative.

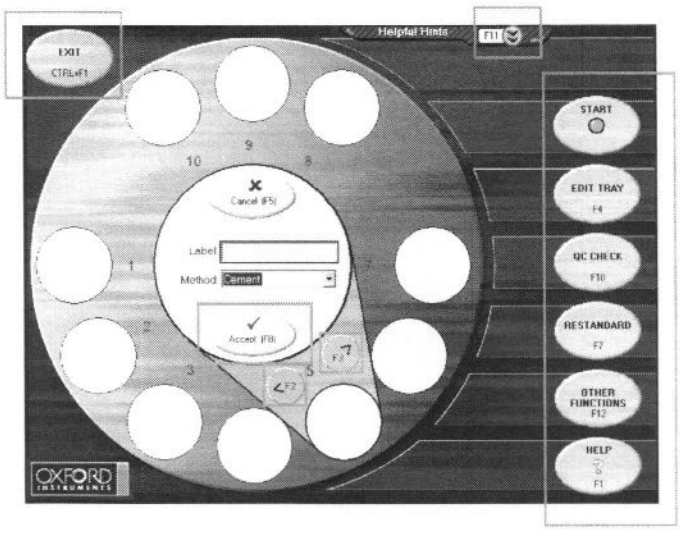
Day to day help is provided within the X-Supreme software through on-screen localised bubble help, and a comprehensive on-line guide (English text) can be viewed.

All X-Supreme functions can be executed using a mouse or the keyboard.
Note: The optional touch screen allows the execution of the functions by tapping directly on the X-Supreme monitor.

Using the mouse

Hover the cursor over the screen feature and single click on the left-hand mouse button. (Note: a right-hand mouse click can be used on many, but not all occasions).

Example: Main screen; selecting Main screen features using the mouse.

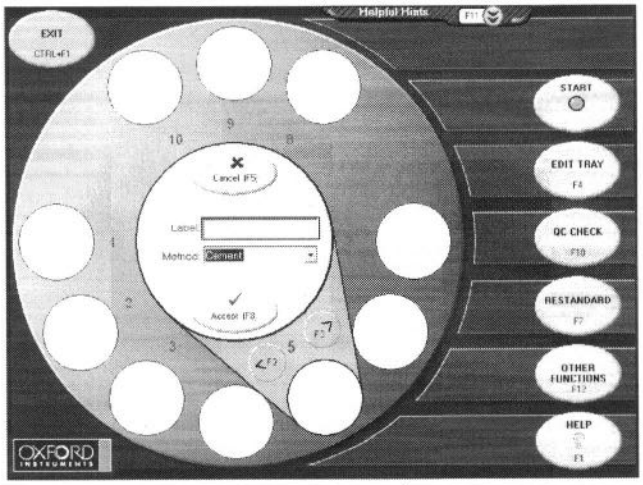


Mouse click on any of these features to activate their function.

Using the keyboard

Many features can be activated using the function keys on the keyboard. Select the function key detailed on the screen feature.

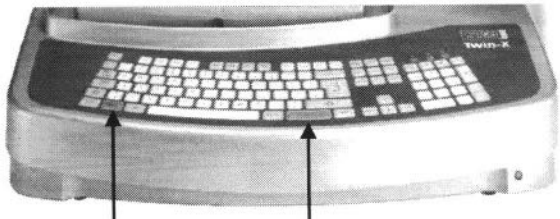
Example: Main screen; selecting Main screen features using the keyboard.



Feature	Function Key
EXIT	*Control button + F1*
EDIT TRAY	*F4*
QC CHECK	*F10*
RESTANDARD	*F7*
OTHER FUNCTIONS	*F12*
Accept	*F8*
Move tray selection anti-clockwise	*F3*
Move tray selection clockwise	*F2*

Starting and Stopping an analysis

Dedicated keys can be found on the X-Supreme integrated keyboard.



Red key:
Stop analysis.

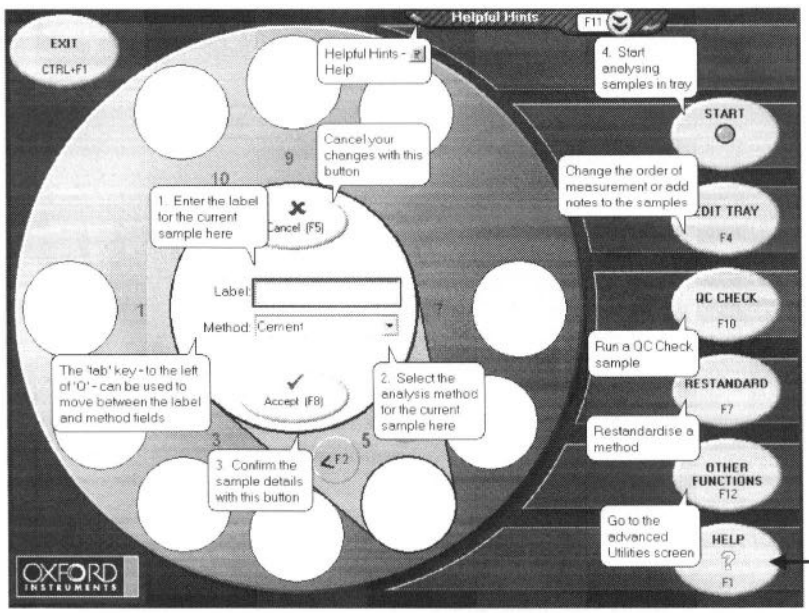
Green key:
Start analysis.

Getting help

On-screen bubble help is available on many screens to explain the function of each screen feature. Click on a question mark symbol or select F1 from the keyboard to activate the bubble help. The question mark symbol may appear on a 'button' or as an icon.

Example: How the bubble help button may appear on different screens

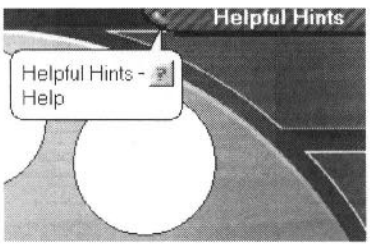
Example: Main screen bubble help

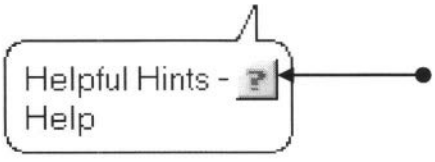


Screen 'Help button'.

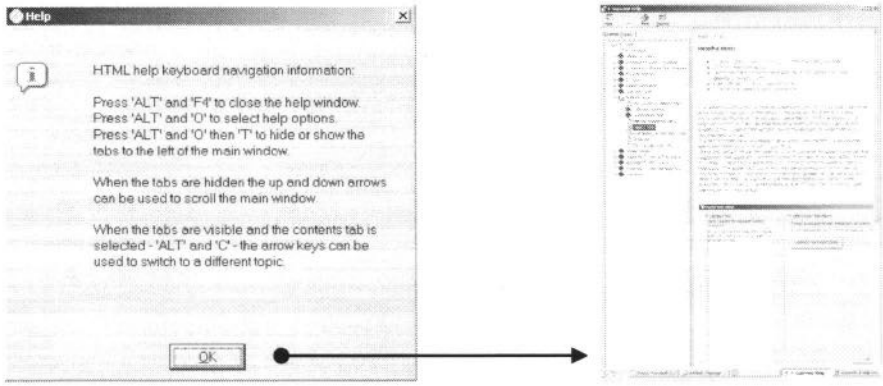
A question mark symbol within an on-screen bubble, indicates that further help on this feature is available.

Example: More help on 'Helpful Hints' is available.

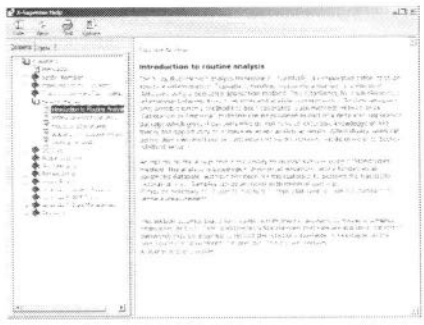




Click on the question mark symbol to open the on-line help file. The on-line help information box will open first. Click 'OK' to open the on-line help file (English text).



Navigate through the help file using the mouse or keyboard.



Mouse: Double click to open a book.
: Single click to open a topic.

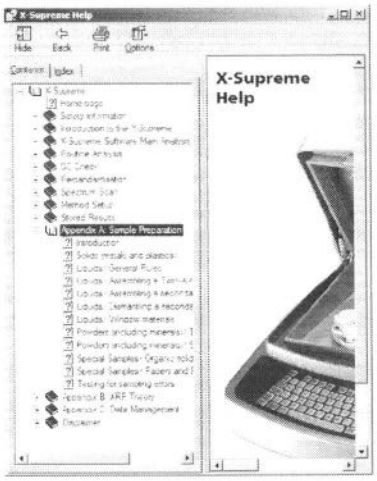
Keyboard: Navigate using a combination of the arrow keys and the 'Enter' button.

Use the 'Search' and 'Index' tabs to find topics of interest.

How to analyse a sample

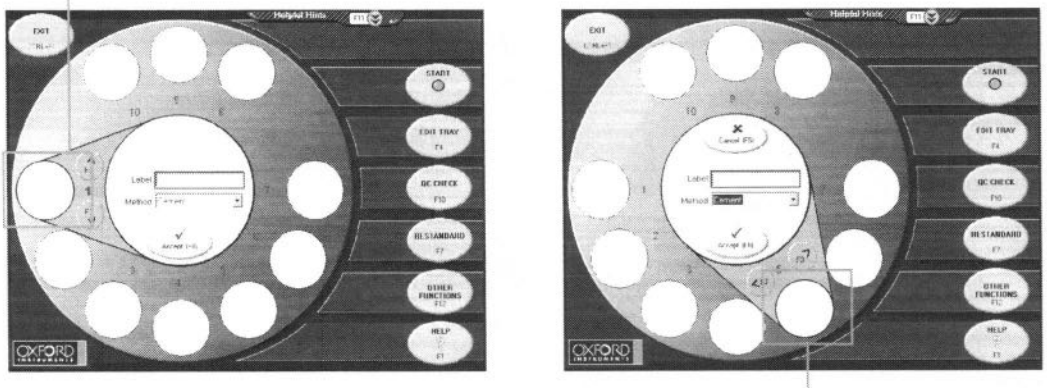
Carefully prepare the sample. Refer to the on-line help and Method sheets, and Application Notes where appropriate.

Example: On-line help, Appendix A, Sample Preparation.



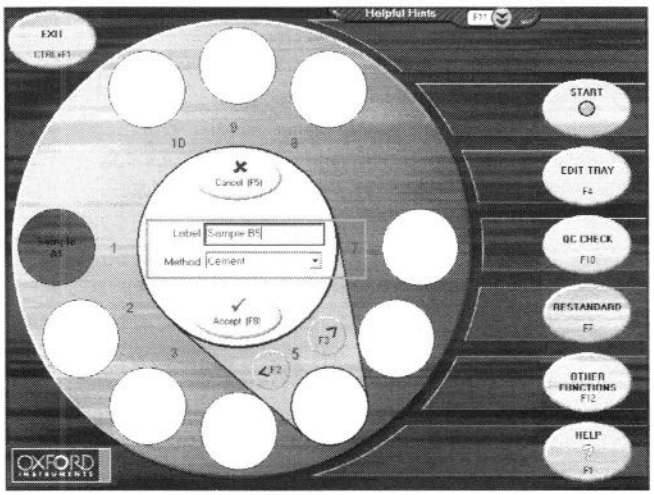
Position the sample in the instrument tray. (n.b. Use a secondary window for liquid sample analysis). Any tray position can be used. Identify the sample on the screen tray with a title.

Example: Tray position 1 selected



Example: Tray position 5 selected

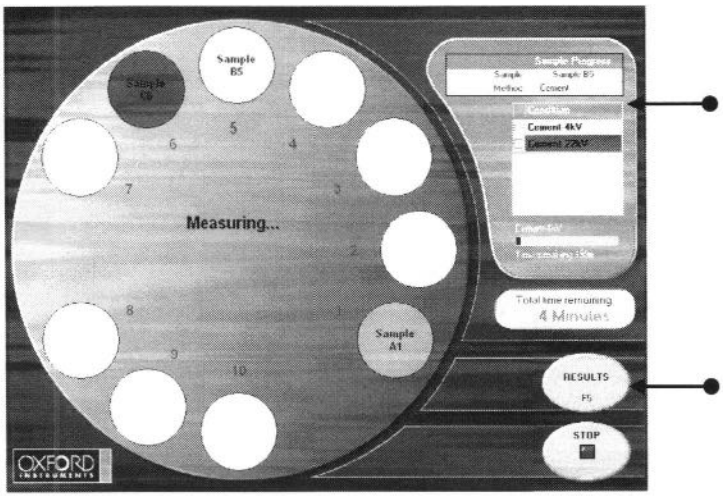
Example: Sample in tray position 1 has the title 'Sample A1'. Click on 'Accept [F8]' (or use keyboard function key F8) to name sample in tray position 5 'Sample B5' and use the method named 'Cement'.



Select a method to analyse the sample. Each sample can use a different method.

Click on 'Start' or use the keyboard to begin the analysis.
The screen tray will rotate to place the sample being measured at the top.
The 'yellow' tray sample status indicates that the sample is being measured
The 'red' tray sample status indicates that the sample has not been measured.
The 'green' tray sample status indicates that the sample has been measured.

Example: One sample has completed its measurement (green), one sample is in the process of being measured (yellow), and one sample is waiting to be measured (red).

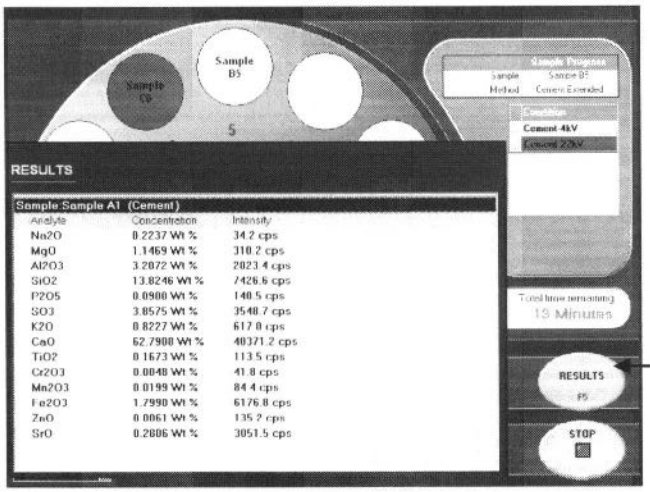


Details of the current measurement are displayed in this section.

Results

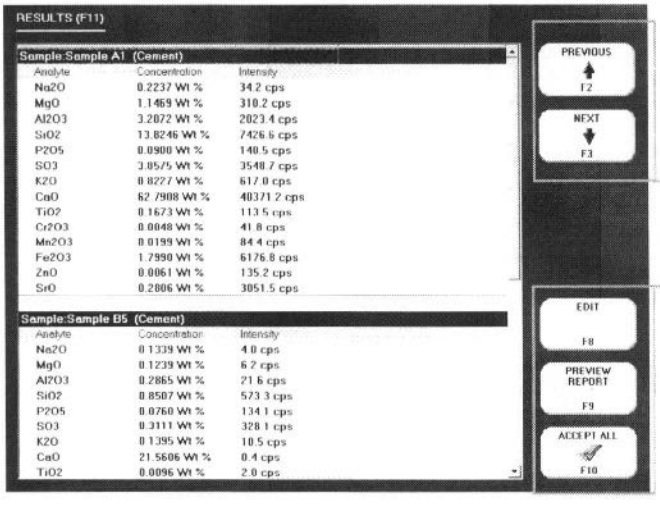
The result of an analysis is displayed automatically when all samples on the tray have been measured. However, the 'result' page can be viewed at any time by clicking 'Results F5' or selecting keyboard function key F5. Only completed results are displayed.

Example: One sample (Sample A1) has been measured and the analysis is continuing. Results (F5) has been selected, to display the completed result.



Select Results (F5) a second time to close the intermediate result page.

Example: The analysis of all the samples has finished and the final results page is displayed.



Use these buttons, or F2 and F3 on the keyboard, to scroll through the results list to

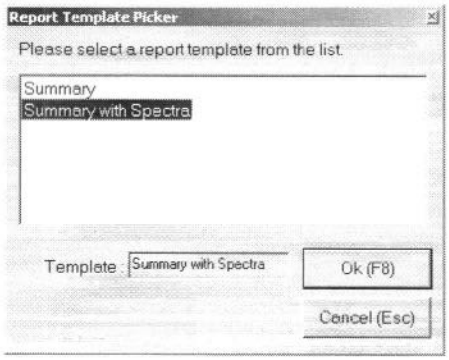
Use these buttons to :

- *Edit the results (F8)*
- *Preview a report (F9)*

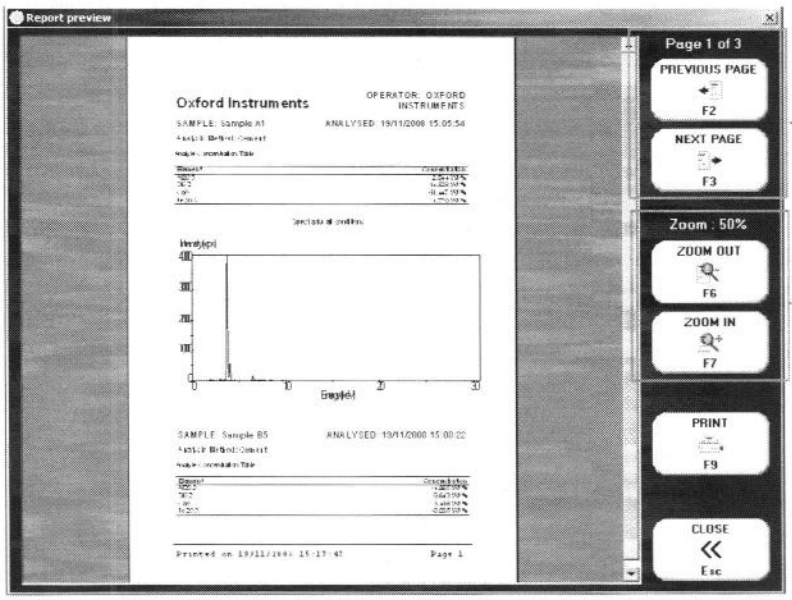
Preview Report (F9)

Choose to preview a summary or a full report (summary with Spectra).

Example: Click on Ok button or select F8 on the keyboard to preview a full report.



Use these buttons to scroll through the report. (Use F2 and F3 on the

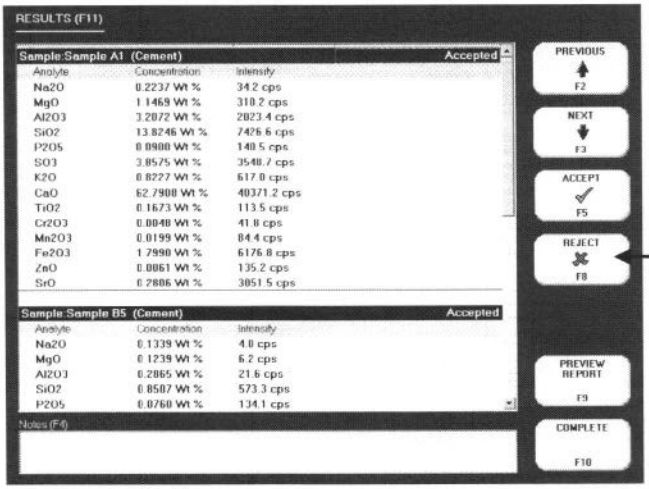


Use these buttons to zoom in or out of a page (Use F6 and F7 on the keyboard).

Example: Click on the 'Print' button or select F9 on the keyboard to print this three (3) page report. Select the 'Close' button, or The 'ESC' key on the keyboard to return to the results page without printing the report.

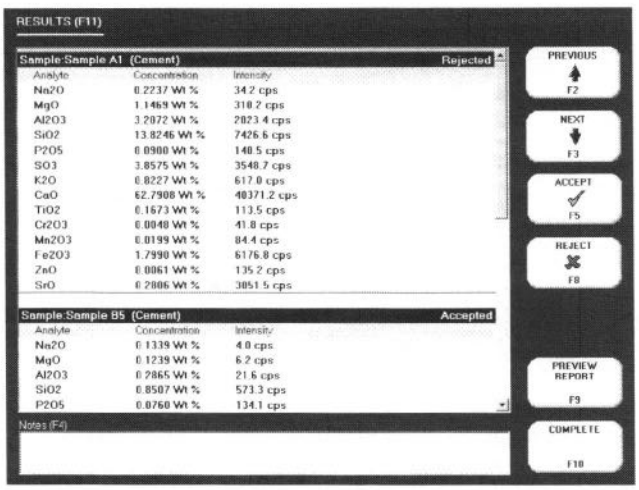
Edit results (F8)

Example: All displayed results will be saved to the results database (Accepted).



To reject a sample from being saved, first choose the sample in the list and then select Reject' (F8).

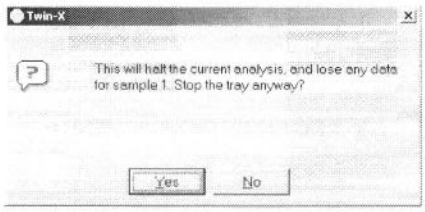
Example: One result has been selected to be rejected from the results database.



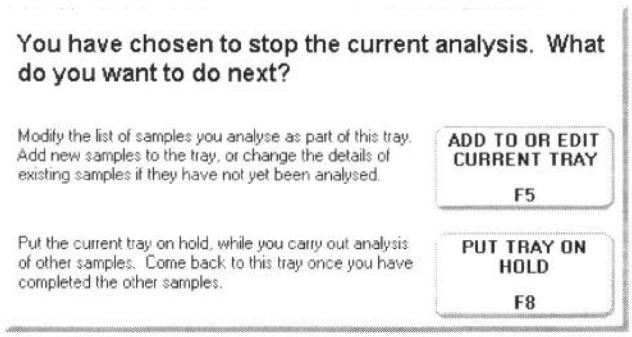
Stopping analysis

The analysis can be stopped before all the samples on the tray have been measured.

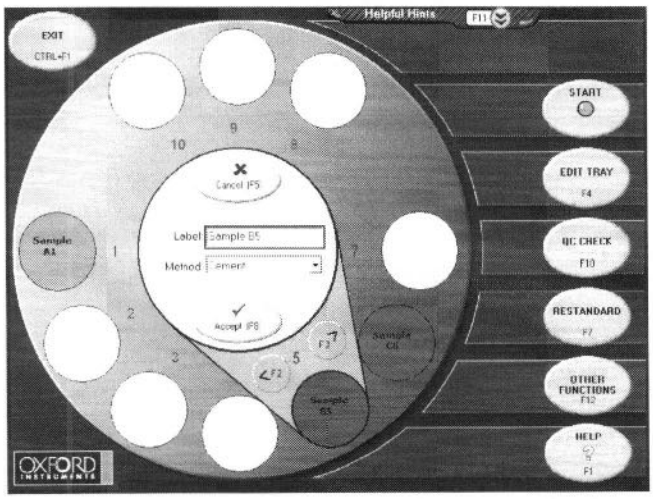
Example: Sample 1 was being measured when the 'Stop' command was given. The user may proceed with halting the measurement by choosing Yes.



The user must now decide to either edit the tray or put the tray on hold.

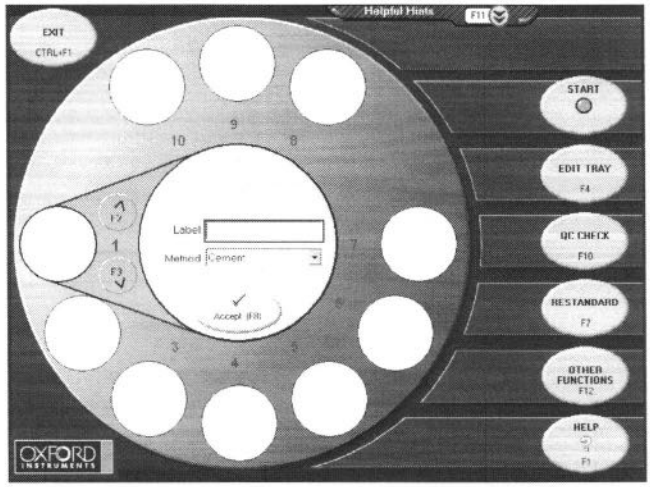


Example: User clicks on 'ADD TO OR EDIT CURRENT TRAY ' or presses function key F5.



Any sample that has not been measured (red colour) can be edited, that is; name changed, method changed or the sample deleted. New samples may be added to the tray.
Click on 'Start' (or use keyboard button) to measure edited tray.

Example: User clicks on 'PUT TRAY ON HOLD' or presses function key F8.

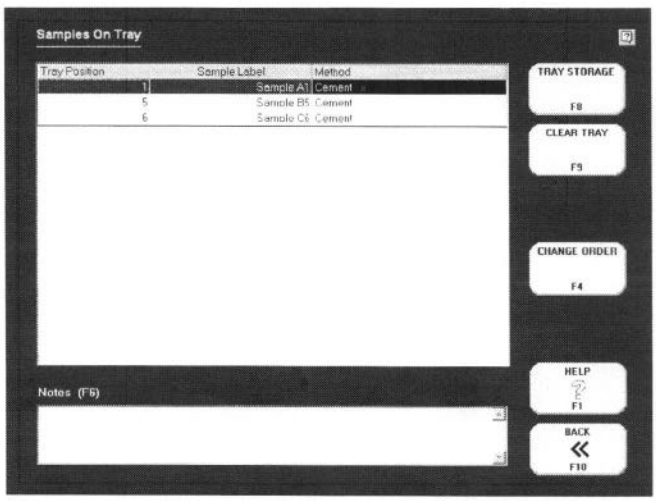


The tray is cleared of all samples. The user may now enter details of a new analysis and measure the new samples. At the end of the new measurement the tray 'on hold' will be displayed for the user to continue from where the measurement was stopped.

Sample measurement—order of measurement

Samples are measured in the order that their details are accepted on the screen tray. The order may be viewed by clicking on 'EDIT TRAY' or pressing function key F4.

Example: The sample in tray position 1 will be measured first. The sample in tray position 5 will be measured next, followed by the sample in tray position 6.

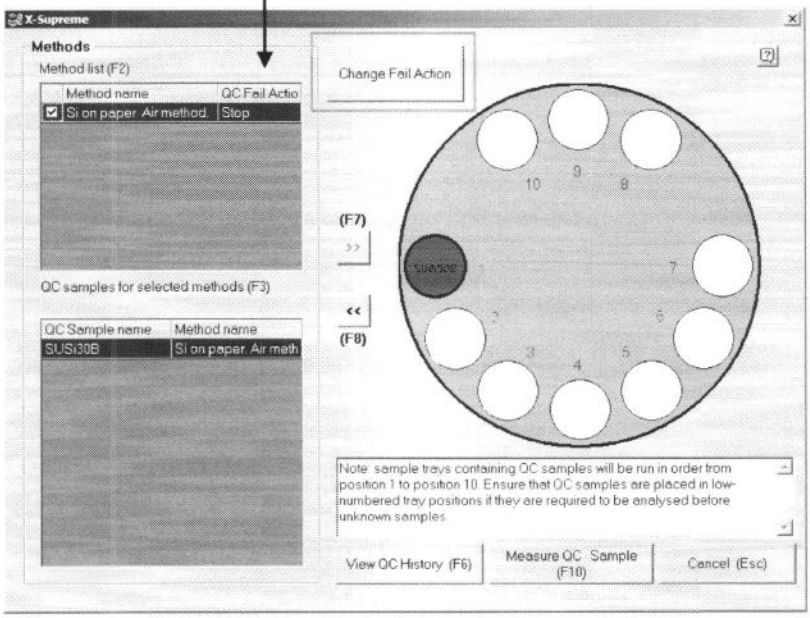


A user with sufficient login access may:

- *Change the order of measurement.*
- *Clear the tray*
- *Store the tray details to a file location.*

QC Check F10

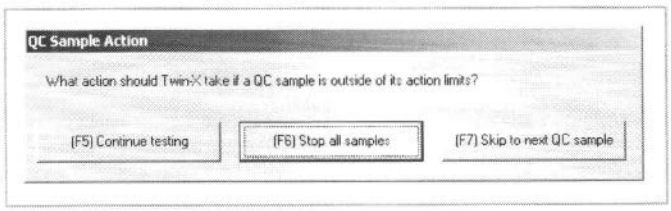
The action to be taken if the QC check sample fails is shown in the 'QC Fail Action' column.



Click on this button to change the action to be taken if a QC check sample fails.

Be careful to use the correct QC check sample.

Example: All measurements will 'Stop' if the QC check sample (SUSi30B) fails.

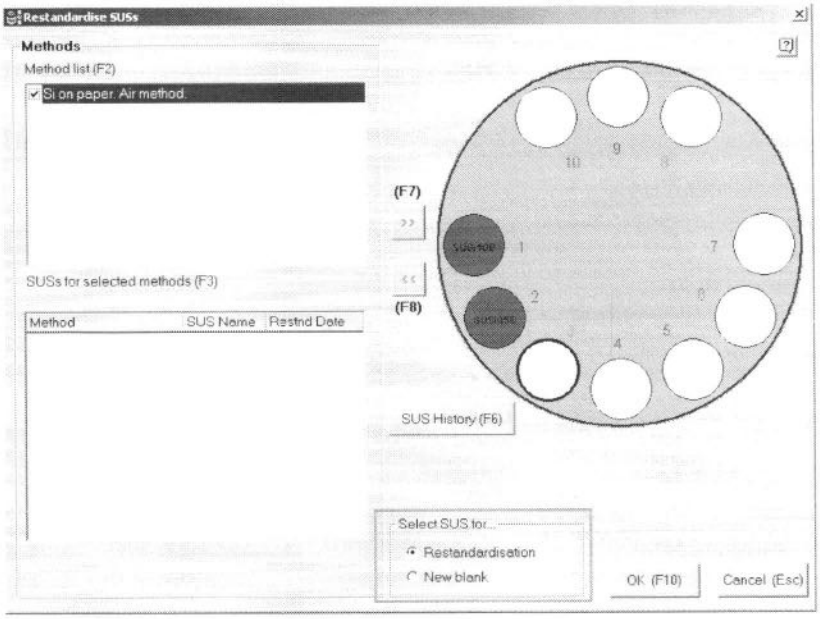


Choose one of three analysis options to take if the QC check sample fails:

- *Continue analysing (F5)*
- *Stop tray (F6)*
- *Measure next QC sample on tray (F7)*

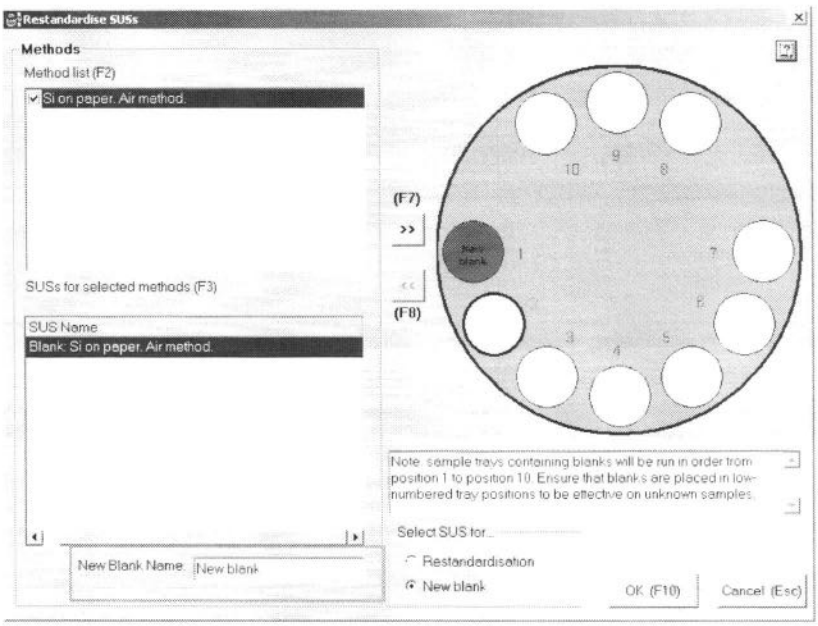
Restandard F7

Example: Select OK (F10) to restandardise the method 'Si on paper. Air method' using Setting up Samples (SUSs) SUSi10B in tray position 1 and SUSi05B in tray position 2.



Select with the radio button to perform a restandardisation or to measure a new blank SUS.

Example: Select OK (F10) to measure a new blank SUS for the method 'Si on paper. Air method' in tray position 1. The name 'New blank' has been assigned to the SUS.



Enter a new name or use the default: "Blank: method name"

DISCLAIMER

Appendix: XRF Theory

An introduction to XRF analysis

X-rays, which form a part of the electromagnetic spectrum (see Figure A1), are generated when atoms of elements are struck either by particles, such as electrons, or by other higher-energy radiation. The process of primary X-ray emission, which takes place inside an X-ray tube, can be considered simply as the production of characteristic X-ray wavelengths of the tube anode element when it is bombarded by a beam of high-energy electrons.

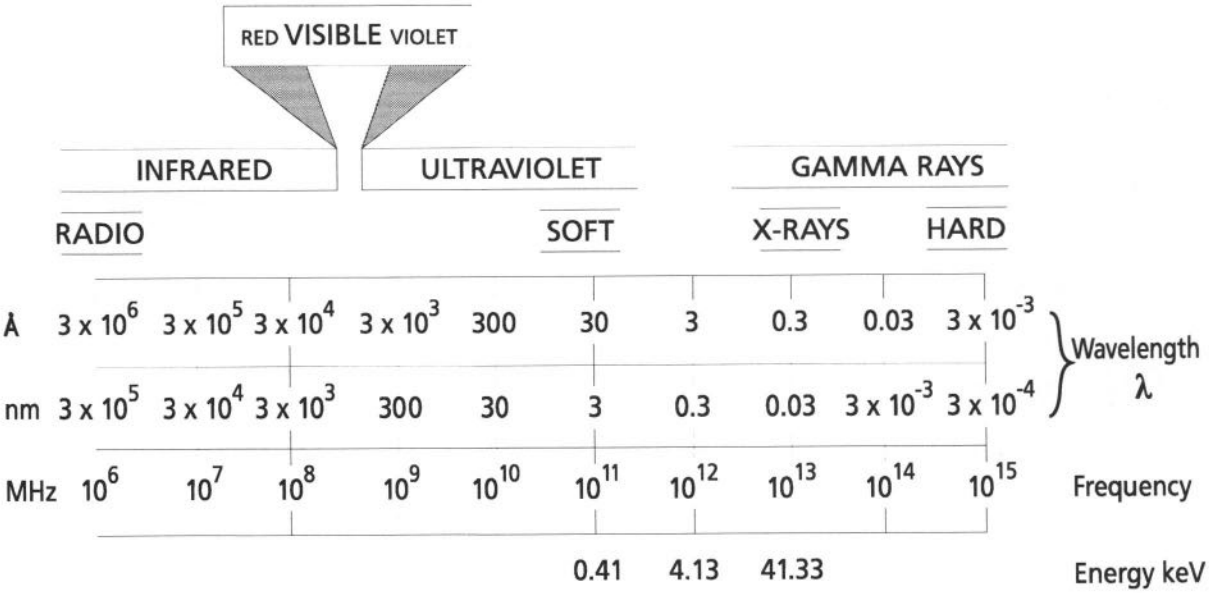


Figure A1: Part of the electromagnetic radiation spectrum

Note: Although the X-ray region can be defined by its frequency in MHz, it is more convenient to use wavelengths specified in either angstrom units (1Å = 10^{-10}m) or the SI unit nanometres (1nm = 10^{9}m), or to use Energy in units of keV.

Characteristic X-rays are often also expressed in terms of energy in keV (inversely proportional to wavelength). This means that the energy of the X-ray emission lines in keV increases with increasing Atomic Number. The relationship between energy and wavelength is as follows:

$$E(keV) = \frac{12.4}{wavelength(\text{Å})}$$

As the atomic number of an element increases, the wavelength of its most sensitive characteristic emission spectral line, known as the K line, decreases. Low atomic number elements are often called 'light elements' and their X-ray radiations are absorbed by air. Conversely, high atomic numbers are called heavy elements.

It is important to recognise that each element has a number of characteristic lines and that these occur as a result of internal electron transitions within the atom. The lines occur as the result of the excess energy liberated when electrons from higher energy levels 'fall down' to take the place of K, L and M electrons which have been ejected by the exciting radiation (Figure A2).

These discrete energy differences give rise to characteristic element lines. These are discussed below and shown in Figure B3. When X-rays are used as a source of radiation to excite other elements the process is known as X-Ray Fluorescence (XRF).

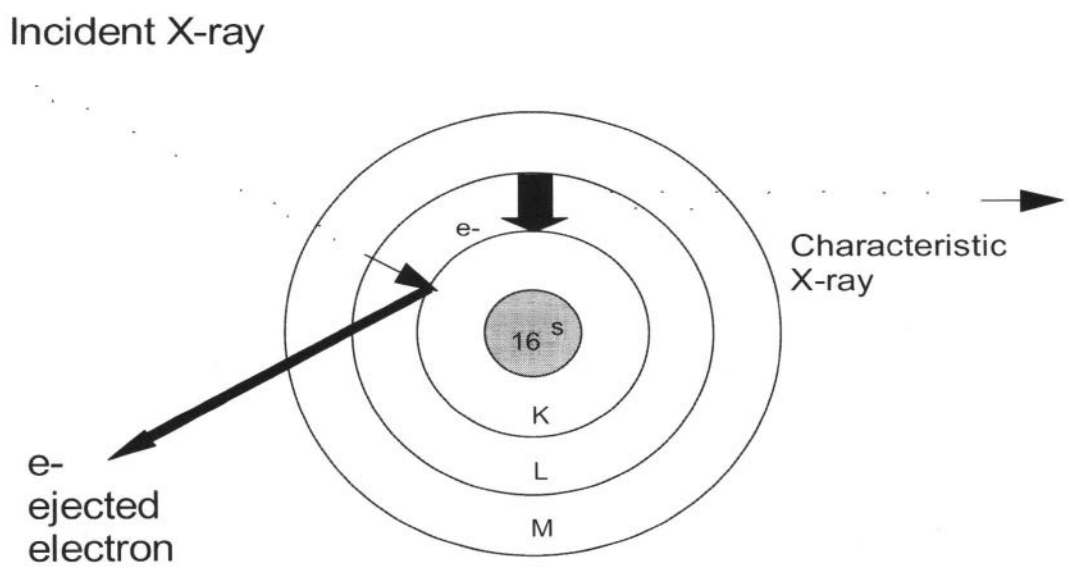


Figure A2: Illustration of internal electron transitions within the Sulfur atom

Figure A3 represents the electron energy levels about the nucleus of a sulfur atom for the K, L and M shells. Each transition results in the emission of an X-ray photon, it's energy being equal to the difference between the two energy levels involved. The emitted K lines have a higher energy than the emitted L lines or the emitted M lines. Wherever possible, XRF analysis uses the K lines because they are the most sensitive.

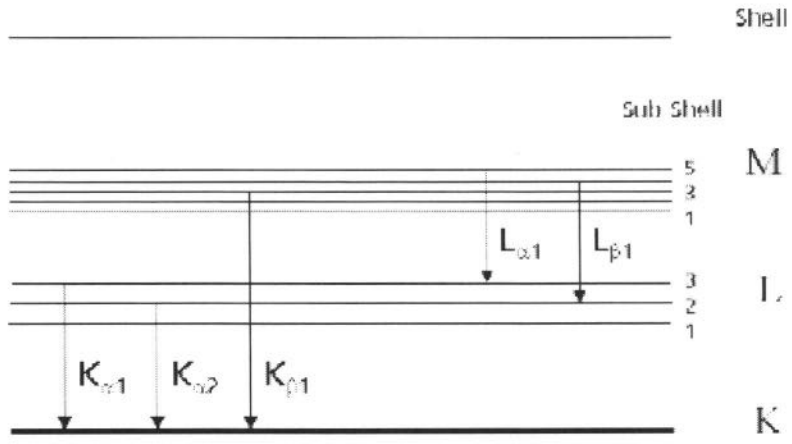


Figure A3: Fluorescence of sulfur atom

Selective excitation and primary beam filtration in EDXRF

Energy dispersive XRF (EDXRF) spectrometers have detectors that see the X-rays of all energies simultaneously. This advantage has to be used carefully to ensure a maximum count rate that the detector will accept.

In the EDXRF spectrometer, therefore, it is important to reduce the percentage of the spectrum taken up by background counts, and this is achieved by selective excitation and primary beam filtration. In practice, this means that the excitation voltage used for a particular range of elements is the voltage that effectively excites the highest energy line and no more. This avoids producing large amounts of continuum background higher in energy than the element with the highest energy that is to be analysed under these conditions. In addition, placing a filter between the X-ray tube and the sample will modify the position of the peak energy available for excitation.

In this way, the X-ray beam reaching the sample has a spectral distribution that favours good excitation and low backgrounds for a particular region of the spectrum. To achieve this, the X-Supreme is fitted with programmable multiple filter changers which provide automatic filter changing between different ranges of elements in multi-element applications. The choice of filter is matched with an appropriate combination of tube voltage and beam current. Such analytical procedures may be stored for automatic repeat use where stored sets of analytical conditions (methods) are re-used.

Absorption of X rays

Knowledge of the nature of X-ray absorption is fundamental to the understanding of nearly everything that happens in X-ray analysis. Two basic facts need to be grasped:

i. X-rays are absorbed according to Beer's law.

$$I_x = I_o e^{-\mu\rho x}$$

Where
I_x = the intensity transmitted through a thickness x
I_o = the initial incident intensity
μ = the mass absorption coefficient
ρ = the density
x = the thickness of the absorber

ii. The value of the mass absorption coefficient changes with wavelength, and a plot of the variation between absorption and wavelength shows maxima followed by a sharp drop - the absorption edge. Figure A4 shows this effect.

Absorption edges occur at each critical excitation wavelength (5.0185 Å), i.e. at the critical excitation energy (2.4700 keV) of Sulfur.

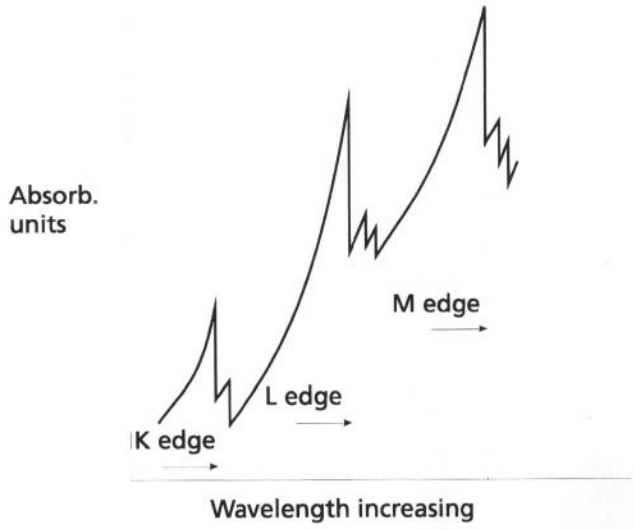


Figure A4: Diagrammatic representation of absorption edges

Elements of higher atomic number are normally called heavy elements and, because they have many electrons, they have a series of absorption edges – K, L, M, etc. The higher the atomic number the higher the energy (and the shorter the wavelength) of the K spectral lines. Conversely, elements of a low atomic number are called light elements and have K spectral lines of low energy (or long wavelength).

From a study of a set of mass absorption tables it is clear that the magnitude of the mass absorption coefficient at the energy being studied plays a principal role. This is the whole basis of inter-element (matrix) correction procedures, which are needed in the analysis of multi-element materials. The make-up of the material or the matrix determines how much of the excited characteristic radiation escapes from the sample: this is measured and related by calibration to the concentration of the element.

Precision

The term 'precision' is used to describe the repeatability of the instrument, which is normally quoted as the standard deviation

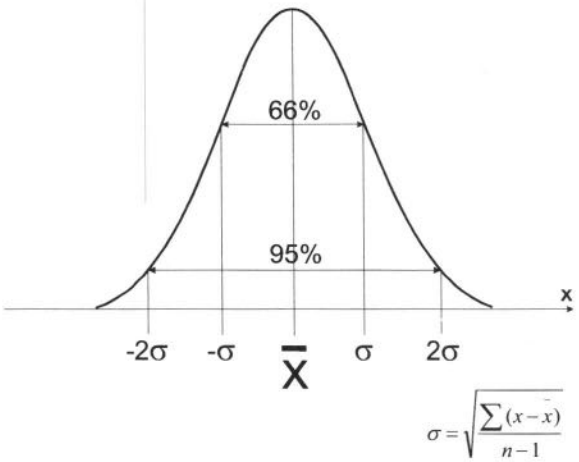


Figure A5: Typical Normal distribution

Figure A5 shows a typical Gaussian distribution, which can be used for any measured value where random events are involved. From this it can be seen that deviation from the mean value (above and below) for approximately 66% of all the measured values is a value which is called sigma (σ). Twice this standard deviation value either side of the mean accounts for 95% of all the measured values, and three times this value either side of the mean takes in 99.97% of all measured values.

These break-points are frequently used to describe various levels of confidence. That is to say, there is a 1 in 20 chance in getting a result with a deviation more than twice the one sigma (σ) value above or below the mean.

With X-ray analysis the measurements are made up of a series of numbers accumulated by counting photons of a particular energy entering a detector. This counting process obeys a Normal distribution and one standard deviation (1σ) is

$$1\sigma = \sqrt{total\ number\ of\ \ photons\ counted}$$

and

total number of photons counted = (C + BEC) * QT

where
C = concentration
BEC = background equivalent concentration
Q = sensitivity (counts per second per concentration unit)
T = counting/analysis time (in seconds)

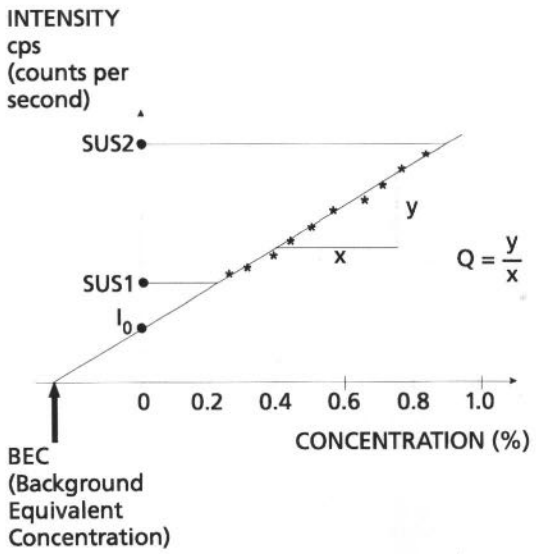


Figure A6: Concentration vs intensity

In Figure A6:

$$1\sigma = \sqrt{(C + BEC) * QT\ counts}$$

however:

QT counts = 1%

so that:

$$1\sigma = \frac{\sqrt{(C + BEC) * QT}}{QT}\% = \frac{\sqrt{(C - A_o)A_1}}{T}$$

$$1\sigma = \sqrt{\frac{(C + BEC)}{QT}}\%$$

Note: A_0 is negative

From this it is seen that:

$$\sigma \text{ at zero} = \sqrt{\frac{BEC}{QT}}\% = \sqrt{\frac{-A_o \times A_1}{T}}$$

Three times this value, i.e. $\sqrt{\frac{BEC}{QT}}$, is known as the limit of detection (LOD).

It should be remembered that precision and time are intimately related. For example, if the requirement is to improve the value for sigma at a particular concentration level by a factor of two, then the counting time must be increased by a factor of four. That is to say, the time must increase by the square of the improvement required.

Accuracy

The terms 'accuracy' and 'precision' are often interchanged and wrongly used. The definitions are quite clear:

- ***Precision*** is the ability of the instrument to *repeat a result* from the same sample.
- ***Accuracy*** is the *correctness* of this result compared with other reference methods of analysis.

A useful way of expressing accuracy is in terms of the standard error (standard deviation of differences between X-ray and given values) of the calibration curve.

From this, it follows that an instrument can have excellent precision or reproducibility but the value being produced could be quite wrong. Poor calibration is a classic cause, possibly resulting from insufficient calibration standards, inaccurate concentration values, errors in sample preparation etc.

OXFORD INSTRUMENTS

OXFORD INSTRUMENTS

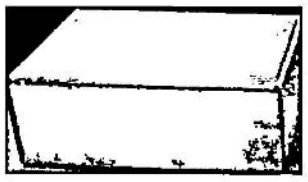
Packing List

Order Type	Serial No.:	X16104
System/X-Supreme	SO:	37480

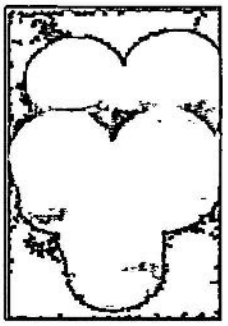
Item	No.	Description	Q'ty	Remarks	Box No.
54-C-XSP	1	X-Supreme instrument	1		Box 1 of 1
	2	Accessories box –TXPAK1	1		
	3	Accessories box –TXPAK2	1		
	4	54-L73	1	Kept in TXPAK2	
	5	54-ZX1223	1	Kept in SUS box of TXPAK1	
	6	54-SUGL50B	1		
	7	54-SUCM03B	1		
	8	54-SUCM04B	1		
	9	MOUSE MAT IA	1	Kept in TXPAK1	
	10	MOUSE - OPTICAL	1	Kept in TXPAK2	
	11	Main IEC cable	2	51-1106-1109 Kept in TXPAK2	
Documents	1	54-XSPOM1000E	1		Box 1 of 1
	2	54-XSMS-03A	1		
	3	54-XSMS-03B	1		
	4	54-XSMS-03C	1		
	5	54-XSMS-03D	1		
	6	54-XRFMS002	1		
	7	54-XRFMS007	1		
	8	54-ZX1267	1		
	9	54-SHIPPING ORIGIN	1		
	10	54-XSU-02-0001-AA	1		
	11	XSP X-Ray Key note	1		
	12	Customer Service Flyer	1	51-5103150	
	13	Win7 operating system note	1		
	14	X-Ray Key	2		
	15	54-QDR	2		
	16	Virus Scan report	1		
	17	XSP Software	1		
Other Items under SO	1	54-Q58	5	Kept in TXPAK2	
	2				
Numbers of Pieces Packed	**36**	**Packed by:** Chen. Bin. Bin		**Checked by:** Allan	
		Date: 28. Dec. 2016		**Date:** 28. Dec. 2016	

TXPAK2 TWINX ACCESSORY KIT CHECKLIST

54-SPARES BOX
WHITE SPARES BOX 355X300X115
QTY 1 ☐



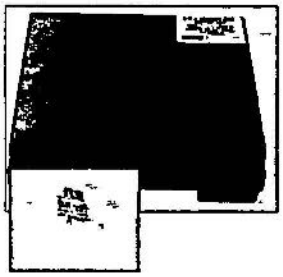
54-LX6879-2
SECONDARY SAFETY WINDOW
QTY 5 ☐



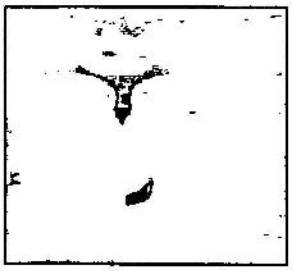
54-QX1008
SAMPLE BOX
QTY 1 ☐

54-LB3181
SUS TRANSIT CASE WARNING LABEL
QTY 1 ☐

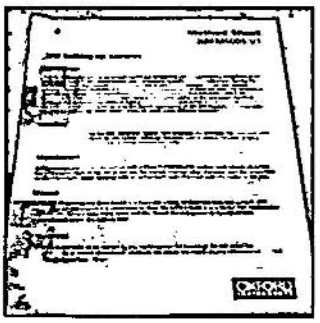
54-QX1009
SILICA GEL DESICCANT 601-057
QTY 1 ☐



54-Q59
41mm SAMPLE CELL
QTY 5 ☐



54-XRFMS005
XRF SUS INSTRUCTIONS
QTY 1 ☐



54-CP6618
Recovery CD

QTY 1 ☐

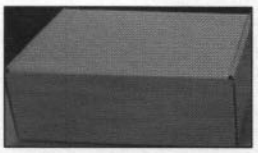
CARDBOARD TUBE 2FT X 2IN DIA
QTY 1 ☐

54-PTE/0399
PERIODIC TABLE BY IAG
QTY 1 ☐

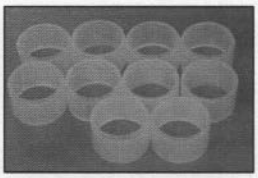
NAME .....
SIGNATURE. .QC STAMP

TXPAK1 TWINX ACCESSORY KIT CHECKLIST

54-SPARES BOX
WHITE SPARES BOX 355X300X115
QTY 1 ☑



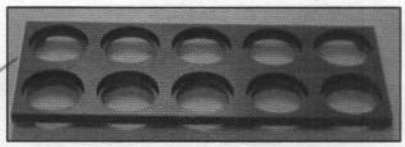
54-LX6879-2
SECONDARY SAFETY WINDOW
QTY 10 ☑



54-LX320
SAFETY WINDOW ASSY TOOL
QTY 1 ☑

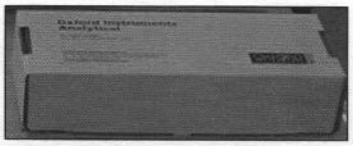
54-LX1054
WINDOW ASSY JIG
QTY 1 ☑

54-LX1032
SAMPLE RACK
QTY 1 ☑



54-PR1023
VAC GREASE
QTY 1 ☑

54-L242
SAMPLE CELLS(SET OF 10)
QTY 1 ☑



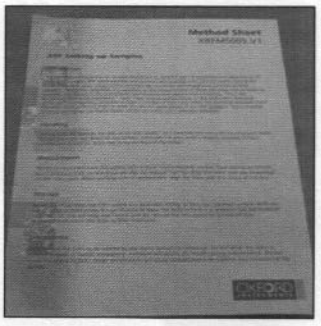
54-CK-100
CONSUMABLES KIT 100
QTY 1 ☑

54-QX1008
SAMPLE BOX
QTY 1 ☑

54-LB3181
SUS TRANSIT CASE WARNING LABEL
QTY 1 ☑

54-QX1009
SILICA GEL DESICCANT 601-057
QTY 1 ☑

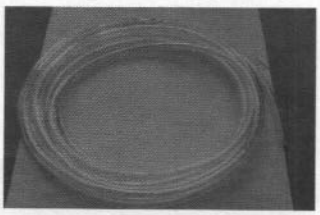
54-XRFMS005
XRF SUS INSTRUCTIONS
QTY 1 ☑



54-CP6618
Recovery CD

QTY 1 ☒

54-PI1023
1/8" CLEAR HOSE
QTY 5M ☑



54-HW8000
CARDBOARD TUBE 2FT X 2IN DIA
QTY 1 ☑

54-PTE/0399
PERIODIC TABLE BY IAG
QTY 1 ☑

NAME chen · Bin · Bin
SIGNATURE.....QC STAMP

54-TXPAK1CKLTV4

Method Sheet

XSMS-03B.V5

Oxford Instruments X-Supreme for the analysis of finished cement

Note: Please refer to the integrated on-screen help provided in the X-Supreme software for additional guidance.

Instrument Configuration

This method is specific to the **X-Supreme**, which must be fitted with a **Tungsten target X-ray tube**, a **Silicon drift detector** (SDD) and a **sample spinner**.

Note: You are strongly advised to follow this method exactly without editing any of the parameters. If you wish to make any changes, you should have established at least one calibration with the standard format so that you can then see the effect of any changes you wish to make.

Accessories

This method needs the following accessories:

- 40mm-diameter pellet holders (Part Number Q59)
- Setting up sample, SUCM03B
- Setting up sample, SUCM04B
- Setting up sample, SUGL50B.

Copying the Oxford Instruments template

All the instrument operating and calibration parameters are defined in the Oxford Instruments pre-defined calibration template **XSMET-03B.V5**. This is stored in the instrument and is "locked" to prevent any alteration. It can be copied as many times as required and each copy will be automatically "unlocked" so that it can be used to measure calibration standards and establish a working method. To make your working copy, select "Other Functions" from the main analysis menu. Go to Method Setup and click on "Manage Methods" (bottom of page). Highlight **XSMET-03B.V5**, right-button click on the mouse and select "Copy". The copy will be called "XSMET-03B.V5 (2)" by default. You can rename the copy by pressing the right button on the mouse, selecting the "Rename" option, and entering the name of your choice for your calibration.

Summary of the method

The method includes two measurement conditions that have been optimised to obtain best sensitivity and limit of detection for all elements present in finished cement. The total measurement time is about seven minutes per sample. The sample spinner is used to compensate for any residual sample heterogeneity. The fixed conditions and regions of interest used in this method are shown in Table 1.

Table 1: Method parameters

Analyte	Region of interest	Condition name	X-ray tube current (µA)	Path	Measurement time (seconds)
Na_2O	Na Ka	Na – S (4kV no filter)	750	He	200
MgO	Mg Ka				
Al_2O_3	Al Ka				
SiO_2	Si Ka				
P_2O_5	P Ka				
SO_3	S Ka				
K_2O	K Ka	K – Sr (22kV A6 filter)	85	Air	100
CaO	Ca Ka				
TiO_2	Ti Ka				
Cr_2O_3	Cr Ka				
Mn_2O_3	Mn Ka				
Fe_2O_3	Fe Ka				
ZnO	Zn (W) Ka				
SrO	Sr Ka				

Preparing the instrument

Before making any measurements in the calibration it is advisable for the instrument to have been switched on for at least two hours to achieve temperature stability. It is also a good idea to make a preliminary spectrum scan using the "Na - S" condition given in Table 1 to ensure that the helium pipework is free of residual air.

Preparation of standards

Calibration standards should be well analysed, production samples with concentrations that evenly span the range of interest for all analytes. Production samples are used so that the mineralogy of standards matches that of unknowns.

Standards and unknowns are measured as 40mm-diameter pressed pellets in the Oxford Instruments pellet holders (Part number Q59). Many cement companies have established techniques for preparing such pellets. Refer to method sheet XRFMS002 to confirm these techniques are suitable for the X-Supreme. When there is no existing method, grind the samples in a swing mill before pelletizing using a hydraulic press (See method sheet XRFMS002). A grinding additive is useful to prevent the sample clogging the mill and to help it bind into a pellet. The best type is one available as tablets of precise weight so that only the sample needs weighing. Oxford Instruments can supply such a grinding/binding aid in tablet form (Part number CM0039).

Calibration

Measure at least ten standards. To do so, select your copy from the methods list, highlight and click "Measure Standards". At that time, you can:

- If the standards data already exist in the database: Answer "No" to the "Do you want to add new standards?" question. Tick the Filter Type required so that your standards appear in the available standards list. Highlight each standard you wish to use for your calibration and click on "Add to calibration" to include it in your calibration standards list. When you have selected all the required standards, click "Next". Position each standard on the screen tray by

highlighting the sample tray position, highlighting the corresponding standard and pressing the ">>" key. Press "Next" when all standards are positioned on the screen tray. Place the standards on the instrument tray and press "Start" to start the measurements.

- If the standards data do not exist in the database: Answer "Yes" to the "Do you want to add new standards?" question. Enter the name of the first standard and press "Next". Enter your standard's concentrations and press "Next" again. You may now enter Standard Qualities criteria for the standard (e.g. matrix type, quality). The criteria entered will be used in the database search engine. When the entry is complete, press "Finish". A green tick appears to show the standard data have been saved. Click "Add New" to enter the next standard. Repeat the same procedure for all standards, then click "Close". Press "Next" and position each standard on the screen tray by highlighting the sample tray position, highlighting the corresponding standard and pressing the ">>" key. Press "Next" when all standards are positioned on the screen tray. Place the standards on the instrument tray and press "Start" to start the measurements.

When all standards have been measured, return to Method Setup and Regress. A linear (no correction) calibration will be set as default for all analytes. Only Fe2O3 requires a correction.

To apply the correction for Fe_2O_3, select "Fe_2O_3" from the analyte drop-down list. Press "Edit Regression" and select "Intensity" from the Model drop-down list. Highlight "Ca K" in the "Available" column and press ">>" to select it for the correction. Press "OK" to apply the correction model.

If the results are not satisfactory (see Table 2: Typical calibration performance for XSMET-03B.V1) you can experiment with deletion and re-measurement of standards or inclusion of additional ones. If you wish to do so, close Regress, save your regression data when prompted and proceed in "Measure Standards".
A History page and a Summary page are available in Regress to help you track changes and assess performance. Make sure that you return to Regress and save the regression after every change you make in the calibration standards.

When you are satisfied with your calibration results, close the Regression menu and answer "Yes" to save the regressed data.

Table 2: Typical calibration performance for XSMET-03B.V5

Analyte	Range (% m/m)	Standard error of calibration (% m/m)	Guaranteed limit of detection (3σ) (% m/m)	Mid-range precision (95% confidence) (% m/m)	Total measurement time (minutes)
Na_2O	0.02 – 1.07	0.04	0.021	0.012	~ 7
MgO	0.81 – 4.48	0.06	0.015	0.03	
Al_2O_3	3.9 – 7.1	0.1	n/a	0.03	
SiO_2	18.6 – 22.4	0.2	n/a	0.07	
P_2O_5	0.02 – 0.31	0.009	0.005	0.003	
SO_3	2.1 – 4.6	0.1	n/a	0.011	
K_2O	0.09 – 1.23	0.04	0.005	0.011	
CaO	57.6 – 67.9	0.5	n/a	0.08	
TiO_2	0.08 – 0.37	0.006	0.003	0.004	
Cr_2O_3	0.002 – 0.06	0.003	0.001	0.001	

Mn_2O_3	0.007 – 0.26	0.006	0.001	0.002	
Fe_2O_3	0.15 – 3.1	0.06	0.003	0.008	
ZnO	0.001 – 0.11	0.001	0.0006	0.001	
SrO	0.02 – 0.64	0.004	0.002	0.001	

The precision was calculated from 10 repeat measurements of NIST standards. The standards were chosen so that the analytes' concentration matches the calibration mid-range.

It is good practice to run validation samples and save their results to ensure you have all the data you need to integrate your X-Supreme into a quality system.

Setting up samples and Restandardisation

This method uses three setting up samples (SUSs) for restandardisation, SUCM03B, SUCM04B and SUGL50B. These act as high and low reference points for each calibration line.

After finishing the regression, proceed directly to "Measure SUSs". The SUSs usage, their measurement current and time are pre-assigned (details in Table 3). Click "Next" and assign the SUSs positions on the tray.

Place the three SUSs on the instrument tray. Close the lid and press "Start" to start the restandardisation measurements.

When the restandardisation is complete (the whole process takes less than twenty minutes), your calibration is ready to be used for routine analysis. To return to the main analysis menu, press "Back".

Table 3: Measurement times and tube current used for SUSs

SUS	Condition	Measurement time (seconds)	Tube current (µA)
SUCM03B	Na – S	200	750
	K – Sr	150	80
SUCM04B	Na – S	200	750
	K – Sr	150	85
SUGL50B	Na – S	200	750
	K – Sr	150	85

Setting up a Quality Control routine

If you wish the X-Supreme to be part of your quality control system, you can assign a quality control (QC) check sample to the calibration method. To do so, go to "Method Setup", select your method then "Define QC Check Sample". You can then specify an existing standard as a QC sample or enter data for a new one. Specify the target concentration for the QC sample, and the warning and action tolerance values. Restandardise when the QC check sample results are outside the tolerance limits.

Parameters for routine analysis

The results format for the screen display is set as "Default" in the method. You can change it if you wish to use other formats. To do so, select your calibration and "Edit Method" in the "Method Setup". Highlight "Save Method" in the method browser (left hand-side column). There you can change and/or add results report output formats.

You can also add automatic calculations to the method (e.g. lime saturation factor), using the Post-Analysis Calculations function in the Method Editor.

After the method is saved, it appears in the methods list available on the main analysis page and can be used for routine analysis. To measure a sample, select its tray position, enter the sample name (Label), select the method you wish to analyse it under from the Method drop-down list, and click on "Accept" to confirm the entry. Place the sample on the tray in the correct position. Repeat this procedure for all samples. When they are ready for measurement, close the instrument's lid and press "Start" to begin the analysis run. Preliminary results will appear after only a few seconds.

Note: You can edit the sample position on the tray when you go to "Edit Tray".

It is good practice to store your calibration data and your sample results in a separate file location for eventual backup.

Method Sheet

XSMS-03D.V5

Oxford Instruments X-Supreme for the analysis of dolomitic limestone

Note: Please refer to the integrated on-screen help provided in the X-Supreme software for additional guidance.

Instrument Configuration

This method is specific to the **X-Supreme**, which must be fitted with a **Tungsten target X-ray tube**, a **Silicon drift detector** (SDD) and a **sample spinner**.

Note: You are strongly advised to follow this method exactly without editing any of the parameters. If you wish to make any changes, you should have established at least one calibration with the standard format so that you can then see the effect of any changes you wish to make.

Accessories

This method needs the following accessories:

- 40mm-diameter pellet holders (Part Number Q59)
- Setting up sample, SUCM04B
- Setting up sample, SUGL50B.

Copying the Oxford Instruments template

All the instrument operating and calibration parameters are defined in the Oxford Instruments pre-defined calibration template **XSMET-03D.V5**. This is stored in the instrument and is "locked" to prevent any alteration. It can be copied as many times as required and each copy will be automatically "unlocked" so that it can be used to measure calibration standards and establish a working method. To make your working copy, select "Other Functions" from the main analysis menu. Go to Method Setup and click on "Manage Methods" (bottom of page). Highlight **XSMET-03D.V5**, right-button click on the mouse and select "Copy". The copy will be called "XSMET-03D.V5 (2)" by default. You can rename the copy by pressing the right button on the mouse, selecting the "Rename" option, and entering the name of your choice for your calibration.

Summary of the method

The method includes two measurement conditions that have been optimised to obtain best sensitivity and limit of detection for all elements present in limestone. The total measurement time is about five minutes per sample. The sample spinner is used to compensate for any residual sample heterogeneity. The fixed conditions and regions of interest used in this method are shown in Table 1.

Table 1: Method parameters

Analyte	Region of interest	Condition name	X-ray tube current (µA)	Path	Measurement time (seconds)
MgO	Mg Ka	Na - K (5kV no filter)	380	Helium	100
Al_2O_3	Al Ka				
SiO_2	Si Ka				
CaO	Ca Ka	Ca - Fe (15kV A4 filter)	50	Air	100
Fe_2O_3	Fe Ka				

Al_2O_3 is corrected for background variation from the Mg and Si peaks. There is no correction for the other elements

Preparing the instrument

Before making any measurements in the calibration it is advisable for the instrument to have been switched on for at least two hours to achieve temperature stability. Also carry out a spectrum scan using the "Na-K" condition shown in Table 1 to ensure the Helium gas purge is free of residual air.

Preparation of standards

Calibration standards should be well analysed, production samples with concentrations that evenly span the range of interest for all analytes. Production samples are used so that the mineralogy of standards matches that of unknowns.

Standards and unknowns are measured as 40mm-diameter pressed pellets in the Oxford Instruments pellet holders (Part number Q59). Many cement companies have established techniques for preparing such pellets. Refer to method sheet XRFMS002 to confirm these techniques are suitable for the X-Supreme. When there is no existing method, grind the samples in a swing mill before pelletizing using a hydraulic press (See method sheet XRFMS002). A grinding additive is useful to prevent the sample clogging the mill and to help it bind into a pellet. The best type is one available as tablets of precise weight so that only the sample needs weighing. Oxford Instruments can supply such a grinding/binding aid in tablet form (Part number CM0039).

Calibration

Measure at least six standards. To do so, select your copy from the methods list, highlight and click "Measure Standards". At that time, you can:

- If the standards data already exist in the database: Answer "No" to the "Do you want to add new standards?" question. Tick the Filter Type required so that your standards appear in the available standards list. Highlight each standard you wish to use for your calibration and click on "Add to calibration" to include it in your calibration standards list. When you have selected all the required standards, click "Next". Position each standard on the screen tray by highlighting the sample tray position, highlighting the corresponding standard and pressing the ">>" key. Press "Next" when all standards are positioned on the screen tray. Place the standards on the instrument tray and press "Start" to start the measurements.
- If the standards data do not exist in the database: Answer "Yes" to the "Do you want to add new standards?" question. Enter the name of the first standard and press "Next". Enter your standard's concentrations and press "Next" again. You may now enter Standard Qualities criteria for the standard (e.g. matrix type, quality). The criteria entered will be used in the

database search engine. When the entry is complete, press "Finish". A green tick appears to show the standard data have been saved. Click "Add New" to enter the next standard. Repeat the same procedure for all standards, then click "Close". Press "Next" and position each standard on the screen tray by highlighting the sample tray position, highlighting the corresponding standard and pressing the ">>" key. Press "Next" when all standards are positioned on the screen tray. Place the standards on the instrument tray and press "Start" to start the measurements.

When all standards have been measured, return to Method Setup and Regress. A linear (no correction) calibration will be set as default for all analytes.

To apply the correction for Al_2O_3, select "Al_2O_3" from the analyte drop-down list. Press "Edit Regression" and select "Additive Int." from the Model drop-down list. Highlight "Mg K" and "Si K"in the "Available" column and press ">>" to select it for the correction. Press "OK" to apply the correction model.

If the results are not satisfactory (see Table 2: Typical calibration performance for XSMET-03D.V5) you can experiment with deletion and re-measurement of standards or inclusion of additional ones. If you wish to do so, close Regress, save your regression data when prompted and proceed in "Measure Standards".

A History page and a Summary page are available in Regress to help you track changes and assess performance. Make sure that you return to Regress and save the regression after every change you make in the calibration standards.

When you are satisfied with your calibration results, close the Regression menu and answer "Yes" to save the regressed data.

Table 2: Typical calibration performance for XSMET-03D.V5

Analyte	Range (% m/m)	Standard error of calibration (% m/m)	Guaranteed limit of detection (3σ) (% m/m)	Mid-range precision (95% confidence) (% m/m)	Total measurement time (minutes)
MgO	18.8 – 21.2	0.21	0.16	0.14	~ 5
Al_2O_3	0.2 – 0.4	0.01	0.013	0.009	
SiO_2	0.3 – 2.2	0.12	0.04	0.02	
CaO	30 – 32.9	0.22	n/a	0.05	
Fe_2O_3	0.1 – 0.6	0.02	0.002	0.002	

The precision was calculated from 10 repeat measurements of a standard containing 20.3% MgO, 0.3% Al_2O_3, 1.2% SiO_2, 31.3% CaO, and 0.2% Fe_2O_3.

It is good practice to run validation samples and save their results to ensure you have all the data you need to integrate your X-Supreme into a quality system.

Setting up samples and Restandardisation

This method uses two setting up samples (SUSs) for restandardisation SUCM04B and SUGL50B. These act as high and low reference points for each calibration line.

After finishing the regression, proceed directly to "Measure SUSs". The SUSs usage, their measurement current and time are pre-assigned (details in Table 3). Click "Next" and assign the

SUSs positions on the tray. Place the two SUSs on the instrument tray. Close the lid and press "Start" to start the restandardisation measurements.

When the restandardisation is complete (the whole process takes less than twenty minutes), your calibration is ready to be used for routine analysis. To return to the main analysis menu, press "Back".

Table 3: Measurement times and tube current used for SUSs

SUS	Condition	Measurement time (seconds)	Tube current (μA)
SUGL50B	Na – K	200	380
	Ca – Fe	200	60
SUCM04B	Na – K	200	340
	Ca – Fe	200	45

Setting up a Quality Control routine

If you wish the X-Supreme to be part of your quality control system, you can assign a quality control (QC) check sample to the calibration method. To do so, go to "Method Setup", select your method then "Define QC Check Sample". You can then specify an existing standard as a QC sample or enter data for a new one. Specify the target concentration for the QC sample, and the warning and action tolerance values. Restandardise when the QC check sample results are outside the tolerance limits.

Parameters for routine analysis

The results format for the screen display is set as "Default" in the method. You can change it if you wish to use other formats. To do so, select your calibration and "Edit Method" in the "Method Setup". Highlight "Save Method" in the method browser (left hand-side column). There you can change and/or add results report output formats.

After the method is saved, it appears in the methods list available on the main analysis page and can be used for routine analysis. To measure a sample, select its tray position, enter the sample name (Label), select the method you wish to analyse it under from the Method drop-down list, and click on "Accept" to confirm the entry. Place the sample on the tray in the correct position. Repeat this procedure for all samples. When they are ready for measurement, close the instrument's lid and press "Start" to begin the analysis run. Preliminary results will appear after only a few seconds.

Note: You can edit the sample position on the tray when you go to "Edit Tray".

It is good practice to store your calibration data and sample results in a separate file location for eventual backup.

Method Sheet

XSMS-03C.V5

Oxford Instruments X-Supreme for the analysis of high-calcium limestone

Note: Please refer to the integrated on-screen help provided in the X-Supreme software for additional guidance.

Instrument Configuration

This method is specific to the **X-Supreme**, which must be fitted with a **Tungsten target X-ray tube**, a **Silicon drift detector** (SDD) and a **sample spinner**.

Note: You are strongly advised to follow this method exactly without editing any of the parameters. If you wish to make any changes, you should have established at least one calibration with the standard format so that you can then see the effect of any changes you wish to make.

Accessories

This method needs the following accessories:

- 40mm-diameter pellet holders (Part Number Q59)
- Setting up sample, SUCM03B
- Setting up sample, SUCM04B.

Copying the Oxford Instruments template

All the instrument operating and calibration parameters are defined in the Oxford Instruments pre-defined calibration template **XSMET-03C.V5**. This is stored in the instrument and is "locked" to prevent any alteration. It can be copied as many times as required and each copy will be automatically "unlocked" so that it can be used to measure calibration standards and establish a working method. To make your working copy, select "Other Functions" from the main analysis menu. Go to Method Setup and click on "Manage Methods" (bottom of page). Highlight **XSMET-03C.V5**, right-button click on the mouse and select "Copy". The copy will be called "XSMET-03C.V5 (2)" by default. You can rename the copy by pressing the right button on the mouse, selecting the "Rename" option, and entering the name of your choice for your calibration.

Summary of the method

The method includes two measurement conditions that have been optimised to obtain best sensitivity and limit of detection for all elements present in limestone. The total measurement time is about five minutes per sample. The sample spinner is used to compensate for any residual sample heterogeneity. The fixed conditions and regions of interest used in this method are shown in Table 1. The corrections applied in the calibration regression are in Table 2.

Table 1: Method parameters

Analyte	Region of interest	Condition name	X-ray tube current (μA)	Path	Measurement time (seconds)
MgO	Mg Ka	Na - K (5kV no filter)	300	Helium	100
Al_2O_3	Al Ka				
SiO_2	Si Ka				
K_2O	K Ka				
CaO	Ca Ka	Ca - Fe (15kV A4 filter)	40	Air	100
Fe_2O_3	Fe Ka				

Table 2: Calibration corrections

Analyte	Correction Model
MgO, SiO_2, CaO, Fe_2O_3	No correction
Al_2O_3	Intensity for Mg to correct for mass absorption
K_2O	Additive Intensity for Ca to correct for changes in background from the Ca peak

Preparing the instrument

Before making any measurements in the calibration it is advisable for the instrument to have been switched on for at least two hours to achieve temperature stability. Also carry out a spectrum scan using the "Na-K" condition shown in Table 1 to ensure the Helium gas purge is free of residual air.

Preparation of standards

Calibration standards should be well analysed, production samples with concentrations that evenly span the range of interest for all analytes. Production samples are used so that the mineralogy of standards matches that of unknowns.

Standards and unknowns are measured as 40mm-diameter pressed pellets in the Oxford Instruments pellet holders (Part number Q59). Many cement companies have established techniques for preparing such pellets. Refer to method sheet XRFMS002 to confirm these techniques are suitable for the X-Supreme. When there is no existing method, grind the samples in a swing mill before pelletizing using a hydraulic press (See method sheet XRFMS002). A grinding additive is useful to prevent the sample clogging the mill and to help it bind into a pellet. The best type is one available as tablets of precise weight so that only the sample needs weighing. Oxford Instruments can supply such a grinding/binding aid in tablet form (Part number CM0039).

Calibration

Measure at least ten standards. To do so, select your copy from the methods list, highlight and click "Measure Standards". At that time, you can:

- If the standards data already exist in the database: Answer "No" to the "Do you want to add new standards?" question. Tick the Filter Type required so that your standards appear in the available standards list. Highlight each standard you wish to use for your calibration and click on "Add to calibration" to include it in your calibration standards list. When you have selected all the required standards, click "Next". Position each standard on the screen tray by highlighting the sample tray position, highlighting the corresponding standard and pressing the ">>" key. Press "Next" when all standards are positioned on the screen tray. Place the standards on the instrument tray and press "Start" to start the measurements.

- If the standards data do not exist in the database: Answer "Yes" to the "Do you want to add new standards?" question. Enter the name of the first standard and press "Next". Enter your standard's concentrations and press "Next" again. You may now enter Standard Qualities criteria for the standard (e.g. matrix type, quality). The criteria entered will be used in the database search engine. When the entry is complete, press "Finish". A green tick appears to show the standard data have been saved. Click "Add New" to enter the next standard. Repeat the same procedure for all standards, then click "Close". Press "Next" and position each standard on the screen tray by highlighting the sample tray position, highlighting the corresponding standard and pressing the ">>" key. Press "Next" when all standards are positioned on the screen tray. Place the standards on the instrument tray and press "Start" to start the measurements.

When all standards have been measured, return to Method Setup and Regress. A linear (no correction) calibration will be set as default for all analytes.

To apply the correction for Al_2O_3, select "Al_2O_3" from the analyte drop-down list. Press "Edit Regression" and select "Intensity" from the Model drop-down list. Highlight "Mg K" in the "Available" column and press ">>" to select it for the correction. Press "OK" to apply the correction model.

To apply the correction for K_2O, select "K_2O" from the analyte drop-down list. Press "Edit Regression" and select "Additive Int." from the Model drop-down list. Highlight "Ca K" in the "Available" column and press ">>" to select it for the correction. Press "OK" to apply the correction model.

If the results are not satisfactory (see Table 3: Typical calibration performance for XSMET-03C.V5) you can experiment with deletion and re-measurement of standards or inclusion of additional ones. If you wish to do so, close Regress, save your regression data when prompted and proceed in "Measure Standards".

A History page and a Summary page are available in Regress to help you track changes and assess performance. Make sure that you return to Regress and save the regression after every change you make in the calibration standards.

When you are satisfied with your calibration results, close the Regression menu and answer "Yes" to save the regressed data.

Table 3: Typical calibration performance for XSMET-03C.V5

Analyte	Range (% m/m)	Standard error of calibration (% m/m)	Guaranteed limit of detection (3σ) (% m/m)	Precision (95% confidence) (% m/m)	Total measurement time (minutes)
MgO	1 – 4.1	0.05	0.04	0.02	~ 5
Al_2O_3	0.6 – 3.3	0.04	0.02	0.02	
SiO_2	2.3 – 10.4	0.08	0.05	0.03	
K_2O	0.1 – 0.8	0.008	0.02	< 0.01	
CaO	42.3 – 52.6	0.4	n/a	0.05	
Fe_2O_3	0.4 – 1.3	0.006	0.02	0.01	

The precision was calculated from 10 repeat measurements of a standard containing 1%m/m MgO, 2.2%m/m Al_2O_3, 6.3%m/m SiO_2, 0.5%m/m K_2O, 48.2%m/m CaO and 0.9%m/m Fe_2O_3.

It is good practice to run validation samples and save their results to ensure you have all the data you need to integrate your X-Supreme into a quality system.

Setting up samples and Restandardisation

This method uses two setting up samples (SUSs) for restandardisation SUCM03B and SUCM04B. These act as high and low reference points for each calibration line.

After finishing the regression, proceed directly to "Measure SUSs". The SUSs usage, their measurement current and time are pre-assigned (details in Table 4). Click "Next" and assign the SUSs positions on the tray. Place the two SUSs on the instrument tray. Close the lid and press "Start" to start the restandardisation measurements.

When the restandardisation is complete (the whole process takes less than fifteen minutes), your calibration is ready to be used for routine analysis. To return to the main analysis menu, press "Back".

Table 4: Measurement times and tube current used for SUSs

SUS	Condition	Measurement time (seconds)	Tube current (µA)
SUCM03B	Na – K	200	390
	Ca – Fe	150	40
SUCM04B	Na – K	200	390
	Ca – Fe	150	40

Setting up a Quality Control routine

If you wish the X-Supreme to be part of your quality control system, you can assign a quality control (QC) check sample to the calibration method. To do so, go to "Method Setup", select your method then "Define QC Check Sample". You can then specify an existing standard as a QC sample or enter data for a new one. Specify the target concentration for the QC sample, and the warning and action tolerance values. Restandardise when the QC check sample results are outside the tolerance limits.

Parameters for routine analysis

The results format for the screen display is set as "Default" in the method. You can change it if you wish to use other formats. To do so, select your calibration and "Edit Method" in the "Method Setup". Highlight "Save Method" in the method browser (left hand-side column). There you can change and/or add results report output formats.

After the method is saved, it appears in the methods list available on the main analysis page and can be used for routine analysis. To measure a sample, select its tray position, enter the sample name (Label), select the method you wish to analyse it under from the Method drop-down list, and click on "Accept" to confirm the entry. Place the sample on the tray in the correct position. Repeat this procedure for all samples. When they are ready for measurement, close the instrument's lid and press "Start" to begin the analysis run. Preliminary results will appear after only a few seconds.

Note: You can edit the sample position on the tray when you go to "Edit Tray".

It is good practice to do the following to store your calibration data and sample results in a separate file location for eventual backup.

Method Sheet

XSMS-03A.V5

Oxford Instruments X-Supreme for the determination of Al_2O_3, SiO_2, CaO and Fe_2O_3 in finished cement

Note: Please refer to the integrated on-screen help provided in the X-Supreme software for additional guidance.

Instrument Configuration

This method is specific to the **X-Supreme**, which must be fitted with a **Tungsten target X-ray tube**, a **Silicon drift detector** (SDD) and a **sample spinner**.

Note: You are strongly advised to follow this method exactly without editing any of the parameters. If you wish to make any changes, you should have established at least one calibration with the standard format so that you can then see the effect of any changes you wish to make.

Accessories

This method needs the following accessories:

- 40mm-diameter pellet holders (Part Number Q59)
- Setting up sample, SUCM03B
- Setting up sample, SUCM04B

Copying the Oxford Instruments template

All the instrument operating and calibration parameters are defined in the Oxford Instruments pre-defined calibration template **XSMET-03A.V5**. This is stored in the instrument and is "locked" to prevent any alteration. It can be copied as many times as required and each copy will be automatically "unlocked" so that it can be used to measure calibration standards and establish a working method. To make your working copy, select "Other Functions" from the main analysis menu. Go to Method Setup and click on "Manage Methods" (bottom of page). Highlight **XSMET-03A.V5**, right-button click on the mouse and select "Copy". The copy will be called "XSMET-03A.V5 (2)" by default. You can rename the copy by pressing the right button on the mouse, selecting the "Rename" option, and entering the name of your choice for your calibration.

Summary of the method

The method includes one single measurement condition, in air path. The total measurement time is just less than seven minutes per sample, and preliminary results can be seen after only a few seconds. The sample spinner is used to compensate for any residual sample heterogeneity. The fixed conditions and regions of interest used in this method are shown in Table 1.

Table 1: Method parameters

Analyte	Region of interest	Condition name	X-ray tube current (μA)	Path	Measurement time (seconds)
Al_2O_3	Al Ka	Al – Fe (10kV no filter)	50	Air	300
SiO_2	Si Ka				
CaO	Ca Ka				
Fe_2O_3	Fe Ka				

Preparing the instrument

Before making any measurements in the calibration it is advisable for the instrument to have been switched on for at least two hours to achieve temperature stability.

Preparation of standards

Calibration standards should be well analysed, production samples with concentrations that evenly span the range of interest for all analytes. Production samples are used so that the mineralogy of standards matches that of unknowns.

Standards and unknowns are measured as 40mm-diameter pressed pellets in the Oxford Instruments pellet holders (Part number Q59). Many cement companies have established techniques for preparing such pellets. Refer to method sheet XRFMS002 to confirm these techniques are suitable for the X-Supreme. When there is no existing method, grind the samples in a swing mill before pelletizing using a hydraulic press (See method sheet XRFMS002). A grinding additive is useful to prevent the sample clogging the mill and to help it bind into a pellet. The best type is one available as tablets of precise weight so that only the sample needs weighing. Oxford Instruments can supply such a grinding/binding aid in tablet form (Part number CM0039).

Calibration

Measure at least ten standards. To do so, select your copy from the methods list, highlight and click "Measure Standards". At that time, you can:

- If the standards data already exist in the database: Answer "No" to the "Do you want to add new standards?" question. Tick the Filter Type required so that your standards appear in the available standards list. Highlight each standard you wish to use for your calibration and click on "Add to calibration" to include it in your calibration standards list. When you have selected all the required standards, click "Next". Position each standard on the screen tray by highlighting the sample tray position, highlighting the corresponding standard and pressing the ">>" key. Press "Next" when all standards are positioned on the screen tray. Place the standards on the instrument tray and press "Start" to start the measurements.
- If the standards data do not exist in the database: Answer "Yes" to the "Do you want to add new standards?" question. Enter the name of the first standard and press "Next". Enter your standard's concentrations and press "Next" again. You may now enter Standard Qualities criteria for the standard (e.g. matrix type, quality). The criteria entered will be used in the database search engine. When the entry is complete, press "Finish". A green tick appears to show the standard data have been saved. Click "Add New" to enter the next standard. Repeat the same procedure for all standards, then click "Close". Press "Next" and position each

standard on the screen tray by highlighting the sample tray position, highlighting the corresponding standard and pressing the ">>" key. Press "Next" when all standards are positioned on the screen tray. Place the standards on the instrument tray and press "Start" to start the measurements.

When all standards have been measured, return to Method Setup and Regress. A linear (no correction) calibration will be set as default for all analytes. Only Fe2O3 requires a correction.

To apply the correction for Fe_2O_3, select "Fe_2O_3" from the analyte drop-down list. Press "Edit Regression" and select "Intensity" from the Model drop-down list. Highlight "Ca K" in the "Available" column and press ">>" to select it for the correction. Press "OK" to apply the correction model.

If the results are not satisfactory (see Table 2: Typical calibration performance for XSMET-03A.V5) you can experiment with deletion and re-measurement of standards or inclusion of additional ones. If you wish to do so, close Regress, save your regression data when prompted and proceed in "Measure Standards".
A History page and a Summary page are available in Regress to help you track changes and assess performance. Make sure that you return to Regress and save the regression after every change you make in the calibration standards.

When you are satisfied with your calibration results, close the Regression menu and answer "Yes" to save the regressed data.

Table 2: Typical calibration performance for XSMET-03A.V5

Analyte	Range (% m/m)	Standard error of calibration (% m/m)	Guaranteed limit of detection (3σ) (% m/m)	Precision (95% confidence) (% m/m)	Measurement time (seconds)
Al_2O_3	3.9 – 7.1	0.2	n/a	0.13	300
SiO_2	18.6 – 22.4	0.3	n/a	0.13	
CaO	57.6 – 67.9	0.5	n/a	0.05	
Fe_2O_3	0.2 – 3.1	0.04	0.008	0.01	

The precision was calculated from 10 repeat measurements of a standard containing 4.3% Al_2O_3, 20.6% SiO_2, 62.3% CaO, and 2.7% Fe_2O_3.

It is good practice to run validation samples and save their results to ensure you have all the data you need to integrate your X-Supreme into a quality system.

Setting up samples and Restandardisation

This method uses two setting up samples (SUSs) for restandardisation, SUCM03B and SUCM04B. These act as high and low reference points for each calibration line.

After finishing the regression, proceed directly to "Measure SUSs". The SUSs usage, their measurement current and time are pre-assigned (details in Table 3). Click "Next" and assign the SUSs positions on the tray.

Place the two SUSs on the instrument tray. Close the lid and press "Start" to start the restandardisation measurements.

When the restandardisation is complete (the whole process takes about sixteen minutes), your calibration is ready to be used for routine analysis. To return to the main analysis menu, press "Back".

Table 3: Measurement times and tube current used for SUSs

SUS	Condition	Measurement time (seconds)	Tube current (µA)
SUCM03B	Al – Fe Cement air path	400	60
SUCM04B	Al – Fe Cement air path	400	50

Setting up a Quality Control routine

If you wish the X-Supreme to be part of your quality control system, you can assign a quality control (QC) check sample to the calibration method. To do so, go to "Method Setup", select your method then "Define QC Check Sample". You can then specify an existing standard as a QC sample or enter data for a new one. Specify the target concentration for the QC sample, and the warning and action tolerance values. Restandardise when the QC check sample results are outside the tolerance limits.

Parameters for routine analysis

The results format for the screen display is set as "Default" in the method. You can change it if you wish to use other formats. To do so, select your calibration and "Edit Method" in the "Method Setup". Highlight "Save Method" in the method browser (left hand-side column). There you can change and/or add results report output formats.

You can also add automatic calculations to the method (e.g. lime saturation factor), using the Post-Analysis Calculations function in the Method Editor.

After the method is saved, it appears in the methods list available on the main analysis page and can be used for routine analysis. To measure a sample, select its tray position, enter the sample name (Label), select the method you wish to analyse it under from the Method drop-down list, and click on "Accept" to confirm the entry. Place the sample on the tray in the correct position. Repeat this procedure for all samples. When they are ready for measurement, close the instrument's lid and press "Start" to begin the analysis run. Preliminary results will appear after only a few seconds.

Note: You can edit the sample position on the tray when you go to "Edit Tray".

It is good practice to store your calibration data and sample results in a separate file location for eventual backup.

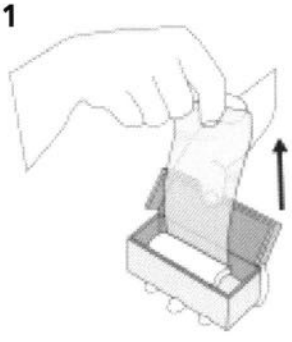
Method Sheet
XRFMS007.V1

Guidelines for sample film handling

This method sheet illustrates the correct procedure for handling X-ray fluorescence (XRF) sample film when preparing cups and secondary safety windows.

Following this procedure will help you avoid touching the film with fingers (reducing contamination of the film), avoid wrinkles and tears in the film (reducing risks of leakage through the film).

1



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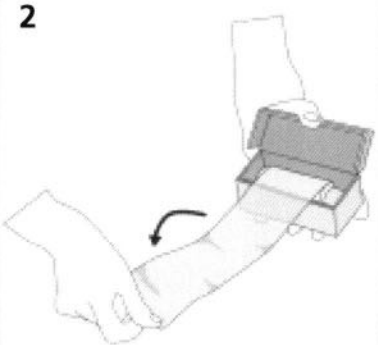

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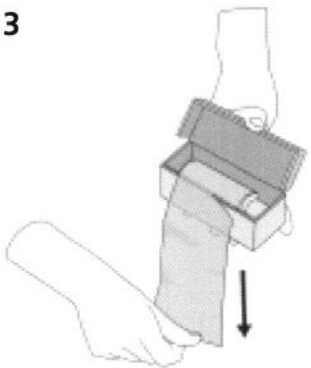
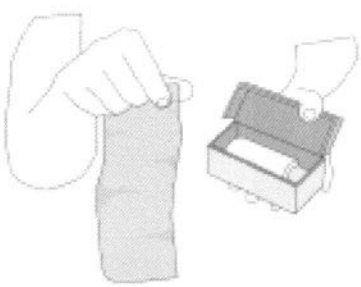
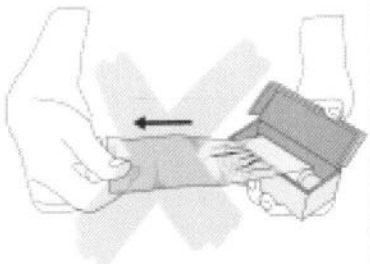


Squeeze the box lightly to hold the roll in place while you tear the film off.

4





Note 1: **When you use a new roll of sample film**, please restandardise your analysis system using setting-up samples (SUSs) prepared in cups and secondary windows (where required) fitted with the new sample film.

Note 2: **If you change your existing film for another film material or thickness**, please re-calibrate your analysis system with standards and setting-up samples prepared in cups and secondary windows (where required) fitted with the new film material.

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Method Sheet
XRFMS005.V1

XRF Setting up Samples

Introduction

Oxford Instruments setting up samples (SUS's) are an essential part of quantitative analysis for all of Oxford Instruments XRF spectrometers and are normally supplied with the instrument. During the initial calibration their measurement provides the sensitivity and background for each analyte element. Thereafter, whenever it is necessary a new measurement of them will allow the software to quantify any change in the instrument's performance and apply a drift correction. This is the procedure for "restandardisation". SUS's have to give signals similar to the calibration standards although it is sometimes necessary to have a number of SUS's and divide the elements between them. We offer many different SUS's to cover the diverse applications of our XRF products. This is possible because we mainly, use fusion beads, which can mimic many different materials.

Handling

Always hold the SUS's by the side. Avoid the "glassy" face (opposite the end with the engraved label). Do not place the face down on any surface. Although the glass bead is slightly recessed, it has a convex surface and its centre may protrude beyond the holder.

Measurement

Before measurement wipe the surface with a tissue moistened with alcohol. Each setting up sample has a reference mark on the rim at the top. For manual insertion align this mark with the arrowhead on the instrument. When working with an autosampler align the mark with the centre of the tray.

Storage

When not in use keep the SUS's sealed in a dessicator sitting on their top (labelled) surface. With the Lab-X range of instruments it is permissible to leave the SUS's in Ports 2 or 4 WHILE THE INSTRUMENT REMAINS ON. this will keep them warm and dry. Should the instrument be turned off (not recommended) return the SUS's to their dessicator.

Precautions

NEVER allow the SUS's to be wetted by any liquid (except for cleaning). Do not allow the SUS's to stand in a damp or humid atmosphere -moisture will attack the beads causing efflorescence. Do not touch the glassy surface - finger perspiration will cause contamination by chlorine and corrosion of the surface.

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Cleaning

Slight tarnishing of the surface can be removed by vigorous polishing using a "cotton bud" wetted with water and covered with an abrasive paste. This paste is prepared with lithium tetraborate powder (similar to the composition of the bead) and water. It is free of any interfering elements. Remove any residue of the paste with a tissue moistened with water followed by wiping by a tissue moistened with alcohol.

Note: Lithium tetraborate powder is available from Oxford Instruments, Part Number QX3004.

Note: "Water" means deionised or distilled water.

Finally

If any changes are permitted to occur to a SUS then this will affect all subsequent analysis and a complete calibration with all the original standards will be necessary.

Certificate No FM29142

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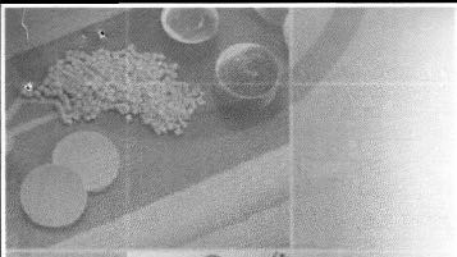


Method Sheet

XRFMS002.v1

Preparation of Pressed Pellets for use with XRF Spectrometers

Introduction

This method sheet gives details of the various techniques used in the production of pressed pellets. Specific information is given for sample matrix types typically determined using the Oxford Instruments' range of XRF spectrometers.

Sample Preparation Equipment

This method requires sample preparation equipment capable of grinding geological material, such as rocks and soils, to a particle size of <50µm. A tungsten carbide ring and puck mill with a minimum capacity of 50cm^3 is recommended. All techniques used in this method sheet use a tungsten carbide grinding surfaces.

A press capable of 25 tonne force and 31.5, 36 or 40mm (part numbers: QX1014, QX1002 and QX1014) briquetting die for preparing pressed powder pellets is necessary, together with:

- Appropriate aluminium backing cups - Part Numbers: QX1014/1000 (31.5mm), QX1001/600 (35mm), QX1015/600 (40mm)

- Wax pellets, Oxford Instruments' part number CM0039 per 5000.

Accessories that may be useful:

Boric acid backing die insert, part number B6057 for use with QX1014 pelletising die and ED2000 only.

Sample Preparation Theory

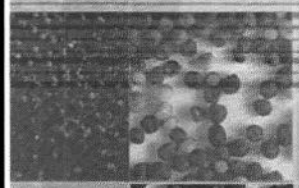
To obtain precise analyses using the technique of pressed pellets it is essential to follow a number of principles governing sample preparation for XRF spectrometry.

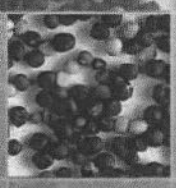
(i) The particle size of a sample has a large effect on element sensitivity.

(ii) The particle packing density of a sample has a large effect on element sensitivity.

(iii)The lower the atomic number the greater the effect sample preparation has on element sensitivity.

The above three principles are related, with the first two controlling the third to a large extent. In all cases the reduction of a sample to an even, reproducible particle size will determine the performance of this method.





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Before taking a sample to be prepared for XRF analysis, bulk and analytical sample size(s) and their relationship to accuracy, precision and detection limits must be assessed. i.e. the analysis of heterogeneous bulk samples cannot be accurately determined from a single small analytical sample.

Using a suitable grinding mill, as advised by Oxford Instruments', the particle size of a geological matrix type sample should be reduced to <50µm particle size (i.e. capable of passing through a 325 mesh or 43µm sieve). This is achieved by milling a suitably-sized sample, in relation to the mill capacity, for a specified time. Various grinding aids can be used during this process.

The milling times are related to the overall hardness of the sample and/or the differential hardness of the mineral phases present.

The resultant powdered sample is transferred to a pellet die and pressed at a minimum of 15 tonnes pressure. Typically, 20 tonnes is used. The sample can be pressed into any one of the following forms:

a) Aluminium backed pellet

b) 40mm steel ring supported - 14mm high.

a) Boric acid backed pellet

b) Non-backed pellet (using liquid binder)

Aluminium backed pellets

This type of pellet is the most commonly used as it is robust and simple to prepare. The manufacturer's instructions should be followed when using milling and pressing equipment.

Table 1 - Special preparation techniques

Matrix type	Sample preparation	Grinding based on 100cm³ mill	Grinding notes
Raw meal cements	Add 5 wax tablets to 20.000g sample	Mill tablets and sample for minimum of 5 minutes	Grinding time will vary with conc. of free quartz.
Finished cement	Add 5 wax tablets to 20.000g sample	Mill tablets and sample for 5 minutes	S intensity will decrease with time.
Silica sand	Add 7 wax tablets to 20.000g sample	Mill tablets and sample for 4 minutes	

Note : The above table of techniques must only be applied to matrix types measured using calibration methods, they are not suitable for the fundamental parameter technique.

Of the three common pellet sizes, the smallest (31.5mm) applies only to the ED2000. The other two (35mm and 40mm) apply to both the Lab-X 3500 and MDX1000 for normal operation. However, only the 40mm size is recommended for routine use where an autosampler is fitted.

Grinding requirements:

Samples should be dry. Any matrix type that has not previously been ground should first undergo a series of tests. These tests will determine the minimum grinding time required to achieve firm, reproducible pellets. Samples should not exceed 50% of the mill capacity (typically 20g). Samples are milled for a known time (starting at 2 minutes) and pressed at 20 tonnes force. Successively longer milling times are used, increasing the time by 30 seconds, to a maximum of 10 minutes. By measuring the countrate (cps) of the critical element(s) that are of interest or required, the optimum grinding time is determined as the point at which the countrate for all these elements changes by the least amount. A plot of intensity versus grinding time will show the optimum time to use. This may be a compromise because not all elements will give a maximum intensity together.

All pellets made in the above test must be robust. If after 10 minutes milling, pellets fail to be robust, then alternative methods of pelletising may be required.

For many matrix types the ground sample can simply be placed into a pellet die and pressed. For a number of specific matrix types this is not possible without adding a grinding/binding agent. Table 1 gives details of matrix types and special sample preparation requirements needed to make aluminium backed pellets.

Sample pressing:

Using an appropriate pelletising die (see page 1) construct the die according to the manufacturer's instructions. Place an appropriately sized Al cup into the die. Pour enough prepared powdered sample into the die such that the depth of powder is approximately twice the depth of the Al cup.

Insert a clean die face and plunger and press at 20 tonnes force. Using the tools provided by the manufacturer, remove the pellet slowly. Ensure that it does not fall onto any surface and crack, and that the analytical surface is not touched. The thickness of the pellet should not typically be less than 3mm thick to produce robust pellets for X-ray analysis.

The die must be cleaned thoroughly between each pellet. Plastic dish-washing scourer's have been found very effective for this. Do not use abrasives or metal objects to clean the dies as these will damage the die surfaces and affect the making of pellets.

The amount of powder used to produce a pellet will vary for each matrix type. Generally, the lighter the matrix the greater the volume of powder that is required. i.e. - silica sand may need 3 to 4 times the volume of the aluminium cup to produce a pellet, whereas metal powder usually only needs the volume of one aluminium cup.

Steel Ring supported pellets

These pellets are produced for use with proprietary automated sample systems. The ring is usually 40mm outside diameter and 14mm high. The rings are designed to be re-usable. The sample preparation is the same as that for the aluminium backed pellets.

Boric Acid backed pellets

Pellets using this process are normally restricted for use with the ED2000 as the boric acid pellet insert provided is only suitable for the 31.5mm die.

The main advantages over aluminium are that the boric acid binds with the sample preventing flaking and breaking at the edges of the pellet and that small samples can be accommodated. However the disadvantages are that they are difficult and time consuming to make and contamination of the sample with boric acid is probable if recovery of the sample from the pellet is required.

Grinding requirements:

Sample preparation is the same as for the aluminium, backed pellets.

Sample pressing:

The Oxford Instruments' boric acid pellet insert is designed to be used with dies supplied through Oxford Instruments'. Put together the sample die according to the manufacturer's instructions in the same way as for aluminium backed pellets. Place the outer ring insert into the die. Pour approximately 5g of sample (will vary for different matrix types) into the insert. Using the solid plunger, gently hand compress the powder whilst holding the outer ring insert firmly in place against the base of the die. Very slowly remove the plunger using a twisting action. The compressed powder must not be disturbed or loosened. Remove the outer ring insert, again by a gentle twisting action. The powder must remain as a disk at the bottom of the die, separated from the sides by the equivalent thickness of the outer ring insert. The disk should not be less than 3mm thick at this time. Pour sufficient boric acid powder over the sample disk to a depth of about 1.5cm ensuring that the space around the sample disk is filled. Place the remaining die components (second die face and plunger) into the die and press at a minimum of 20 tonnes force. Remove the pellet as per the manufacturer's instructions. Clean all the sample preparation equipment. Pay special attention to the boric acid pellet insert i.e. ensure that the plunger and insert freely slide into one another.

Pellets using liquid binder

The making of this type of pellet is covered by a separate method sheet (XRFMS001) as there are solutions to prepare. The use of liquid binder in the production of pellets is required in a number of specific circumstances. These are as follows:

1. Samples which are to be measured using the technique of Fundamental Parameters (FPt) and will not form pellets without some form of binder.
2. The elements of interest are diluted beyond acceptable levels if other grinding/binding methods of sample preparation are used (i.e. see table 1).

Grinding requirements:

These are the same as for the aluminium, backed pellets except that grinding aids or binding agents must not be used.

Sample pressing:

These are specified in the separate method sheet (XRFMS001) and are different from those of other techniques.

Useful information

The most common reason for the failure to produce a robust pellet is the particle size of the sample. A good test for particle size is referred to as the 'finger test'. Your fingers are capable of sensing even µm particle size differences. Take a small portion of the powdered sample, taking account of any harmful aspects of the sample (place sample into a thin plastic bag if necessary) and roll the powder firmly between index finger and thumb. If the sample feels and remains gritty then the sample is too coarse. Discard this test sample and mill the sample for a longer time. The correct particle size will be determined when no appreciable grittiness is felt using the above test, i.e. the sample feels like talc and slips between the index finger and thumb.

A less common problem in failing to produce a pellet is that the die faces that come into contact with the pellet are badly scratched. This causes the pellet to stick to the die and on release the pellet surface is damaged. Die

faces can easily be re-polished. To reduce this problem tungsten carbide die faces can be used as these are rarely scratched (the standard die faces are stainless steel).

The internal bore of the die will become scored over a period of time as the die faces grind sample against them. This is the factor that determines the life of the die. Once the scoring becomes severe, removing the pellet will cause it to catch on the damaged die surfaces breaking the pellet or distorting the aluminium backing cup. This will cause the pellet to crumble around the edges or break after removal and the die must be replaced.

Samples should be dried before pelletising. If the sample is damp then it is likely to stick to the die face. Removal usually destroys the pellet. Alternatively damp pellets if left to dry will often crumble.

Pelletising ashed (i.e. ignited at >900° C) samples can be problematical. Material such as cements and carbonates are chemically unstable in their ignited form and will degrade (e.g. absorb water) in a short time.

To limit these effects pellets should be measured as soon as possible after pelletising and stored in a desiccator.

The die must not be used as a heat mould. Coarse plastic granules cannot be pressed in this type of die. Placing the die in a furnace at high temperature, i.e. >300° C, will cause distortion and ruin the die.

Many pelletising dies are available. In selecting a suitable die, consideration should be given to the following,; Die material, i.e. it must not be corroded by the samples, or by water - stainless steel is usually preferred, It must be engineered to high tolerances, i.e. the die faces must fit tightly into the die to reduce scoring, It must be able to be used with common XRF accessories such as aluminium backing cups.

For further information on pressed pellet sample preparation, users are recommended to use the following reference book:

Handbook of X-ray Spectrometry, methods and techniques. Edited by R E Van Grieken & A A Markowicz. Practical spectroscopy series V.14. ISBN 0-8247-8483-9.

Certificate No FM29142

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OXFORD INSTRUMENTS

X-Supreme

RADIATION SAFETY FEATURES

Part number 54-ZX1267

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X-Supreme
February 2009

Introduction

The X-Supreme 8000 is an extension to the range of Twin-X benchtop XRF analysers manufactured by Oxford Instruments.

The Twin-X can contain one or two analysis heads; each head is fitted with a TF Series Potted X-ray tube manufactured by Oxford Instruments X-Ray Technology Inc. The available X-ray tube target materials are Pd and Ti. One of the analysis heads has a gas filled proportional counter and the other has a small PIN detector.

On the X-Supreme, which uses the same chassis as the Twin-X, the PIN detector has been replaced by a Si Drift Detector (SDD). The SDD is similar in size to the PIN and is not considered part of the radiation shielding. The configuration is such that only one analysis head can be fitted to the instrument which can either be the standard Twin-X head with gas filled proportional counter or the head fitted with SDD. Additionally W has been added to the available X-ray tube target materials to extend the range of applications that can be offered. The W tube has the same general construction and is made by the same manufacturer as the existing Pd and Ti target tubes.

A sample is irradiated with X-rays from a X-ray tube and secondary X-rays characteristic of the elements present in the sample are emitted and collected in a detector.

The X-ray tube is contained within a fully shielded and interlocked radiation enclosure. The top section of the enclosure has a lid that is opened by the operator for sample loading. Safety features of the lid design are very similar to those adopted on the Oxford Instruments' ED2000. The lower section of the enclosure is locked and only accessible to an Oxford trained engineer, the design of this lower section is very similar to that used on the Oxford Instruments' Lab-X 3000. Both ED2000 and Lab-X 3000 have been in production for more than 10 years and have an excellent track record for radiation safety.

Stray radiation measurements were carried out using a Series 900 mini-monitor with 44B probe. This is a very sensitive monitor and every Twin-X is tested before leaving the factory to ensure that there is no more than 300cps, measured a zero distance, from any accessible surface of the instrument – this equates to a maximum dose rate of 0.1µSv/hr at zero distance.

An X-Supreme fitted with an analysis head containing a W target X-ray tube and SDD was operated at the maximum power of 30kV 100µA and with no primary beam filtration. The maximum leakage measured zero distance from any accessible surface of the instrument was no greater than background (<10cps on mini-monitor).

X-ray Generation

The X-ray tube is a TF series manufactured by Oxford Instruments X-ray Technology. A section through the tube is shown in drawing LX6856-3. It is operated with positive anode (target) and grounded filament. The target can be Pd, Ti or W depending on the application. The tube is potted with silicon rubber and as such is not radiation shielded. The majority of X-rays generated within the tube are emitted from the beryllium exit window, however, at higher anode voltages some X-rays are emitted through the glass/rubber body of the tube.

As on Twin-x, the X-ray tube is operated up to a maximum anode voltage of 30kV and maximum power of 3W i.e. 100µA at 30kV - cooling is provided by a small fan. The HV supply is a XMP30P3/15 manufactured by Spellman which is also rated at 30kV 3W maximum. Filament current is generated independently on the Tube Control board.

Mechanical

The mechanical construction of X-Supreme is shown on drawing LZ1161. Sheet 1 gives a general overview showing where the radiation enclosure is located within the instrument and also shows the position of the two X-ray warning lamps. Sheet 2 is the general assembly of the radiation enclosure with Sheets 3 and 4 giving details within the enclosure.

The X-ray tube is fixed at its window flange to a brass mounting block. Primary X-rays from the beryllium exit window pass through an aperture before irradiating the sample. The X-ray tube/detector assembly is fixed to the underside of an aluminium top plate. The other side of the top plate supports a multi-position sample turntable. The top plate is fixed between the lower section of the radiation enclosure and a radiation loop. A metal lid is attached to the main chassis of the instrument by a rear pivot rod and when in its closed position completely overlaps the radiation loop with minimal clearance. The radiation loop and lower section of the radiation enclosure are secured to the main chassis of the instrument with tamperproof screws. A Guardmaster Trojan 5 safety switch is fixed to the front of the radiation loop by tamperproof screws. The actuator for the safety switch is welded to the lid and is the primary safety interlock of the instrument. An additional Guardmaster Imp safety switch with roller actuator is fixed to the chassis and is activated by a cam soldered to the lid. This second safety switch acts as a backup to the primary interlock switch.

Both lid safety switches are "closed" when the lid is down. The contacts are forced open when the lid is lifted - this is to avoid the possibility of the contacts welding. Also they fail to safety if, for example, any of the connecting wires get detached. The X-ray warning lamp uses the auxiliary normally open contacts of the front safety switch.

The main components of the radiation enclosure are all at least 1.5mm thick mild steel. The metal lid has a plastic top cover secured with tamperproof screws which prevents accidental removal of the nut holding the rear pivot rod in place.

The lower section of the radiation enclosure which contains the X-ray tubes has internal baffles which allow cables to pass from the enclosure without leakage of radiation scattered from the sample or emitted from the unshielded body of the X-ray tube.

During operation there is no requirement for access to the lower section of the radiation enclosure. Also all internal fixings which relate to radiation shielding are secured from within the enclosure and cannot be removed or dismantled from outside. The radiation enclosure has a bottom cover which is locked and the key is only supplied to approved and trained engineers. The design of the bottom cover includes overlapping joints to prevent scattered radiation from escaping.

The lock cam plate acts on an interlock micro-switch and the plate must be fully engaged in the lock switch bracket before the key can be removed. When the micro-switch contacts are opened, by inserting and turning the key to remove the cover, the HV supply to the X-ray tube is switched off. The HV supply can only be switched back on from the instrument keypad after the interlock is remade.

An additional micro-switch is activated when the radiation enclosure bottom cover is correctly in position. This interlock switch is in series with the filament drive of the X-ray tube and therefore is completely independent of the HV interlock. The bottom cover micro-switches are normally open so if they are disconnected they fail to safety. Also they are mounted in such a way as to be difficult to override without the cover in place.

The filament leads from the tube is only 50mm long and therefore it is impossible to operate the X-ray tubes when it is removed from the enclosure.

Safety Interlock Circuit Description

A schematic diagram of the electrical interlock circuit is shown in drawing ZX1166-1.

The X-ray HV supply derives its power from +15V and outputs are controlled by a low voltage input signal Vprog.

The primary interlock is the lid front safety switch. When the lid is opened this switch removes the +15V from the HV supply and also grounds Vprog via relay RL2 therefore preventing X-rays from being generated. The secondary interlock is the safety switch at the rear of the lid. When the lid is opened power

is disconnected from RL1, 3 and 4 which also removes the +15V from the HV supply.

Internal access to the radiation enclosure is by removal of the bottom cover which is locked. Turning the key to open the cover breaks a micro-switch, which provides an input to U5 and has the effect of de-energising RL1, 3 and 4 therefore removing power to the HV supply. The micro-switch input to the logic is taken to +5V through a pull up resistor so that the circuit fails to safety if the micro-switch is disconnected. When the radiation enclosure cover is removed this also opens another micro-switch, which is in series with the filament drive of the X-ray tube again preventing X-rays from being generated.

When all the interlocks are in place, a microcontroller on the X-ray Tube Control board provides a signal to turn on HV power supply 1 via Q3 and RL1 (when F5 Analysis Head is fitted) or HV power supply 2 via Q4 and RL3 (when SDD Analysis Head is fitted).

The operator has a visual indication of the generation of X-rays within the instrument by two LED panel warning lamps on the top cover. The warning lamps are part of the interlock system and the circuit detects current flowing through the lamps. To initialise the system a short duration start pulse is triggered by either X-ray tube 1 (if proportional counter head is fitted) or X-ray tube 2 (SDD head is fitted) enable signal which activates the relay drivers provided that both the lid and cover interlock switches are in the closed (safe) position. The relays provide the +15V to the appropriate HV supply but additional contacts simultaneously complete the warning lamp circuit. Actual warning lamp current maintains the interlock relays after the start pulse has finished provided the current remains above the required threshold. If one of the interlocks is interrupted, X-ray generation will be disabled and will not continue when the interlock is restored until the operator restarts the measurement thereby generating another start pulse.

Operation

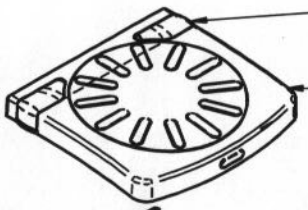
X-ray tubes are widely used in XRF analysers but these usually operate at higher potentials and power than on the X-Supreme and often remain energised between measurements to maintain instrument stability. A consequence of using a low power tube is that it can be switched off when not needed, a condition which obviously gives zero external dose rate. A typical measurement sequence will take about three minutes and users will probably analyse about twenty samples per day.

Operation of the X-Supreme is largely under the control of an inbuilt microprocessor. After loading one or more samples onto the turntable and closing the lid, the operator starts an automatic sequence whereby the motorised turntable transports the sample to either of the X-ray tube/detector positions. The high voltage and emission current for the X-ray tube is then ramped to predetermined levels. The analysis is then carried out and the X-ray tube switched off before returning the turntable to its initial position. If the lid is lifted before the measurement is complete the interlocks switch off the X-rays. Any fault condition in the sequence also turns off the X-rays and the cause of the malfunction is displayed.

X-Supreme Radiation Safety Documentation

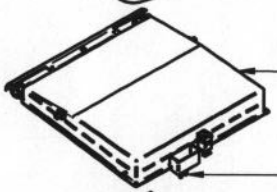
ZX1161-1 Drawing	Twin-X Radiation
ZX1166-1	Safety Interlock Circuit
LX6856-3 Outline	TF Series X-ray Tube

ISSUE	AF	1	11/09/02
DATE			



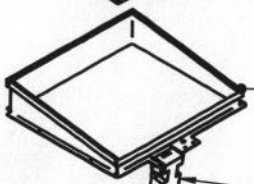
X-RAY WARNING LAMPS x 2

TOP COVER (Foam moulding)
(Secured to turntable metal lid
using System Zero tamperproof screws)



TURNTABLE METAL LID

GUARDMASTER SWITCH ACTUATOR
(Secured with nuts welded in place)

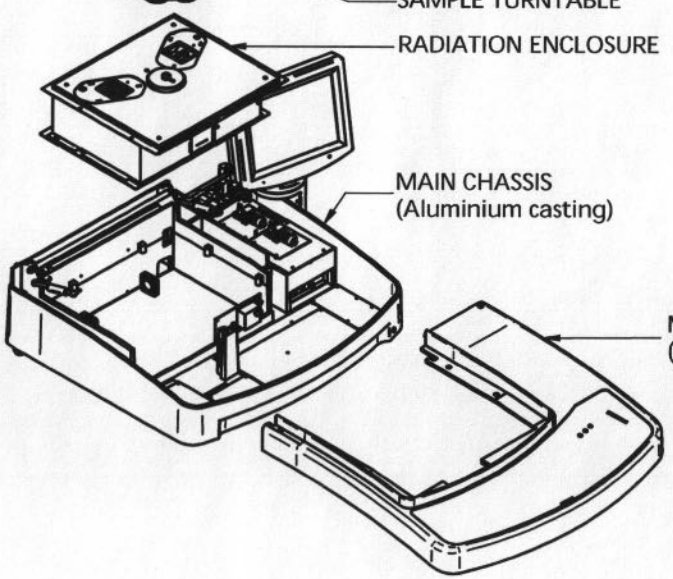


RADIATION LOOP
(Secured to radiation enclosure using
System Zero tamperproof screws)

GUARDMASTER SAFETY SWITCH
(Secured to radiation loop using
System Zero tamperproof screws)

SAMPLE SPINNERS

SAMPLE TURNTABLE

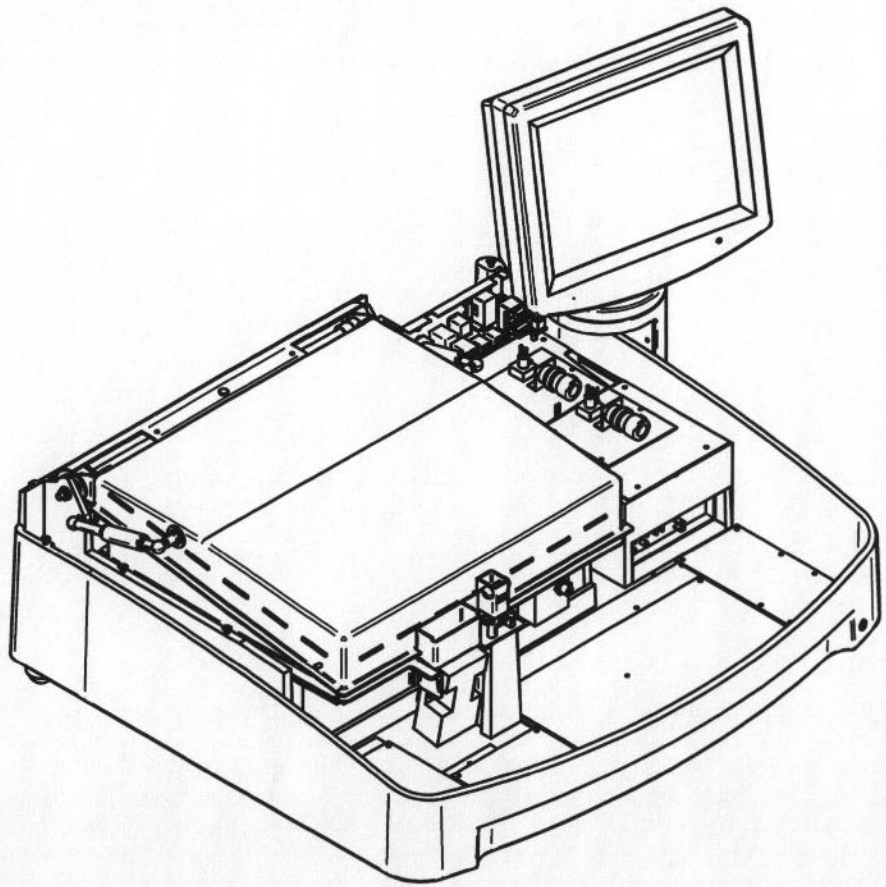


RADIATION ENCLOSURE

MAIN CHASSIS
(Aluminium casting)

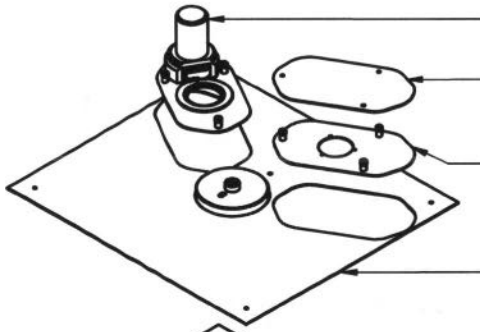
MAIN COVER
(Foam moulding)

VIEW OF WARNING LAMP COVER



OXFORD INSTRUMENTS ANALYTICAL LTD	This drawing is the property of OXFORD INSTRUMENTS LIMITED, and is issued on condition that it is not copied, reprinted or discussed either wholly or in part to a third party without the prior written consent from, OXFORD INSTRUMENTS LTD, Analytical Ltd.	THIRD ANGLE PROJECTION IF IN DOUBT ASK	DRAWN: P. SLIGHT	DATE: 11/09/02	SHEET 1 OF 4
			TITLE: TWIN-X RADIATION DRAWING		DRAWING No: ZX1161-1

ISSUE	AF DATE	1	11/09/02



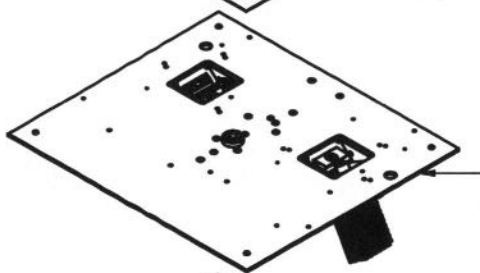
SAMPLE SPINNER ASSEMBLY

BLANKING PLATE
1.2mm aluminium alloy

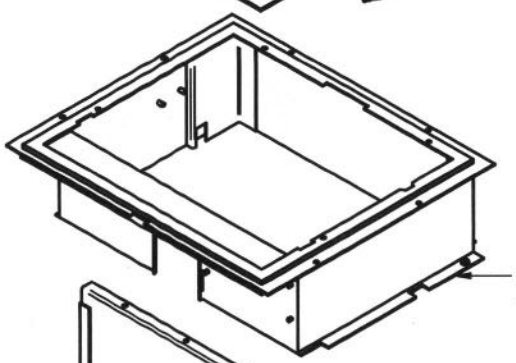
SAMPLE PLANE MOUNTING PLATE
1.2mm aluminium alloy

NOTE:
When both analysis heads are fitted either a sample spinner assembly or sample plane mounting plate are used. If only 1 head is fitted a blanking plate is used.

TRIM PLATE
1.2mm aluminium alloy

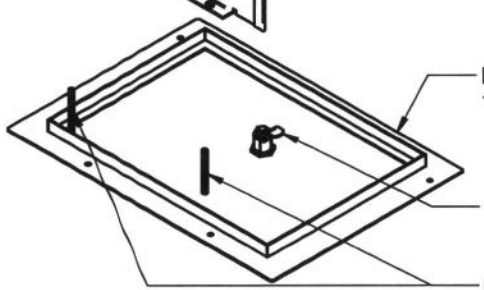


TOP PLATE
3mm aluminium alloy



RADIATION ENCLOSURE
1.5mm mild steel

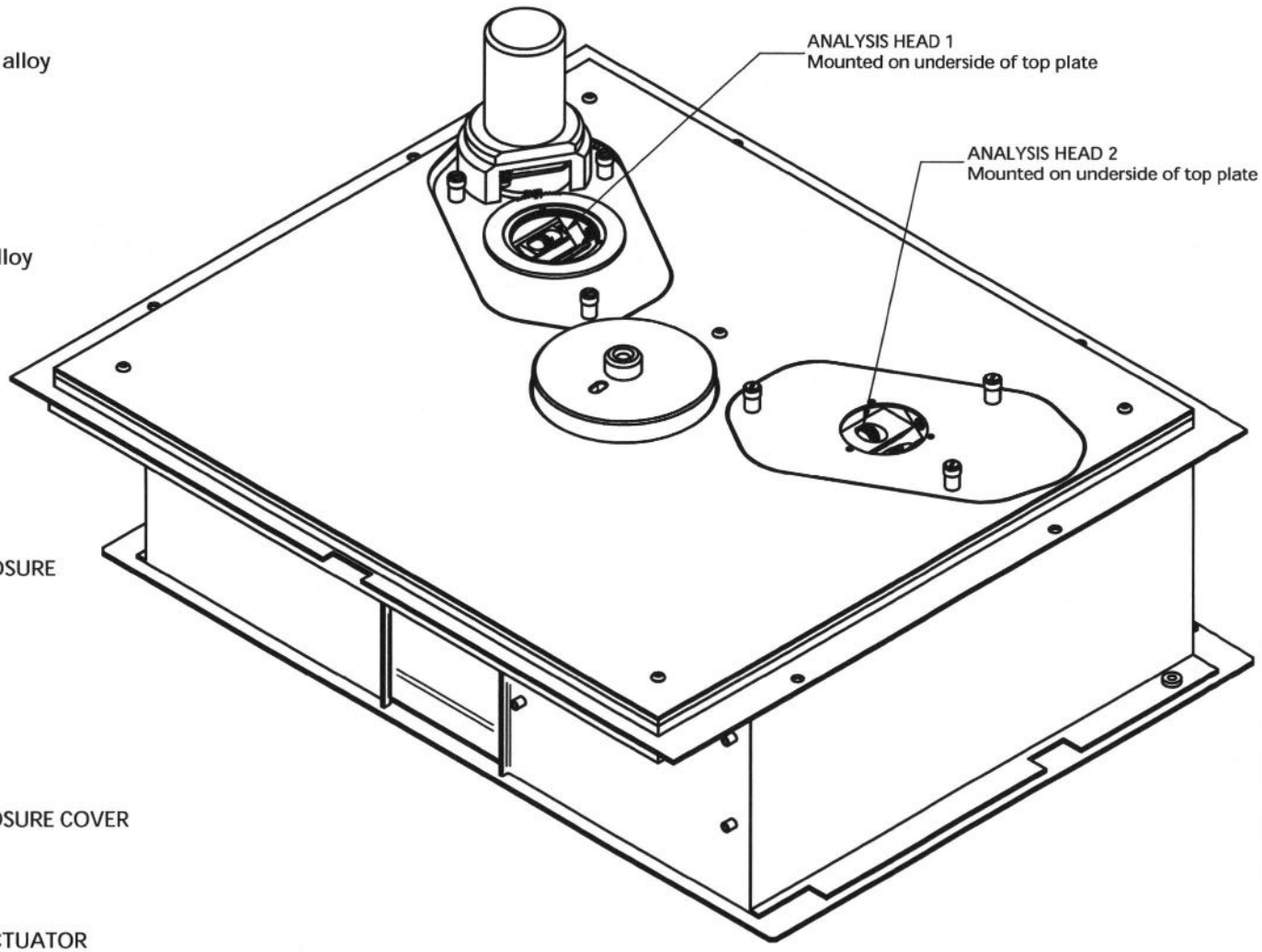
BAFFLE PLATE
1.5mm mild steel



RADIATION ENCLOSURE COVER
1.5mm mild steel

HV INTERLOCK ACTUATOR

FILAMENT INTERLOCK ACTUATORS



ANALYSIS HEAD 1
Mounted on underside of top plate

ANALYSIS HEAD 2
Mounted on underside of top plate

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THIRD ANGLE PROJECTION

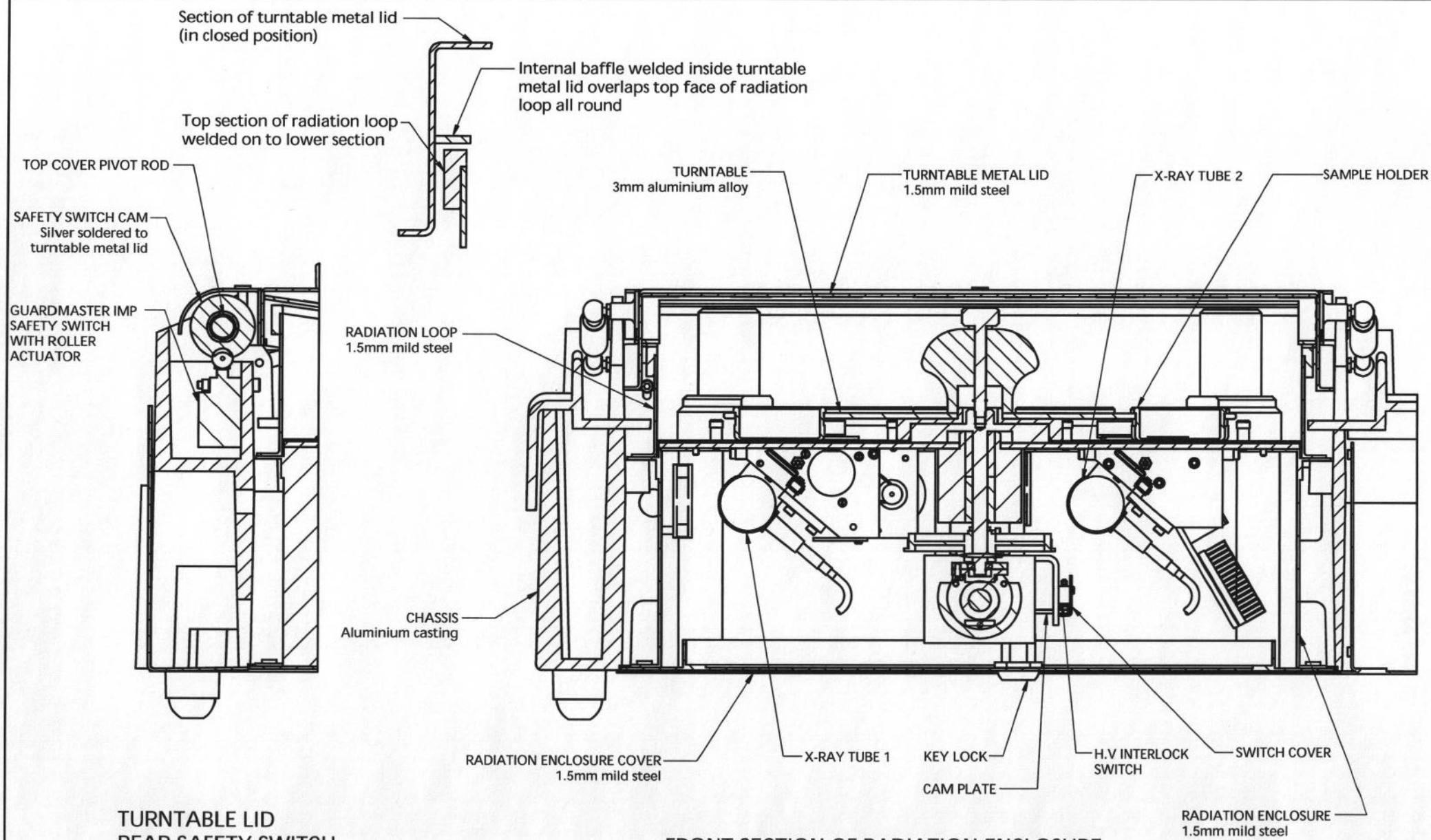
IF IN DOUBT ASK

DRAWN: P. SLIGHT | DATE: 11/09/02 | SHEET 2 OF 4

TITLE: TWIN-X RADIATION DRAWING

DRAWING No: ZX1161-1

ISSUE	AF DATE	1	11/09/02

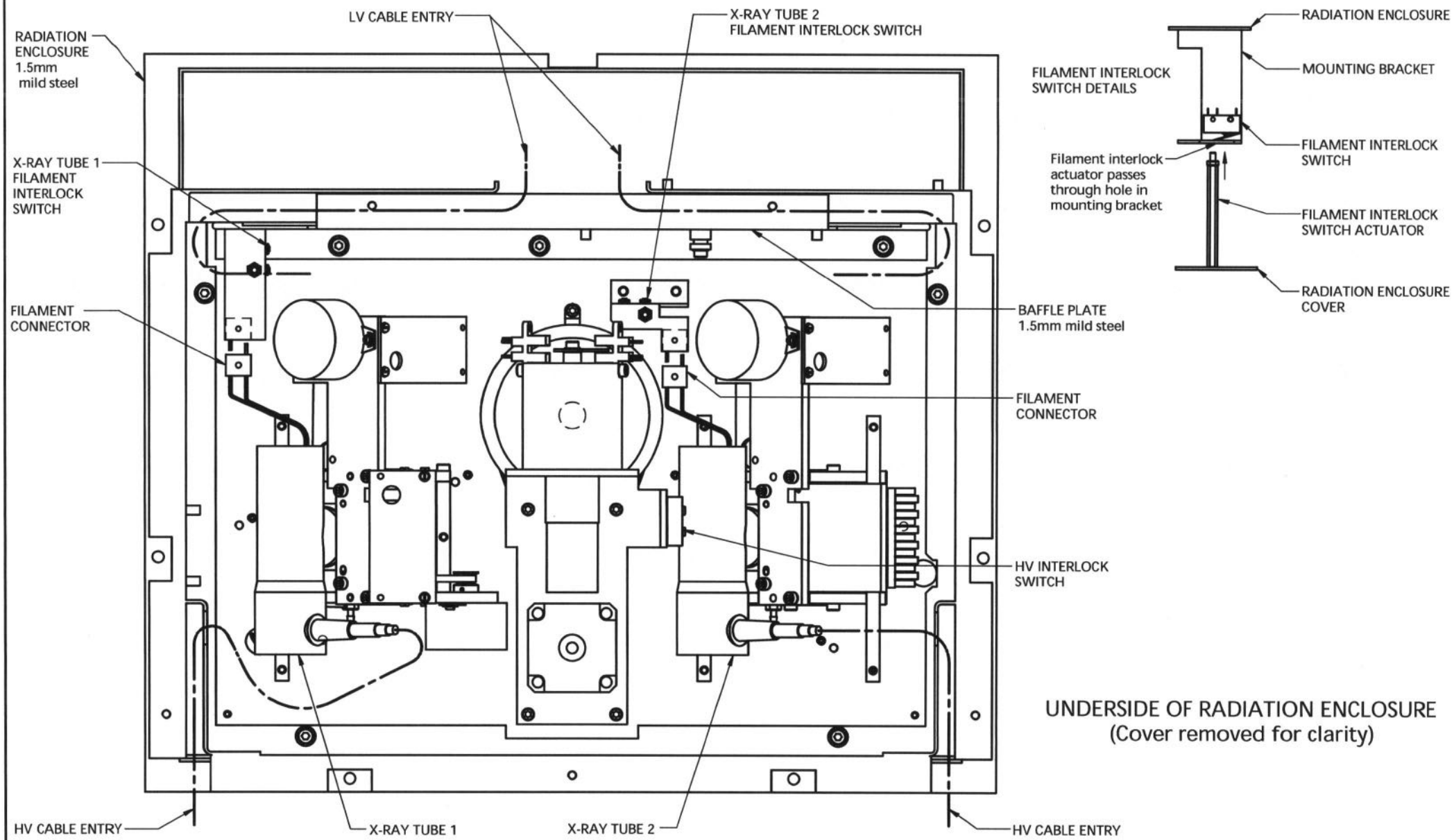


TURNTABLE LID
REAR SAFETY SWITCH

FRONT SECTION OF RADIATION ENCLOSURE

OXFORD INSTRUMENTS ANALYTICAL LTD		THIRD ANGLE PROJECTION IF IN DOUBT ASK	DRAWN: P. SLIGHT	DATE: 11/09/02	SHEET 3 OF 4
			TITLE: TWIN-X RADIATION DRAWING		DRAWING No: ZX1161-1

ISSUE	AF DATE	1	11/09/02



UNDERSIDE OF RADIATION ENCLOSURE
(Cover removed for clarity)

RADIATION ENCLOSURE COVER REMOVED FOR CLARITY

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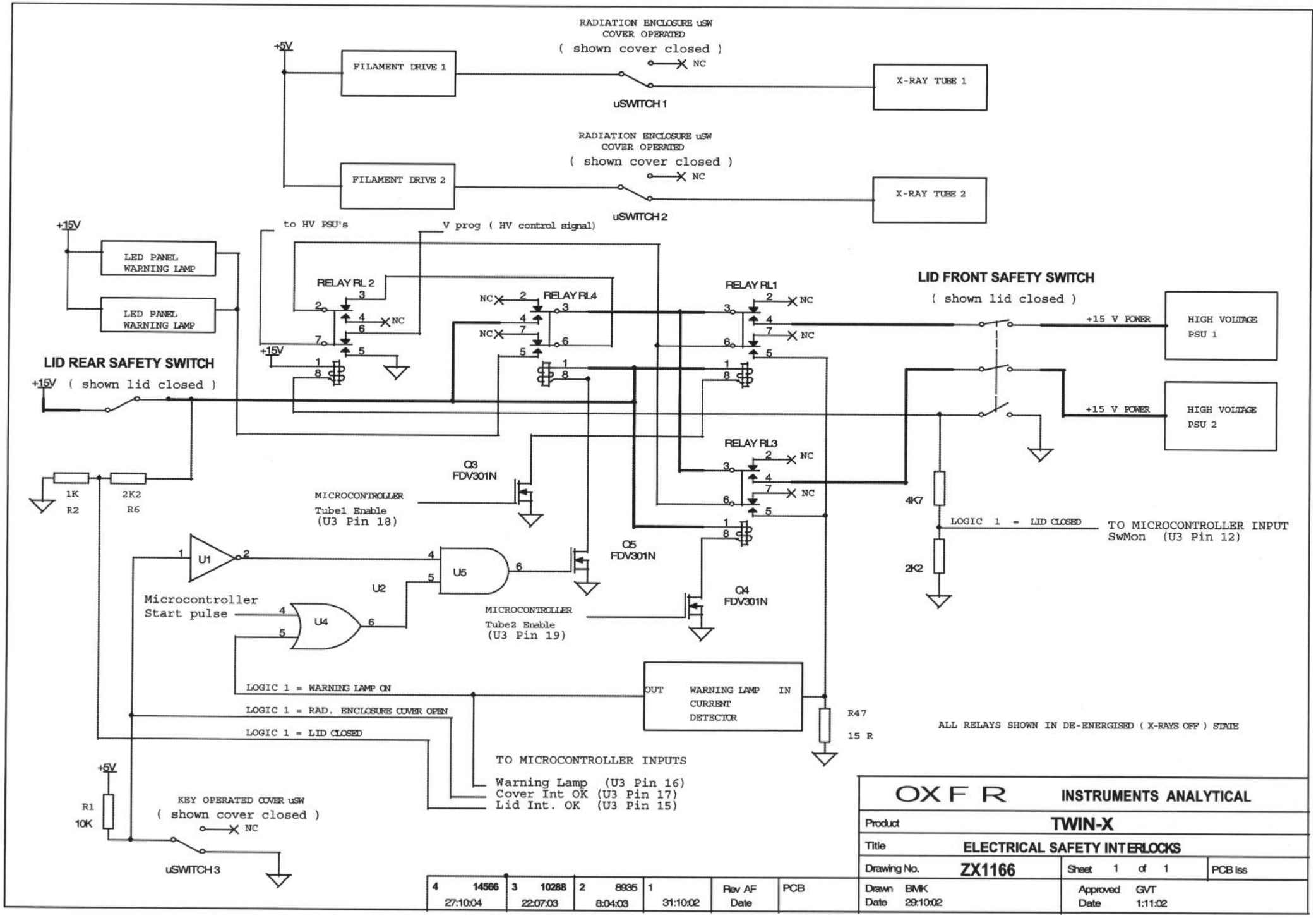
THIRD ANGLE PROJECTION
IF IN DOUBT ASK

DRAWN: P. SLIGHT	DATE: 11/09/02	SHEET 4 OF 4
TITLE: TWIN-X RADIATION DRAWING		DRAWING No: ZX1161-1



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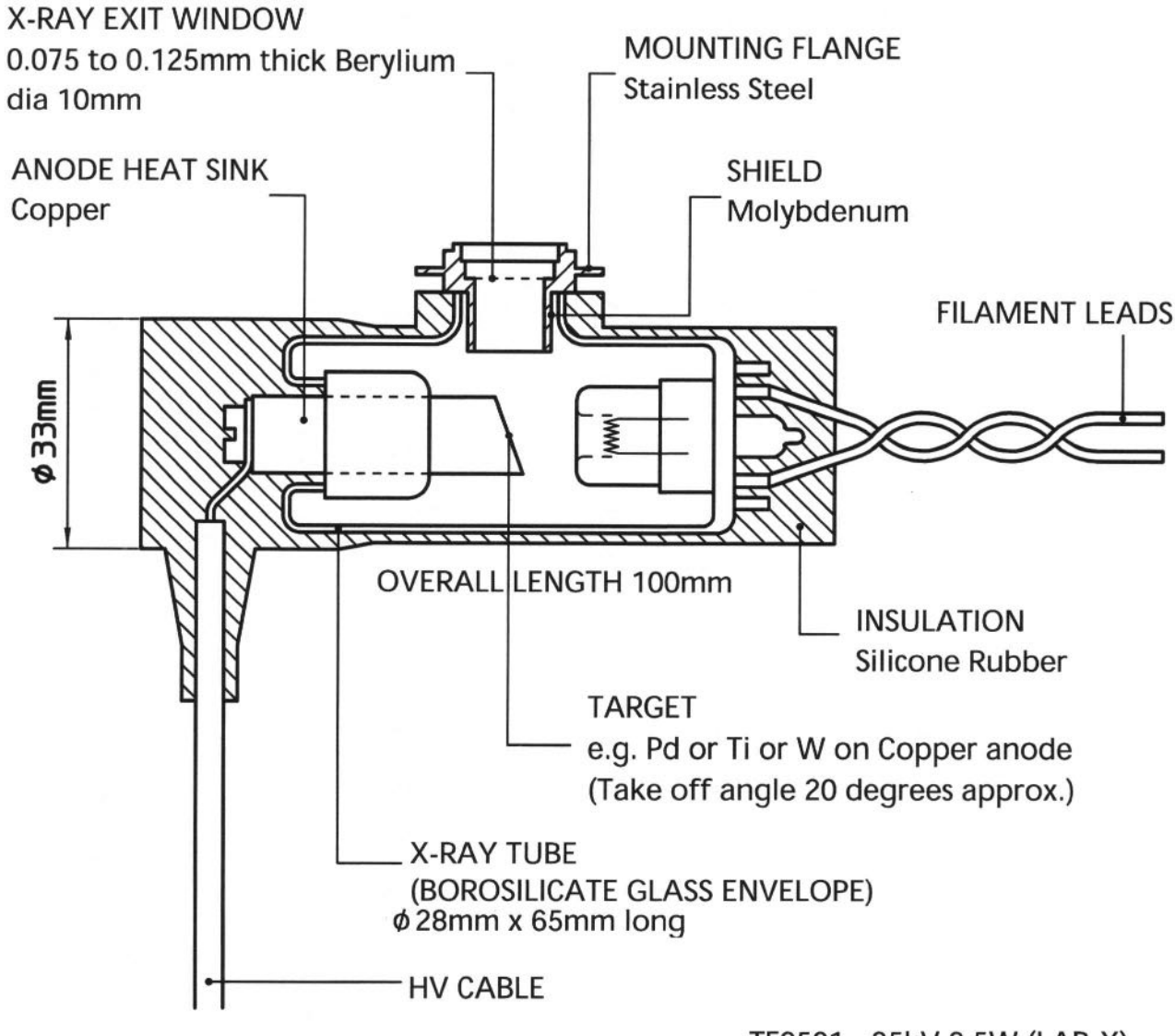
OXFR INSTRUMENTS ANALYTICAL

Product	TWIN-X		
Title	ELECTRICAL SAFETY INTERLOCKS		
Drawing No.	ZX1166	Sheet 1 of 1	PCB Iss
Drawn BMK Date 29:10:02		Approved GVT Date 1:11:02	

ISS. AF DATE	1. 09.04.97	2. 06.11.02	3. 16.02.09						



TF2501 - 25kV 2.5W (LAB-X)
30kV 3.0W (Twin-X)

TF1001 - 10kV 1.0W (Lab-X)

CROSS SECTION OF TF SERIES X-RAY TUBE
MANUFACTURED BY OXFORD INSTRUMENTS
X-RAY TECHNOLOGY GROUP

DRAWN	DATE	ORIG. SCALE
TAN	09.04.97	1:1

OXFORD INSTRUMENTS
INDUSTRIAL ANALYSIS GROUP

TITLE
TF SERIES X-RAY TUBE
OUTLINE DRAWING

DRAWING No.
LX6856-3

Product Information -

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www.oxford-instruments.com

Fitment of additional key switch to the X-Supreme 8000 instrument

June 2011

Please note that the X-Supreme8000 now incorporates and additional key switch, (located on the front of the instrument), which must be in the "enable" position, before the X-Supreme can make any X-ray measurements. Note: Full details on the operation of the key switch are described in the user manual

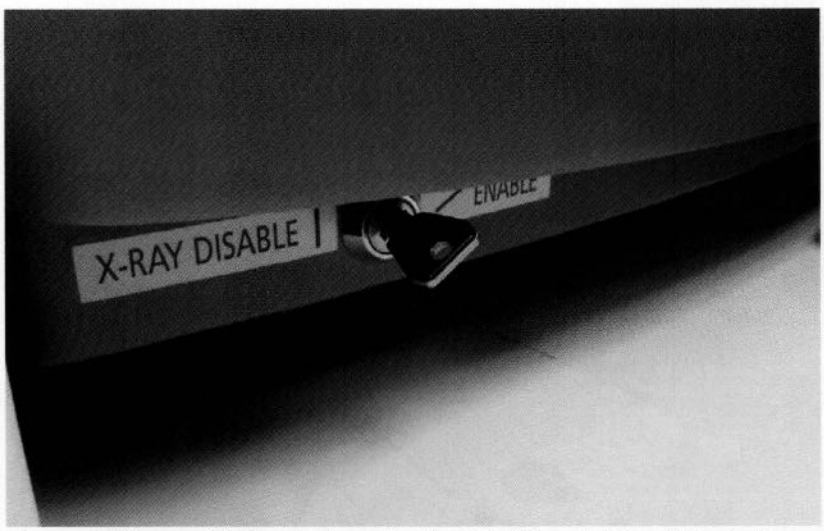

Figure 1: Keyswitch located on the front left hand side of the instrument.

If it is required to prevent any measurements being taken on the X-Supreme, then the key can be rotated to the "X-ray disable" position and removed from the instrument. Note: The key cannot be removed when in the "enable" position.

Thank you

Ref:XRF-PI-1002
Date January 2010

Oxford Instruments Analytical Limited
Registered office Tubney Woods
Abingdon, Oxon OX13 5QX
Registered in England, number 1468532
A subsidiary of Oxford Instruments plc

Product Information

Industrial Analysis
Email: industrial@oxinst.com
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OXFORD INSTRUMENTS

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X-Supreme 8000: introduction of Microsoft ™ Windows 7 operating system

Date: July 2014

This is to advise that the X-Supreme supplied is now fitted with Microsoft™ Windows 7 operating system which replaces the previous Windows XP operating system.

There is no change in the functionality of the X-Supreme operating software and the only difference seen by users will be when shutting down the software.

On shutdown with Windows XP the message "It is now safe to switch off your computer" is displayed and the user switches off the X-Supreme at the back of the instrument. For Windows 7 on shutdown no message is displayed so in this case simply switch off the X-Supreme at the back of the instrument as before.

Thank You

Oxford Instruments Industrial Products Limited
Registered office Tubney Woods
Abingdon, Oxon OX13 5QX
Registered in England, number 1468532
A subsidiary of Oxford Instruments plc

Product Information

Oxford Instruments (Shanghai) Co., Ltd
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Minhang District, Shanghai, 201109, China
Tel: (021) 64908280 Fax: (021) 64904042
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www.oxford-instruments.com

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X-Supreme 8000 Software Virus Scan report

Dear Customer

To ensure the highest level of software security, one of the final X-Supreme8000 instrument production checks is a full anti-virus software scan. The following gives the details:

INSTRUMENT SERIAL NUMBER:	**X16104**
ANTI-VIRUS SOFTWARE:	**4.89.0**
DATE & TIME SCANNED:	**27.Dec.2016**
TOTAL FILES SCANNED:	**32387**
VIRUS FOUND:	**0**

The above check ensures that the X-Supreme8000 is supplied virus free. Note: It is the responsibility of the user to ensure any USB devices used are virus free.

FOR AND ON BEHALF OF
OXFORD INSTRUMENTS (SHANGHAI) CO., LTD.

VERIFIED BY:

Scanned by: Chen Bin Bin	**Reviewed by:** Yang Yong
Date: 27. Dec. 2016	**Date:** 27. Dec. 2016

X-Supreme Measurement System

X-ray Radiation Survey Form
Doc.No: 51-XSU-02-0001-AA

Introduction:

This survey form must be completed as the last step of the final test before packing the instrument for dispatch. An instrument must not be handed over to a customer until all radiation safety checks pass successfully.

Required tools:

Instrument Ludlum Model 3 Survey meter
With probe model 44-7 or model 44-9.
Nylon sample when testing. Part number: 54-ZX1223-1.

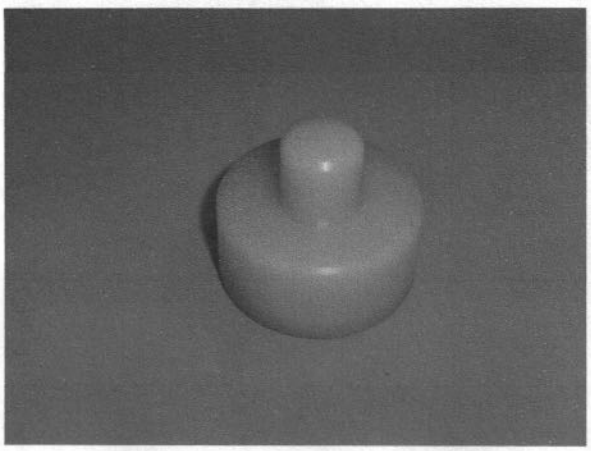
Test Instructions:

- Set the system under test to maximum power, 30kV, 100µA.
- Use Nylon sample when testing.
- Readings are taken at all numbered observation points (see photos on next page). The highest reading for each side is recorded in the test table.
- The distance between the probe and the instrument must be less than 30 mm when the reading is taken.

- **NOTE! Highest reading (background + emission) must be less than 0.3 mR/hr at 30 mm.**

NOTE:
This safety inspection is being provided as a reference in relation to the annual certification, repair, or initial installation on your x-ray system. It may not replace your particular state's requirements for radiation safety. Contact your state agency for further information.

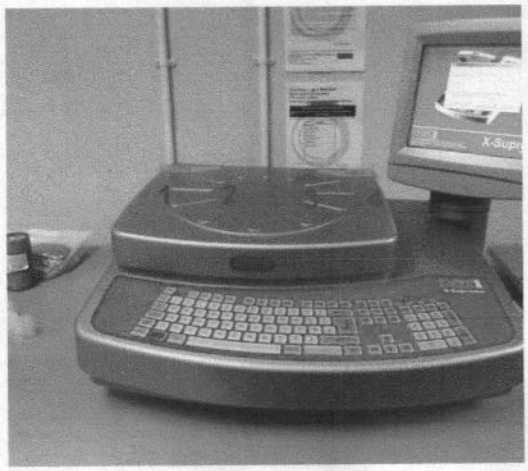
Photo of nylon test sample:

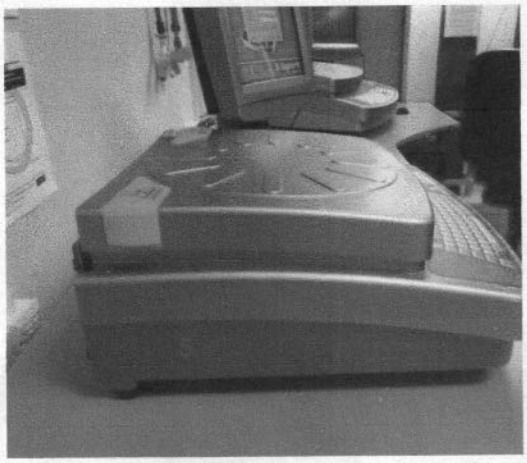


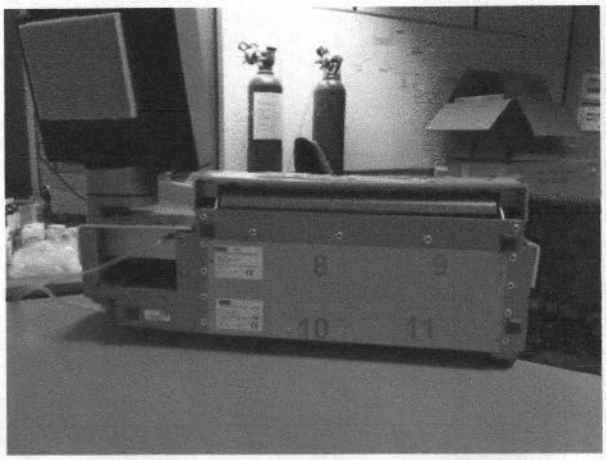
X-Supreme Measurement System

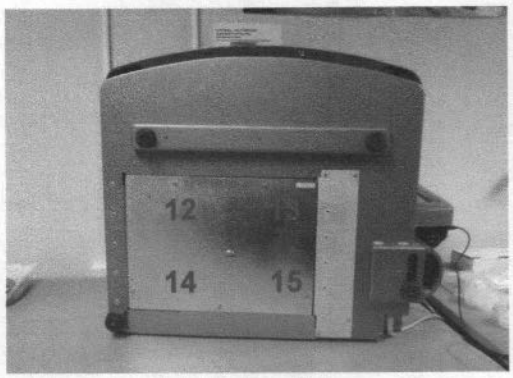
X-ray Radiation Survey Form
Doc.No: 51-XSU-02-0001-AA

X-ray Serial #:	122742	
X-ray model #:	X-Supreme	
SurveyMeter Manufacturer:	Ludlum Measurements INC.	
Survey Meter Model #:	Model 3	
Survey Meter Serial #:	274643	
Survey Meter Due Date:	27.Oct.2017	
Technician:	YY	
Survey Date:	27.Dec.2016	
Lid Interlock OK (yes/no):	Yes	
Highest reading top:	0.04mR/hr	**Max 0.3 mR/hr**
Highest reading back:	0.02mR/hr	**Max 0.3 mR/hr**
Highest reading left side:	0.03mR/hr	**Max 0.3 mR/hr**
Highest reading bottom:	0.02mR/hr	**Max 0.3 mR/hr**









Form 1 - Version 4.

OXFORD INSTRUMENTS

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Manufacturer's Warranty Activation Form

Note: This form must be filled out and returned to Oxford Instruments to activate the manufacturer's warranty.

(Please Complete in English)

Please select: Installation or Pre-Install?	**Installation**	Yes / No	**Pre-Installation**	Yes / No
Type of instrument:				
Instrument serial number:				
Installation Accepted & Warranty begins:	__ __/ __ __/ __ __ __ __ **DD / MM / YYYY**			

End Customer details:

Contact name:	
Company name & address:	
E-mail address:	
Telephone number:	

The instrument is now fully operational following the installation and the analyser is working according to specifications. The user has received all relevant software and hardware training and is able to operate the instrument successfully.

Site / Office / Location of Instrument:	
Representative Name (please print):	
Representative / Installation Engineer Name (please print) & signature:	
Customers representative Name (please print) & signature:	

Please return by scanned email attachment to ia.installations@oxinst.com or fax a copy of this form
Fax Number: +49 2825 5358146

***IMPORTANT**: It is essential that this document is completed by the end customer directly after the installation and faxed or emailed to Oxford Instruments in order to both activate and protect your manufacturer's warranty.*

Form 2 - Version 4.

OXFORD INSTRUMENTS

The Business of Science™

Quality Deviation Report (QDR)

(Please Complete in English)
Sheet 1 of 2

This **Quality Deviation Report** (QDR) should be completed by the installation engineer to record **any issues experienced or observed during installation** of an Oxford Instruments Analyzer. This document is for OI employees and distributors. The customer does not need to sign this paperwork and should not receive a copy. Feedback recorded on this form will trigger corrective action therefore it is most important to fill this form out accurately with as much detail as possible to help with the investigation and subsequent corrective action.

Type of instrument:	
Instrument serial number:	
Company name (End Customer):	
Contact name (End Customer):	
Company address:	

Please mark only one appropriate statement with an "X" in the box:

- **MODIFIED** - The instrument is now operational following the installation. The entire system is now working according to the specification, but there were issues to address to achieve total customer satisfaction. The user has received all relevant software and hardware training and is able to operate the instrument successfully. Further notes were made and can be found in the attached Quality Deviation Report (QDR). ☐

- **FAILED** - The instrument failed to operate successfully during the installation. The Quality Deviation Report contains the details and is attached. The time on site was either extended due to the failure or a return visit is required ☐

Site / Office / Location of Instrument:	
Representative Name (please print):	
Representative / Installation Engineer Name (please print) & signature:	
Installation Engineer e-mail address:	

Please return by scanned email attachment to ia.installations@oxinst.com or fax a copy of this form +49 2825 5358146

Form 2 - Version 4.

OXFORD INSTRUMENTS

The Business of Science™

Quality Deviation Report (QDR)

Sheet 2 of 2

Type of instrument:	
Instrument serial number:	
Installation date / period (from / to):	
Representative:	
End-Customer:	

Deviation Description (please specify any issue(s) to be resolved)

Hardware: Fault description	**Outstanding Action Required:**
Software: Fault description	**Outstanding Action Required:**
Analytical Performance: Fault description	**Outstanding Action Required:**
Sales / Support issue: Fault description	**Outstanding Action Required:**
Incomplete delivery: Fault description	**Outstanding Action Required:**

Please return by scanned email attachment to ia.installations@oxinst.com or fax a copy of this form +49 2825 5358146